ABSTRACT

JASROTIA, ASWINI KUMAR SINGH. Construction and Testing of Implant Carrier Particles for Validation of Multiphase Aseptic Processes. (Under the direction of Josip Simunovic and K.P. Sandeep)

Aseptic processing of low-acid foods containing large particles is an emerging technology. Most multiphase process validation methods employ simulated particles to contain residence time tags, thermo-sensitive implants and/or bio-loads for temperature detection, time-temperature integration, and bactericidal efficacy confirmation. Such particles need to have conservative (fast-moving and slow-heating) characteristics to compare them with real food particles for thermal treatment. This study was conducted to fabricate and test (by heat penetration studies) conservative simulated particles which serve as carriers for thermo-sensitive implants and bio-loads in the validation procedure required for aseptic processing of shelf stable low-acid multiphase foods. A custom developed CPD (Conservative Particle Design) software was used to determine the minimum wall thickness (~ 2 mm) and cavity dimensions of ½ inch cubic particles for validation of aseptic processing of foods containing ½ inch cubic potato, carrot and other vegetable pieces. These particles were fabricated from PP (polypropylene) and PMP (polymethylpentene) polymers and they exhibited conservative heat penetration characteristics when compared with various real food particles. Duplicate samples of simulated and real food particles were fitted with thermocouples and heated (< 127 ˚ C) under pressurized (autoclave, 24 psi) conditions. The method developed in this study can be used for experimental validation of the safety of aseptic processing of multiphase foods and would reduce the cost and complexity of process documentation and filing with regulatory agencies and bring aseptic multiphase foods closer to commercial reality.

CONSTRUCTION AND TESTING OF IMPLANT CARRIER PARTICLES FOR VALIDATION OF MULTIPHASE ASEPTIC PROCESSES

by

ASWINI KUMAR SINGH JASROTIA

A thesis submitted to the Graduate Faculty of
North Carolina State University
in partial fulfillment of the
requirements for the Degree of
Master of Science

FOOD SCIENCE

Raleigh

2004

APPROVED BY:

_____	_____
Dr. Josip Simunovic	Dr. K.P. Sandeep
(Chair of Advisory Committee)	(Co-Chair of Advisory Committee)

_____
Dr. John H. van Zanten

DEDICATION

To my mother
Sudesh Jasrotia

And

To my sisters
Suman, Rajni, Deepak and Kirti

BIOGRAPHY

Aswini Kumar Singh Jasrotia was born on April 30, 1979 in Mumbai, India. He originally hails from the beautiful state of Jammu and Kashmir, and grew up in costal cities of Jamnagar and Vishakhapatnam in India. He graduated from Indian Institute of Technology – Kharagpur in 2001, with a Bachelor in Technology (Honors) degree in Agricultural and Food Engineering. He was awarded t he Institute Silver Medal for being the academic topper during his undergraduate studies in the department of Agricultural and Food Engineering at Indian Institute of Technology – Kharagpur. After his graduation he joined Infosys Technologies Limited – a multinational software company as a software engineer in Aug 2001 and worked with Infosys Technologies Limited for one and half years, where he stood first in the software training program. In the spring of 2003 he began his Master of Science degree program in the Department of Food Science at North Carolina State University at Raleigh, NC and currently works with Dr. Josip Simunovic and Dr. K.P. Sandeep as a research assistant in the same department. His research focuses on aseptic processing of low-acid multiphase foods.

ACKNOWLEDGEMENTS

There are many people who have been a part of my graduate education as, friends, teachers and colleagues. It is a pleasure to thank each one of them, who made this thesis possible. First of all, I would like to thank my advisor and thesis committee chair Dr. Josip Simunovic who has consistently found time to give expert advice and help in conducting the experiments and steer me clear of fruitless paths in my research. I pay my deep gratitude to Dr. K.P. Sandeep for his constructive criticisms and editorial candor which no doubt has greatly improved the quality of this thesis.

I would like to thank my committee member Dr. John H. van Zanten for his understanding and advice. I have always felt greatly inspired to work with him. I am also greatly indebted to Dr. Kenneth R. Swartzel, for his great motivation and encouragement provided throughout this study.

My sincere thanks are extended to Dr. T.K. Palazoglu for his indispensable involvement in past research work relevant to current study, which laid foundation for successful completion of this research work.

My special thanks go to Jack Canady and Gary D. Cartwright for their help and advice in setting up the equipments most needed for this research work. I am greatly thankful to Bernard Eckhardt for providing all needed help on computer related issues. He was always available for the timely help, which is greatly appreciated.

I would like to thank former and current food engineering graduate students, especially Pablo, Cristina, Dorin, Ediz, Jeff, Supriyo, Zeynep, Andriana, Deepti, Arun and Junhua for all the support and good times. I wish to thank many faculty members at Indian

Institute of technology – Kharagpur, India, for providing guidance and support, important in pursuing my current graduate study.

I am deeply indebted to my family for giving me the love and needed encouragement throughout my studies. Without their support and absolute confidence in me, this study would not have been possible.

Finally, I wish to thank all my wonderful friends here at Raleigh and in India, for providing me help and encouragement at all times.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Chapter 1

INTRODUCTION

Aseptic processing and packaging has been an important area of research in food engineering for the past several decades. During aseptic processing, food is sterilized in a heat exchanger such as tubular heat exchanger, helical heat exchanger, scraped-surface heat exchanger, microwave or ohmic-heater. The packaging material is sterilized separately and both the food and the package are brought together during a continuous filling operation. This process allows products once considered perishable (such as milk, soy beverages, juice, and nectars) to be distributed and stored without refrigeration for periods up to six months or more. Most other processes use preservatives and/or refrigeration to achieve a long shelf life.

Aseptic processing has significant quality advantages over conventional thermal techniques such as retort or hot-filling. Retort canning typically requires products to be heated in the container for 20 to 50 minutes. Hot-filling uses the heat of the product to sterilize both the product and the package, a process which takes 1-3 minutes for heating and another 7-15 minutes for cooling. In contrast, aseptically processed liquid foods and beverages are sterilized outside the package using an ultra-high temperature process. The food product is rapidly heated, and then cooled before filling. The product is heated for a certain time (3 to 15 s) and temperature (195-285 °F) which can be tailored to obtain the best quality product, while ensuring safety. This fast heating and cooling process substantially reduces the energy use and nutrient loss associated with conventional sterilization. Compared to traditional canning techniques, aseptic processing renders a

substantial reduction in the time necessary for sterilization. In-addition, aseptically packaged products exhibit more natural texture, color, and taste. In the U.S., aseptically packaged products include milk, juices, tomato products, soups, broths, tofu, soy beverages, wines, liquid eggs, whipping cream, and teas. In Europe, a much wider variety of aseptically packaged products are available such as fresh milk, beverages, wine and aseptically processed fruit, vegetables & dairy products.

In canning, the product is sterilized inside a can after it has been packed. The thermal treatment for a canned food product depends on the size of the can. Product in a larger can would therefore need a greater amount of processing time to receive the required lethality. As a result, the quality (nutritional value) of the product would decrease as the size of can increases. In-addition, the desired texture, color, and taste of such a product is lost. The required thermal treatment for such a food product would further increase if it contains particulates, resulting in a further decrease in the quality of the product. However, thermal treatment of a food product during aseptic processing does not depend on the size of the package. Therefore, aseptically processed foods receive more uniform heat treatment than a food processed in a retort. In-addition, overcooking of the product is avoided and quality is retained.

The pH of a food product is a critical factor in determining the type of process to be adopted and the class of viable microorganisms of concern (Sandeep and Puri, 2001). Food products are divided into high-acid foods ($pH \leq 4.6$) and low-acid foods ($pH > 4.6$). A low-acid food (21 CFR 113.3) is defined as any food, other than alcoholic beverages,

with a finished equilibrium pH greater than 4.6 and a water activity greater than 0.85. These foods include butter, cheeses, grains, seeds, most nuts, dried fruit, fresh eggs, pears, papayas, sweet apples, raspberries, strawberries, bananas, prunes, and raisins. Acidified foods (21 CFR 114.3) are defined as those low-acid foods, which have had their pH reduced to 4.6 or lower by the addition of acids or acid foods. Examples include, pickles, pickled vegetables, salsa, pumpkin butter, sweet potato butter, barbecue sauces, chow-chow, and relishes. Any product that uses a combination of vinegar or other acid, acid foods (such as tomatoes) and vegetables (cucumbers, beets onions, ramps, cabbage, artichokes, cauliflower, squash, peppers (hot or sweet), beans, tropical fruits or any other vegetable) to achieve a pH < 4.6 are acidified foods.

Clostridium botulinum is an anaerobic, gram-positive, spore-forming rod that is heat resistant and produces a potent neurotoxin (U.S. Food and Drug Administration, 1992). For low-acid foods, the anaerobic conditions that prevail in aseptic processing are ideal for the growth of *Clostridium botulinum*. However, its growth is inhibited in acid environments (pH ≤ 4.6). Due to the high heat resistance of the toxin-producing spores of *Clostridium botulinum*, this microorganism is considered to be the target microorganism for commercial sterility of processed foods. Commercial sterility would be achieved for a food if it receives a heat treatment which statistically achieves a 12D reduction for a test organism which is more heat resistant than *Clostridium botulinum* (type E). "Commercial sterility of thermally processed food" (21 CFR 113.3) means the condition achieved:

(a) By the application of heat which renders the food free of:

(i) Microorganisms capable of reproducing in the food under normal non-refrigerated conditions of storage and distribution; and

(ii) Viable microorganisms, including spores, of public health significance; or

(b) By the control of water activity and the application of heat, which renders the food free of microorganisms capable of reproducing in the food under normal non-refrigerated conditions of storage and distribution.

The need for a process that is an alternative to the traditional canning process to produce low-acid particulate foods is increasing, since consumers are becoming more demanding about the quality, variety, and convenience of the food they eat. Aseptic processing is one such alternative. The technology and knowledge-base for aseptic processing of liquid foods has been well developed and approved by regulatory agencies. However, solving the issues involved in aseptic processing of low-acid particulate foods has been the area of interest for researchers during the past several years. All food particulates in such a product must receive the required thermal treatment to ensure the safety of the product while minimizing loss in quality. The geometric center of a food particle is the critical point of interest since it is always the slowest heating point. The challenge in such a system is to quantify the thermal treatment received by the food particulate at its center.

To establish an aseptic process for multiphase food products, regulatory agencies require the processor to identify and select a sterilizing F_0 value for the product, develop a conservative model that reliably predicts the total lethality of the heat process, quantitatively verify the lethality delivered by means of bio-indicators, and list the critical factors of the process and the procedures to be used for controlling them. Researchers in

the past have attempted to measure the temperature at the center of a particle during its thermal treatment in a continuous flow system using different methods. One such method is a thermal memory cell – a unit that has no power source but has a thermal memory. The cell is based on diffusion of a variety of metals through a silicon base. Different metals diffuse at different temperature-dependent rates. Other researchers have used magnetic sensors to measure the changing magnetic field strength of a magnet placed inside a simulated food particle. The magnetic field of a magnet changes along with the change in the temperature of its surroundings. As these simulated particles flow in a continuous system, the magnetic properties change. These changing magnetic fields created around the particle are sensed using magnetic sensors placed outside the tube through which the product flows. Some other researchers have been able to measure the temperature histories within particles which were restricted in free movements. These methods serve as a first step towards can estimating the F_0 (lethality) value received at the center of the food particle and they need to be validated by biological measurement methods.

Due to various challenges in accurately determining the temperature histories at the center of a particle flowing in a continuous system, alternatives have been considered. Researchers in the past have suggested the use of conservative mathematical models to predict the total lethality of the heat process during aseptic processing of low-acid food particulates foods. The most critical factors identified in mathematical models were particle size and shape, fluid-to-particle heat transfer coefficient, and the residence time distribution (RTD) in the holding tube. RTD measurement is needed because of the

difficulties in non-invasive measurement of internal temperatures of particle during continuous flow. If the temperature of the cold-spot could be measured at the end of the heater and the hold tube, RTD measurements would be unnecessary.

Other mathematical models have been used to estimate the minimum residence time required under the specific conditions necessary to deliver the produce the desired lethality. The minimum residence time requirement determined by the model had to be coupled with RTD data for designing the holding tube length of the aseptic system. It is necessary to ensure that largest and the fastest traveling particle will have a residence time at least as small as the minimum residence time determined by the model.

Another problem associated with aseptic processing of particulate foods has been the lack of understanding of the heat transfer between fluid and particles during continuous flow. Determination of fluid-to-particle heat transfer coefficient (h_{fp}) poses a unique challenge of monitoring the temperature of moving particles without restricting the flow of the particle. Various methods have been developed to solve this problem. This includes both invasive and non-invasive methods. Invasive methods include the thermocouple and the moving thermocouple methods while non-invasive methods include the relative velocity method, microbiological method, use of time-temperature integrators, remote temperature sensor method, liquid crystal method, melting point indicator method, and phase change technique.

Biological validation of a scheduled process for traditionally processed canned foods is not required if the filed process is based on properly designed heat penetration tests. For aseptically processed particulate products, however, biological validation of the process is necessary because the design of the aseptic process is not based on heat penetration tests due to the unavailability of the time-temperature history of the product. Biological validation of an aseptic process for a particulate food product involves the use of biological indicators that travel through the system inside a particle.

The National Center for Food Safety and Technology (NCFST) and the Center for Aseptic Processing and Packaging Studies (CAPPS) sponsored a workshop in November 1995 and March 1996 to reach a consensus on concerns that needed to be resolved to develop an aseptic process for multiphase food products. In the NCFST-CAPPS workshop, various methods for determining the RTD measurements were discussed. Some such methods are magnetic methods, magnetic resonance imaging, history methods, ultrasonic methods, and salt tracer method. The workshop also focused on issues related to the lethality received by the "critical particle". The "critical particle" is the particle that heats up the slowest (due to its low thermal diffusivity) and receives least thermal treatment because it is the fastest or because of a combination of both. The goal is to "prove" that the "critical" part of the critical particle received the required thermal treatment.

The research groups at the workshop agreed that a distribution-free method was the most appropriate method to reliably determine the fastest particle within the system.

They recommended the determination of residence time of 299 particles to determine the fastest particle residence time. Tetra Pak, Inc., used the case study from the workshop as a guideline and filed a thermal process for condensed cream of potato soup (a low-acid particulate food). The company received a no-objection letter from FDA in May 1997. Despite interest in the food industry, many economical issues have not encouraged them to develop such a process. For example fillers which can handle large particles or viscous foods are not economical. In addition, the validation process is not only time consuming (six to nine months) but also expensive (~ US $100, 000). In the validation process, each particle is identified by a magnetic tag contained in the particle and must pass through the entire system before the next one can be inserted to avoid misidentification of the particles (due to cross-over or overlap). To avoid this huge cost, a Magnetic I. D. / Timed Insertion Particle (MID/TIP) system – that uses multiple magnetic identification tags to determine particle residence times have been developed by researchers.

Thus, based on results from the NCFST-CAPPS workshop it was evident that identification of the "critical pariticle" was essential for sucessful filing (with FDA) of a low-acid particulate food product. the identification particle with slow heating and fast flowing characteristics is important in developing convervative methods to predict the lethality delivered at its center. Monitoring and validating a continuous thermal sterilization process requires the use of these simulated food particles, which must exhibit conservative flow and thermal characteristics. A conservative design of the carrier particle is targeted to ensure the saftey of the product. By conservative design, it is meant that the thermal protection provided by the simulated particle to its center is at least

equivalent to or greater than the thermal protection provided by the target food particle to its cold spot (typically its geometric center) under identical heating conditions. Thus, the current study was undertaken with the goal of developing a validated systematic approach for the construction of simulated particles that would assure conservative thermal behavior (slow-heating behavior) and near-neutral buoyancy (fast-flowing behavior in a particultate food product).

Chapter 2

REVIEW OF LITRATURE

2.1 Aseptic Processing

A typical aseptic process involves sterilization of the product by a heat-hold-cool approach followed by filling and sealing into pre-sterilized containers under aseptic conditions. Product heating in aseptic systems is performed directly (steam infusion and steam injection) or indirectly (plate, tubular, or scraped surface heat exchangers), whereas the containers are sterilized by superheated steam and/or Hydrogen Peroxide (Lopez, 1987).

Aseptic processing originated from the need to solve problems associated with conventional 'in-container' sterilization of foods such as low rate of heat penetration to the slowest heating point in the container, the long processing times required to deliver the required lethality, destruction of nutritional and sensory characteristics of the food, low productivity, and high energy costs (Smith *et al*., 1990). The field of aseptic processing for liquid foods is well developed, whereas aseptic processing of particulate foods faces many technical issues. In aseptic processing of low-acid particulate foods, some major hurdles are present in process establishment. Unlike a conventional retort process for which heat penetration studies are easily applied for process evaluation, there is no technique is available to measure the center temperature of particles that are sterilized during continuous flow.

2.1.1 History of Aseptic Processing

Aseptic processing was initiated in 1927 at the American Can Company (Mitchell, 1988). The result was the development of the heat, cool, fill (HCF) process for liquid and semi-liquid foods. The product was heated to 300 °F in less than 15 s, immediately cooled and filled into sterile cans (Ball and Olson, 1957). The process was not a commercial success because of the associated high cost of the equipment, inflexibility with respect to can size, and the frequent problems occurring with the can filing machines.

The Avoset process (which is no longer in operation) was another milestone in the development of aseptic processing. In this process the filling and sealing area were heated to eliminate contamination and further protection was accomplished by ultraviolet lamps (Mitchell, 1988). Sterilization was achieved by direct steam injection to achieve temperature of 260-280 °F. The product was packaged in containers that were retorted or sterilized by hot air.

Real progress in the commercial development of aseptic processing technology of foods began with the invention of the Dole-Martin process in the late 1940s in California by the Dole Engineering Company (Lopez, 1987). The process could be used for the sterilization of any low- or high-acid products. It was not a technical success as the package, a metal can, had nothing to differentiate it from conventional ‘in-container’ sterilized foods (Dennis, 1992). Product heating and cooling was achieved by heat exchangers based on the principles of high temperature short time sterilization, while the

containers were sterilized by superheated steam (Mitchell, 1988). The first commercial Dole system was installed in the early 1950s for the production of split pea soup and sauces. Another development in the early 1960s in Europe was the use of hydrogen peroxide to sterilize flexible packaging materials for packaging (Lopez, 1987).

In the U.S., use of hydrogen peroxide to sterilize packaging materials was approved by the FDA only in February 1981 (Cousin, 1993). This approval provided the opportunity for the use of laminates for consumer packages. Aseptic containers can now be produced in many different sizes and shapes, from small packages to large 100,000 gallon bulk storage tanks. The bag-in-box technology has significantly grown over the years and its thrust has been in units ranging from 2 to 300 gallons.

In the 1960s, TetraPak introduced chemically sterilized packages. Introduction of drink boxes was another important development for aseptic packaging. Drink boxes are one of many aseptic packaging materials available. A drink box combines thin layers of paper, plastic, and aluminum to form a unique, high-performance beverage and liquid-foods container that is compact, lightweight, and tough. Paper (70 %) provides stiffness, strength, and the efficient brick shape to the package. Polyethylene (24 %) on the innermost layer forms the seals that make the package wafer-tight. A protective polyethylene coating on the exterior keeps the package dry and provides a unique surface for printing nutritional information and graphic design. Aluminum (6 %) forms a barrier against light and oxygen. This ultra-thin layer of foil eliminates the need for refrigeration and prevents spoilage without using preservatives.

2.1.2 Advantages and Disadvantages of Aseptic Processing

Compared to traditional canning, where products are heated in the container for 20 to 50 minutes, aseptic processing involves flash heating and cooling followed by aseptic packaging which substantially reduces the energy-use and nutrient loss associated with conventional sterilization. As a result, aseptically packaged products can retain a higher degree of nutritional value, natural texture, color, and taste while using less energy. Typical organoleptic properties (color and texture) and heat sensitive nutrients are better retained due to the high temperatures and short processing times involved. Compared to canning, aseptic processing can a) destroy microorganisms in a considerably shorter time and pack the product in sterile environment, b) fill finished product into containers of different types and sizes, c) substantially increase quality (including increased retention of nutrients, improvement in the overall sensory score, and reduction in the extent of non-enzymatic browning), d) enable the final product to be microwaveable, and e) save cost by the use of plastics as opposed to using expensive metal containers that have to withstand high temperatures and pressure (David, 1992; Toledo and Chang, 1990). The advantage of aseptic processing with regard to packaging is also important. A wide variety of shapes and materials can be chosen for aseptic packaging, whereas in conventional processing, metal cans and glass jars are the major choices. Aseptic processing avoids recontamination of the sterile food because of the use of pre-sterilized packages.

Though aseptic processing offers better quality and packaging advantages for processed foods, it has some drawbacks. The limitations include higher cost involved with increasing speeds of fillers, higher cost of equipment, and the sophisticated automation required for process control. The workers in an aseptic plant must be better trained and skilled (than for conventional retort processing) in accordance with the laws of the regulatory agencies, which increases the cost for this process significantly. In addition, the use of aseptic process for specific product types and destroying heat-resistant enzymes are some other limitations (Ramaswamy *et al.*, 1997).

2.2 Aseptic Processing of Particulate Foods

Currently, aseptic processing and packaging in North America is primarily limited to liquid foods, but there is considerable interest for the extension of the technology to low-acid liquid foods containing large particulates (Dignan *et al.,* 1989; Heldman, 1989; Toledo and Chang, 1990; Lund and Singh, 1993).

In the 1980s, FDA received two filings for aseptically produced low-acid foods containing particles. Both filings were withdrawn after discussion between the FDA and the process filers about the establishment of a scheduled process (Larkin, 1997). In 1995, a workshop was held by the National Center for Food Safety and Technology (NCFST) and Center for Aseptic Processing and Packaging Studies (CAPPS) (CAPPS and NCFST Workshop, 1995, 1996) to resolve issues regarding the proper establishment of an aseptic process for multiphase food products. The workshop focused

on RTD of particles, mathematical modeling of the process, biological validation, statistical design and analysis of the RTD data. The consensus statements developed in the workshop were summarized in a series of symposium papers on "Continuous Multiphase Aseptic Processing of Foods" (Larkin, 1997; Sastry, 1997; Digeronimo *et al*., 1997; Damiano, 1997).

The outcome of the CAPPS and NCFST workshop served as a guideline for a study by Tetra Pak Inc. (Palaniappan and Sizer, 1997) and the National Food Processors Association (NFPA), which led to the first FDA no-rejection letter for a multiphase process filing (Larkin, 1997). The product used by Tetra Pak, Inc. was a diced potato soup containing 15% diced fresh potato in a modified starch solution. Using the following formula, they concluded that 299 particles must be timed in order to determine the residence time of the fastest particle with a 95% confidence level.

$$N = \log (1-C) / \log (1-P) \qquad (2.1)$$

Where N is the population size, C is the confidence level (95%), and P is the fastest particle fraction (1%). They used simulated potato particles with embedded magnets, and adjusted the density so that it was less than the density of potato particles and closer to that of the carrier fluid. Since particles having a density similar to the density of the carrier fluid move faster along the holding tube, this would yield a conservative approach in the determination of residence times (less than the residence times of real potato particles). Real potato particles were also included in the RTD study to verify that the simulated particles yielded conservative residence time values. Magnetic coils were placed at the entrance and exit of the final heater, the 58.4 m-long

holding tube, and the pre-cooling unit. The electromotive force generated by the magnet-containing simulated particles was recorded by a data acquisition system, and the residence times in each section were obtained using the entrance and exit times. A finite difference program was used to calculate the temperatures of the fluid and the particle, and the accumulated lethality values were determined. The model was biologically validated using chicken-alginate cubes inoculated with *Clostridium sporogenes* PA 3679, which again had a density that would give residence times smaller than those of real potato particles. The process proved to be safe based on the results of the mathematical model and the biological validation. This study has become a milestone in the history of aseptic processing in the U.S., as it was the first successful filing of an aseptically processed low-acid particulate food. It will serve as an established protocol for other food processors who want to produce low-acid aseptic products (Palaniappan and Sizer, 1997).

Aseptic process of low-acid foods containing particulates can offer many safety and quality attributes, but has not been able to get popularity in the commercial circle. This is attributed to uncertainties in the approval of the process and cost of process validation involved in continuous thermal processing of particulate low-acid foods (Larkin, 1997). Due to differences and lack of appropriate knowledge of microbiological, chemical and biochemical reaction rates during thermal destruction, an engineering analysis is necessary to determine an optimum thermal treatment to obtain a safe and high quality product (Jung and Fryer, 1999). In addition, each new filing with FDA is unique and a processors cannot expect that there would ever be instructions for optimum treatment determination applicable to every product (Larkin, 1997).

2.2.1 Issues Involved in Aseptic Processing of Particulate foods

The early problems facing the establishment of a process for continuous heat-hold-cool sterilization of low-acid liquid foods containing particulates were: a mechanical means of physical handling in order to maintain proper distribution and particle integrity and the assurance of commercial sterility with minimal quality loss (de Ruyter and Brunet, 1973). The first problem was successfully handled with equipment such as scraped surface heat exchangers (SSHEs) or tubular heat exchangers with a displacement pump (Lee and Singh, 1990). The problem of assuring a commercially sterile product with minimizing loss in quality however remains unsolved.

The real challenge, however, is the establishment of a microbiologically safe process for particulate foods, without sacrificing the essential quality factors. An appropriate thermal process can be determined if accurate time-temperature data measured at the slowest heating point within the largest and fastest particle flowing through the aseptic system is available. Thermal process establishment for liquid foods undergoing aseptic processing is simple because the temperature of the liquid can be measured at the point of interest (Rao, 1992). Heat penetration measurements for a food particle traveling through an aseptic processing system is difficult and not practical at the present time without restricting the free movement of food particles (Sastry, 1986; Lee and Singh, 1990; Heldman, 1992; Lund and Singh, 1993; Maesmans *et al.,* 1994). Due to challenges encountered in measuring the temperature of moving particles in aseptic processing systems, several alternatives have been considered such as biological validation techniques (Heppell, 1985a, b; Berry, 1989; Tobback *et al.,* 1992; Cacase *et*

al., 1994), moving thermocouple methods (Sastry, 1992; Zitoun and Sastry, 1994a), liquid crystal technique (Stoforos and Merson, 1991; Balasubramaniam and Sastry, 1994c; Zitoun and Sastry, 1994b), time temperature integrators (Hendrickx *et al.,* 1992) melting point indicators (Mwangi *et al.,* 1993), and relative velocity methods (Balasubramaniam and Sastry, 1994b).

These methods apply, with some limitations, to heat transfer in continuous two-phase flow. Biological validation is considered to be the most reliable technique to address safety issues, but the process is tedious and not entirely reliable (Pflug *et al.,* 1990). In the absence of experimentally determined values of temperature, mathematical modeling is a useful alternative for establishing an aseptic process for particulate fluids (Heldman, 1992). Establishing processes for continuous sterilization of low-acid particulate foods require consideration of the following aspects (Dignan *et al.,* 1989):

1. Identification and selection of an appropriate sterilization value
2. Development of a conservative model to predict the sterilization value achieved (in the holding tube) at the slowest heating location of a given particle
3. Quantitative microbiological validation of the lethality delivered and
4. Identification of critical factors and procedures to be used for controlling these factors.

de Ruyter and Brunet (1973), Manson and Cullen (1974), Dail (1985), Heppell (1985 a, b), Sastry (1986), Chandarana *et al.* (1990) and Lee *et al.* (1990 a, b) were among several researchers who have tried to address these issues involved in aseptic

processing of food particulates. The most critical factors in mathematical models were identified as particle size and shape, fluid-to-particle heat transfer coefficient, and the residence time distribution (RTD) in the heat exchangers and the holding tube (Dignan *et al.,* 1989; Heldman, 1989, 1992; Rao, 1992). In a continuous flow system, not all particles of the product remain in the processing equipment for the same time. The particles or parts close to the wall (boundary layer) flow at a much slower speed than those traveling near the center of the pipe (Danckwerts, 1953). Therefore, there is a distribution of residence times through the heat-hold-cool sections of the aseptic system, with portions of the total flow spending either less or more time than the mean residence time (Burton, 1988).

2.2.2 Equipment

The main components of an aseptic processing system are a product supply tank, sanitary pump, deareator, heat exchanger, holding tube, aseptic surge tank, back pressure device, flow diversion valve, and a sterilized packaging zone. Positive displacement pumps such as rotary or reciprocating pumps are used in aseptic processing depending on the pressure drop across the system, viscosity of the product, and type and size of particles. With small particle sizes (< 1/8 ") and low pressure drops (< 150 psi), a rotary positive displacement pump is preferred. When the product contains particulates up to 3 " in size, a reciprocating piston pump is preferred (Lund and Singh, 1993).

Dearation removes excessive air before the sterilization process. It is especially important for products that are under risk of oxidation reactions. Dearation reduces

fouling in heat exchangers and maintains constant filling conditions by preventing foaming (Carlson, 1996). The back pressure valve provides a pressure higher than the boiling pressure of the product. An aseptic surge tank provides sterile product to the packaging unit. If a problem is encountered in sterilization process, packaging can be carried on independently using the product stored in the tank. In-addition, when the fillers are not working, the sterilized product can be stored in the aseptic surge tank.

Direct heating or cooling or indirect heating or cooling are applied for heating or cooling the product. In direct heating, the product is sterilized with steam either by steam injection or steam infusion. A steam injector is used to inject steam to sterilize the product in steam injection method. In steam infusion method, the product is dispersed into the steam by spraying or by falling sheets (Lund and Singh, 1993). In direct heating, heat transfer occurs due to condensation of the steam which results in some dilution of the product. This brings the need for an additional operation, flash cooling, to remove the excess water. The advantages of direct heating are very rapid heating of the product and dearation of the product in flash cooling. However, high cost of the equipment, requirement for an additional operation, and the risk of losing desirable volatiles are the disadvantages of direct heating. In indirect heating, plate heat exchangers, tubular heat exchangers, and scraped surface heat exchangers are used.

The characteristics of the equipment have a great influence on the efficiency of the process resulting in different heating rates in different heat exchangers (Heldman, 1992). The choice of the heat exchanger is based on the characteristics of the product.

Plate heat exchangers are inexpensive and easier to clean. These types of heat exchangers can also be used for preheating or precooling of the product. Plate heat exchangers are used for liquid products with low viscosity (juices and juice drinks). For viscous products and products containing large particles, one should not use a plate heat exchanger because of the high pressure drop involved in pumping viscous fluids. In addition, particles can build up between the plates and block the flow passages (Mitchell, 1988). In tubular heat exchangers, the product and heating or cooling medium are separated by concentric tubes. These heat exchangers can be double tube or triple tube types. Tubular heat exchangers can handle more viscous products than plate exchangers. Tubular heat exchangers have only a few gaskets and no moving parts, thereby minimizing the maintenance costs and enhancing microbial safety (Lund and Singh, 1993). However, high pressure drops, low rates of regeneration, and tendency for fouling are the drawbacks of tubular heat exchangers (Bhamidipati and Singh, 1995). Tubular heat exchangers are more versatile than plate heat exchangers in that they can be used for products that are more viscous than juices and juice drinks. Shell-and-tube or tube-in-tube heat exchangers are two types of heat exchangers that are used in the food industry.

Scraped surface heat exchangers (SSHE) appear to be the best choice for more viscous products and foods containing particulates. A scraped-surface unit consists of a jacketed cylinder housing scraping blades on a rotating shaft. The rotating action of the scraping blades prevents fouling on the heat exchanger surfaces and improves heat transfer rates. One disadvantage of these units is that because of the excessive physical action of the blades, particles are likely to be damaged (Carlson, 1991). In fact, a recent

study it has been shown that significant damage to the particles could occur due to mechanical effects in a SSHE when combined with the thermal effects, i.e. softening of the product texture. Therefore, a compromise is made so as to obtain the maximum heat transfer possible with an acceptable level of particle damage (Carlson, 1991). Another problem related with the use of scraped surface heat exchangers is the non-uniform particle density distribution in vertical scraped surface heat exchangers.

Coiled heat exchangers are have the ability to provide high heat exchange rates, improved mixing of the product, and thus uniformity of heating. In helical coils, a flow in radial direction, called secondary flow, develops due to centrifugal force. The secondary flow helps mixing of fluid elements in the radial direction. Due to more kinetic energy dissipation in helical tubes, secondary flow, and thus the pressure drop, increases. Investigating the pressure drop and related pumping requirements before using helical heat exchangers is thus critical (Coronel and Sandeep, 2003).

The most important section of an aseptic processing system is the holding tube since commercial sterility is achieved in this section. External heat cannot be applied at any point along the holding tube. The holding tube must be sloped upwards at least 0.25" per foot of the tube in order to avoid air pockets and help drainage of the product back to the heat exchanger (Lund and Singh, 1993). The regulatory agency gives credit for the lethality (F_0) accumulated only in holding section. A back pressure device is used to maintain the pressure in the holding tube above the vapor pressure of the product to prevent flashing. The temperature of the product at the exit of the holding is monitored

and used as the process temperature when determining the adequacy of the process. To ensure safety, and to get the credit for required lethality of microorganisms, the processor should consider only the time that the product spends in the holding tube and the temperature at the holding tube exit. Thus, the heat received by the product in the hat exchanger can only be a safety measure. It has been shown that such an approach results in significant over-processing, as the thermal treatment received in the heat exchanger will cause a temperature distribution within the particles leaving the heat exchanger (Chandarana and Gavin, 1989). Thus, holding tube design must take into account this thermal contribution which otherwise would result in poorer product quality due to unnecessarily long holding times.

After the holding section, the product passes on to the cooling section where it is cooled prior to filling into sterile containers in an aseptic environment. Heat, radiation, and chemicals, and their combination might be used to sterilize the package. Hydrogen peroxide (H_2O_2) is the only chemical allowed by FDA that can be used to sterilize packages. H_2O_2 is used in concentrations varying from 25 - 35 %. The packaging material is either sprayed with H_2O_2 or dipped in a H_2O_2 bath. H_2O_2 residual in the packaging material is limited to 0.5 ppm after packaging (Floros, 1993). The aseptic processing equipment must be sterilized using clean-in-place (CIP) methods. Aseptic processing of high-acid foods involves a CIP cycle consisting of hot water, alkali, and hot water. For low-acid foods it consists of hot water, alkali, hot water, acid, and hot water. High efficiency particulate air (HEPA) filters are used to sterilize the air flow system, where air is treated through the filters as it circulates (Carlson, 1996).

2.2.3 Effect of Processing on Product Quality

Producing a microbial safe product with a high quality is the goal of an aseptic process. In aseptic processing, quality factors such as color, flavor, texture, and nutritional value are preserved as high temperatures are used for a short period of time, unlike conventional processing where the food is exposed to lower temperatures for a longer period of time. Better quality is achieved since a high temperature-short time (HTST) treatment destroys microorganisms at a higher rate than the degradation of quality attributes. Theoretically this can be explained by the fact that thermal degradation reactions of vitamins or pigments have higher decimal reduction times (D value) and higher thermal resistance constants (z value) compared to microbial inactivation.

Chlorophylls, carotenoids, anthocyanins, and betalains constitute most of the pigments present in foods. Decomposition of chlorophyll to pheophytin and further to pyropheophytin results in an olive-brown color. In order to retain the original color in retort and aseptic processing of green vegetables, addition of zinc was shown to be promising. The changes in flavor, color or other quality attributes in aseptically processed foods is related to food composition and the effect of high heat on them. Trans-to-cis isomerization of carotenoid pigments may occur readily and especially at high temperatures, resulting in a reduction in their bioavailability as vitamin A, which is a precursor, resulting loss of nutritional value. HTST treatments favor retention of the natural color of foods containing chlorophylls and carotenoids. However, it has been shown that poor storage conditions affect nutrient and sensory quality of aseptically

processed products in a far greater way (Schwartz, 1992). For example, poor oxygen barrier properties of aseptic packages causes oxidation of vitamin C and pro-vitamin A carotenoids. Also, hydrogen peroxide that might have remained in the container after packaging can result in loss of color, especially in products containing anthocyanins. Thus, packaging aseptically processed foods with minimal headspace and storing them at low temperature reduces color losses (Schwartz, 1992). In-addition, temperature abuse can cause vitamin C degradation during storage of the product (Sizer *et al.*, 1988).

Carbohydrates may undergo Maillard browning reactions, gelatinization of starch, and starch retrogradation. Non-enzymatic browning (Maillard) reactions occurs between a reducing sugar and an amine group (from proteins) during aseptic processing and storage, with the extent of browning depending of the type of sugar (monosaccharide or disaccharide) and the heat treatment. Enzymatic browning can be controlled to some degree by careful batching of the raw ingredients prior to aseptic processing (Schwartz, 1992). Lipids are sensitive to oxygen and may develop textural and flavor changes due to oxidation reactions.

Heat treatment results in protein denaturation which is a desirable characteristic as enzymes causing loss in quality are deactivated. The nutritional property of a protein is not affected by its denaturation (Schwartz, 1992). Enzymes can cause off-flavor development (proteases, peroxidase, lipase, lipoxygenase, and catalase), texture changes and loss of consistency (amylase, cellulose, and polygalacturanase), color degradation (chlorophyllase, polyphenoloxidase, and peroxidase), and nutritional changes (ascorbic

acid oxidase and thiaminase). Peroxidase is highly temperature resistant and is therefore used as an indicator of adequate heat treatment. Heat treatment may partially denature the structure of an enzyme which results in reactivation of the enzymes during storage of the product. Peroxidase exhibits such a property during storage of aseptically processed peas in starch slurry (Schwartz, 1992). Amylase, which is responsible for thinning of starch based products, is another such heat resistant enzyme and is produced by microorganisms. Therefore, microbial load and enzyme structure are important in designing a process. In particulate products, heat resistant enzymes might be contained within the tissues of particles, and thus a stronger treatment to deactivate those enzymes may be necessary.

High heat treatments can result in off-flavor development. For example, the cooked flavor, in UHT milk, is why consumers choose pasteurized milk over aseptically processed milk (Schwartz, 1992). In aseptic processing of orange juice, some essential volatiles are lost during the vacuum concentration operation. Lost aroma and essential oils are then added prior to aseptic packaging. It was found that the main factor causing quality loss during storage of aseptic orange juice was not the interaction of the food with the packaging material, but the oxidation reaction and chemical degradation of flavoring components (Sizer *et al.*, 1988). However, Schwartz (1992) reported that the loss of D-Limonene flavor compound in orange juice was due to its adsorption onto the packaging film.

A review study by David (1992) summarized the differences in nutrient (ascorbic acid and thiamin) retention between retorted and aseptically processed soups. It was observed that 91 % of ascorbic acid was retained in aseptically packed tomato soup, whereas 59 % of ascorbic acid remained in conventionally packed product. In addition, to the retention of thiamin (82 %) was higher in aseptically packed chicken soup when compared to a similar product (27 %).

Fluid-to-particle heat transfer coefficient (h_{fp}) is a critical factor in aseptic processing of particulate foods to determine the extent of microbial destruction and nutrient degradation. A conservative mathematical model (Palazoglu and Sandeep, 2002) was developed to determine the effect of h_{fp} on lethality and nutrient retention. This study involved a computer program that could be used to calculate the lethality accumulated and the volume average nutrient (thiamine and lysine) retention during the heat treatment of food particles. For small particles ($r \leq 0.0075$ m), even for an infinite h_{fp}, thiamine retention was acceptable. However, for larger particles, increasing h_{fp} above a certain value resulted in reducing the overall nutrient content. It was reasoned that near-surface regions of the particles reached higher temperatures resulting in a decrease in the volume-average nutrient retention.

A study was conducted by Chandarana and Gavin (1989) to determine the retention of nutrients for aseptically processed heterogeneous foods. A mathematical model was developed to predict the heat transfer into particulate foods. The impact of thermal processing on the nutrient retention and the effect on the microbial load was

accessed. It was found that for large particles, the retention of thiamine and peroxidase was lower when compared to those for smaller size particles. In addition, it was observed that some spoilage enzymes could remain active within smaller particles due to the lower surface area available for enzyme inactivation. A study conducted by Storofos (1992) discussed the effects of various product and processing parameters on process optimization based on maximum nutrient retention. It was found that in small particles (diameter < 0.0127 cm) more thiamine was retained after processing when compared to large particles. In addition, upon decreasing fluid viscosity, thiamine retention increased. This increase may be due to the higher Reynolds number which results in higher heat transfer coefficients, and shorter processing times to accumulate the required lethality. No effect of particle concentration on thiamine retention was observed.

Inactivation of *Clostridium botulinum*, (target microorganism for low-acid foods) has an activation energy of 70 kcal/mole. Some of the reactions important in terms of food quality have the following activation energies -- Thiamine degradation: 27 kcal/mole, Vitamin A degradation: 14.6 kcal/mole, Maillard browning: 27 kcal/mole. These low activation energies indicate that these reactions are less temperature sensitive, i.e. the higher the temperature, the less damage product will receive (Nielsen *et al*., 1993).

2.3 Thermal Processing Kinetics and Microbial concerns

Inactivation of the microbes causing quality changes and of health concern is the mail goal of all thermal and non-thermal processing of foods. If a homogeneous

population of viable spores is subjected to a constant temperature, T, the rate of death of spores generally follows a first-order chemical reaction in which the reactant (viable spores) decreases exponentially or logarithmically with time t, and is given by (David, 1996):

$$\frac{-dN}{dt} = k_T N \tag{2.2}$$

This is equivalent in terms of common logarithms (to base 10) to:

$$\frac{-d(\log_{10} N)}{dt} = \frac{1}{D_T} \tag{2.3}$$

where $D_T = 2.303 / K_T$. The heat resistance parameter D_T is a characteristic of the type of spore, pH and water activity (a_w) of the heating medium, and laboratory protocol and procedures under consideration and is found experimentally to vary with temperature according to:

$$D_T = D_{T_{ref}} . 10^{(T_{ref} - T)/z} \tag{2.4}$$

where D_T is the D value at temperature T, T_{ref} is an arbitrary reference temperature, $D_{T_{ref}}$ is the D value at T_{ref} and z is a temperature dependent characteristic of the microorganism assumed to be constant over normal processing conditions. The temperature during the heating process can be know as function of time, T(t). When the initial and final count of the target microorganism is known (N_i and N_f), the thermal process can be evaluated over the starting and the end time (t_i and t_f). The integral equation for this process can be represented by considering integration of previous two equations and it can be easily seen that it leads to two expressions for the F value:

$$F^{z}_{T_{ref}} = D_{T_{ref}}(\log N_i - \log N_f) = \int_{t_i}^{t_f} 10^{(T(t)-T_{ref})/z}\,dt \tag{2.5}$$

F value of a heat process is the heating time at a specific temperature to achieve a required reduction in population of microorganisms. Here, N_f is a "safe" probabilistic final concentration of spores established from public healthy or economic spoilage rate considerations. The definition of a required F value and the process F value can thus be stated as:

$$(F^{z}_{T_{ref}})_{req'd} = F_{req'd} = D_{T_{ref}}(\log N_i - \log N_f) \tag{2.6}$$

$$(F^{z}_{T_{ref}})_{process} = F_{process} = \int_{t_i}^{t_f} 10^{(T(t)-T_{ref})/z}\,dt \tag{2.7}$$

Here F value at temperature T indicates that the entire integrated time-temperature effect on the spores is equivalent to the F value at the single temperature T_{ref}. Common reference temperatures are 250 °F for low-acid foods and 212 or 200 °F for high-acid and intermediate or acidified products (David, 1996). The lethality (L) of a thermal process is the ratio of the process F value to the required F value. Therefore:

$$L = \frac{(F^{z}_{T_{ref}})_{process}}{(F^{z}_{T_{ref}})_{req'd}} \tag{2.8}$$

Lethality must be at least unity for commercial sterility (Merson and Leonard, 1979). Traditionally, for *C. botulinum* spores, researchers have experimentally used N_i less than or equal to 10^{12} spores per milliliter with end point of N_f less than one spore per milliliter or probability of non-sterile unit given a $D_{250^{o}F}$ of 0.1 to 0.2 min and a z of 18 °F, giving rise to the 12D requirement for *Clostridium botulinum:*

$$F_{req'd} = D_{T_{ref}}(\log 10^{12} - \log 1) = 12D \tag{2.9}$$

Clostridium botulinum is an anerobe and is the target organism for canned foods because 1) when it grows, it can produce a deadly toxin, 2) It can be isolated from soil or water, and is present practically universially, and 3) it can produce spores, which are notably tolerant to heat, chemicals, and irradiation (Gavin and Weddig, 1995).

A thermal treatment process can only be optimized by integrating microorganism and nutrients/enzyme destruction kinetics with residence time distribution and heat transfer modeling (Ramaswamy *et al.*, 1997). One of the earliest works in this area is by Manson and Cullen (1974). In this study, a single point F value, integrated F value within the particulate, and total F value of the container were numerically computed for a system of swept surface heat exchangers. Effects of residence time, flow behavior index of the fluid, particle size, and particle concentration were found to be important in determining process lethality.

Sandeep *et al.* (1999) developed a finite difference program to compute the lethality of a process by taking residence times, particle concentration, and kinetics of the microbial reaction into consideration. Overall heat transfer coefficient and temperatures of the fluid and particles were calculated using another computer program. The effects of h_{fp}, residence time distribution (RTD), particle size, and concentration on overall heat transfer coefficient and fluid temperature were determined. It was concluded that an increase in particle size decreased the process F_0 value due to the longer time required to accumulate lethality at the center of a particle. Increase in particle concentration also decreased F_0, because higher particle concentrations resulted in less overall thermal

diffusivity, and hence a lower heating rate of the mixture. Wider RTD of particles resulted in lower F_0 values. It was pointed out that narrow RTD translated into a more uniform process and hence a higher value of lethality. As fluid-to-particle heat transfer coefficient increased, the center of the particle achieved the required time-temperature combination faster and more lethality was accumulated.

Bhamidipati and Singh (1994) studied the role of thermal time distributions on process effectiveness. A mathematical model was developed to obtain the temperatures and lethality values for fluid elements at different locations in a tubular heat exchanger. It was pointed out that although the lowest F_0 value is important in terms of process safety, the entire lethality distribution will give the information on destruction of nutrients. According to the results, at higher temperatures, spread of the thermal time distribution was higher which resulted in low nutrient retention. It was also found that the smaller the radius of the tube, the narrower the distribution and higher the degree of nutrients retained.

Yang *et al.* (1992) modeled the lethal effects of heat accumulated at the center of particles. Temperatures of the fluid and particle and lethality at the coldest point of particles were calculated based on residence time data. However, the analysis was not applied for multiple particle systems. Distribution of F_0 at the center was found to be wider as the h_{fp} decreased, which did not agree with the results of the study by Bhamidipati and Singh (1994).

Chandarana and Gavin (1989) compared three approaches to scheduling a commercial process for aseptically processed heterogeneous foods. These approaches – total system, F_0 hold, and hold only were considered. By using an explicit finite difference method, a mathematical model was developed to calculate fluid and particle temperatures and the lethality values accumulated at the center of particles. As far as the hold tube length calculations were concerned, the "total" and "F_0 hold" approaches did not result in significant differences. However, "hold only" approach resulted in a requirement of a longer tube length, since the temperature change and lethality accumulated in the heat exchanger were disregarded. In this approach, the F_0 at the center of a particle could go up to 78 minutes for a target value of 6.0 minutes which would result in over processing of the particles. However, as particle size increased, this effect was less pronounced.

In another study by Chandarana *et al.* (1989) the "F_0 hold" approach was used for a thermal process establishment. In this approach, it was accepted that the particle does not accumulate lethality in the heat exchanger but there is a temperature rise. By a mathematical model, effects of particle loading, particle shape, and thermal diffusivity of particles on lethality were studied. Two significant factors -- residence time distribution and h_{fp} were taken into consideration. The effects of these factors were modeled. Resulting required length of holding tubes to reach a predetermined target F_0 value at the center of the particle were presented.

David and Shoemaker (1985) designed a transducer, consisting of a thermocouple and an amplifier, to measure and collect lethality rates of a thermal process. A computerized data acquisition system was used to evaluate the transducer. Output from the transducer was recorded with a strip chart recorder and the area under the recorded curve was integrated to determine the F value of the process. Both the hardware and software of this system was easier to modify for different experimental setups than other commercial data loggers. However, its use for continuous systems needs further investigation.

Gratzek and Toledo (1993) determined the optimum process temperatures for maximum product quality. Enzyme destruction kinetics and volume-averaged cook values were modeled by Simpson's numerical integration technique. The overall cook value was found to decrease with decreasing particle size and increasing temperature. Optimum processing temperature increased as particle size decreased when enzyme inactivation was not considered. When peroxidase inactivation was taken into account, the temperatures for optimum cook values were lower for smaller particles.

2.4 Residence Time Distribution (RTD)

The residence time of a fluid is the time that it spends within the system boundaries (Bhamidipati and Singh, 1995). In continuous systems, each fluid element spends different lengths of time flowing through the heat exchanger or the holding section. Therefore, residence time distribution (RTD) is used to describe the time that

different elements spend in the system and the lethality of the process can be predicted according to the lethality received by each volume element of the product (Chambers and Nelson, 1993).

Residence time distribution (RTD) of particles in a continuous flow system is one critical factor in aseptic processing. Some particles in such a system remain longer than others, which is attributed to the slow moment of the particles close to wall or in a dead space when compared to those traveling along the centerline (Ramaswamy *et al*., 1995b). Residence time distribution of particles must be as narrow as possible in order to reduce the degree of over-processing and obtain a uniformly heated product.

Particle properties such as shape, size, and particle density affect its residence in a continuous system. If the particle and carrier fluid densities are similar, the particle is suspended and moves along the center of the tube, where the fluid velocity is the highest. A denser particle would stay longer in the holding tube as it would be dragged along the bottom wall.

RTD of a particle in a continuous system can be described by the E- and F-functions. The E-function is the fraction of the total material that exits the system. The F-function is the integral of the E-curve, obtained by plotting the E-function against time, and represents the fraction of a material at the exit of the system with a residence time t or less (Lareo *et al*., 1997).

2.4.1 Measurement Techniques of RTD in Two-Phase Flow

There are several studies which can be used to determine the residence time distribution of liquid or multiphase media. Reasons for measurement of RTD include determination of the fastest-moving particle, which is necessary for (a) designing a process using mathematical modeling to ensure commercial sterility, and (b) biological validation of a model. The biological test must contain a sample particle that represents the fastest-moving particle (Larkin, 1997).

RTD measurements are required because of the difficulty in non-invasive measurements of internal temperatures of particles during continuous flow. If a technique could be developed to measure the temperature of the slowest heating at the end of the heating and the holding tube for a multiphase continuous system, RTD measurements would be unnecessary (Larkin, 1997). Several methods were approved for determining the RTD of particles in a multiphase food in the NCFST-CAPPS workshop (CAPPS and NCFST Workshop, 1995, 1996). Among various types of methods used in determination of RTD some are optical methods, magnetic methods, magnetic resonance imaging, history methods (chemical markers and thermal memory cell), ultrasonic methods (doppler scattering), and salt tracers.

Optical methods include particle tracking velocimetery (Zitoun, 1996) and other visualization techniques. Magnetic methods involve introduction of tagged particles containing small magnets (Chandarana and Unverferth, 1996) which are detected by a voltage generated within coils at selected locations of the process equipment (Faraday's

law of electromotive induction). In this method, small magnets are embedded in particles to detect their presence at inlet and outlet of the system. Copper coils were wrapped around stainless steel tubing at the detection points. Passage of a magnet through the coils causes a sudden change in the EMF signal which is recorded by a data acquisition system. The elapsed time between the signal at the entry and the exit of the tube is sthe residence time of the particle. This method is however inapplicable to multiple particle systems due to its incapability to differentiate between multiple particles passing through the coils at the same time.

Magnetic resonance imaging has been used for flow visualization in food systems (Manavel *et al*., 1993). In ultrasonic methods, RTD of particles is determined by Doppler scattering of ultrasound waves by the moving particles. In the salt tracer method, RTD is detected by electrical conductivity measurements similar to approaches applied in chemical engineering for pure fluids.

In several multiphase studies, simulated food particles such as polystyrene spheres, alginate beads, and rubber cubes have been used (Sandeep and Zuritz, 1995; Palazoglu and Sandeep, 2002, 2004; Fairhurst *et al*., 1999; Dutta and Sastry, 1990a, b). Real food particles such as carrot and potato cubes (Ramaswamy *et al.*, 1992; Alhamdan and Sastry, 1997; Abdelrahim *et al.*, 1997) have also been used for multiphase studies. Residence time studies are important for the designing the holding tube in a continuous system. However it has been observed that if a thermal process is designed according to residence time estimates in the holding tube, excessive thermal treatment and loss of

texture and nutritional qualities would result (Chandarana and Unverferth, 1996). This study concentrated on an aseptic processing system consisting of a horizontal SSHE heater and cooler, and a stainless steel holding tube. The system operating temperatures were as high as 135 -140 ^{o}C, at which the residence time data was collected. Although a high particle concentration (15 % w/w) was used, no channeling or leapfrog effects were observed. The residence time data fit both Gamma and Lognormal probability distributions. These distributions allowed estimating the residence time of the fastest moving particle.

2.4.2 Effect of RTD on Sterilization Efficiency

The effect of RTD on lethality can be expressed by the following equation (Chambers and Nelson, 1993)

$$\frac{M}{M_{in}} = \int_0^{\infty} \exp(-k_m t)\, E(t)\, dt \qquad (2.10)$$

Where M and M_{in} are the spore concentrations after a time t and at initial time, respectively, k_m is the rate constant for spore destruction, and E(t) is the residence time distribution.

2.4.3 Factors Influencing RTD

The RTD of food particles is affected by factors related to the particles themselves (type, shape, size, density, and concentration), the carrier fluid (type, density, flow rate, concentration, and rheological properties), and the aseptic system such as configuration, temperature, mutator speed, and pumps (Ramaswamy *et al.,* 1995a, b).

Particles similar in density to the carrier fluid spend more time in the center of the flow and have shorter residence times. At the same conditions, for cube, a cylinder, and a sphere which have the same characteristic dimensions, the required length of the holding tube flows the order of cube > cylinder > sphere (Chandarana *et al.* 1989). In addition, smaller particulates require significantly shorter holding tubes. Reducing the particulate heat penetration distance in at least one dimension often results in significantly lower processing requirement (Mason and Cullen, 1974).

Sandeep *et al.* (1997a, 1997b, 1999) and Sandeep and Zurtiz (1991, 1993, 1995) studied the influence of flow rate, particle size and particle concentration on residence time distrubnution. Their results are summarized in Table 1.

Table 1. The effect of different parameters on RT_{mean}, RT_{min}, RT_{std}, and F_0*

Increase of variable	Changes in RT_{mean} and RT_{min}	Change in RT_{std}	Change in F_0
Viscosity	Decrease	Increase	No data
Flow rate	Decrease	Decrease	Decrease
Particle concentration	Increase or decrease depending on the experimental conditions	Decrease	Decrease
Particle size	Increase or decrease depending on the experimental conditions	Decrease	Decrease
h_{fp}	No data	No data	Increase

*Compiled from Sandeep and Zurtiz (1991, 1993, 1995); Sandeep *et. al.* (1997a, 1997b, 1999)

2.5 Mathematical Modeling

Since particle temperatures cannot be measured during continuous flow, mathematical modeling helps to deign the holding tube required for desired thermal processing. Modeling helps in overcoming the trial and error process for determining the hold tube size. With out modeling there could be substantial increase in cost of the experiments. In-addition, trial and error method can lead to inadequately simulated commercial practice, leading to disastrous consequences. Modeling simplifies identifying product and process parameters, which have a critical influence on the safety of the processed product. Thus critical control points can be determined while by varying the product and system parameters in the simulation (Sastry, 1997). Mathematical model can detect any unusual behavior during processing by comparing real physical behavior to the model. In addition, mathematical model assist in adjustments needed to correct process validation when a deviation occurs.

2.5.1 Steps in Modeling

A mathematical model is desired to know parameters such as the particle and fluid velocities, particle positions, orientations, and fluid particle temperature distributions. This is however not feasible due to the limited understanding of the basic physics, lack of precise knowledge of all initial conditions, and limitations in computational abilities. Thus it is important to simplify the assumptions while taking into account the worst scenarios when arriving at an acceptable model.

Most mathematical models (Sastry, 1986; Chandarana and Gavin, 1989), involve the following steps:

1. Prediction of Fluid Temperature: An energy balance on the fluid phase can be used to predict the fluid temperature. Energy enters or leaves the system via the walls and heat exchanger in form of heat.
2. Prediction of Representative Particle Temperature: The heat conduction equation is solved for a particle that is considered the representative of the system. This would be the slowest heating particle in the system. If the lethality for this particle is achieved then rest of the particles in the system can be considered to have acquired the required thermal treatment.
3. Iteration: Since the fluid temperature in step 1 is dependent on the particle temperature predicted in step 2 and vice versa, steps 1 and 2 are iterated until the temperature converges.

Above method simulates the fluid temperature distribution throughout the heater. It is then possible to conduct a separate set of simulations to identify the worst-case scenario by solving the heat conduction equation for the slowest-heating particle. For this, the previously determined temperature is used in a time-dependent convective boundary condition (Sastry, 1986; Chandarana and Gavin, 1989), along with the physical properties, characteristics, and time scales of the slowest-heating particle. In addition, Worst-case scenario simulations are needed to determining the holding tube size.

2.5.2 Determination of Fluid-to-particle Heat Transfer coefficient (h_{fp})

Aseptic processing of particulate foods has been hindered due to lack of the understanding of heat transfer between fluid and the particles in a continuous thermal processing system. The understanding becomes critical as it poses a difficult task of measuring the fluid-to-heat transfer coefficient without restricting the moment of the particles (Zareifard and Ramaswamy, 1999). Heat transfer from the fluid to the particles is a function of the thermal and rheological properties of the fluid and relative motion between the particle and the fluid. The boundary layer between the particle and the fluid is represented by a convective heat transfer coefficient (h_{fp}) at the interface (Chandarana *et al.*, 1989). The value of h_{fp} depends on many factors such as fluid viscosity and temperature, fluid-to-particle relative velocity, particle size, shape and location (Ramaswamy *et al.*, 1997).

Ramaswamy and Zareifard (2000) used a calorimetric technique to evaluate the effects of flow rate, viscosity, particle size, and temperature on fluid-to-particle heat transfer coefficient in a holding tube. A calorimeter was used to determine the bulk average temperature of a particle. Knowing the medium temperature, initial particle temperature, residence time, and the bulk temperature of the particle, fluid-to-particle heat transfer coefficient was calculated. An interesting result of this study was the effect of temperature on h_{fp}. It was observed that as the temperature increased h_{fp} decreased. The reason behind this was attributed to the fact that at lower temperature ranges (50 - 70 ^{o}C), particle velocity decreased as temperature increased due to a decrease in fluid viscosity and loss in drag force.

Various techniques have been used to estimate h_{fp} under flow conditions. The methods that resist the particle motion are termed invasive methods, while non-invasive methods involve free flow of a particle. Invasive methods include the thermocouple (Zuritz *et al.*, 1987), and the moving thermocouple (Sastry *et al*., 1990) technique. In the thermocouple method, temperature of a stationary particle in a flowing stream is measured, whereas in the moving thermocouple, a particle with a thermocouple attached is moved through the system at predetermined velocities. Both methods provide the opportunity of recording the particle temperature and using opaque carrier fluids. However, since the thermocouple attached to the particle restricts the particle movement, the h_{fp} calculated will deviate from its real value (Masesmans *et al.*, 1992; Sastry and Cornelius, 2002).

Non-invasive methods consist of the relative method (Balasubramaniam and Sastry, 1994b), microbiological method (Hersom and Shore, 1981), use of time-temperature integrators (Weng *et al*., 1992), remote temperature sensor method (Balasubramaniam and Sastry, 1996b), liquid crystal method (Stoforos and Merson, 1991), melting point indicator method (Mwangi *et al*. 1993), and phase change technique.

The relative velocity method involves determination of the fluid-to-particle relative velocities, where very fine particles (tracers) are introduced into the system, and the relative notion between the tracers and the particle is recorded (Balasubramaniam and Sastry, 1994b). This method involves introducing a test particle into the fluid medium in a transparent section and videotaping it as it moves through the system. The video is

replayed to determine the relative velocity by finding the time required for a selected tracer particle to pass over a transducer particle. Relative velocity data is used to back-calculate h_{fp} by various dimensionless correlations (Sastry and Cornelius, 2002).

Since the fine particles are assumed to travel with the fluid velocity, the difference between the velocities of the tracers and the particle yields the fluid-to-particle relative velocity, h_{fp} can be calculated using one of the empirical Nusselt number correlations given in the literature. One such correlation was suggested by Ranz and Marshall for external flow over a spherical body (1952):

$$Nu = 2.0 + 0.6\,Re_p^{0.5}\,Pr^{0.33} \tag{2.11}$$

Balasubramaniam and Sastry (1996b) utilized coil circuit sensors around a pipe to sense the temperature-dependent resonation of a quartz crystal placed in a particle. The method is known as the remote temperature sensor method. The particle flow is not restricted, but density compensation is a problem due to the far greater density of quartz crystals. In addition, since quartz crystals cannot withstand high temperatures, the method is not applicable at aseptic processing temperatures.

A noninvasive method which was developed by Storofos and Merson (1991) is the use of liquid crystals as temperature sensors. The method involves application of a liquid crystal to the surface of a transducer particle and observing the color change as a function of temperature. Color-temperature response is calibrated, and the time-temperature-history can be obtained by digital image analysis. The method requires the use of transparent tubes and carrier fluids. Since the color change is reversible, liquid

crystals can be used multiple times. The experimental system consisted of a cylindrical acrylic container filled with aqueous suspensions of encapsulated liquid crystals. Color changes of liquid crystal coated particles were determined as the container was heated up from an initial temperature of 20 ^{o}C. There are two basic equations to be solved to determine h_{fp}. The first one is an overall energy balance equation on the container using a lumped parameter approach – energy exchanged between the heating medium and the liquid in the container is equal to the energy accumulated within the fluid and the particle. The second one is the energy balance on the particles – convective heat transfer from the liquid in the container to the particles is equal to the energy accumulated within the particles.

Zitoun and Sastry (1994b) also used the liquid crystal method in order to investigate h_{fp} between carboxymethlycellulose (CMC) solution and aluminum hollow spherical particles. Flow rate, viscosity, and particle size were the experimental parameters. The authors compared the results of the liquid crystal method to those from the particle tracking velocimetry method. It was found that h_{fp} increased with increasing flow rate, decreasing viscosity and decreasing particle size in both of the methods.

Melting point indicators are similar to liquid crystals in that it changes color with temperature. However, the color change of melting point indicators is irreversible, and occurs at a specific temperature. The method requires transparent tubes and clear carrier fluids, and also transparent particles if the melting point indicator is placed inside a particle (Mwangi *et al.,* 1993). In another technique, phase changes are involved during particulate flow. Studies have been conducted to determine h_{fp} utilizing the change in

particle mass during flow and equating the energy supplied to the particle to the energy of phase change.

A new approach to determine h_{fp} is the use of magnetic resonance imaging. Although this method has some advantages in terms of being non-invasive and being applicable to multiple particles, there are some disadvantages such as the need to immobilize the particle and lack of residence time information on the particles which are being monitored (Sastry and Cornelius, 2002).

Heppell (1985b) used a microbiological method to determine h_{fp}. In this method, spores of *Bacillus stearothermophilus* were entrapped in calcium alginate beads and introduced into the system. The number of surviving spores at the end of the process was determined. Reduction in bacterial count is attributed to a time-temperature history, which could be used to determine h_{fp} by a mathematical model. This method is noninvasive, and can be applied to real foods and processing systems. However, only a single end point microbiological determination is used in constructing the entire time-temperature history, which may result in errors (Sastry and Cornelius, 2002).

Chemical and enzymatic indicators have been proposed as alternatives for hfp estimation as well as for process validation (Ramaswamy *et al*., 1996). Kim and Taub (1993) discussed the possible application of compounds that are produced within the food as a marker to validate the sterility. Three carbohydrate compounds formed within carrots, meats, and juice drinks upon thermal treatment were of interest in this study. It

was suggested that monitoring the change in the concentration profiles of these compounds can be used to calculate h_{fp}. In spite of being noninvasive, lack of residence time data and uncertainty of the location of the critical particle may restrict the use of this method.

A recent method to measure h_{fp} is the use of ablation (Tessner *et al*., 2001). In this method, the change in mass of ice was used as an ablation probe. The amount of solid ablated was directly proportional to the energy flux across the fluid-solid interface with constant h_{fp}. A lot of research has been conducted on determination of fluid-to-particle heat transfer coefficient. However, if it was possible to measure the time-temperature history of the cold spot of the critical particle, then measurement of h_{fp} would not be of concern.

Åström and Bark (1994) showed that a 50% change in heat transfer coefficient resulted in significant effect on the required holding tube length. Therefore, an accurate determination of h_{fp} is necessary for process design and optimization. As can be seen from the above discussion, each method has its own limitation, and there is no universal method that can be used under all conditions. Thus, the accurate determination of h_{fp} in continuous flow systems still remains a challenge.

2.5.3 Acceptable features of model

The mathematical model developed must have minimum acceptable features at the heater section, hold tube, and the cooling section. For heater section a thermally

mixed systems is considered. Some examples of such a system is a pure fluids in turbulent flow or solid-liquid mixtures in swept-surface heat exchangers, where radial temperature variations are eliminated by mixing. In such a system (1) energy balance must be conducted on the fluid, combined with temperature measurement at the inlet and outlet of the heater; (2) the value of the fluid-particle convective heat transfer coefficient h_{fp} that yields the best agreement with measured inlet and outlet fluid temperatures must be determined by trial-and-error method or by some numerical procedure; and (3) a check must be conducted to ensure that the estimate of h_{fp} is realistic (Balasubramaniam and Sastry 1994a, b, 1996a, b; Cacace *et al.*, 1994; Alhamdan, 1995; Godonna *et al.* 1996; and others). When the heater section does not have a uniform thermal mixing other conditions are applied for minimum acceptance features of the model which verifying fluid temperature predictions along the tube axis with measuring the fluid temperatures at intermediate points along the heater.

2.6 Biological Validation

Biological validation of scheduled process for traditionally processed canned foods is not required if the filed process is based on properly designed heat penetration tests (Chandarana, 1992). For aseptically processed particulate products, however, biological validation of the process in necessary, simply because the design of the aseptic process is not based on heat penetration tests due to the unavailability of the time-temperature history of the product. In-addition to this, biological validation of mathematical model in aseptic processing is crucial to support the validity of the

mathematical model and to document the lethality delivered to the slowest heating zone of the particle.

Biological validation of an aseptic process for a particulate food product involves the use of biological indicator that travel through the system inside a particle. It is recovered intact at the exit of the system and tested quantitatively to determine the heat treatment received by the particle. An acceptable quantitative biological validation verifies that the target F_0 value has been delivered to the cold spot (Pflug *et al.*, 1990).

To access the heat delivered to the center of a large particle, a biological thermocouple system can be used. In the biological thermocouple method, suspended spores are encapsulated in a carried and do not come into contact with the food product (Marcy, 1997). Hence, the measurement is not affected by the pH, oxidation-reduction potential, and nutrient condition of the food (Pflug *et al.*, 1990). After recovering the carrier including the bacterial spores, the surviving spores are quantified. Using the calibration curve obtained from the plotting the F_0 value vs. surviving spores, the sterilization value achieved at the center of the particle is determined. The advantages of this method are as follows: (1) if the carrier is recovered then all the spores are recovered with it; (2) the calibration curve can be set up with bacterial spores in their carriers independent of the food product; (3) the carrier including the spores is located near the geometric center of the particle; (4) since a calibration curve procedure is used, the number of survivors can be determined for a given F_0 value (Pflug *et al.*, 1990).

Simulated particles, instead of the actual food particles, inoculated with bacterial spores have also been used for biological validation. Again, as in the use of actual food particles must be quantified when using this method to determine the effective initial spore concentration. Another issue that must be addressed is that the conditions (water activity, oxidation-reduction potential, ionic concentration, and type of nutrient) must be identical during calibration and validation. In contrast to actual food particles, simulated particles normally have a uniform inoculum distribution, and their size is uniform and controlled. Pflug *et al.* (1990) stated that when particle (actual and simulated) inoculated with bacterial spores are used; the sterilization value obtained will be an integrated sterilization value rather than the sterilization value at the center of the particle.

Hersom and Shore (1981) placed spores in capillary glass beads, which were then embedded within carrot cubes. Segner *et. al.* (1989) inoculated turkey cubes with spores of *Clostridium sporogenes* using the "knotted string" method. The method of inoculation was reported to be an effective way of placing the inoculum as close to the geometric center of the particle as possible. The use of alginate gels as simulated particle has been studied by Sastry *et al.* (1988) who suspended bacterial spores in an alginate solution and immobilized them upon gelation of the alginate. Bhamidipati and Singh (1996) also used alginate spheres as model food particles and reported that the thermal response characteristics of the simulated particles were similar to those of real food particles.

In an attempt to avoid the problems associated with using bacterial spores and to reduce effort and time of evaluation, some researchers used compounds that undergo

reduction upon heat treatment. Bhamidipati and Singh (1996) used peroxides as a bio-indicator in alginate spheres to verify a mathematical model developed for aseptic processing evaluation. They used the D-values of peroxidase determined in TDT cans at temperatures from 100 to 130 °C in aseptic processing simulations. Their model calculated the mass-averaged peroxidase concentration assuming an even distribution of peroxidase throughout each particle at the beginning of the process and that the peroxidase throughout each particle at the beginning of the process and the peroxidase inactivation followed the same kinetics as the TDT cans. They compared the predicted valued with the spectrophotometrically obtained experimental values, and reported that the difference was not significant ($\alpha = 0.05$) for 110 and 120 °C. However, they observed significant differences at 130 °C ($\alpha = 0.05$), which they attributed to the correlation used for calculating the h_{fp} being less accurate at higher temperatures. Predicted values were generally higher indicating that the simulation would yield a safe prediction while most likely resulting in over-processing. They suggested that alginate sphere with peroxidase would be suitable model system for an aseptic process evaluation.

Kim and Taub (1993) identified three compounds that formed from carbohydrates and proteins upon heating broccoli, beef and fruits and vegetables. They suggested that the formation of these compounds can be used as an indicator of the degree of heat penetration within a particle at aseptic processing temperatures, and since their formation followed the first-order kinetics, the determination of the marker concentration in sections of particles would provide information on lethality distribution within the particle.

Bernard *et al.* (1990) explained the steps a processor should follow when filing an aseptic process with the appropriate regulatory agency. Once the sterilization process has been established, inoculated packs of product are processed for confirmation of the system.Then, lethality delivered to the system is calculated and compared to the results of the inoculated pack study. Microorganisms which can be used in verification of the process are chosen according to the sterilizing medium -- for superheated steam *Bacillus stearothermophilus* is chosen as the test microorganism.

Sadeghi and Swartzel (1987) proposed to use encapsulated and immobilized calibration materials to establish the equivalent point method developed by Swartzel (1982). The equivalent point technique describes thermal systems with one time and temperature, independent of an activation energy. In Sadeghi's study, sucrose solution was immobilized first in calcium alginate spheres and then in polymethylmethacrylate. Immobilized materials were encapsulated in stainless steel and aluminum cylinders. Thermal characteristics of these particles were determined after processing them in retort and obtaining concentration data. Residence time distribution was established based on concentration changes. Further application of this procedure to continuous systems has potential for determination of slowest and fastest moving particles as well as the effects of residence time, shear stress, Reynolds and Nusselt numbers on concentration changes, and hence time-temperature equivalent points and lethality.

Ramaswamy *et al.* (1996) used immobilized bovine pancreas trypsin (type III) to verify a finite difference model developed to determine the fluid-to-particle heat transfer

coefficient in a holding tube. The enzyme was immobilized in a steel capsule and embedded in cylindrical potato particles. The retention of enzyme estimated from the finite difference
program was compared to the results of actual enzyme assays. Predicted and measured values were in good agreement. As a result, using encapsulated enzyme as a method of verification of heat transfer coefficients was shown to be promising.

Use of chemical indicators to validate aseptic processing has also been studied. Examining chemical compounds in foods have been shown to be a potential alternative for assessment of the lethality accumulated in the particle. The chemical indicator to be used is chosen according to simplicity and effectiveness of the instrumentation, sensitivity of the indicator to changing environmental conditions, and thermal stability of the indicator at aseptic processing conditions (Ramaswamy *et al*., 1997). Although microbiological verification will finally be required to establish the process, using chemical indicators can save time and serve as a starting point (Bhamidipati and Singh, 1996).

In the case study for condensed cream of potato soup, biological verification of an aseptic process was discussed (CAPPS and NCFST Workshop, 1995, 1996). It was pointed out that the test microorganism should have a D value which is not too low (to ensure there are survivors retrieved) and not too high (to eliminate improbable results). Two inoculation methods were discussed -- count reduction and inoculated pack methods. In count reduction method, a known number of microorganisms are implanted into the center of a food particle. After the particle passes through the aseptic processing

system, number of surviving spores is determined, and the lethality of the process is established. In this method, additional labor to collect inoculated particles and enumerate the survivors is required. In the inoculated pack method, relatively larger number of particles is inoculated to ensure that an inoculated particle is in the containers to be incubated. The disadvantage of this method is the longer times required for incubation.

Thermal properties of food are important to predict the heat transfer rates in the food materials under various processing conditions. For validation of multiphase aseptic process of low-acid food, real food particles and simulated food particles are compared for thermal behaviors at the geometric center of the food materials. Thermal properties such as specific heat, thermal conductivity, and thermal diffusivity are important for studying heating behaviors for various food particles. Early heat transfer analyses for heating or cooling food products required uniform values of thermal properties. These analyses were typically oversimplified and inaccurate. Present-day analytical techniques such as the finite-element method are much more sophisticated and can account for non-uniform thermal properties, which change with time, temperature, and location as the food product is heated or cooled. This greatly increases the demand for more accurate thermal property data as it is important to know how thermal properties change during a heating process (Sweat, 1995). Thermal diffusivity is one of the important properties describing the thermal behavior of food. Thermal diffusivity is the ability of a food to conduct heat to adjacent molecules, serving as a measurement of the quantity of heat absorbed by a food at a given temperature (Dickerson and Read, 1968).

2.7 Developments in techniques for validation of multiphase aseptic processes

Validation techniques for multiphase aseptic processing are in their development stage in the U.S. Validation is a very important issue concerning multiphase aseptic processing, and is employed for ensuring the safety of the aseptic process. In Europe, aseptically processed chunky soups – as well as various stews and sauces – have been a household staple for nearly 30 years. In the U.S, however, multiphase processing of food prepared by aseptic methods must be scientifically validated. Recent research in this area has focused to reduce the cost of validation for such process (Morris-Lee, 2004).

A recent aseptic system developed by NCFST uses magnetic resonance to measure residence time and temperatures for the fastest moving and largest particulates by placing small magnets in their center and recording the signals at various points in the system (Higgins, 2004). Signal data then is extrapolated to determine temperature. However, since the electronic noise in a commercial plant interferes with the signals, it makes validation difficult. A possible solution is to insert micro thermometers into the particles, and then use X-rays to capture temperature readings. Thus, recent work on micro thermometry has attracted attention. The technology was developed for the use in medical science (Reiffel, 2001; Higgins, 2004). Expanding fluid thermometers are implanted in a body – for example in and adjacent to a cancerous tumor – and the temperatures are recorded using x-ray imaging. However, this can also be used for real time-temperature measurements in food particulates for validation process. Thermometers of 100 to 200 microns in size were placed within the human body near a cancerous tumor before radiation treatment. This technology provides technicians with

highly accurate, real-time temperature readings for the surrounding tissue, to minimize the damage to healthy organs.

Beller (1993) developed a method and apparatus for the in-process measurement of internal particulate temperature utilizing ultrasonic tomography techniques to determine the speed of sound through a specimen material. In this technique, ultrasonic pulses are transmitted through a material (multi-phase), over known paths and the ultrasonic pulse transit times through all sectors of the specimen are measured to determine the speed of sound. The speed of sound being a function of temperature, it is possible to establish the correlation between speed of sound and temperature, throughout a cross-section of the material, which correlation is programmed into a computer to provide for a continuous in-process measurement of temperature throughout the specimen.

Ghiron and Litchfield (1997) demonstrated that by inserting small paramagnetic particles in the interior of simulated and real food particles, the local temperature can be measured. The measurements were done by directing the food material flow through a magnetic field and sensing the voltages induced in a pickup coil by the motion of the magnetized particles. An aseptic food processing system including a non-invasive temperature measurement apparatus capable of processing low acid particulate foods was later demonstrated by Ghiron and Litchfield (1998). A magnetic particle is inserted into a food particle in the aseptic system and undergoes heating corresponding to a cold zone in the food product being processed. After heating, the particle is passed by one or more magnetic sensors along the food flow path. The magnetic strength of the particle is in

proportion to its magnetic susceptibility, which is recorded by the sensors, as the particle flows. The particle material is chosen to be paramagnetic at processing temperature ranges, and produces a magnetization in a sensor coil in the sensor which is proportional to the magnetic susceptibility. Because the susceptibility varies with temperature, actual temperature measurements of cold zone heating within the food particles can be obtained. The system may include a particle injector to inject the particles at a desired time, sensors to detect the temperature, data storage and process control based upon detected temperatures and a particle extractor.

A method based on change in proton precession frequency with temperature was compared with temperature measurement by T1-weighted imaging (Kantt *et. al.*, 1997). Temperature maps of a gel and of cooked and raw red potatoes were developed during heating from 20 to 60 °C. To measure change in proton precession frequency, a steady state free precession sequence allowed 2-dimensional acquisitions in 8 s with a spatial resolution of 0.88 mm^2. Temperature resolution with chemical shift imaging, ranged from 0.3 to 3 °C, decreasing with increasing temperature due to a decrease in signal to noise ratio. The difference by this method and thermocouples was < 5 °C. T1-weighted spin-echo images showed artifacts due to an inhomogeneous T1 distribution within the potatoes, indicating chemical shift was more reliable for inhomogeneous foods.

Kantt *et. al.* (1998) used Magnetic Resonance Imaging (MRI) was to obtain two-dimensional temperature maps in potatoes undergoing aseptic processing. The change in precession frequency of protons served as the temperature indicator. Larger particles (6.9

and 3.84 cm^3) exhibited a ΔT ($T_{surface}$ - T_{center}) of up to 22 ± 0.4 ºC, 45 s after exiting the heat exchanger with the ΔT (T_{outlet} - T_{inlet}) of the carrier fluid in the heat exchanger at 30 to 45 °C. No changes in temperature were measured between the center and the surface of particles with size less than 2.05 cm^3 (pumped at < 22.7 l/min). The average fluid to particle convective heat transfer coefficient (h_{fp}) in the heat exchanger and holding tube was calculated using a finite element method. h_{fp} ranged from 600 to 2500 W/m^2 °K for large particles (6.9 cm^3 cubes) and was greater than 3000 W/m^2 °K for the smaller particles.

Saksena (2001) developed a non-contact system for effectively approximating the internal temperature of a food being cooked on a grill or griddle. Non-contact measurement devices such as ultrasound or infrared were directly installed onto the cooking apparatus. These devices were use to monitor the status of the food being cooked, or to control the heat input to the cooking surface using a feedback loop. The non-contact measurement devices may be permanently positioned above the cooking surface or may be mounted to arms that can be pivoted, or otherwise moved into position over the cooking surface.

Guiavarc'h *et. al.* (2002a) studied the thermal stability of *Bacillus licheniformis* – which can breakdown amylose at low moisture content – during isothermal experiments performed in a temperature range 113 to 125 °C. Thermal inactivation was monitored by measuring the decrease in enthalpy during thermal denaturation and/or by measuring the decrease in enzymatic activity on p-nitrophenyl – a-D-maltoheptaoside, or on starch as a

substrate. Based on enthalpy readings, an enzymatic system with a z-value of 10.4 °C was determined, at a relative humidity of 81% (at 4 °C). A theoretical study showed that this system could be used as a Time Temperature Integrator (TTI) to monitor the safety of sterilization processes of numerous food products.

Heat denaturation kinetics of *Bacillus licheniformis* R-amylase, equilibrated at 81% equilibrium relative humidity at 4 °C (BLA81), was studied by Guiavarc'h *et. al.* (2002b). The study was conducted in isothermal and non-isothermal conditions by monitoring the decrease in enthalpy associated with the thermal denaturation of the enzyme. Due to its low water content, BLA81 denaturation could be studied in the range of 118-124 °C. Two batches of BLA81 were successfully validated under non-isothermal conditions allowing the determinations of F_0 in the range of 1-15 min. In a second step, BLA81 was used as a time-temperature integrator (TTI) to investigate potential differences of process values received by freely moving spherical particles as compared to a centrally fixed particle (single-position impact) inside cans containing brine water. Results showed that the F_0 value received by freely moving particles can be from 5.6% (4 rpm) to 19.7% (8 rpm) smaller than the F_0 value received by the centrally fixed sphere. This means that evaluating the process value by means of a particle fixed at the critical point in a package can lead to potential overestimations of the F_0 values with possible hazardous safety implications. This study highlighted the potentials of TTI technology to record the time-temperature history for monitoring the safety of thermally processed foods.

Monitoring and validation of continuous thermal pasteurization and sterilization processes require the use of simulated food particles which must exhibit conservative flow and thermal characteristics. A magnetically detectable particle and related methods, systems, and devices are provided for obtaining a temperature measurement for a batch or a continuous stream of material (Palazoglu *et. al.*, 2004). Simulated food particles (having the same shape and size of food particles or biomaterial present in the processed product) are designed and fabricated from plastic polymer materials to meet these design criteria, where the determination of wall thickness of the carrier particle becomes very critical for the safety and quality of the product to be processed. Using the Conservative Particle Design (CPD) software in the design stage of a process, the appropriate (minimum) wall thickness, which provides the highest product quality possible, while ensuring the safety of the processed product was determined (Palazoglu *et. al.*, 2004).

A method was developed by Simunovic *et. al.* (2004) for obtaining a temperature measurement for a batch or a continuous stream of material. The method includes a particle capable of generating a signal that changes at a pre-determined temperature, inserting the particle into the batch or continuous stream, and detecting a change in the signal from the particle to thereby obtain a temperature measurement for the batch or continuous stream.

In addition to temperature measurements, residence time determination is an important part of aseptic process validations. There are various models in use by in industry for calculating residence time for aseptic processing of particulate foods. One

such model is Asepti CAL which has been developed by FMC FoodTech (Higgins, 2004). In a recent research work a method to determine residence time measurement of a particulate-containing food product while passing the product as a continuous stream through a thermal processing apparatus was developed (Swartzel and Simunovic, 1999, 2000, 2003, 2004). The method involves inserting at least one particle tagged with at least one magnetic implant into the food stream at pre-selected intervals. In addition, it includes a method for detecting at least one magnetic implant using at least one magnetic sensor located at a detection point downstream from the location of particle insertion. This system can be used to determine the time of passage of at least one detectable particle in the stream using output from at least one sensor, and generating a residence time measurement for the stream using the time of passage for the at least one detectable particle. The study also describes a method for use of multiple magnetic sensors for detecting the implants, and describes the detectable particle used for carrying implant.

A workshop (CAPPS and NCFST Workshop, 1995, 1996) was conducted to discuss the issues involved in multiphase aseptic processing of food. In an attempt to validate a multiphase aseptic process for the first time, a system and method was filed (with FDA) by Tetra Pak for thermal processing of diced potato (in modified food starch). Density-adjusted chicken-alginate cubes were prepared for use in the bio-validation step. These particles were shown to have a residence time which was sufficient for receiving desired thermal treatment. It received a no-objection letter from FDA (Palaniappan and Sizer, 1997) and was an important milestone in the area of multiphase aseptic processing. However, the cost of validation of this process is very high.

Therefore, reducing the cost for validation techniques has been a key area of focus in multiphase aseptic processing.

Thus, from the recent research work it is clear that study of heat received by a simulated food particle is crucial in multiphase aseptic processes. It is also clear that there has been an emphasis on developing methods to determine time-temperature history, and residence time distribution, for such particles in a multiphase aseptic system. However, a technique to build such simulated food particles that exhibits a "conservative behavior" is still not available. Conservative behavior of a particle in this study would mean that the particle heats up the slowest (due to its low thermal diffusivity) and receives least thermal treatment (because it is the fastest). These particles are of utmost importance in understanding a multiphase aseptic process because they represent the real food particles in aseptic system, which gathers the least thermal treatment. Thus, if we ensure that this conservative particle receives the minimum required thermal treatment, we would ensure that all the particles in the processed food receive a thermal treatment equal to or greater than the simulated food particle, thereby ensuring the safety of the product. Systematic procedures for design, fabrication, and testing of simulated food particles that would be used for experimental validation of multiphase aseptic processing in foods are desired. Availability and reduced cost of such particles would reduce the complexity of process documentation and filing with regulatory agencies and bring aseptically multiphase foods closer to commercial reality. The study of conservative behavior of this "critical particle" is the key to the present research. The current study was thus undertaken with the goal of developing a validated systematic approach for the construction of simulated particles

that would ensure conservative thermal behavior (slow-heating behavior) and near-neutral buoyancy (fast-flowing behavior).

2.8 Objectives

The main objectives of the current research are:

- To develop a systematic approach for construction of nearly neutrally buoyant simulated particles with magnetic implants that exhibit conservative thermal behavior
- To experimentally validate the conservative thermal behavior of particle constructed using the above methodology

SYMBOLS

D-value	decimal reduction time	min
F_0	sterilization value	min
h_{fp}	fluid-to-particle heat transfer coefficient	W/m^2-K
T	temperature	°C
t	time	min
z-value	temperature change required to change D-value by a factor of ten	°C

Subscripts

min	minimum
ref	reference
std	standard deviation

Abbreviations

CAPPS	Center for Advanced Processing and Packaging Studies
CFR	Code of Federal Regulation
CIP	clean in place
CPD	Conservative Particle Design
EMF	electromotive force
FDA	Food and Drug Administration
HEPA	high efficiency particulate air
HTST	high temperature-short time
MID/TIP	Magnetic I. D. / Timed Insertion Particle
MRI	magnetic resonance imaging
NCFST	National Center for Food Safety and Technology
PMP	polymethylpentene
PP	polypropylene
RT	residence time
RTD	residence time distribution
SSHE	scraped surface heat exchangers
TTI	time temperature integrator
UHT	ultra high temperature

REFERENCES

21 CFR 113.3. In "Thermally processed low-acid foods packaged in hermetically sealed containers". FDA Code of federal regulation. Title 21. Part 113.

21 CFR 114.3. In "Acidified foods". FDA Code of federal regulation. Title 21. Part 114.

Abdelrahim, K.A., Ramaswamy, H.S., and Marcotte, M. 1997. Residence time distribution of meat and carrot cubes in the holding tube of an aseptic processing system. Lebensmittel-Wissenschaft und -Technologie. Vol. 30: 9-22.

Alhamdan, A. 1995. Particle residence time distribution and bulk heat transfer coefficients of two-phase flow in scraped surface heat exchanger and holding tubes. Ph.D. dissertation, Ohio State University, Columbus, OH.

Alhamdan, A., Sastry, S.K. 1997. Residence time distribution of food and simulated particles in a holding tube. Journal of Food Engineering. Vol. 34: 271-292.

Åström, A., Bark, G. 1994. Heat transfer between fluid and particles in aseptic processing. Journal of Food Engineering. Vol. 21(1): 97-125.

Balasubramaniam, V.M., Sastry, S.K. 1994a. Liquid-to-particle heat transfer in continuous flow through a horizontal scraped surface heat transfer in continuous flow through a horizontal scraped surface heat exchanger. Food and Bio-products Processing, Transactions of the Institution of Chemical Engineers. Vol. 71(C): 189-196.

Balasubramaniam, V.M., Sastry, S.K. 1994b. Convective heat transfer at particle-liquid interface in continuous tube flow at elevated fluid temperatures. Journal of Food Science. Vol. 58: 675-681.

Balasubramaniam, V.M., Sastry, S.K. 1994c. Liquid-to-particle heat transfer in non-Newtonian carrier medium during continuous tube flow. Journal of Food Engineering. Vol. 23: 169-187.

Balasubramaniam, V.M., Sastry, S.K. 1996a. Liquid-to-particle heat transfer in continuous tube flow: Comparison between experimental techniques. International Journal of Food Science and Technology. Vol. 31: 177-187.

Balasubramaniam, V.M., Sastry, S.K. 1996b. Estimation of convective heat transfer between fluid and particle in continuous flow using a remote temperature sensor. Journal of Food Process Engineering. Vol. 19: 233-240.

Ball, C.O., Olson, F.C.W. 1957. Sterilization in Food Technology: theory, practice and calculations. McGraw-Hill Book Company Inc., New York. pp. 1-26.

Beller, L.S. 1993. Ultrasonic tomography for in-process measurements of temperature in a multi-phase medium. U.S. Patent No. 5,181,778. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Bernard, D.T., Gavin III, V.N., Shafer, B.D., Stevenson, K.E., Unverferth, J.A., and Chandarana, D.I. 1990. Validation of aseptic processing and packaging. Food Technology. Vol. 44(2): 119-122.

Berry, M.R. 1989. Predicting the fastest particle residence time. First International Conference on Aseptic Processing Technologies. Indianapolis, IN, March 19-21.

Bhamidipati, S., Singh, R.K. 1994. Thermal time distributions in tubular heat exchangers during aseptic processing of fluid foods. Biotechnology Progress. Vol. 10: 230-236.

Bhamidipati, S., Singh, R.K. 1995. Design considerations in aseptic processing of foods. In "Food process design and evaluation" (Edited by R.K. Singh). Technomic Publishing Co. Inc., Lancaster, PA. pp. 89-124.

Bhamidipati, S., Singh, R.K. 1996. Model system for aseptic processing of particulate foods using peroxidase. Journal of Food Science. Vol. 61(1): 171-175, 179.

Burton, H. 1988. Ultra-high-temperature processing of milk and milk products. Elsevier Applied Science, London. pp. 141-145, 171-173.

CAPPS and NCFST Workshop. 1995, 1996. Case study for condensed cream of potato soup from the aseptic processing of multiphase foods workshops. November 14-15, March 12-13.

Cacase, D., Palmieri, L., Pirone, G., and Dipollina, G. 1994. Biological validation of mathematical modeling of the thermal processing of particulate foods: The influence of heat transfer coefficient determination. Journal of Food Engineering. Vol. 23: 51-68.

Carlson, V.R. 1991. Enhancement of heat transfer in heat exchangers for aseptic processing. ASAE, Paper No. 916608. St. Joseph, MI.

Carlson, V.R. 1996. Food Plant Organization- Present Operations. In "Aseptic processing and packaging of food, a food industry perspective". Edited by David, J.R.D., Graves, R.H., Carlson, V.R. CRC Press, Inc., Boca Raton, FL. pp. 51-95.

Chambers, J.V., Nelson, P.E. 1993. Principles for Aseptic Processing and Packaging. The Food Processors Institute, Washington, D.C. pp. 22-45.

Chandarana, D.I. 1992. Acceptance procedures for aseptically processed particulate foods. In "Advances in aseptic processing technologies". Edited by Singh, R. K., Nelson, P.E. Elsevier Applied Sciences, London. pp. 261-278.

Chandarana, D.I., Gavin, A. 1989. Establishing thermal processes for heterogeneous foods to be processed aseptically: a theoretical comparison of process development. Journal of Food Science. Vol. 54(1): 198-204.

Chandarana, D.I., Gavin, A., and Wheaton, F.W. 1989. Simulation of parameters for modeling aseptic processing of foods containing particulates. Food Technology. Vol. 43(3): 137-143.

Chandarana, D.I., Gavin, A., and Wheaton, F.W. 1990. Particle fluid interface heat transfer coefficient under UHT conditions at low particle/fluid relative velocities. Journal of Food Process Engineering. Vol. 13: 191-206.

Chandarana, D.I., Unverferth, J.A. 1996. Residence time distribution of particulate foods at aseptic processing temperatures. Journal of Food Engineering. Vol. 28: 349-360.

Coronel, P., Sandeep, K.P. 2003. Pressure drop and friction factor in helical heat exchangers under nonisothermal and turbulent flow conditions. Journal of Food Process Engineering. Vol. 26: 285-302.

Cousin, M.A. 1993. Microbiology of aseptic processing and packaging. In Principles of Aseptic Processing and Packaging, 2nd edition. (editors: P.E. Nelson and J.V. Chambers). The Food Processors Institute, Washington, D.C. pp. 47-86.

Dail, R. 1985. Calculation of required hold time of aseptically processed low-acid foods containing particulates utilizing the Ball method. Journal of Food Science. Vol. 50: 170-173.

Damiano, D. 1997. Issues involved in producing a multiphase food product. In workshop targets continuous multiphase aseptic processing of foods. Food Technology. Vol. 51(10): 56, 60-62.

Danckwerts, P.V. 1953. Continuous flow systems. Distribution of residence times. Chemical Engineering Science. Vol. 1: 1-13.

Dickerson, R.W., Jr., Read, R.B., Jr. 1968. Calculation and measurement of heat transfer in foods. Food Technology. Vol. 22:1533.

Digeronimo, M., Garthright, W., and Larkin, J.W. 1997. Statistical Design and Analysis. In "Workshop targets continuous multiphase aseptic processing of foods". Food Technology. Vol. 51(10): 52-56.

David, J.R.D. 1992. Aseptic processing of foods: Market advantages and microbiological risks. In "Advances in aseptic processing technologies" (Edited by Singh, R.K., Nelson, P.E.). Elsevier Applied Sciences, London, England. pp. 189-216.

David, J.R.D. 1996. Principles of Thermal Processing and Optimization. In "Aseptic processing and packaging of food, a food industry perspective". Edited by David, J.R.D., Graves, R.H., Carlson, V.R. CRC Press, Inc., Boca Raton, FL. pp. 4-8.

David, J.R.D., Shoemaker, C.F.1985. A transducer for the direct measurement of rates of lethality during thermal processing of foods. Journal of Food Science. Vol. 50: 223-225.

Dennis, C. 1992. HTST processing - scientific situation and perspective of the industry. In Processing and Quality of Foods. Vol. 1. (editors: P. Zeuthen *et al*.). Elsevier Applied Science, London. pp. 1.36-1.50.

de Ruyter, P.W., Brunet, R. 1973. Estimation of process conditions for continuous sterilization of foods containing particulates. Food Technology. Vol. 27: 44-51.

Dignan, D.M., Berry, M.R., Pflug, I.J., and Gardine, T.D. 1989. Safety considerations in establishing aseptic processes for low acid foods containing particulates. Food Technology. Vol. 43(3): 118-121, 131.

Dutta, B., Sastry, S.K. 1990a. Velocity distributions of food particle suspensions in holding tube flow: distribution characteristics and fastest-particle velocities. Journal of Food Science. Vol. 55(6): 1703-1710.

Dutta, B., Sastry, S.K. 1990b. Velocity distributions of food particle suspensions in holding tube flow: experimental and modeling studies on average particle velocities. Journal of Food Science. Vol. 55(5): 1448-1453.

Fairhurst, P.G., Barigou, M., Fryer, P.J., and Pain, J.P. 1999. Particle passage time distributions in vertical pipe flow of solid-liquid food mixtures. Transactions of the Institution of Chemical Engineers. Vol. 77(C): 293-301.

Floros J.D. 1993. Aseptic packaging technology. In "Principles of aseptic processing and packaging". Edited by Chambers J.V., and Nelson, P.E. The Food Processors Institute, Washington D.C. Ch. 5.

Gadonna, J.P., Pain, J.P., and Barigou, M. 1996. Determination of the convective heat transfer coefficient between a free particle and a conveying fluid in a horizontal pipe. Food and Bio-products Processing, Transactions of the Institution of Chemical Engineers. Vol. 74(C): 27-39.

Gavin, A., Wedding, L.M. 1995. Canned Foods: Principles of thermal process control, acidification and container closure evaluation (sixth edition). The Food Processors Institute, Washington, D.C. pp. 7-17, 107-117.

Ghiron, K.M., Litchfield, J.B. 1997. Magnetic thermometry in the aseptic processing of foods containing particulates. Journal of Applied Physics. Vol. 81(8): 4321.

Ghiron, K.M., Litchfield, J.B. 1998. Local magnetic temperature measurement for aseptic processing. U. S. Patent No. 5,722,317. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Gratzek, J.P., Toledo, R.T. 1993. A critical analysis of continuous sterilization of liquid foods containing particulates. ASME, Heat Transfer Division. Vol. 254: 17-23.

Guiavarc'h, Y., Deli, V., Van, L.A., and Hendrickx, M. 2002a. Development of an enzymic time temperature integrator for sterilization process based on *Bacillus licheniformis* a-amylase at reduced water content. Journal of Food Science. Vol. 67(1): 285-291.

Guiavarc'h, Y., Dintwa, E., Van, L., Ann., Z.F., and Hendrickx, M. 2002b. Validation and use of an enzymic time temperature integrator to monitor thermal impacts inside a solid/liquid model food. Biotechnology Progress. Vol. 18(5): 1087-1094.

Heldman, D.R. 1989. Establishing aseptic thermal processes for low acid foods containing particulates. Food Technology. Vol. 43(3): 122-124.

Heldman, D.R. 1992. Innovative concepts in equipment design for aseptic processing. In Advances in Food Engineering. (editors: R.P. Singh and M.A. Wirakartakusumah). CRC Press Inc., Boca Raton, FL. pp. 309-318.

Hendrickx, M., Weng, Z., Maesmans, G., and Tobback, P. 1992. Validation of a time temperature integrator for thermal processing of foods under pasteurization conditions. International Journal of Food Science and Technology. Vol. 27: 21-31.

Heppell, N.J. 1985a. Comparison of the residence time distributions of water and milk in an experimental UHT sterilizer. Journal of Food Process Engineering. Vol. 4: 71-84.

Heppell, N.J. 1985b. Measurement of liquid-solid heat transfer coefficient during continuous sterilization of food-stuffs containing particles. 4th International Congress on Engineering and Food, Edmonton, Alberta, Canada, July 7-10.

Hersom, A.C., Shore, D.T. 1981. Aseptic processing of foods comprising sauce and solids. Food Technology. Vol. 35(5): 53-62.

Higgins, K.T. 2004. Will aseptic heat up. In "Food Engineering". Vol. 76 (2): 43-46.

Jung, A., Fryer, P.J. 1999. Optimising the quality of safe food: Computational modeling of a continuous sterilization process. Chemical Engineering Science. Vol. 54: 717-730.

Kantt, C.A., Schmidt, S.J., Sizer, C.E., Palaniappan, S., and Litchfield, J.B. 1998. Temperature mapping of particles during aseptic processing with magnetic resonance imaging. Journal of Food Science. Vol. 63(2): 305-311.

Kantt, C.A., Webb, A.G., Litchfield, J.B., 1997. Temperature measurement of foods using chemical shift magnetic resonance imaging as compared with T1-weighted temperature mapping. Journal of Food Science. Vol. 62(5): 1011-1016.

Kim, H., Taub, I.A. 1993. Intrinsic chemical markers for aseptic processing of particulate foods. Food Technology. Vol. 47(1): 91-97, 99.

Lareo, C., Fryer, P.J., and Barigou, M. 1997. The fluid mechanics of two-phase solid-liquid food flows: A review. Transactions of the Institution of Chemical Engineers. Vol. 75(C): 73-105.

Larkin, J.W. 1997. Continuous multiphase ascetic processing of foods. Food Technology. Vol. 51(10): 43-44.

Lee, J.H., Singh, R.K. 1990. Mathematical models of scraped surface heat exchangers in relation to food sterilization. Chemical Engineering Communication. Vol. 87: 21-51.

Lee, J.H., Singh, R.K., and Chandarana, D.I. 1990a. Sensitivity analysis of aseptic process simulation for foods containing particulates. Journal of Food Process Engineering. Vol. 12: 295-315.

Lee, J.H., Singh, R.K., and Chandarana, D.I. 1990b. Determination of lethality and process time in a continuous sterilization system containing particulates. Journal of Food Engineering. Vol. 11: 67-92.

Lopez, A. 1987. A complete course in canning and related processes. The Canning Trade Inc., Timonium, MD. Vol. II. pp. 225-229.

Lund, D.B., Singh, R.K. 1993. The system and its elements. In "Principles of aseptic processing and packaging", 2nd Edition. (editors: P.E. Nelson and J.V. Chambers). The Food Processors Institute, Washington, D.C. pp. 3-30.

Maesmans, G., Hendrickx, M., DeCordt, S., Fransis, A., and Tobback, P. 1992. Fluid-to-particle heat transfer coefficient determination of heterogeneous foods: a review. Journal of Food Processing and Preservation. Vol. 16: 26-69.

Maesmans, G., Hendrickx, M., De Cordt, S., Vant Loey, A., Noronha J., and Tobback, P. 1994. Evaluation of process value distribution with time temperature integrators. Food Research International. Vol. 27: 413-423.

Manavel, J.E., Powell, R.L., McCarthy, M.J., and McCarthy, K.L. 1993. Magnetic resonance imaging of multiphase systems. In "Particulate Two-Phase Flow". (Editor M. Roco). Butterworth-Heinemann, Boston. pp. 127-140.

Manson, J.E., Cullen, J.F. 1974. Thermal process simulation for aseptic processing of foods containing discrete particulate matter. Journal of Food Science. Vol. 39: 1084-1089.

Marcy, J.E. 1997. Biological validation. Food Technology. Vol. 51(10): 44-48.

Merson, R.L., Leonard, S.J., 1979. Principles of thermal processing – FST 150 Class notes. Department of Food Science and Technology, University of California at Davis, Davis.

Mitchell, E.L. 1988. A review of aseptic processing. Advances in Food Research. Vol. 32: l-37.

Morris-Lee, J. 2004. CAPPS develops validation technologies for multiphase aseptic processing. In Aseptic processing and packaging. Witter Publishing Corporation, NJ. Vol. 1(2): pp. 5, 14-21.

Mwangi, J.M., Rizvi, S.S.H., and Datta, A.K. 1993. Heat transfer to particles in shear flow: Application in aseptic processing. Journal of Food Engineering. Vol. 19(1): 55-74.

Nielsen, S.S., Marcy, J.E., and Sadler, G.D. 1993. Chemistry of aseptically processed foods. In "Principles of aseptic processing and packaging". Edited by Chambers, J.V., and Nelson, P.E. The Food Processors Institute, Washington, D.C.

Palaniappan, S., Sizer, C. 1997. Aseptic process validated for foods containing particulates. Food Technology. Vol. 51(8): 60-68.

Palazoglu, T.K., Sandeep, K.P. 2002. Assessment of the effect of fluid-to-particle heat transfer coefficient on microbial and nutrient destruction during aseptic processing of particulate foods. Journal of Food Science. Vol. 67(9): 3359-3364.

Palazoglu, T.K., Sandeep, K.P. 2004. Effect of tube curvature ratio on the residence time distribution of multiple particles in helical tubes. Lebensmittel-Wissenschaft und -Technologie. Vol. 37: 387-393.

Palazoglu, T.K., Simunovic, J., Sandeep, K.P. and Swartzel, K.R. 2004. Methods, Systems and Devices for Evaluation of Thermal Treatment. European Patent No. WO2004067786. European Patent Office.

Pflug, I.J., Berry, M.R., and Dignan, D.M. 1990. Establishing the heat-preservation process for aseptically-packaged low-acid food containing large particulate, sterilized in a continuous heat-hold-cool system. Journal of Food Protection. Vol. 53: 312-321.

Qashou, M., Nix., G.H., Vachon, R.I. and Lowery, G.W. 1970. Thermal conductivity values for ground beef and chuck. Food Technology. Vol. 24: 493.

Ramaswamy, H.S., Abdelrahim, K.A., Marcotte, M., and Calvier, P. 1995a. Residence time distribution (RTD) characteristics of meat and carrot cubes in starch solutions in a vertical scraped surface heat exchanger (SSHE). Food Research International. Vol. 28(3): 273-284.

Ramaswamy, H.S., Abdelrahim, K.A., Simpson, B.K., and Smith, J.P.1995b. Residence time distribution (RTD) in aseptic processing of particulate foods: a review. Food Research International. Vol. 28(3): 291-310.

Ramaswamy, H.S., Awuah, G.B., and Simpson, B.K. 1996. Biological verification of fluid-to-particle interfacial heat transfer coefficients in a pilot scale holding tube simulator. Biotechnology Progress. Vol. 12: 527-532.

Ramaswamy, H.S., Awuah, G.B., and Simpson, B.K. 1997. Heat transfer and lethality considerations in aseptic processing of liquid / particle mixtures: a review. Critical Reviews in Food Science and Nutrition. Vol. 37(3): 253-286.

Ramaswamy, H.S., Pannu, K., Simpson, B.K., and Smith, J.P. 1992. An apparatus for particle-to-fluid relative velocity measurement in tube flow at various temperatures under nonpressurized flow conditions. Food Research International. Vol. 25: 277-284.

Ramaswamy, H.S., Zareifard, M.R. 2000. Evaluation of factors influencing tube-flow fluid-to-particle heat transfer coefficient using a calorimetric technique. Journal of Food Engineering. Vol. 45: 127-138.

Ranz, W.E., Marshall, W.R., Jr. 1952. Evaporation from drops. Chemical Engineering Progress. Vol. 48: 141-146.

Rao, M.A. 1992. Aseptic processing of foods. In Biotechnology and Food Process Engineering. (editors: M.A. Rao and H.G. Schwartzberg). Marcel Dekker, Inc. New York. pp. 247-275.

Reiffel, L. 2001. X-ray imaged implanted thermometers. U.S. Patent No. 6,250,800. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Sadeghi, F., Swartzel, K.R. 1987. Kinetic Studies of Calibration Materials for Thermal Evaluation of Food Systems. Ph.D. Dissertation, North Carolina State University, Raleigh.

Saksena, A. 2001. Systems and method for non-invasive assessment of cooked status of food during cooking. U.S. Patent No. 6,299,920. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Sandeep, K.P., Puri, V.M. 2001. Aseptic processing of liquid and particulate foods. In Food Processing Operations Modeling: Design and Analysis. (editor: J. Irudayaraj). Marcel Drekker, Inc. New York. pp. 37-41.

Sandeep, K.P., Zuritz, C.A. 1991. Residence time distribution of multiple particles in non-Newtonian tube flow. ASAE, Paper No. 916549. St. Joseph, MI.

Sandeep, K.P., Zuritz, C.A. 1993. Residence time distribution in holding tubes: dimensionless correlations and statistical analyses. ASAE, Paper No. 936577, St. Joseph, MI.

Sandeep, K.P., Zuritz, C.A. 1995. Residence times of multiple particles in non-newtonian holding tube flow: effect of process parameters and development of dimensionless correlations. Journal of Food Engineering. Vol. 25: 31-44.

Sandeep, K.P., Zuritz, C.A., and Puri, V.M. 1997a. Mathematical modeling and experimental studies on RTD and heat transfer during aseptic processing of non-Newtonian suspensions. Seventh International Congress on Engineering and Food, Brighton, England., April 13-17.

Sandeep, K.P., Zuritz, C.A., and Puri, V.M. 1997b. Residence time distribution of particles during two-phase non-Newtonian flow in conventional as compared with helical holding tubes. Journal of Food Science. Vol. 62(4): 647-652.

Sandeep, K.P., Zuritz, C.A., and Puri, V.M. 1999. Determination of lethality during aseptic processing of particulate foods. Transactions of the Institution of Chemical Engineers. Vol. 77(C): 11-17.

Sastry, S.K. 1986. Mathematical evaluation for process schedules for aseptic processing of low acid foods containing discrete particulates. Journal of Food Science. Vol. 51: 1323-1328.

Sastry, S.K. 1992. Liquid to particle heat transfer coefficient in aseptic processing. In Advances in Aseptic Processing Technologies. (editors: R.K. Singh and P.E. Nelson) Elsevier Applied Science, London. pp. 63-72.

Sastry, S.K. 1997. Measuring Residence Time and Modeling the System. In "Workshop targets continuous multiphase aseptic processing of foods". Food Technology. Vol. 51(10): 44-48.

Sastry, S.K., Cornelius, B.D. 2002. Aseptic processing of foods containing solid particulates. John Wiley and Sons Inc., New York. pp. 1-3, 130-181.

Sastry, S.K., Li, S.F., Patel, P., Konanayakam, M., Bafna, P., Doores, S., and Beelman, R.B. 1988. A bioindicator for verification of thermal processes for particulate foods. Journal of Food Science. Vol. 53(5): 1528-1532.

Sastry, S. K., Lima, M., Brim, J., Brunn, T., and Heskitt, B.F. 1990. Liquid-to-particle heat transfer during continuous tube flow: Influence of flow rate and particle to tube diameter ratio. Journal of Food Process Engineering. 13(3): 239-253.

Schwartz, S.J. 1992. Quality considerations during aseptic processing of foods. In "Advances in aseptic processing technologies". Edited by Singh, R.K., Nelson, P.E. Elsevier Applied Sciences, London. pp. 245-259.

Segner, W.P., Ragusa, T.J., Marcus, C.L., and Soutter, E.A. 1989. Biological evaluation of heat transfer simulation for sterilizing low-acid large particulate foods for aseptic packaging. Journal of Food Processing and Preservation. Vol. 13(4): 257-274.

Simunovic, J., Swartzel, K. R. and Adles, E. 2004. Method and system for conservative evaluation, validation and monitoring of thermal processing. U. S. Patent No. 6,776,523. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Sizer, C.E., Waugh, P.L., Edstam, S., and Ackermann, P. 1988. Maintaining flavor and nutrient quality of aseptic orange juice. Food Technology. Jun 1989. pp.152-159.

Smith, J.P., Ramaswamy, H.S., and Simpson, B.K. 1990. Developments in food packaging technology, Part I: Processing/cooking considerations. Trends in Food Science and Technology. Vol. 1: 106-109.

Storofos, N.G. 1992. An overview of aseptic processing of particulate foods. Food Science and Human Nutrition. Edited by Charalambous, G. Elsevier Science Publishers Company Inc., New York. pp. 665-677.

Stoforos, N.G., Merson, R.L. 1991. Measurement of heat transfer coefficients in rotating liquid/particulate systems. Biotechnology Progress. Vol. 7: 267-271.

Swartzel, K.R. 1982. Arrhenius kinetics as applied to product constituent losses in ultra high temperature processing. Journal of Food Science. 47.

Swartzel, K.R., Simunovic, J. 1999. Method and system for residence time measurement of simulated food particles in continuous thermal food processing and simulated particles for use in same. U. S. Patent Application No. 5,932,813. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2000. Method for conservatively evaluating continuous thermal treatment process for a particulate-containing food product stream. U. S. Patent No. 6,015,231. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2003. Plurality of particles made of a detectable magnetic implant and a carrier in combination with a plurality of magnetic field sensors. U.S. Patent 6,536,947. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2004. System for measuring residence time for a particulate containing food product. U.S. Patent No. 6,766,699. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Sweat, V.E. 1995. Thermal properties of foods. In Engineering properties of foods. (editors: M.A. Rao and S.S.H. Rizvi). Marcel Dekker, Inc. New York. pp. 99-138.

Tessner, W.A., Farkas, B.E., and Sandeep, K.P. 2001. Use of ablation to determine the convective heat transfer coefficient in two-phase flow. Journal of Food Process Engineering. Vol. 24: 315-330.

Tobback, P., Hendrickx, M.E., Weng, Z., Maesmans, G.J., and De Cordt, S.V. 1992. The use of immobilized enzymes as time temperature indicator system in thermal processing. In Advances in Food Engineering (editors: R.P. Singh and M.A. Wirakartakusumah). CRC Press Inc., Boca Raton, FL. pp. 561-574.

Toledo, R.T., Chang, S.Y. 1990. Advantages of aseptic processing of fruits and vegetables. Food Technology. Vol. 44: 72-78.

U.S. Food and Drug Administration. 1992. Foodborne Pathogenic Microorganisms and Natural Toxins Handbook. Center for Food Safety and Applied Nutrition. U.S. Food and Drug Administration. (http://www.cfsan.fda.gov/~mow/chap2.html).

Weng, Z., Hendrickx, M., Maesmans, G., and Tobback, P. 1992. The use of time-temperature-integrator in conjunction with mathematical modeling for determining liquid/particle heat transfer coefficients. Journal of Food Engineering. Vol. 16: 197-214.

Yang, B.B., Nunes, R.V., and Swartzel, K.R. 1992. Lethality distribution within particles in the holding section of an aseptic processing system. Journal of Food Science. Vol. 57(5): 1258-1265.

Zareifard, M.R., Ramaswamy, H.S. 1999. A calorimetric approach for evaluation of fluid-to-particle heat transfer coefficient under tube-flow conditions. Lebensmittel-Wissenschaft und Technologie. Vol. 32: 495-502.

Zitoun, K.B. 1996. Continuous flow of solid-liquid food mixtures during ohmic heating: Fluid interstitial velocities, solid area fraction, orientation and rotation. Ph.D. dissertation. Ohio State Univesity. Columbus, OH.

Zitoun, K.B., Sastry, S.K. 1994a. Convective heat transfer coefficient for cubic particles in continuous tube flow using the moving thermocouple method. Journal of Food Process Engineering. Vol. 17: 229-241.

Zitoun, K.B., Sastry, S.K. 1994b. Determination of convective heat transfer coefficient between fluid and cubic particles in continuous tube flow using noninvasive experimental

techniques. Journal of Food Process Engineering. Vol. 17: 209-228.

Zuritz, C.A., McCoy, S., and Sastry, S. 1987. Convective heat transfer coefficients for non-Newtonian flow past food-shaped particulates. ASAE Paper No. 876538, ASAE, St Joseph, MI.

Chapter 3

MANUSCRIPT I

Construction and Testing of Conservative Thermal Properties of Simulated Food Particles for use in Validation of Multiphase Aseptic Processing

A.K.S. Jasrotia
J. Simunovic
K.P. Sandeep
T.K. Palazoglu
K.R. Swartzel

Department of Food Science, North Carolina State University

Please address all correspondence to:

K.P. Sandeep
129 Schaub Hall, Box 7624
North Carolina State University
Raleigh, NC 27695
Phone: 919-515-2957
Fax: 919-515-7124
E-mail: kp_sandeep@ncsu.edu

Paper No. FSR-04-XY of the Journal Series of the Department of Food Science, North Carolina State University, Raleigh, NC 27695-7624.

ABSTRACT

Validation of multiphase aseptic processing for production of shelf-stable low-acid foods requires simulated food particles to contain implants such as magnets or bio-loads for determining the time-temperature history, and bactericidal efficacy confirmation. These simulated particles need to have conservative (fast-moving and slow-heating) characteristics as compared to real food particles. Conservative Particle Design (CPD) software was used to determine the minimum wall thickness (~2 mm) and maximum implant weight of the simulated food particles for validation of aseptic processing of foods containing ½ " cubic potato and carrot pieces. These particles were fabricated from polypropylene and polymethylpentene polymers. Thermocouples were fitted in duplicate samples of simulated and real food particles and heated up to 127 °C under pressurized (24 psi) conditions. These simulated particles exhibited conservative heat penetration characteristics when compared to carrot and potato particles. The method and the data acquisition program developed in this study can be used as a tool for the construction and the experimental validation of such critical particles, which are essential for the validation of multiphase aseptic processes.

Keywords: validation, multiphase aseptic processing, time-temperature history, conservative particle design, critical particle

INTRODUCTION

Aseptic processing and validation of aseptic processing for multiphase foods has attracted the attention of researches world wide for over three decades. Compared to traditional canning, where products are heated in the container for 20 minutes to several hours, aseptic processing involves rapid heating and cooling followed by aseptic packaging which substantially reduces the energy use and nutrient loss associated with conventional sterilization. Aseptically processed chunky soups – as well as various stews and sauces – have been a household staple in Europe for nearly 30 years (Morris-Lee, 2004). In Europe, microbial testing of aseptically processed multiphase foods at the end of their shelf life is performed to find the safety of the product. However, in the U.S., multiphase processing of food must be validated before the product can be produced.

Recent research works in aseptic processing of multiphase foods is mainly conducted to reduce the cost of validation for such processes. Significant research work has been already done over many years to make multiphase aseptic processing technology commercial (Dignan *et al.,* 1989; Heldman, 1989; Toledo and Chang, 1990; Lund and Singh, 1993). However, current validation techniques for multiphase aseptic processing are costly and thus far from attempts to bring shelf-stable low-acid aseptic particulate food products in the U.S. (Larkin, 1997; Higgins, 2004).

The technology and knowledge-base for aseptic processing of liquid foods has been well developed and approved by regulatory agencies. However, solving the issues involved in aseptic processing of low-acid particulate foods has been the area of interest for researchers during the past several decades. The geometric center of a food particle is the critical point of interest since it is the slowest heating point. Therefore, the challenge

in such a system is to quantify the thermal treatment received by the food particulate at its center. The thermal treatment in any thermal process can be quantified using the F value (for a given z and T_{ref} values) definition (David, 1996):

$$F^{z}_{T_{ref}} = \int_{t_i}^{t_f} 10^{(T(t)-T_{ref})/z} dt \qquad (12)$$

where, T is the temperature (time function), T_{ref} is the reference temperature (usually 121.1 °C), t_i and t_f are initial and final process time respectively and z (in °C) is a temperature dependent characteristic of the microorganism and is assumed to be constant over normal processing conditions. In the above equation, z is defined as the increase in temperature to decrease the decimal reduction time of the target microorganism by one log scale and F_0 represents the F value for T_{ref} = 121.1 °C and z = 10 °C (*Clostridium botulinum*). To establish an aseptic process for multiphase food products, regulatory agencies require the processor to identify and select a sterilizing F_0 value for the product, develop a conservative model that reliably predicts the total lethality of the heat process, quantitatively verify the lethality delivered by means of bio-indicators, and list the critical factors of the process and the procedures to be used for controlling them (Dignan *et al.,* 1989). Researchers in the past have attempted to measure the temperature at the center of a particle during its thermal treatment in a continuous flow system using different methods. Heat penetration measurements for a food particle traveling through an aseptic processing system is difficult and not practical at the present time without restricting the free movement of food particles (Sastry, 1986; Lee and Singh, 1990; Heldman, 1992; Lund and Singh, 1993; Maesmans *et al.,* 1994). Due to challenges encountered in measuring the temperature of moving particles in aseptic processing systems, several alternatives have been considered such as biological validation techniques (Heppell,

1985a, b; Berry, 1989; Tobback *et al.,* 1992; Cacase *et al.,* 1994), moving thermocouple methods (Sastry, 1992; Zitoun and Sastry, 1994a), liquid crystal technique (Stoforos and Merson, 1991; Balasubramaniam and Sastry, 1994a; Zitoun and Sastry, 1994b), time temperature integrators (Hendrickx *et al.,* 1992; Guiavarc'h *et. al.* 2002a; Guiavarc'h *et. al.*, 2002b) melting point indicators (Mwangi *et al.,* 1993), and relative velocity methods (Balasubramaniam and Sastry, 1994b).

Determination of fluid-to-particle heat transfer coefficient (h_{fp}) poses a unique challenge of monitoring the temperature of moving particles without restricting the flow of the particle (Chandarana *et al.*, 1989; Zareifard and Ramaswamy, 1999). The value of h_{fp} depends on many factors such as fluid viscosity and temperature, fluid-to-particle relative velocity, particle size, and shape (Ramaswamy *et al.*, 1997, Tessner *et al*., 2001). Various invasive and non-invasive methods (Zuritz *et al.*, 1987; Sastry *et al*., 1990; Masesmans *et al.*, 1992; Sastry, 1997; Sastry and Cornelius, 2002) methods have been developed to determine h_{fp}.

In aseptically processed particulate product biological validation of the process is necessary because the design of the aseptic process is not based on heat penetration tests due to the unavailability of the time-temperature history of the product (Chandarana, 1992). Biological validation of an aseptic process for a particulate food product involves the use of biological indicators that travel through the system inside a particle (Pflug *et al.*, 1990; Marcy, 1997). In an important workshop (CAPPS and NCFST Workshop, 1995, 1996), a consensus was reached on concerns that needed to be resolved to develop an aseptic process for multiphase food products. In this workshop, various methods for determining the RTD measurements were discussed. In an attempt to validate a

multiphase aseptic process for the first time a filing was done by Tetra Pak (1997) for diced potato soup with FDA. The product chosen by Tetra Pak was diced potato soup in a carrier of modified food starch. Density-adjusted chicken-alginate cubes were prepared for use in the bio-validation step. These particles were also used to show that their residence time was sufficient for receiving required heat treatment. The filing received a no-objection letter from FDA (Palaniappan and Sizer, 1997). However, the cost of validation of this process is very high. Therefore, reducing the cost for validation techniques has been one of the key areas of focus in multiphase aseptic processing research.

Monitoring and validation of continuous thermal pasteurization and sterilization process typically requires the use of simulated food particles, which must exhibit conservative flow and thermal characteristics. In several multiphase studies, simulated food particles such as polystyrene spheres, alginate beads, and rubber cubes have been used (Sandeep and Zuritz, 1995; Palazoglu and Sandeep, 2002, 2004; Fairhurst *et al*., 1999; Dutta and Sastry, 1990a, b). Real food particles such as carrot and potato cubes (Ramaswamy *et al.*, 1992; Alhamdan and Sastry, 1997; Abdelrahim *et al.*, 1997). Due to various challenges in accurately determining the temperature histories at the center of a particle flowing in a continuous system, alternatives have been considered. Researchers in the past have suggested the use of conservative mathematical models to predict the total lethality of the heat process during aseptic processing of low-acid food particulates foods. The most critical factors identified in mathematical models were particle size and shape, fluid-to-particle heat transfer coefficient, and the residence time distribution (RTD) in the holding tube. RTD measurement is needed because of the difficulties in

non-invasive measurement of internal temperatures of particle during continuous flow. If the temperature of the cold-spot could be measured at the end of the heater and the hold tube, RTD measurements would be unnecessary (Sastry, 1986; Chandarana and Gavin, 1989; Sastry, 1997). In a recent research work, a method of generating residence time measurement of a particulate-containing food product while passing the product as a continuous stream through a thermal processing apparatus was developed (Swartzel and Simunovic, 1999, 2000, 2003, 2004).

Small paramagnetic particles in the interior of simulated and real food particles were used, to measure the local temperatures within the particles (Ghiron and Litchfield., 1997; Ghiron and Litchfield, 1998). Beller (1993) developed a method and apparatus for the in-process measurement of internal particulate temperature utilizing ultrasonic tomography techniques to determine the speed of sound through a specimen material. Micro thermometry has also been proposed for real time-temperature measurements in food particulates for validation process (Reiffel, 2001; Higgins, 2004). In another study, a method based on change in proton precession frequency with temperature was compared with temperature measurement by T1-weighted imaging (Kantt *et. al.*, 1997). Magnetic Resonance Imaging (MRI) was used to obtain two-dimensional temperature maps in potatoes undergoing aseptic processing (Kantt *et. al.*, 1998). The change in precession frequency of protons served as the temperature indicator. Saksena (2001) developed a non-contact system and method was provided for effectively approximating the internal temperature of food being cooked upon a cooking surface. A magnetically detectable particle and related methods, systems, and devices are provided for generating a temperature measurement for a batch or a continuous stream of material (Palazoglu *et.*

al., 2004). In another study, a method was developed for generating a temperature measurement for a batch or a continuous stream of material (Simunovic *et. al.*, 2004).

Thus from the recent research work it is seen that simulated food particle have been widely used in multiphase aseptic processes and a lot of emphasis has been given on developing methods to determine time-temperature history, and residence time distribution, for such particles in a multiphase aseptic system. However, no systematic approach is available or published for fabrication of simulated particles used to carry a variety of thermo-sensitive implants used in these measurements. A systematic procedure for design, fabrication and testing of simulated food particles to be used for validation of aseptic processing of multiphase foods to generate particles with conservative flow and thermal behavior (fast-moving and slow-heating) is thus needed and is the goal of current study. Wide availability and reduced cost of such particles would reduce the complexity of process documentation and filing with regulatory agencies and bring aseptic multiphase foods closer to commercial reality.

MATERIALS AND METHODS

Experimental setup

Figure 3.1 shows the components of the experimental setup. A bench top autoclave (Pelton Crane Omni-Clave OCM Sterilizer, Kroslak Enterprises Inc., Riverview, FL) was used. This equipment is used for providing a sterilization environment for surgical and clinical instruments and can reach pressures up to 24 psi and temperatures up to 127 °C. A stuffing box was used to enable the coupling of the

autoclave with the thermocouple system. This box must be fixed air tight so that it does not leak at high pressures.

The thermocouples used were teflon-coated type T thermocouples (Marlin Manufacturing Corporation, Cleveland, OH). WebDAQ/100™ (Capital Equipment Corp., Bedford, NH) was used to acquire time-temperature data coupled with two thermocouple adapter boards (Capital Equipment Corp, Bedford, NH) designed to provide a solution to the connecting thermocouples for measuring temperatures. Time-temperature data were acquired at a sampling rate of 3000 Hz with output rate of 10 Hz.

Conservative Particle Design

Construction of simulated food particles (polymer-based) used in the current study was based on the Conservative Particle Design (CPD) software. The CPD software (Palazoglu ***et. al.***, 2004) can be used to determine the design parameters (namely wall-thickness and implant weight) for cubic, spherical, and cylindrical particles made of a selected polymer (thermal diffusivity of which is lower than that of the food particle being simulated). This particle gives thermal protection to its cavity greater than or equal to the thermal protection provided by the food to its geometric center with identical shape and dimensions. This particle can be used as an magnetic implant carrier when developing a continuous sterilization flow system for validation of aseptic processing for particulates foods.

In CPD, this is achieved by simulating concurrent heating of a theoretical polymer particle and a theoretical food particle under identical sterilization-level conditions. For this both the particles are divided into hypothetical grids. During this simulated sterilization treatment calculations, F_0 value accumulated in each grid is determined.

Since thermal diffusivity of most (not all) polymer particles is lower (i.e., they heat slower) than most food particles of equal size and shape, the accumulation of F_0 values will be slower and will not penetrate as deep in a simulated particle.

Design selection is made once the center of the food particle has reached the target F_0 (e.g. 3 min), the F_0 map Figure 3.2 of the concurrently heated simulated food particle is examined to determine the points (depth) to which the penetration of target F_0 value or lower has progressed under these identical heating conditions – all points deeper within the particle beyond this level will therefore be thermally protected at least as well or better than the protection provided by the food particle to its geometric center. The depth of the penetration of this pre-selected F_0 value into the particle establishes the most important construction criterion – particle wall thickness. By removing the material within the "equal protection zone" established in this way, a cavity is generated within each fabricated particle that is supposed to be at least as equal or better thermally protected than the centers of real food particles, i.e., an implant carrier cavity or a "cold spot" carrier cavity. Theoretically, anything placed in that cavity will under the identical heating conditions receive less thermal treatment than the center of a real food particle of identical shape and dimensions. Therefore, if for example, we place a time-temperature recording device within the particle cavity generated in this manner, and process a multiphase stream of product until the time-temperature exposure to the device placed in this cavity accumulates an F_0 value of three minutes or more, we will be assured that *everything else* within the system will have received *at least* this same treatment level.

Simulated particles must also to be constructed to provide a fast flowing behavior (in addition to this slow heating behavior). This is achieved separately but concurrently

by adjusting their effective density to a value slightly lower than the carrier fluid density – this is applicable to vertical and slightly inclined (as required by regulatory agencies) heat exchangers and hold tubes only, for all other configurations this "critical density" needs to be determined experimentally. In CPD, one could also input the process variables such as the initial particle temperature, ambient temperature, heat transfer coefficient, desired F_0 and density of the target particle. This is to ensure that the particle is not denser than the fluid in the system and is thus having desired flow properties for the RTD measurements.

The CPD software is used for determining the starting point for the construction of the conservative particles. The CPD software requires the user to choose the type of food (carrot, potato, beef or my food), kind of material that would be used for the construction of the simulated particle (nylon, polypropylene (PP), teflon, polymethylpentene (PMP) or my plastic material), shape of the particle (spherical, cubical or cylindrical) and the size of the food and simulated particle (half thickness for cube). Present study deals with the cubical shape particles. The results are generated as a lethality chart that shows the demarked region in a quarter portion of the particle (Figure 3.2). The curved line shows the region inside which, the particle received F_0 value of less than three minutes (12D reduction process for *Clostridium botulinum*).

Calibration of thermocouples

Calibration of the thermocouple system is required due to WebDAQ/100™ adapter and thermocouple installation. Calibration was performed by measuring all 24 channel temperature readings at ice cold water and boiling water. A mercury thermometer (Ertco Ever Ready Thermometer Company, Inc., Dubuque, IA) was used to

measure the ice cold and boiling water temperature, to compare them with the measured values of thermocouples at the same time. The thermometer has a lower scale limit of 2 °F and could measure temperatures in the range of 0-220 °F. The correction data for calibration is stored in a file which is later read by the data acquisition system to integrate the corrections for each channel in a real time acquisition. This is done by storing the measured temperature of all the thermocouple channels at the two ends (ice and boiling water) which would be used to correct the measured temperature at the run time and display the corrected temperature and F_0 values on the Data Acquisition Program. To understand the calibration methodology it would be useful to understand that if a plot a graph between the measured temperature (x-axis) and the actual temperature (y-axis) at ice cold bath and boiling water for each thermocouple channel in the system, we can obtain equation for determining the actual temperature for that particular channel, if the measured temperature is known. The measured temperature is acquired by the Data Acquisition Program over the network from the WebDAQ/100™ system. Figure 3.3 shows this simple method of determining the actual temperature for a thermocouple channel. It can been seen from Figure 3.1 that if X_1 = 0 °C (ice), X_2 = 100 °C (boiling water), Y_1 = measured temperature of thermocouple at X_1 and Y_2 = measured temperature of thermocouple at X_2 is know, then for any give X (measured temperature) the Y value (actual temperature) can be determined using a linear relationship assumption. A linear relation between Y and X was assumed since the variation of measured to actual values over the range Y_1 to Y_2 was not significant as can be seen from Table 3.1. The working ranges for the current system were 25-127 °C, in which linear relation holds well.

Thus the equation for determining the actual temperature (Y), at any reading time point for a particular channel would be:

$$Y = \left(\frac{Y_2 - Y_1}{X_2 - X_1} \right)(X - X_1) + Y_1 \tag{13}$$

Data Acquisition Program

TestPoint v4.1™ software (Capital Equipment Corp, Bedford, NH) was used to develop the customized programs to acquire temperature data. It provides high-level analog-to-digital (A/D) functions to simplify data acquisition.

The Data Acquisition Program (using in TestPoint v4.1™) developed to monitor the two parameters – temperature and F_0 value, in both the center of real food particles and within the simulated particle "cold-spot carrier" cavity was developed. These calibrated temperature and F_0 values are plotted by the program on the monitor screen at real time. The program connects to a WebDAQ/100™ accessible over a network, in conjunction with the thermocouple measuring system to generate the temperature, and F_0 value data files for a 24 channel WebDAQ/100™ system. The WebDAQ/100™ system included two WebDAQ/100™ thermocouple adapter boards to convert analog temperature reading to digital data.

The F_0 measurement logic incorporated in the program is shown in Figure 3.4. From equation (1) we assume a function L(t) – the lethality rate as:

$$L(t) = 10^{(T(t) - T_{ref})/z} \tag{14}$$

The integral of equation (3), over the processing time range of t_i to t_f, which is the area under L(t) on Figure 3.4 would thus determine the F_0 value for the heat process – T(t) . Due to the limitations of the TestPoint v4.1™ software, an exact integral cannot be

calculated as the measured time-temperature data is recorded at certain frequency (depending on the sampling rate/output rate of WebDAQ/100™). From Figure 3.4 it can be seen that the shaded areas under step wise function (points ABCD…) can be calculated as:

$$(F_0)_{t+\Delta t} = (F_0)_t + (L(t) * \Delta t) \quad (15)$$

where $(F_0)_t$ is F_0 at time t. Here, T_{ref} = 121.1 °C and z = 10 °C. Also:

$$F_0 = 0 \text{ at } t_i = 0 \quad (16)$$

From Figure 3.4 it can be seen that the area of the shaded region would be less than the integral in equation (1). Given the limitations of the programming software, this would be beneficial, since we would be conservative in F_0 calculation and thus would ensure the product safety. The T(t) is the corrected temperature of anyone of the thermocouple channel in consideration, and is calculated using equation (2). A flow diagram explaining general steps involved in Data Acquisition System is shown in Figure 3.5. All these calculations are carried out at the run time within the Data Acquisition System, and the results for corrected temperature and F_0 values are displayed on the monitor screen Figure 3.6.

Experimental runs

The particles fabricated using the above tools and methodology were used to perform all the experimental runs. Duplicate particles made of PP and PMP with cube size of half inch were used in the study. Thermocouples were fitted inside the cavity of simulated particles and at approximate center of the real food particles. The runs were performed with pressures close to 24 psi and temperatures of up to 127 ^oC. This acquired F_0 and temperature data was analyzed using Microsoft® Excel (Microsoft® Corporation,

Redmond, WA). The plots for F_0, temperature vs. time, for different runs were prepared and the conservative behavior evaluated.

RESULTS AND DISCUSSION

The output data for the Data Acquisition Program was stored in temperature and F_0 value files. The F_0 values measured and recorded in this file was compared with calculated F_0 data obtained using the measured temperature data file. The result for this comparison is shown in Table 3.1. This comparison is performed to make sure that the program is calculating the F_0 values correctly using its measured temperature data file at run time, as explained in equations (1) to (5). It can be seen that the program calculates these values as expected incorporating the correction from the calibration file. Also the program assigns a value of zero for F_0 at start of the experimental, as mentioned in equation (5).

Conservative Particle Design (CPD) software was used to generate the simulated particle design for comparison with potato and carrot of ½ inch cube size. CPD determined wall thickness and implant weight required for the polypropylene (PP) and polymethylpentene (PMP) particles. Thermophysical properties values of polymers, PP and PMP and food materials, carrot and potato are shown in Table 3.2. It can be seen that the thermal diffusivity values for PP and PMP are lower than those of carrot and potato, implying that these polymers would heat slower than carrot and potato. Also PMP has a higher thermal diffusivity than PP, therefore heat penetration would be faster for PMP when compared to PP. Parameters such as thermal conductivity, specific heat and density

of carrot, potato, PP, and PMP used as input values to generate design for PP and PMP. Input process variable listed in Table 3.3 were not changed for any of the design configurations in this study. These are conditions that were assumed to occur in a normal experimental setup for this study.

Wall thickness and implant weight determined by CPD for fabricating the simulated particles for different combination of polymers and food materials, are shown in Table 3.4. These particles are expected to exhibit conservative properties and are representative of the least thermally treated particle in the system. This is tested experimentally in different runs. It is assumed that these particles would be used in validation for a continuous flow medium of average density equal to that of water (target particle density in Table 3.3 as 1000 kg/m^3). This would make sure that the particle is neutrally buoyant while thermally protecting its "cavity" equal to or better than the rest of the particles in the system.

Figure 3.8 shows the temperature acquisition data for the run with PP, PMP, carrot and potato. The data clearly shows that the simulated food particles heated slower than the food particle, showing a thermally conservative behavior. Many similar runs were done and it was seen that the temperature in the cavity of the simulated food particles always remained lower than that at the center of the real food particles. To make this clear, various F_0 charts were drawn. These contained replicates of PP and PMP to see the consistency in the behavior of simulated particles. These are shown in Figure 3.9 and Figure 3.10 respectively. As can be seen, the F_0 accumulated by carrot and potato is always higher than that for both PP and PMP. Also many similar runs were performed and the data analyzed. Every single time the slow heating behavior of these particles was

proved. This clearly shows that the particle cavity is thermally better protected than the center of real food particles. In fact CPD take into account the calculations for maximum weight of the implant to be neutrally buoyant and ensures that the designed particles are the fastest and thus acquire the least lethality at it cavity or the geometric center.

CONCLUSIONS

A method for fabrication and validation of implant-carrier simulated particles with conservative thermal properties for monitoring and validation of continuous thermal processing of multiphase food products has been developed and implemented. The results demonstrate the procedure and feasibility of implementing a conservative carrier particle design for process monitoring and validation. To use these particles for validation of multiphase aseptic processes we need to be confident that they will provide a conservative thermal protection when used for validation of any aseptic multiphase process under similar process variables. For this the fabricated particles were experimentally validated and the experimental data was analyzed using Microsoft® Excel (Microsoft® Corporation, Redmond, WA).

The developed Data Acquisition Program can be used by a user to calculate F_0 as per the requirement for safety (microbial destruction) or quality (nutrient loss) calculations. It incorporates the corrections to the measured temperature readings at run time and also calculates the F_0 values, displaying them on the monitor screen. In addition, the temperature data file could also be used as standalone to calculate F_0 or other parameters with tools like Microsoft® Excel (Microsoft® Corporation, Redmond, WA).

Developed tools and methods could be used by food processors and equipment providers to collect process validation data needed for process safety documentation and filing with regulatory agencies as well as comparison of various products, processes and equipment properties.

ACKNOWLEDGEMENTS

Support for the research study undertaken here, resulting in the publication of Paper No. FSR-04-XY of the Journal Series of the Dept. of Food Science, NCSU, Raleigh, NC 27695-7624, from USDA-NRICGP and North Carolina Agricultural Research Service is greatly acknowledged.

The use of trade names in this publication does not imply endorsement by the North Carolina Agricultural Research Service of the products named nor criticism of similar ones not mentioned.

SYMBOLS

D-value	decimal reduction time	min
F_0	sterilization value	min
h_{fp}	fluid-to-particle heat transfer coefficient	W/m^2-K
T	temperature	°C
t	time	min
z-value	temperature change required to change D-value by a factor of ten	°C

Subscripts

ref	reference

Abbreviations

CAPPS	Center for Advanced Processing and Packaging Studies
CPD	Conservative Particle Design
FDA	Food and Drug Administration
NCFST	National Center for Food Safety and Technology
PMP	polymethylpentene
PP	polypropylene
RTD	residence time distribution

REFERENCES

Abdelrahim, K.A., Ramaswamy, H.S., and Marcotte, M. 1997. Residence time distribution of meat and carrot cubes in the holding tube of an aseptic processing system. Lebensmittel-Wissenschaft und -Technologie. Vol. 30: 9-22.

Alhamdan, A., Sastry, S.K. 1997. Residence time distribution of food and simulated particles in a holding tube. Journal of Food Engineering. Vol. 34: 271-292.

Balasubramaniam, V.M., Sastry, S.K. 1994a. Liquid-to-particle heat transfer in non-Newtonian carrier medium during continuous tube. flow. Journal of Food Engineering. Vol. 23: 169-187.

Balasubramaniam, V.M., Sastry, S.K. 1994b. Convective heat transfer at particle-liquid interface in continuous tube flow at elevated fluid temperatures. Journal of Food Science. Vol. 58: 675-681.

Beller, L.S. 1993. Ultrasonic tomography for in-process measurements of temperature in a multi-phase medium. U.S. Patent No. 5,181,778. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Berry, M.R. 1989. Predicting the fastest particle residence time. First International Conference on Aseptic Processing Technologies. Indianapolis, IN, March 19-21.

CAPPS and NCFST Workshop. 1995, 1996. Case study for condensed cream of potato soup from the aseptic processing of multiphase foods workshops. November 14-15, March 12-13.

Cacase, D., Palmieri, L., Pirone, G., and Dipollina, G. 1994. Biological validation of mathematical modeling of the thermal processing of particulate foods: The influence of heat transfer coefficient determination. Journal of Food Engineering. Vol. 23: 51-68.

Chandarana, D.I. 1992. Acceptance procedures for aseptically processed particulate foods. In “Advances in aseptic processing technologies”. Edited by Singh, R.K., Nelson, P.E. Elsevier Applied Sciences, London. pp. 261-278.

Chandarana, D.I., Gavin, A. 1989. Establishing thermal processed for heterogeneous foods to be processed aseptically: A theoretical comparison of process development methods. Journal of Food Science. Vol. 54(1): 198-204.

Chandarana, D.I., Gavin, A., and Wheaton, F.W. 1989. Simulation of parameters for modeling aseptic processing of foods containing particulates. Food Technology. Vol. 43(3): 137-143.

David, J.R.D. 1996. Principles of Thermal Processing and Optimization. In "Aseptic processing and packaging of food, a food industry perspective". Edited by David, J.R.D., Graves, R.H., Carlson, V.R. CRC Press, Inc., Boca Raton, FL. pp. 4-8.

Dignan, D.M., Berry, M.R., Pflug, I.J., and Gardine, T.D. 1989. Safety considerations in establishing aseptic processes for low acid foods containing particulates. Food Technology. Vol. 43(3): 118-121, 131.

Dutta, B., Sastry, S.K. 1990a. Velocity distributions of food particle suspensions in holding tube flow: distribution characteristics and fastest-particle velocities. Journal of Food Science. Vol. 55(6): 1703-1710.

Dutta, B., Sastry, S.K. 1990b. Velocity distributions of food particle suspensions in holding tube flow: experimental and modeling studies on average particle velocities. Journal of Food Science. Vol. 55(5): 1448-1453.

Fairhurst, P.G., Barigou, M., Fryer, P.J., and Pain, J.P. 1999. Particle passage time distributions in vertical pipe flow of solid-liquid food mixtures. Transactions of the Institution of Chemical Engineers. Vol. 77(C): 293-301.

Ghiron, K.M., Litchfield, J.B. 1997. Magnetic thermometry in the aseptic processing of foods containing particulates. Journal of Applied Physics. Vol. 81(8): 4321.

Ghiron, K.M., Litchfield, J.B. 1998. Local magnetic temperature measurement for aseptic processing. U. S. Patent No. 5,722,317. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Guiavarc'h, Y., Deli, V., Van, L.A., and Hendrickx, M. 2002a. Development of an enzymic time temperature integrator for sterilization process based on *Bacillus licheniformis* a-amylase at reduced water content. Journal of Food Science. Vol. 67(1): 285-291.

Guiavarc'h, Y., Dintwa, E., Van, L., Ann., Z.F., and Hendrickx, M. 2002b. Validation and use of an enzymic time temperature integrator to monitor thermal impacts inside a solid/liquid model food. Biotechnology Progress. Vol. 18(5): 1087-1094.

Heldman, D.R. 1989. Establishing aseptic thermal processes for low acid foods containing particulates. Food Technology. Vol. 43(3): 122-124.

Heldman, D.R. 1992. Innovative concepts in equipment design for aseptic processing. In Advances in Food Engineering. (editors: R.P. Singh and M.A. Wirakartakusumah). CRC Press Inc., Boca Raton, FL. pp. 309-318.

Hendrickx, M., Weng, Z., Maesmans, G., and Tobback, P. 1992. Validation of a time temperature integrator for thermal processing of foods under pasteurization conditions. International Journal of Food Science and Technology. Vol. 27: 21-31.

Heppell, N.J. 1985a. Comparison of the residence time distributions of water and milk in an experimental UHT sterilizer. Journal of Food Process Engineering. Vol. 4: 71-84.

Heppell, N.J. 1985b. Measurements of the liquid-solid heat transfer coefficient during continuous sterilization of food-stuffs containing particles. Presented at the 4th Congress on Engineering and Foods. Edmonton, Alberta, Canada, July 7-10.

Higgins, K.T. 2004. Will aseptic heat up. Food Engineering. Vol. 76(2): 43-46

Jasrotia, A.K.S., Simunovic, J., Swartzel, K.R., and Sandeep, K.P. 2003. Multiphase aseptic processing: selection and detection of magnetic particle implants for monitoring residence time and time-temperature history. AIChE Annual Meeting, San Francisco, CA: November 16-21: 107by.

Kantt, C.A., Schmidt, S.J., Sizer, C.E., Palaniappan, S., and Litchfield, J.B. 1998. Temperature mapping of particles during aseptic processing with magnetic resonance imaging. Journal of Food Science. Vol. 63(2): 305-311.

Kantt, C.A., Webb, A.G., Litchfield, J.B., 1997. Temperature measurement of foods using chemical shift magnetic resonance imaging as compared with T1-weighted temperature mapping. Journal of Food Science. Vol. 62(5): 1011-1016.

Larkin, J.W. 1997. Continuous multiphase aseptic processing of foods. Food Technology. Vol. 51(10): 43-44.

Lee, J.H., Singh, R.K. 1990. Mathematical models of scraped surface heat exchangers in relation to food sterilization. Chemical Engineering Communication. Vol. 87: 21-51.

Lund, D.B., Singh, R.K. 1993. The system and its elements. In Principles of Aseptic Processing and Packaging, 2nd Edition. (editors: P.E. Nelson and J.V. Chambers). The Food Processors Institute, Washington, D.C. pp. 3-30.

Maesmans, G., Hendrickx, M., DeCordt, S., Fransis, A., and Tobback, P. 1992. Fluid-to-particle heat transfer coefficient determination of heterogeneous foods: a review. Journal of Food Processing and Preservation. Vol. 16: 26-69.

Maesmans, G., Hendrickx, M., De Cordt, S., Vant Loey, A., Noronha J., and Tobback, P. 1994. Evaluation of process value distribution with time temperature integrators. Food Research International. Vol. 27: 413-423.

Marcy, J.E. 1997. Biological validation. Food Technology. 51(10): 44-48.

Morris-Lee, J. 2004. CAPPS develops validation technologies for multiphase aseptic processing. In Aseptic processing and packaging. Witter Publishing Corporation, NJ. Vol. 1(2): pp. 5, 14-21.

Mwangi, J.M., Rizvi, S.S.H., and Datta, A.K. 1993. Heat transfer to particles in shear flow: Application in aseptic processing. Journal of Food Engineering. Vol. 19(1): 55-74.

Palaniappan, S., Sizer, C. 1997. Aseptic process validated for foods containing particulates. Food Technology. Vol. 51(8): 60-68.

Palazoglu, T.K., Sandeep, K.P. 2002. Assessment of the effect of fluid-to-particle heat transfer coefficient on microbial and nutrient destruction during aseptic processing of particulate foods. Journal of Food Science. Vol. 67(9): 3359-3364.

Palazoglu, T.K., Sandeep, K.P. 2004. Effect of tube curvature ratio on the residence time distribution of multiple particles in helical tubes. Lebensmittel-Wissenschaft und - Technologie. Vol. 37: 387-393.

Palazoglu, T.K., Simunovic, J., Sandeep, K.P. and Swartzel, K.R. 2004. Methods, systems and devices for evaluation of thermal treatment. European Patent No. WO2004067786. European Patent Office.

Pflug, I.J., Berry, M.R., and Dignan, D.M. 1990. Establishing the heat-preservation process for aseptically-packaged low-acid food containing large particulate, sterilized in a continuous heat-hold-cool system. Journal of Food Protection. Vol. 53: 312-321.

Ramaswamy, H.S., Awuah, G.B., and Simpson, B.K. 1997. Heat transfer and lethality considerations in aseptic processing of liquid / particle mixtures: a review. Critical Reviews in Food Science and Nutrition. Vol. 37(3): 253-286.

Ramaswamy, H.S., Pannu, K., Simpson, B.K., and Smith, J.P. 1992. An apparatus for particle-to-fluid relative velocity measurement in tube flow at various temperatures under nonpressurized flow conditions. Food Research International. Vol. 25: 277-284.

Reiffel, L. 2001. X-ray imaged implanted thermometers. U.S. Patent No. 6,250,800. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Saksena, A. 2001. Systems and method for non-invasive assessment of cooked status of food during cooking. U.S. Patent No. 6,299,920. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Sandeep, K.P., Zuritz, C.A. 1995. Residence time of multiple particles in non-newtonian holding tube flow: effect of process parameters and development of dimensionless correlations. Journal of Food Engineering. Vol. 25: 31-44.

Sastry, S.K. 1986. Mathematical evaluation for process schedules for aseptic processing of low acid foods containing discrete particulates. Journal of Food Science. Vol. 51: 1323-1328.

Sastry, S.K. 1992. Liquid to particle heat transfer coefficient in aseptic processing. In Advances in Aseptic Processing Technologies. (editors: R.K. Singh and P.E. Nelson) Elsevier Applied Science, London. pp. 63-72.

Sastry, S.K. 1997. Measuring residence time and modeling the system. In Workshop targets continuous multiphase aseptic processing of foods. Food Technology. Vol. 51(10): 44-48.

Sastry, S.K., Cornelius, B.D. 2002. Aseptic processing of foods containing solid particulates. John Wiley and Sons Inc., New York. pp. 1-3, 130-181.

Sastry, S. K., Lima, M., Brim, J., Brunn, T., and Heskitt, B.F. 1990. Liquid-to-particle heat transfer during continuous tube flow: Influence of flow rate and particle to tube diameter ratio. Journal of Food Process Engineering. Vol. 13(3): 239-253.

Simunovic, J., Swartzel, K. R. and Adles, E. 2004. Method and system for conservative evaluation, validation and monitoring of thermal processing. U.S. Patent No. 6,776,523. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Singh, R.P., Heldman, D.R. 2001. Introduction to Food Engineering. 3rd edition. Academic Press, London, U.K.

Stoforos, N.G., Merson, R.L. 1991. Measurement of heat transfer coefficients in rotating liquid/particulate systems. Biotechnology Progress. Vol. 7: 267-271.

Swartzel, K.R., Simunovic, J. 1999. Method and system for residence time measurement of simulated food particles in continuous thermal food processing and simulated particles for use in same. U.S. Patent Application No. 5,932,813. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2000. Method for conservatively evaluating continuous thermal treatment process for a particulate-containing food product stream. U.S. Patent No. 6,015,231. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2003. Plurality of particles made of a detectable magnetic implant and a carrier in combination with a plurality of magnetic field sensors. United States Patent 6,536,947. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2004. System for measuring residence time for a particulate containing food product. U.S. Patent No. 6,766,699. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Tessner, W.A., Farkas, B.E., and Sandeep, K.P. 2001. Use of ablation to determine the convective heat transfer coefficient in two-phase flow. Journal of Food Process Engineering. Vol. 24: 315-330.

Tobback, P., Hendrickx, M.E., Weng, Z., Maesmans, G.J., and De Cordt, S.V. 1992. The use of immobilized enzymes as time temperature indicator system in thermal processing. In Advances in Food Engineering (editors: R.P. Singh and M.A. Wirakartakusumah). CRC Press Inc., Boca Raton, FL. pp. 561-574.

Toledo, R.T. 1991. Fundamentals of food engineering. 2nd edition. Chapman and Hall, New York, NY.

Toledo, R.T., Chang, S.Y. 1990. Advantages of aseptic processing of fruits and vegetables. Food Technology. Vol. 44: 72-78.

Zareifard, M.R., Ramaswamy, H.S. 1999. A calorimetric approach for evaluation of fluid-to-particle heat transfer coefficient under tube-flow conditions. Lebensmittel-Wissenschaft und Technologie. Vol. 32: 495-502.

Zitoun, K.B., Sastry, S.K. 1994a. Convective heat transfer coefficient for cubic particles in continuous tube flow using the moving thermocouple method. Journal of Food Process Engineering. Vol. 17: 229-241.

Zitoun, K.B., Sastry, S.K. 1994b. Determination of convective heat transfer between fluid and cubic particles in continuous tube flow using noninvasive experimental techniques. Journal of Food Process Engineering. Vol. 17: 209-228.

Zuritz, C.A., McCoy, S., and Sastry, S. 1987. Convective heat transfer coefficients for non-Newtonian flow past food-shaped particulates. ASAE Paper No. 87-6538, ASAE, St Joseph, MI.

Table 3.1 Measured F_0 values in Data Acquisition Program files compared with the calculated F_0 using temperature data file from Data Acquisition Program

Sample of the temperature and F_0 output file data			Calculated values using temperature output file data	
Time, t in s	Temperature, T in °C	F_0 in s	L(t)	$(F_0)_t$ in s
1085.66556	122.53641	112.00231	1.39200	112.00231
1085.76552	122.53641	112.14146	1.39200	112.14146
1087.26492	122.63412	114.22863	1.42368	114.22863
1087.36488	122.63412	114.37095	1.42368	114.37095
1087.46484	122.63412	114.51326	1.42368	114.51326

Table 3.2 Thermophysical properties of polymers and food materials*

Material	Density (kg/m^3)	Thermal conductivity (W/m-K)	Specific heat (J/kg-K)	Thermal diffusivity (m^2/s)
Polypropylene	910	0.13	2343	6.0972e-08
Polymethylpentene	833	0.17	1968	1.0370e-07
Carrot	1040	0.606	3864	1.5080e-07
Potato	1090	0.554	3517	1.4451e-07

*Complied from Abdelrahim *et. al.*, 1997; Palaniappan and Sizer, 1997; Singh and Heldman, 2001; Toledo, 1991

Table 3.3 Process variable inputs to CPD for particle design generation

Initial temperature of particle	20 °C
Ambient temperature	140 °C
Heat transfer coefficient	1000 W/m²K
Desired F_0	3 min
Target particle density	1000 kg/m³

Table 3.4 Wall thickness and implant weight results for different combinations of food material and polymers

Food material	Polymer	Wall thickness (mm)	Implant weight (g)
Carrot	Polypropylene	1.27	.934
Carrot	Polymethylpentene	2.54	.632
Potato	Polypropylene	1.27	.934
Potato	Polymethylpentene	2.54	.632

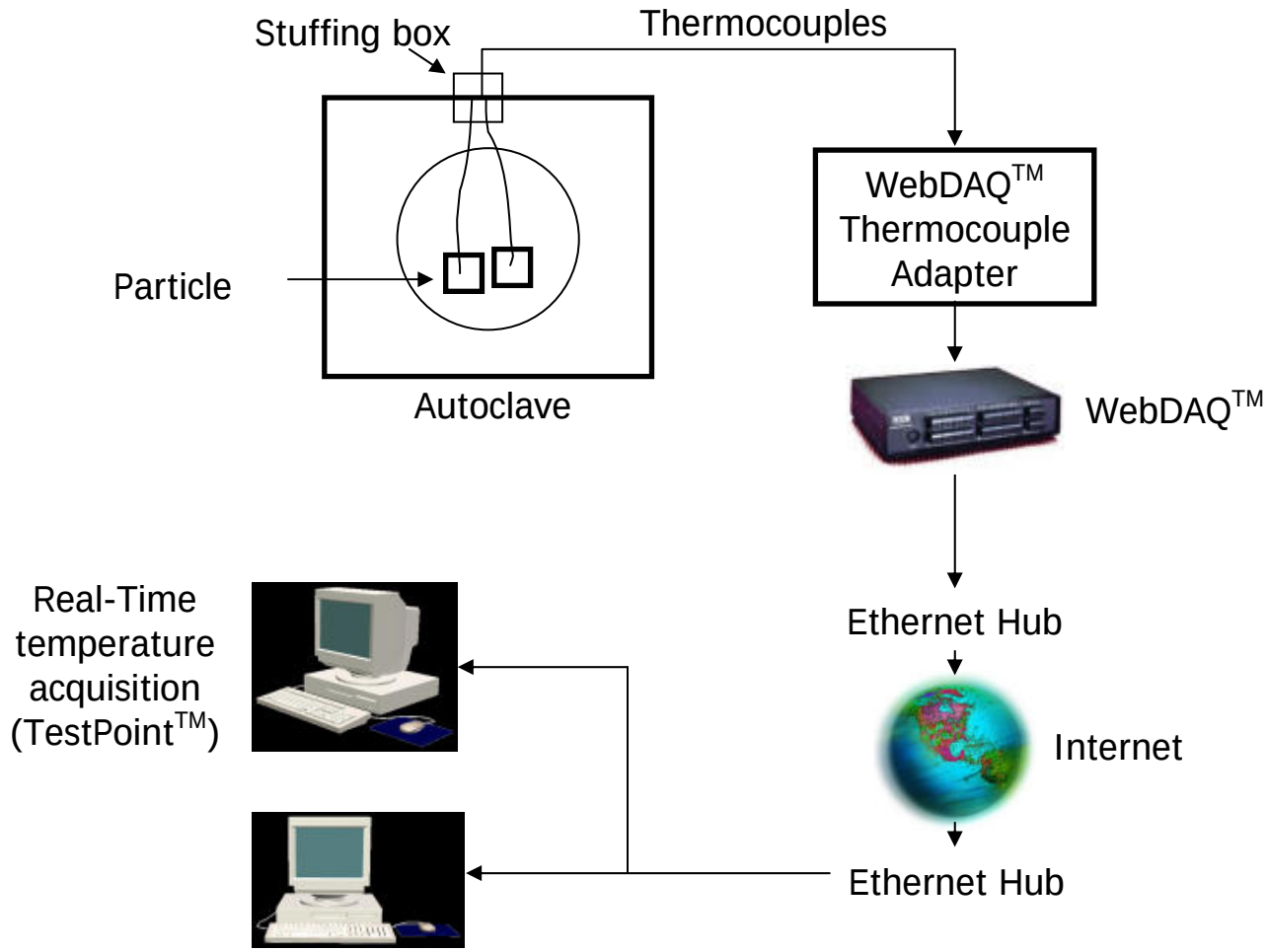
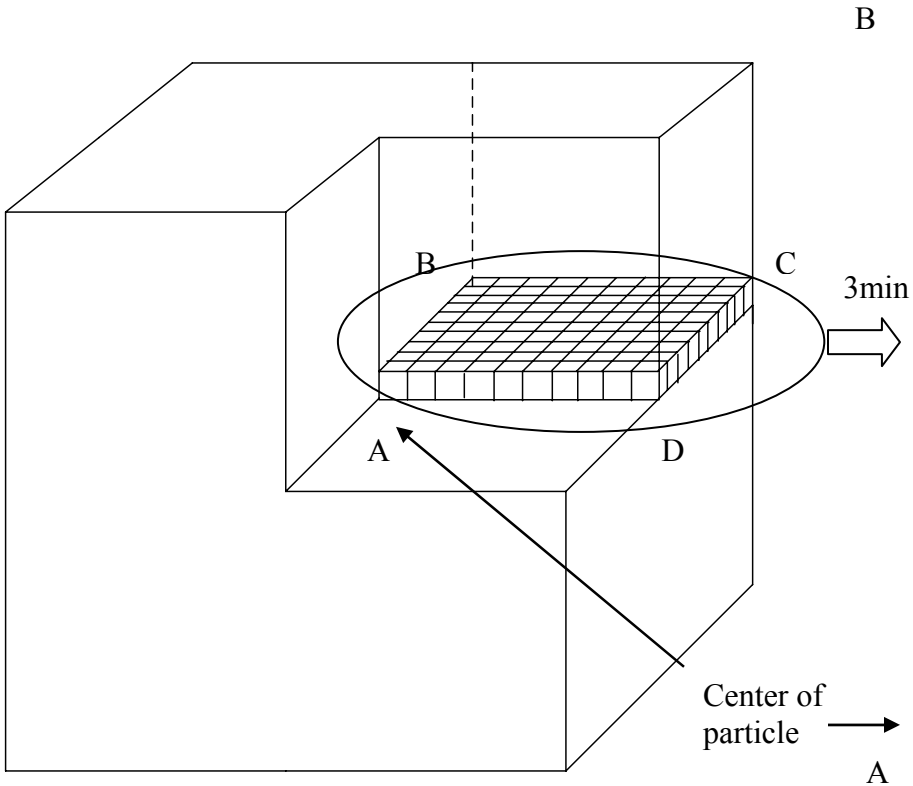


Figure 3.1 Complete experimental setup to acquire real-time time-temperature history and plot temperature and F_0 graphs on the monitor (at run-time)



42	43	45	48	53	59	68	81	100	132
14	15	16	18	21	26	33	44	63	100
6.1	6.4	7.1	8.4	10	13	18	27	44	81
2.9	3.1	3.6	4.3	5.7	7.9	11	18	33	68
1.5	1.7	2	2.5	3.4	5	7.9	13	26	59
0.9	1	1.2	1.5	2.2	3.4	5.7	10	21	53
0.6	0.7	0.8	1.1	1.5	2.5	4.3	8.4	18	48
0.4	0.5	0.6	0.8	1.2	2	3.6	7.1	16	45
0.3	0.4	0.5	0.7	1	1.7	3.1	6.4	15	43
0.3	0.3	0.4	0.6	0.9	1.5	2.9	6.1	14	42

Figure 3.2 F_0 map showing the quarter cube of a particle and the F_0 values at different points inside the cube

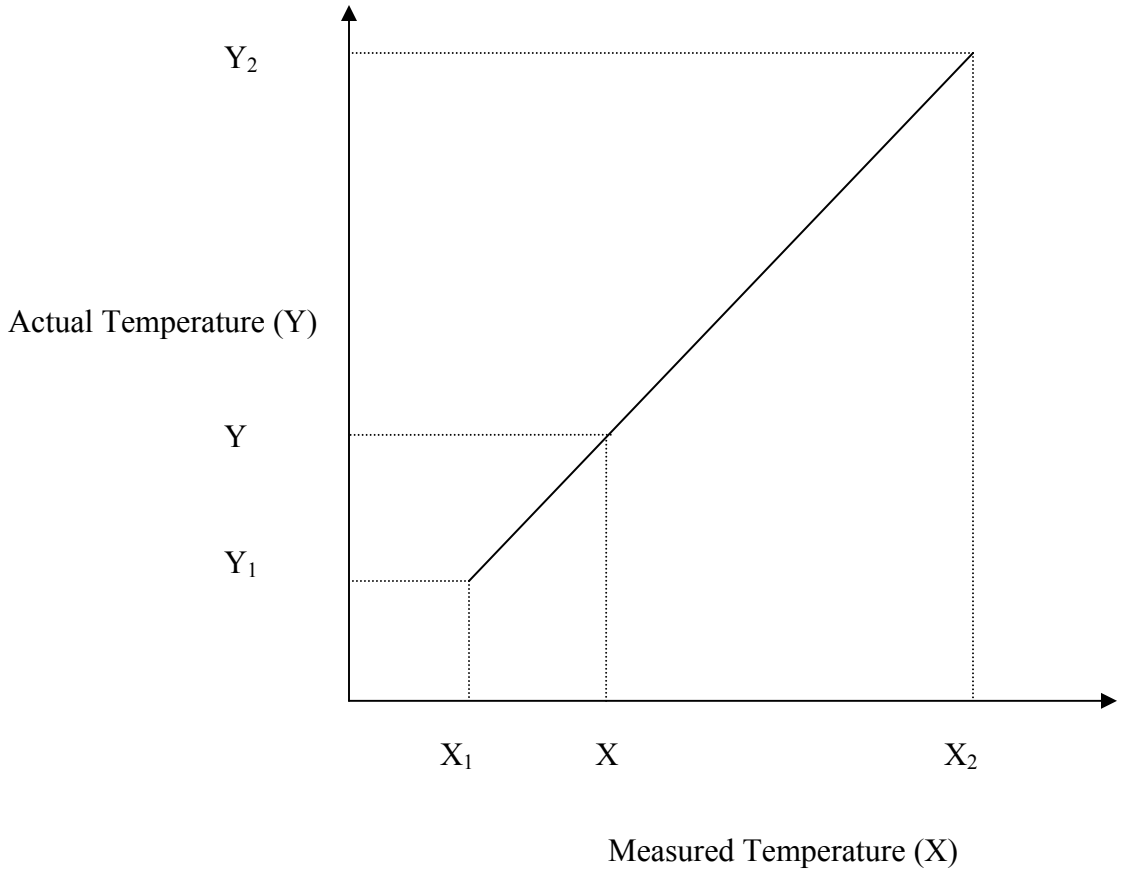


Figure 3.3 Linear relation drawn for determining the actual temperature of a thermocouple (Y) at a measured temperature (X) using the calibration data (Y_1 and Y_2) at ice cold (X_1) and boiling point (X_2)

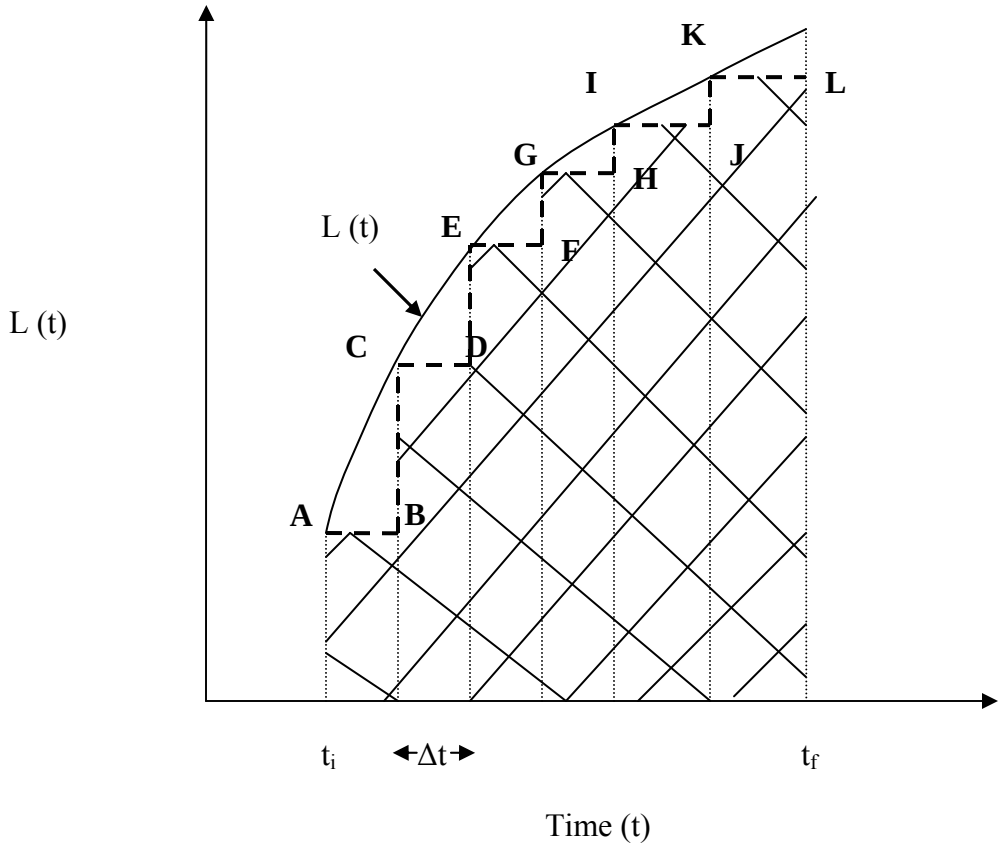


Figure 3.4 Shaded area under the stepwise point function ABCD… which was used by Data Acquisition Program for F_0 value calculations

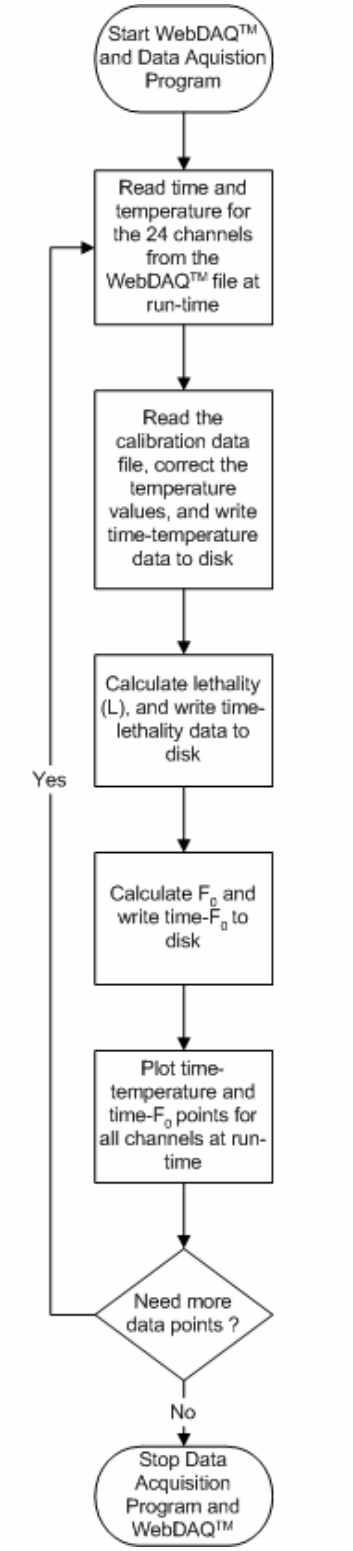


Figure 3.5 Flow diagram showing a brief logic for Data Acquisition Program

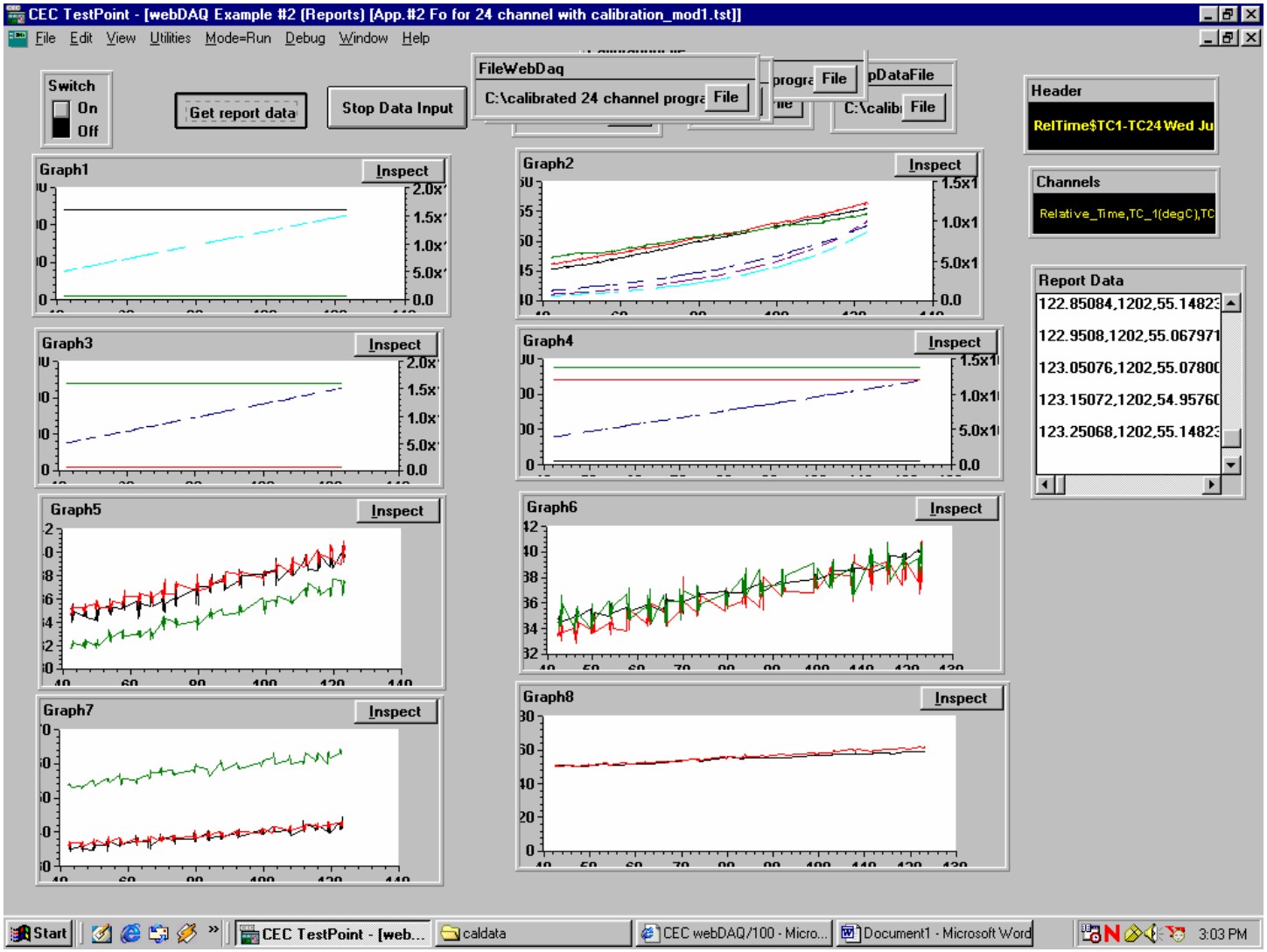


Figure 3.6 A Screen shot of the Data Aquistion Program showing the real-time acquired temperature and F_0 values with their graphs

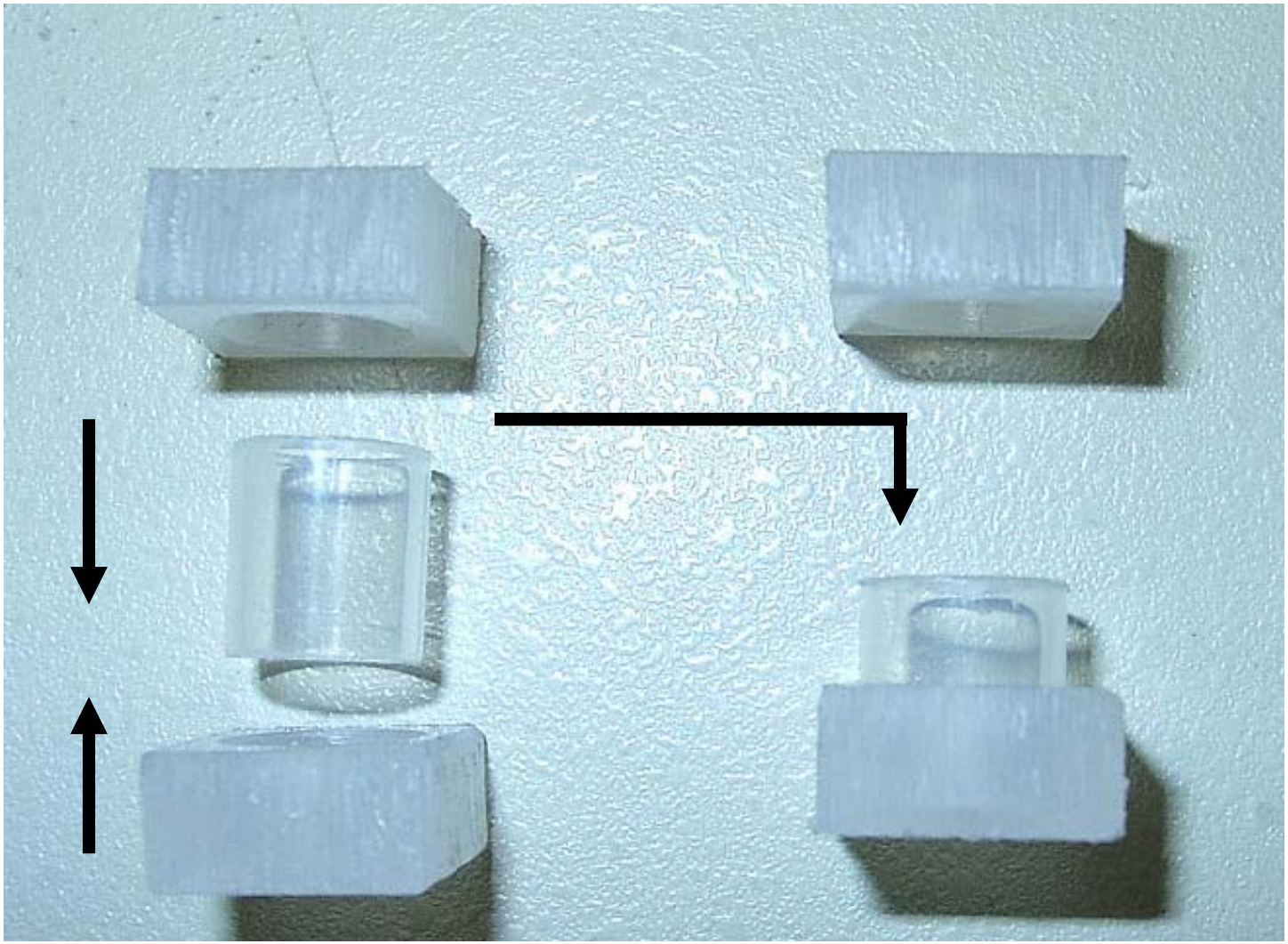


Figure 3.7 A simulated food particle made of PP, showing the two half of the cube (with cavity) and the cylindrical tube, which are joined to complete the particle

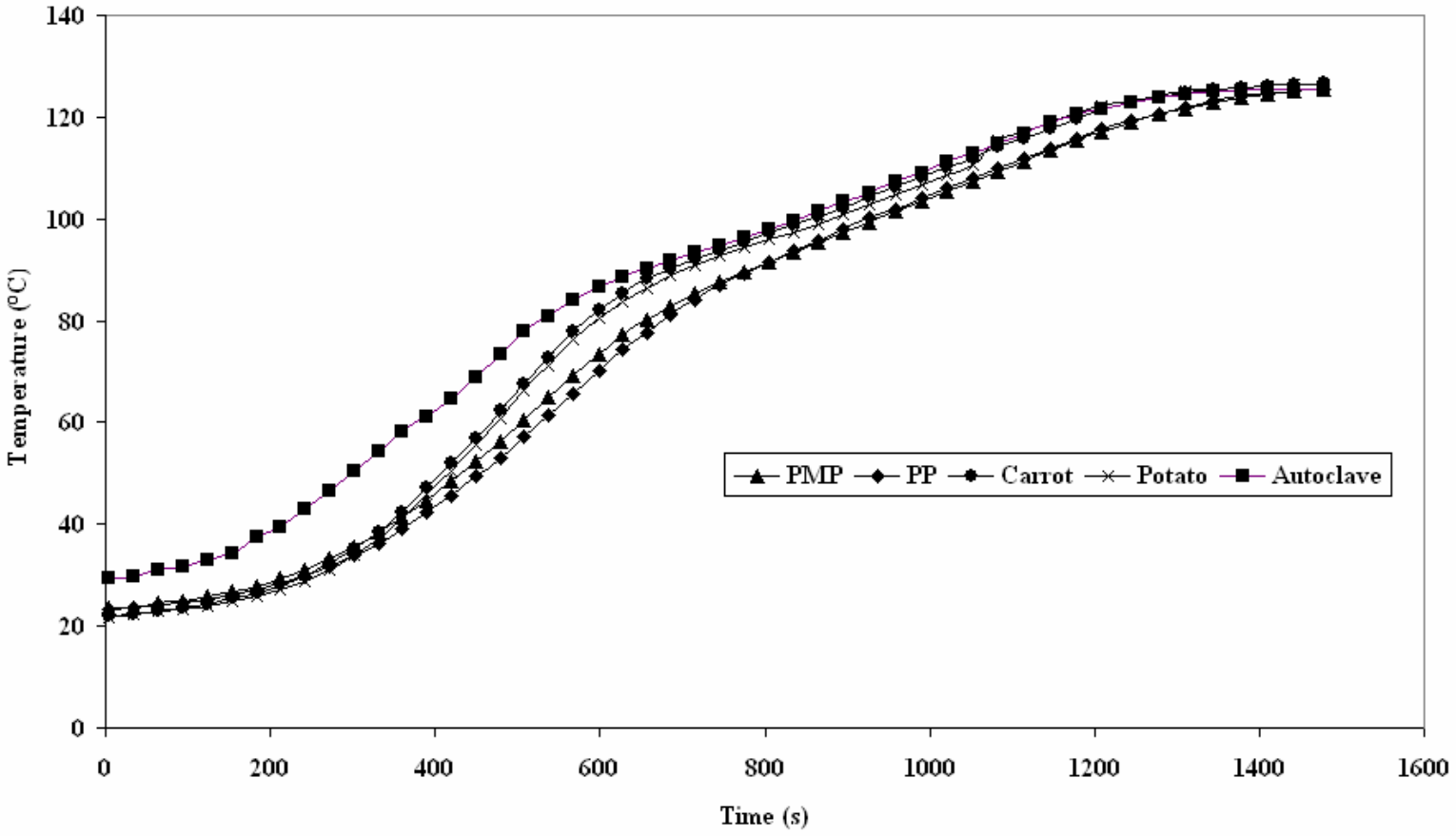


Figure 3.8 Temperature acquisition data for potato, carrot, PP and PMP under high pressure and temperature conditions

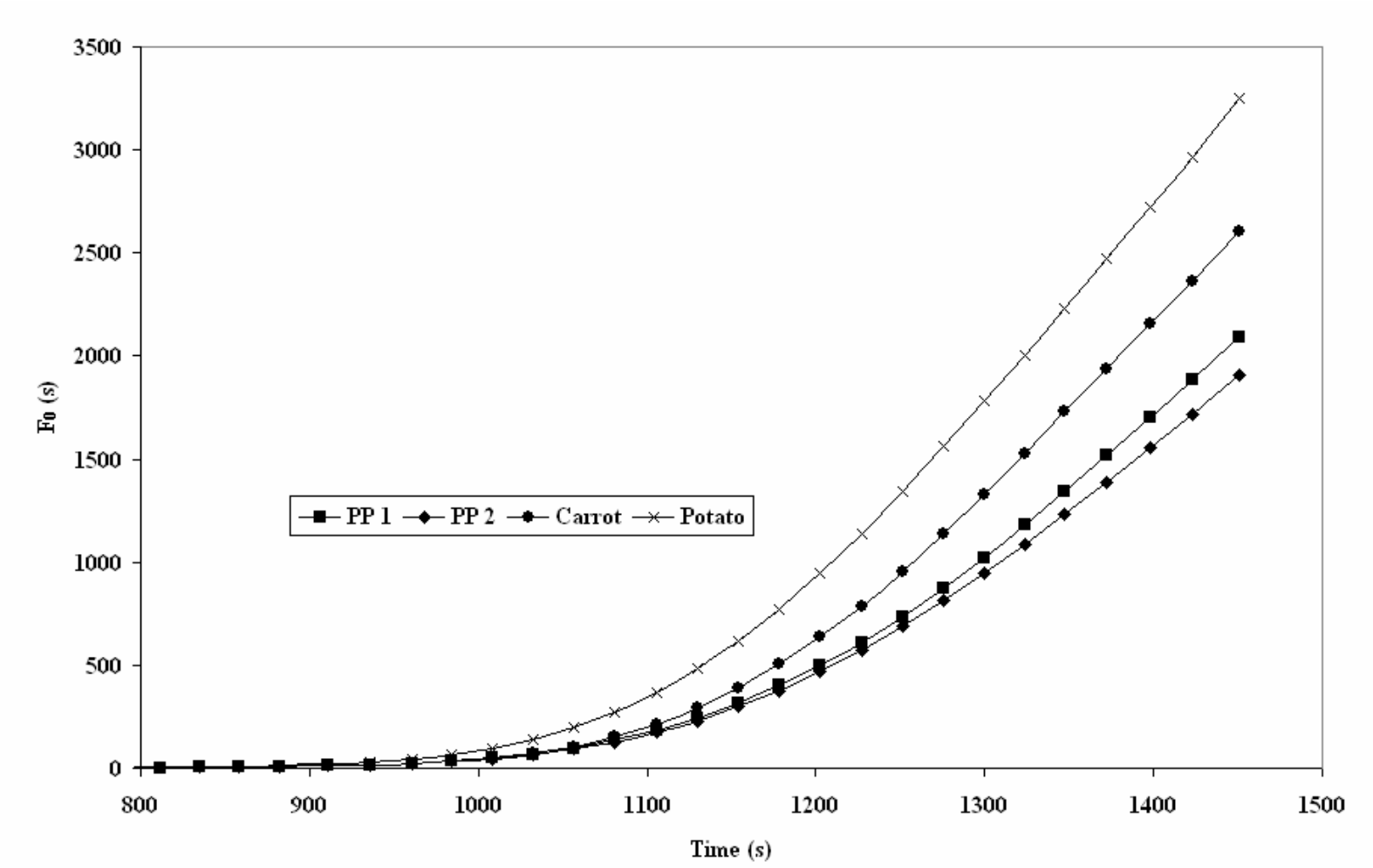


Figure 3.9 F_0 values for carrot and potato with PP under high pressure and temperature conditions

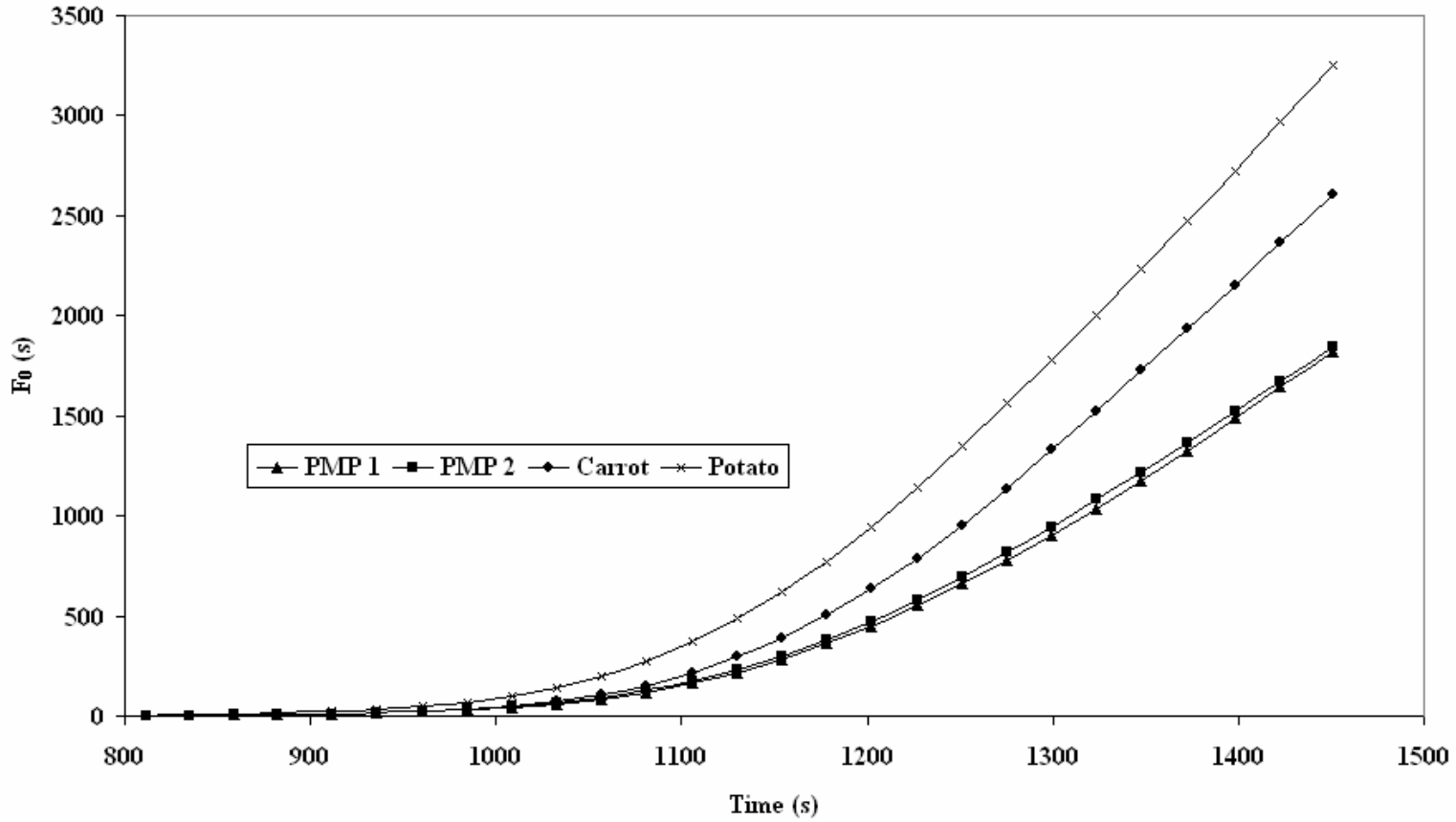


Figure 3.10 F_0 values for carrot and potato with PMP under high pressure and temperature conditions

Chapter 4

MANUSCRIPT II

Thermal Characteristics of Simulated Polymer and Food Particles: Criteria for Establishment of Conservative Properties for use in Multiphase Aseptic Process Validation

A.K.S. Jasrotia
J. Simunovic
K.P. Sandeep
T.K. Palazoglu
K.R. Swartzel

Department of Food Science, North Carolina State University

Please address all correspondence to:

K.P. Sandeep
129 Schaub Hall, Box 7624
North Carolina State University
Raleigh, NC 27695
Phone: 919-515-2957
Fax: 919-515-7124
E-mail: kp_sandeep@ncsu.edu

Paper No. FSR-04-XY of the Journal Series of the Department of Food Science, North Carolina State University, Raleigh, NC 27695-7624.

ABSTRACT

Simulated food particles with conservative (fast-moving and slow-heating) properties are required for validation of multiphase aseptic processing for production of shelf-stable low-acid foods. These validations require simulated particles to contain residence time tags, thermo-sensitive implants and/or bio-loads for temperature detection, time-temperature integration, and bactericidal efficacy confirmation. Conservative Particle Design (CPD) software was used to determine the wall thickness and implant weight required for conservative behavior of such particles made with polypropylene and polymethylpentene, when compared to ½ “cube size carrot and potato. Thermocouples were fitted in simulated and real food particles and heated up to 127 °C under pressurized (24 psi) conditions. Other food materials were then tested to compare their thermal heating behavior with respect to these simulated food particles. In-addition heat penetration studies on simulated food particles made with polypropylene and polymethylpentene (wall thickness 1 mm and 2mm), with different configuration (changing tube insert diameter) were performed to observe thermal heat penetration changes with changing tube insert and wall thickness. Both 1mm and 2 mm particles showed that decreasing the insert tube diameter increases conservative thermal property of the simulated food particle. This would allow a food processor to employ these designed particles and choose a right configuration to validate an aseptic process for a multiphase food containing (any or) all the above tested food materials.

Keywords: validation, multiphase aseptic processing, time-temperature history, conservative particle design, critical particle

INTRODUCTION

Aseptic processing offers many advantages such as improved quality of the product and less labor and time required for the process. Unlike, traditional canning, where products are heated in the container for several minute or hours, aseptic processing involves rapid heating and cooling in a sterile environment, followed by aseptic packaging which substantially reduces the energy use and nutrient loss associated with conventional sterilization. In Europe, where aseptically processed foods containing particulates like stews and sauces, have been a household staple for over three decades (Morris-Lee, 2004), microbial testing of such foods at the end of their shelf life is performed to find the safety of the product. However, in the U.S., multiphase processing of food must be validated before the product can be produced commercially. The main hindrance in commercializing aseptic processing of multiphase foods has been the costly validation techniques (Larkin, 1997; Higgins, 2004). Thus significant research has been done and is still needed for reducing the cost of validation for multiphase aseptic processing of foods (Dignan *et al.,* 1989; Heldman, 1989; Toledo and Chang, 1990; Lund and Singh, 1993

The geometric center of a food particle is the critical point of interest since under conventional heating conditions, it is the slowest heating point. Therefore, the challenge in such a system is to quantify the thermal treatment received by the food particulate at its center. The thermal treatment in any thermal process can be quantified using the F value (for a given z and T_{ref} values) definition (David, 1996):

$$F_{T_{ref}}^{z} = \int_{t_i}^{t_f} 10^{(T(t)-T_{ref})/z} dt \qquad (17)$$

where, T is the temperature (time function), T_{ref} is the reference temperature (usually 121.1 °C), t_i and t_f are initial and final process time respectively and z (in °C) is a temperature dependent characteristic of the microorganism and is assumed to be constant over normal processing conditions. In the above equation, z is defined as the increase in temperature to decrease the decimal reduction time of the target microorganism by one log and F_0 represents the F value for T_{ref} = 121.1 °C and z = 10 °C (*Clostridium botulinum*). Time-temperature histories at the center of the food particles have been measured for thermal treatment in a continuous flow system using different methods. A multiphase aseptic processor must identify and select a sterilizing F_0 value for the product, develop a conservative model that reliably predicts the total lethality of the heat process, quantitatively verify the lethality delivered, and list the critical factors of the process and the procedures to be used for controlling them (Dignan *et al.,* 1989). Heat penetration measurements for a food particle traveling through an aseptic processing system is difficult and not practical at the present time without restricting the free movement of food particles (Sastry, 1986; Lee and Singh, 1990; Heldman, 1992; Lund and Singh, 1993; Maesmans *et al.,* 1994). Due to challenges encountered in measuring the temperature of moving particles in aseptic processing systems, several alternatives have been considered such as biological validation techniques (Heppell, 1985a, b; Berry, 1989; Tobback *et al.,* 1992; Cacase *et al.,* 1994), moving thermocouple methods (Sastry, 1992; Zitoun and Sastry, 1994a), liquid crystal technique (Stoforos and Merson, 1991; Balasubramaniam and Sastry, 1994a; Zitoun and Sastry, 1994b), time temperature integrators (Hendrickx *et al.,* 1992; Guiavarc'h *et. al.* 2002a; Guiavarc'h *et. al.*, 2002b)

melting point indicators (Mwangi *et al.,* 1993), and relative velocity methods (Balasubramaniam and Sastry, 1994b).

Determination of fluid-to-particle heat transfer coefficient (h_{fp}) poses a unique challenge of monitoring the temperature of moving particles without restricting the flow of the particle (Chandarana *et al.*, 1989; Zareifard and Ramaswamy, 1999; Ramaswamy *et al.*, 1997, Tessner *et al*., 2001; Zuritz *et al.*, 1987; Sastry *et al*., 1990; Masesmans *et al.*, 1992; Sastry, 1997; Sastry and Cornelius, 2002). In aseptically processed particulate product biological validation of the process is necessary because the design of the aseptic process is not based on heat penetration tests due to the unavailability of the time-temperature history of the product (Chandarana, 1992). Biological validation of an aseptic process for a particulate food product involves the use of biological indicators that travel through the system inside a particle (Pflug *et al.*, 1990; Marcy, 1997). In an important workshop (CAPPS and NCFST Workshop, 1995, 1996) various methods for determining the RTD measurements were discussed. In an attempt to successfully validate a multiphase aseptic process for the first time a filing was done by Tetra Pak (1997) for diced potato soup with FDA. The filing received a no-objection letter from FDA (Palaniappan and Sizer, 1997). However, the cost of validation of this process is very high. Therefore, reducing the cost for validation techniques has been one of the key areas of focus in multiphase aseptic processing research.

Monitoring and validation of continuous thermal pasteurization and sterilization process typically requires the use of simulated food particles, which must exhibit conservative flow and thermal characteristics. In several multiphase studies, simulated food particles such as polystyrene spheres, alginate beads, and rubber cubes have been

used (Sandeep and Zuritz, 1995; Palazoglu and Sandeep, 2002, 2004; Fairhurst *et al*., 1999; Dutta and Sastry, 1990a, b). Real food particles such as carrot and potato cubes (Ramaswamy *et al.*, 1992; Alhamdan and Sastry, 1997; Abdelrahim *et al.*, 1997). Due to various challenges in accurately determining the temperature histories at the center of a particle flowing in a continuous system, alternatives have been considered. Researchers in the past have suggested the use of conservative mathematical models to predict the total lethality of the heat process during aseptic processing of low-acid food particulates foods. In-addition, RTD measurement is needed because of the difficulties in non-invasive measurement of internal temperatures of particle during continuous flow (Sastry, 1986; Chandarana and Gavin, 1989; Sastry, 1997). In a recent research work, a method of generating residence time measurement of a particulate-containing food product while passing the product as a continuous stream through a thermal processing apparatus was developed (Swartzel and Simunovic, 1999, 2000, 2003, 2004).

Small paramagnetic particles in the interior of simulated and real food particles (Ghiron and Litchfield., 1997; Ghiron and Litchfield, 1998), in-process measurement of internal particulate temperature utilizing ultrasonic tomography (Beller, 1993), method based on change in proton precession frequency with temperature(Kantt *et. al.*, 1997), Magnetic Resonance Imaging (MRI) (Kantt *et. al.*, 1998) and micro-thermometry (Reiffel, 2001; Higgins, 2004) have been proposed for real time-temperature measurements in food particulates for validation. Saksena (2001) developed a non-contact system and method was provided for effectively approximating the internal temperature of food being cooked upon a cooking surface. In another study (Palazoglu *et. al.*, 2004; Simunovic *et. al.*, 2004) a magnetically detectable particle and related methods,

systems, and devices are provided for generating a temperature measurement for a batch or a continuous stream of material.

Thus, developing a conservative particle and testing its thermal behaviors for different design parameters is important in validation of aseptic multiphase foods. In a current study a systematic procedure for designing simulated food particles with conservative flow and thermal behavior (fast-moving and slow-heating) was implemented and different food materials were compared with these designed particles by performing heat-penetration studies. Additionally, changes in wall thickness were shown to change the thermal properties of these simulated food particles. A systematic approach for designing and experimentally confirming the conservative behavior is thus of great importance to determine time-temperature history, and residence time distribution, for such particles in a multiphase aseptic system. Availability of such particles at reduced cost would reduce the complexity of process documentation and filing with regulatory agencies and bring aseptic multiphase foods closer to commercial reality.

MATERIALS AND METHODS

Experimental setup

Figure 4.1 shows the components of the experimental setup. A bench top autoclave (Pelton Crane Omni-Clave OCM Sterilizer, Kroslak Enterprises Inc., Riverview, FL) was used. This equipment is used for providing a sterilization environment for surgical and clinical instruments and was used at pressures up to 24 psi

and temperatures up to 127 °C. A stuffing box was used to enable the coupling of the autoclave with the thermocouple system. This fitting must be equipped with an appropriate gasket, so that it does not leak at high pressures.

The thermocouples used were teflon-coated type T thermocouples (Marlin Manufacturing Corporation, Cleveland, OH). WebDAQ/100™ (Capital Equipment Corp., Bedford, NH) was used to acquire time-temperature data coupled with two thermocouple adapter boards (Capital Equipment Corp, Bedford, NH); designed to provide a solution to the connecting thermocouples for measuring temperatures. Time-temperature data were acquired at 3000 Hz with an output rate of 10 Hz.

Conservative Particle Design

Construction of simulated food particles (polymer-based) used in the current study was based on the Conservative Particle Design (CPD) software. The CPD software (Palazoglu *et. al.*, 2004) can be used to determine the design parameters (namely wall-thickness and implant weight) for cubic, spherical, and cylindrical particles made of a selected polymer (thermal diffusivity of which is lower than that of the food particle being simulated). This particle provides a level of thermal protection to its cavity greater than or equal to the thermal protection provided by the food particle with identical shape and dimensions to its geometric center. This simulated particle can then be used as an magnetic implant carrier when developing a continuous sterilization flow system for validation of aseptic processing for particulates foods.

In CPD, this is achieved by simulating concurrent heating of a theoretical polymer particle and a theoretical food particle under identical sterilization-level conditions. For this, both particles are divided into hypothetical grids. During these simulated

sterilization treatment calculations, F_0 value accumulated in each grid is determined. Since thermal diffusivity of most (not all) polymer particles is lower (i.e., they heat slower) than most food particles of equal size and shape, the accumulation of F_0 values will be slower and will not penetrate as deep in a simulated particle.

Design selection is made once the center of the food particle has reached the target F_0 (e.g. 3 minutes), the F_0 map (Figure 4.2) of the concurrently heated simulated food particle is examined to determine the points (depth) to which the penetration of target F_0 value or lower has progressed under these identical heating conditions – all points deeper within the particle beyond this level will therefore be thermally protected at least as well or better than the protection provided by the food particle to its geometric center. The depth of the penetration of this pre-selected F_0 value into the particle establishes the most important construction criterion – particle wall thickness. By removing the material within the "equal protection zone" established in this way, a cavity is generated within each fabricated particle that is supposed to be at least as equal or better thermally protected than the centers of real food particles, i.e., an implant carrier cavity or a "cold spot" carrier cavity. Theoretically, anything placed in that cavity will under the identical heating conditions receive less thermal treatment than the center of a real food particle of identical shape and dimensions. Therefore, if for example, we place a time-temperature recording device within the particle cavity generated in this manner, and process a multiphase stream of product until the time-temperature exposure to the device placed in this cavity accumulates an F_0 value of three minutes or more, we will be assured that *everything else* within the system will have received *at least* this same treatment level.

Simulated particles must also to be constructed to provide a fast flowing behavior (in addition to this slow heating behavior). This is achieved separately but concurrently by adjusting their effective density to a value slightly lower than the carrier fluid density – this is applicable to vertical and slightly inclined (as required by regulatory agencies) heat exchangers and hold tubes only, for all other configurations this "critical density" needs to be determined experimentally. In CPD, one can also input the process variables such as the initial particle temperature, ambient temperature, heat transfer coefficient, desired F_0 and density of the target particle. This is to ensure that the particle is not denser than the fluid in the system and is thus having desired flow properties for the RTD measurements.

The CPD software is used for determining the starting point for the construction of the conservative particles. The CPD software requires the user to choose the type of food (carrot, potato, beef or other types of food particles, provided their appropriate property data is available), kind of material that would be used for the construction of the simulated particle (nylon, polypropylene (PP), teflon, polymethylpentene (PMP) or other plastic materials with known heat penetration properties), shape of the particle (spherical, cubical or cylindrical) and the size of the food and simulated particle (half thickness for cube). Present study deals with the cubical shape particles. The results are generated as a lethality chart that shows the demarked region in a one-eighth portion of the particle (Figure 4.2). The line shows the region inside which, the simulated particle received an accumulated F_0 value of less than three minutes (12D reduction process for *Clostridium botulinum*).

Calibration of thermocouples

Calibration of the thermocouple system is required due to WebDAQ/100™ adapter and thermocouple installation. Calibration was performed by measuring all 24 channel temperature readings at ice cold water and boiling water. A mercury thermometer (Ertco Ever Ready Thermometer Company, Inc., Dubuque, IA) was used to measure the ice cold and boiling water temperature, to compare them with the measured values of thermocouples at the same time. The thermometer had a lower scale limit of 2 °F and could measure temperatures in the range of 0-220 °F. The correction data for calibration is stored in a file which is later read by the data acquisition system to integrate the corrections for each channel during a real time acquisition. This is done by storing the measured temperature of all the thermocouple channels at two calibration points (ice and boiling water) which would be used to correct the measured temperature at the run time and display the corrected temperature and F_0 values on the Data Acquisition Program. To understand the calibration methodology, graph between the measured temperature (x-axis) and the actual temperature (y-axis) at ice cold bath and boiling water for each thermocouple channel in the system can be drawn and an equation is obtained for determining the actual temperature for that particular channel, if the measured temperature is known. The measured temperature is acquired by the Data Acquisition Program over the network from the WebDAQ/100™ system. Figure 4.3 shows this simple method of determining the actual temperature for a thermocouple channel. It can been seen from Figure 3.1 that if X_1 = 0 °C (ice), X_2 = 100 °C (boiling water), Y_1 = measured temperature of thermocouple at X_1 and Y_2 = measured temperature of thermocouple at X_2 is know, then for any give X (measured temperature) the Y value (actual temperature) can be determined using a linear relationship assumption. A linear

relation between Y and X was assumed since the variation of measured to actual values over the range Y_1 to Y_2 was not significant as can be seen from Table 4.1. The working ranges for the current system were 25-127 °C, in which linear relation holds well.

Thus the equation for determining the actual temperature (Y), at any reading time point for a particular channel would be:

$$Y = \left(\frac{Y_2 - Y_1}{X_2 - X_1} \right)(X - X_1) + Y_1 \tag{18}$$

Data Acquisition Program

TestPoint v4.1™ software (Capital Equipment Corp, Bedford, NH) was used to develop the customized programs to acquire temperature data. It provides high-level analog-to-digital (A/D) functions to simplify data acquisition.

The Data Acquisition Program (using in TestPoint v4.1™) developed to monitor the two parameters – temperature and F_0 value, in both the center of real food particles and within the simulated particle "cold-spot carrier" cavity was developed. These calibrated temperature and F_0 values are plotted and displayed real time. The program polls the WebDAQ/100™ data acquisition device, accessible over a network, to generate the temperature measurements, and F_0 value data files for a 24 channel acquisition system. The WebDAQ/100™ system also included two thermocouple adapter boards to convert analog temperature reading to digital data.

The F_0 measurement logic incorporated in the program is illustrated by Figure 4.4 From equation (1) we assume a function L(t) – the lethality rate as:

$$L(t) = 10^{(T(t) - T_{ref})/z} \tag{19}$$

The integral of equation (3), over the processing time range of t_i to t_f, which is the area under L(t) on Figure 4.4 would thus determine the F_0 value for the heat process – T(t) . Due to the limitations of the data acquisition and measurement system, an exact integral cannot be calculated as the measured time-temperature data is recorded at certain frequency (depending on the sampling rate/output rate of WebDAQ/100™). From Figure 4.4 it can be seen that the shaded areas under step wise function (points ABCD…) can be calculated as:

$$(F_0)_{t+\Delta t} = (F_0)_t + (L(t) * \Delta t) \tag{20}$$

where $(F_0)_t$ is F_0 at time t. Here, T_{ref} = 121.1 °C and z = 10 °C. Also:

$$F_0 = 0 \text{ at } t_i = 0 \tag{21}$$

From Figure 4.4 it can be seen that the area of the shaded region would be less than the integral in equation (1). Given the limitations of the programming software, this would be beneficial, since we would be conservative in F_0 calculation and thus would ensure the product safety. The T(t) is the corrected temperature of any single thermocouple channel in consideration, and is calculated using equation (2). A flow diagram explaining general steps involved in Data Acquisition System is shown in Figure 4.5. All of these calculations are carried out at run time within the Data Acquisition System, and the results for corrected temperature and F_0 values are displayed on the monitor screen Figure 4.6.

Experimental Measurements

The particles fabricated using the above tools and methodology were used to perform all the experimental runs. Duplicate particles made of PP and PMP with cube size of half inch were used in the study. Thermocouples were fitted inside the cavity of

simulated particles and at approximate center of the real food particles. The runs were performed with pressures close to 24 psi and temperatures of up to 127 ^{o}C. These particles were then compared for their conservative behavior with various food materials, other than potato and carrot. The food materials tested were zucchini, yellow squash, okra, purple eggplant, lima beans and string beans. PMP with wall thickness 1 mm and 2 mm made using four and three different insert tube layers respectively were used to test the thermal property and behavior changes, as wall thickness and tube insert varies. Acquired F_0 and temperature data were analyzed using Microsoft® Excel (Microsoft® Corporation, Redmond, WA) spreadsheet software.

RESULTS AND DISCUSSION

The output data for the Data Acquisition Program was stored in temperature and F_0 value files. The F_0 values measured and recorded in this file were compared with calculated F_0 data obtained using the measured temperature data file. The result for this comparison is shown in Table 4.1. This comparison is performed to make sure that the program is calculating the F_0 values correctly using its measured temperature data file at run time, as explained in equations (1) to (5). It can be seen that the program calculates these values as expected incorporating the correction from the calibration file. Also the program assigns a value of zero for F_0 at start of the experimental, as mentioned in equation (5).

Conservative Particle Design (CPD) software was used to generate the simulated particle design for comparison with potato and carrot of ½ inch cube size. CPD determined wall thickness and implant weight required for the polypropylene (PP) and

polymethylpentene (PMP) particles. Thermo-physical properties values of polymers, PP and PMP and food materials, carrot and potato are shown in Table 4.2. It can be seen that the thermal diffusivity values for PP and PMP are lower than those of carrot and potato, implying that these polymers would heat slower than carrot and potato. Also PMP has a higher thermal diffusivity than PP, therefore heat penetration would be faster for PMP when compared to PP. Parameters such as thermal conductivity, specific heat and density of carrot, potato, PP, and PMP used as input values to generate design for PP and PMP. Input process variable listed in Table 4.3 were not changed for any of the design configurations in this study. These are conditions that were assumed to occur in a normal experimental setup for this study.

Wall thickness and implant weight determined by CPD for fabricating the simulated particles for different combination of polymers and food materials, are shown in Table 4.4. These particles are expected to exhibit conservative properties and are representative of the least thermally treated particle in the system. This is tested experimentally in different runs. It is assumed that these particles would be used in validation for a continuous flow medium of average density equal to that of water (target particle density in Table 4.3 as 1000 kg/m^3). This would make sure that the particle is neutrally buoyant while thermally protecting its "cavity" equal to or better than the protection received by the rest of the real food particle's geometric center in the system.

Figure 4.8 and Figure 4.9 shows the F_0 acquisition data for the run with PP, PMP, carrot and potato. The data clearly shows that the simulated food particles heated slower than the food particle, showing a thermally conservative behavior. Many similar runs were performed and it was seen that the temperature in the cavity of the simulated food

particles always remained lower than that at the center of the real food particles. As can be seen, the F_0 accumulated by carrot and potato is always higher than that for either PP or PMP. Also many similar runs were performed and the data analyzed. Therefore, the slow heating behavior of these particles was proved.

Figure 4.10 to Figure 4.13 show the F_0 value comparisons for different food materials when compared with the above particles designed for potato and carrot. These foods and their physical dimensions are listed in Table 4.5. It can be seen that PP heats faster than PMP in all the runs. Also PMP behaves conservatively for all the food materials tested except Yuka (Figure 4.12). PP shows an overlapping nature for okra and purple eggplant and less conservative nature for zucchini, and therefore cannot not be used for validation of these food materials (Figure 4.10). Figure 4.11 show that PP did not behave conservatively for beans and green pepper, however PMP does show thermally conservative behavior. Figure 4.12 shows that PP particles did not have conservative characteristics when compared against sweet potato, colocasia, yuka, and beet root where as, PMP is conservative for all of them. Very similar results are shown for PP and PMP in Figure 4.13.

Varying inserts (joining two half of particle) with different inner diameters were used to change the conservative behavior of the particle. Figure 4.14 shows the thermal penetration behavior of PMP particles made of 1 mm wall thickness with inserts of inner diameters of 0.312, 0.187, 0.170 and 0.125 inches. This would help in studying and adjusting the conservative properties for these simulated particles by varying the dimensions of the internal connecting inserts, thereby effectively varying the particle wall thickness protecting the internal, implant-carrying cavity. If these new particles show a

conservative behavior with respect to the previously designed particles, then they can be used for all those food materials that are conservative to the original particles. Figure 4.15 shows the thermal penetration behavior of PMP particles made of 2 mm wall thickness with inserts of inner diameters of 0.250, 0.170 and 0.125 inches. It can be seen that as the particle insert diameter (and therefore the internal cavity size) decreased from 0.312 to 0.125 for 1 mm wall thickness PMP particles, an increase in the conservative behavior of the particles was observed (Figure 4.14). Similar trend was observed for 2 mm wall thickness PMP particles (Figure 4.15), when the diameter was decreased from 0.250 to 0.125 inches. This suggests that the cavity of the particles is better protected with increase in thickness of the insert (lesser inner diameter). Thus food materials like yucca, which heated slower than the original PMP particles can be tested with 1 mm or 2 mm particles with inserts of smaller diameter. This would help in choosing a configuration of particle that would be conservative and thus suitable for aseptic processing of food containing yucca particulates. Also if a configuration shows conservative thermal property for all the food materials in a product, then this particular particle configuration ~~alone~~ can be used to validate multiphase aseptic processing containing multiple particle components.

CONCLUSIONS

A method for fabrication and validation of implant-carrier simulated particles with conservative thermal properties for monitoring and validation of continuous thermal processing of multiphase food products has been developed and implemented. The results

demonstrate the procedure and feasibility of implementing a conservative carrier particle design for process monitoring and validation. To use these particles for validation of multiphase aseptic processes we need to be confident that they will provide a conservative thermal protection when used for validation of any aseptic multiphase process under similar processing conditions. For this the fabricated particles were experimentally validated and the experimental data was analyzed using Microsoft® Excel (Microsoft® Corporation, Redmond, WA).

The developed Data Acquisition Program can be used to calculate F_0 as per the requirement for safety (microbial destruction) or quality (nutrient loss) calculations. It incorporates the corrections to the measured temperature readings at run time and also calculates the F_0 values, displaying them on the computer display in real time. In addition, the temperature data file could also be used as a post-process record to calculate F_0 or other parameters with tools like Microsoft® Excel (Microsoft® Corporation, Redmond, WA).

A method to compare conservative thermal behavior for different food materials along with a method to change the thermal properties to adjust the conservative behavior was developed and illustrated. This would allow a food processor to employ these designed particles and choose the appropriate configurations to validate an aseptic process for a multiphase food containing different food materials. Developed tools and methods could be used by food processors and equipment providers to collect process validation data needed for process safety documentation and filing with regulatory agencies; as well as comparison of various products, processes and equipment properties.

ACKNOWLEDGEMENTS

Support for the research study undertaken here, resulting in the publication of Paper No. FSR-04-XY of the Journal Series of the Dept. of Food Science, NCSU, Raleigh, NC 27695-7624, from USDA-NRICGP and North Carolina Agricultural Research Service is greatly acknowledged.

The use of trade names in this publication does not imply endorsement by the North Carolina Agricultural Research Service of the products named nor criticism of similar ones not mentioned.

SYMBOLS

D-value	decimal reduction time	min
F_0	sterilization value	min
h_{fp}	fluid-to-particle heat transfer coefficient	W/m^2-K
T	temperature	°C
t	time	min
z-value	temperature change required to change D-value by a factor of ten	°C

Subscripts

ref	reference

Abbreviations

CAPPS	Center for Advanced Processing and Packaging Studies
CPD	Conservative Particle Design
FDA	Food and Drug Administration
NCFST	National Center for Food Safety and Technology
PMP	polymethylpentene
PP	polypropylene
RTD	residence time distribution

REFERENCES

Abdelrahim, K.A., Ramaswamy, H.S., and Marcotte, M. 1997. Residence time distribution of meat and carrot cubes in the holding tube of an aseptic processing system. Lebensmittel-Wissenschaft und -Technologie. Vol. 30: 9-22.

Alhamdan, A., Sastry, S.K. 1997. Residence time distribution of food and simulated particles in a holding tube. Journal of Food Engineering. Vol. 34: 271-292.

Balasubramaniam, V.M., Sastry, S.K. 1994a. Liquid-to-particle heat transfer in non-Newtonian carrier medium during continuous tube. flow. Journal of Food Engineering. Vol. 23: 169-187.

Balasubramaniam, V.M., Sastry, S.K. 1994b. Convective heat transfer at particle-liquid interface in continuous tube flow at elevated fluid temperatures. Journal of Food Science. Vol. 58: 675-681.

Beller, L.S. 1993. Ultrasonic tomography for in-process measurements of temperature in a multi-phase medium. U.S. Patent No. 5,181,778. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Berry, M.R. 1989. Predicting the fastest particle residence time. First International Conference on Aseptic Processing Technologies. Indianapolis, IN, March 19-21.

CAPPS and NCFST Workshop. 1995, 1996. Case study for condensed cream of potato soup from the aseptic processing of multiphase foods workshops. November 14-15, March 12-13.

Cacase, D., Palmieri, L., Pirone, G., and Dipollina, G. 1994. Biological validation of mathematical modeling of the thermal processing of particulate foods: The influence of heat transfer coefficient determination. Journal of Food Engineering. Vol. 23: 51-68.

Chandarana, D.I. 1992. Acceptance procedures for aseptically processed particulate foods. In “Advances in aseptic processing technologies”. Edited by Singh, R.K., Nelson, P.E. Elsevier Applied Sciences, London. pp. 261-278.

Chandarana, D.I., Gavin, A. 1989. Establishing thermal processed for heterogeneous foods to be processed aseptically: A theoretical comparison of process development methods. Journal of Food Science. Vol. 54(1): 198-204.

Chandarana, D.I., Gavin, A., and Wheaton, F.W. 1989. Simulation of parameters for modeling aseptic processing of foods containing particulates. Food Technology. Vol. 43(3): 137-143.

David, J.R.D. 1996. Principles of Thermal Processing and Optimization. In "Aseptic processing and packaging of food, a food industry perspective". Edited by David, J.R.D., Graves, R.H., Carlson, V.R. CRC Press, Inc., Boca Raton, FL. pp. 4-8.

Dignan, D.M., Berry, M.R., Pflug, I.J., and Gardine, T.D. 1989. Safety considerations in establishing aseptic processes for low acid foods containing particulates. Food Technology. Vol. 43(3): 118-121, 131.

Dutta, B., Sastry, S.K. 1990a. Velocity distributions of food particle suspensions in holding tube flow: distribution characteristics and fastest-particle velocities. Journal of Food Science. Vol. 55(6): 1703-1710.

Dutta, B., Sastry, S.K. 1990b. Velocity distributions of food particle suspensions in holding tube flow: experimental and modeling studies on average particle velocities. Journal of Food Science. Vol. 55(5): 1448-1453.

Fairhurst, P.G., Barigou, M., Fryer, P.J., and Pain, J.P. 1999. Particle passage time distributions in vertical pipe flow of solid-liquid food mixtures. Transactions of the Institution of Chemical Engineers. Vol. 77(C): 293-301.

Ghiron, K.M., Litchfield, J.B. 1997. Magnetic thermometry in the aseptic processing of foods containing particulates. Journal of Applied Physics. Vol. 81(8): 4321.

Ghiron, K.M., Litchfield, J.B. 1998. Local magnetic temperature measurement for aseptic processing. U. S. Patent No. 5,722,317. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Guiavarc'h, Y., Deli, V., Van, L.A., and Hendrickx, M. 2002a. Development of an enzymic time temperature integrator for sterilization process based on *Bacillus licheniformis* a-amylase at reduced water content. Journal of Food Science. Vol. 67(1): 285-291.

Guiavarc'h, Y., Dintwa, E., Van, L., Ann., Z.F., and Hendrickx, M. 2002b. Validation and use of an enzymic time temperature integrator to monitor thermal impacts inside a solid/liquid model food. Biotechnology Progress. Vol. 18(5): 1087-1094.

Heldman, D.R. 1989. Establishing aseptic thermal processes for low acid foods containing particulates. Food Technology. Vol. 43(3): 122-124.

Heldman, D.R. 1992. Innovative concepts in equipment design for aseptic processing. In Advances in Food Engineering. (editors: R.P. Singh and M.A. Wirakartakusumah). CRC Press Inc., Boca Raton, FL. pp. 309-318.

Hendrickx, M., Weng, Z., Maesmans, G., and Tobback, P. 1992. Validation of a time temperature integrator for thermal processing of foods under pasteurization conditions. International Journal of Food Science and Technology. Vol. 27: 21-31.

Heppell, N.J. 1985a. Comparison of the residence time distributions of water and milk in an experimental UHT sterilizer. Journal of Food Process Engineering. Vol. 4: 71-84.

Heppell, N.J. 1985b. Measurements of the liquid-solid heat transfer coefficient during continuous sterilization of food-stuffs containing particles. Presented at the 4th Congress on Engineering and Foods. Edmonton, Alberta, Canada, July 7-10.

Higgins, K.T. 2004. Will aseptic heat up. Food Engineering. Vol. 76(2): 43-46

Jasrotia, A.K.S., Simunovic, J., Swartzel, K.R., and Sandeep, K.P. 2003. Multiphase aseptic processing: selection and detection of magnetic particle implants for monitoring residence time and time-temperature history. AIChE Annual Meeting, San Francisco, CA: November 16-21: 107by.

Kantt, C.A., Schmidt, S.J., Sizer, C.E., Palaniappan, S., and Litchfield, J.B. 1998. Temperature mapping of particles during aseptic processing with magnetic resonance imaging. Journal of Food Science. Vol. 63(2): 305-311.

Kantt, C.A., Webb, A.G., Litchfield, J.B., 1997. Temperature measurement of foods using chemical shift magnetic resonance imaging as compared with T1-weighted temperature mapping. Journal of Food Science. Vol. 62(5): 1011-1016.

Larkin, J.W. 1997. Continuous multiphase aseptic processing of foods. Food Technology. Vol. 51(10): 43-44.

Lee, J.H., Singh, R.K. 1990. Mathematical models of scraped surface heat exchangers in relation to food sterilization. Chemical Engineering Communication. Vol. 87: 21-51.

Lund, D.B., Singh, R.K. 1993. The system and its elements. In Principles of Aseptic Processing and Packaging, 2nd Edition. (editors: P.E. Nelson and J.V. Chambers). The Food Processors Institute, Washington, D.C. pp. 3-30.

Maesmans, G., Hendrickx, M., DeCordt, S., Fransis, A., and Tobback, P. 1992. Fluid-to-particle heat transfer coefficient determination of heterogeneous foods: a review. Journal of Food Processing and Preservation. Vol. 16: 26-69.

Maesmans, G., Hendrickx, M., De Cordt, S., Vant Loey, A., Noronha J., and Tobback, P. 1994. Evaluation of process value distribution with time temperature integrators. Food Research International. Vol. 27: 413-423.

Marcy, J.E. 1997. Biological validation. Food Technology. 51(10): 44-48.

Morris-Lee, J. 2004. CAPPS develops validation technologies for multiphase aseptic processing. In Aseptic processing and packaging. Witter Publishing Corporation, NJ. Vol. 1(2): pp. 5, 14-21.

Mwangi, J.M., Rizvi, S.S.H., and Datta, A.K. 1993. Heat transfer to particles in shear flow: Application in aseptic processing. Journal of Food Engineering. Vol. 19(1): 55-74.

Palaniappan, S., Sizer, C. 1997. Aseptic process validated for foods containing particulates. Food Technology. Vol. 51(8): 60-68.

Palazoglu, T.K., Sandeep, K.P. 2002. Assessment of the effect of fluid-to-particle heat transfer coefficient on microbial and nutrient destruction during aseptic processing of particulate foods. Journal of Food Science. Vol. 67(9): 3359-3364.

Palazoglu, T.K., Sandeep, K.P. 2004. Effect of tube curvature ratio on the residence time distribution of multiple particles in helical tubes. Lebensmittel-Wissenschaft und - Technologie. Vol. 37: 387-393.

Palazoglu, T.K., Simunovic, J., Sandeep, K.P. and Swartzel, K.R. 2004. Methods, systems and devices for evaluation of thermal treatment. European Patent No. WO2004067786. European Patent Office.

Pflug, I.J., Berry, M.R., and Dignan, D.M. 1990. Establishing the heat-preservation process for aseptically-packaged low-acid food containing large particulate, sterilized in a continuous heat-hold-cool system. Journal of Food Protection. Vol. 53: 312-321.

Ramaswamy, H.S., Awuah, G.B., and Simpson, B.K. 1997. Heat transfer and lethality considerations in aseptic processing of liquid / particle mixtures: a review. Critical Reviews in Food Science and Nutrition. Vol. 37(3): 253-286.

Ramaswamy, H.S., Pannu, K., Simpson, B.K., and Smith, J.P. 1992. An apparatus for particle-to-fluid relative velocity measurement in tube flow at various temperatures under nonpressurized flow conditions. Food Research International. Vol. 25: 277-284.

Reiffel, L. 2001. X-ray imaged implanted thermometers. U.S. Patent No. 6,250,800. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Saksena, A. 2001. Systems and method for non-invasive assessment of cooked status of food during cooking. U.S. Patent No. 6,299,920. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Sandeep, K.P., Zuritz, C.A. 1995. Residence time of multiple particles in non-newtonian holding tube flow: effect of process parameters and development of dimensionless correlations. Journal of Food Engineering. Vol. 25: 31-44.

Sastry, S.K. 1986. Mathematical evaluation for process schedules for aseptic processing of low acid foods containing discrete particulates. Journal of Food Science. Vol. 51: 1323-1328.

Sastry, S.K. 1992. Liquid to particle heat transfer coefficient in aseptic processing. In Advances in Aseptic Processing Technologies. (editors: R.K. Singh and P.E. Nelson) Elsevier Applied Science, London. pp. 63-72.

Sastry, S.K. 1997. Measuring residence time and modeling the system. In Workshop targets continuous multiphase aseptic processing of foods. Food Technology. Vol. 51(10): 44-48.

Sastry, S.K., Cornelius, B.D. 2002. Aseptic processing of foods containing solid particulates. John Wiley and Sons Inc., New York. pp. 1-3, 130-181.

Sastry, S. K., Lima, M., Brim, J., Brunn, T., and Heskitt, B.F. 1990. Liquid-to-particle heat transfer during continuous tube flow: Influence of flow rate and particle to tube diameter ratio. Journal of Food Process Engineering. Vol. 13(3): 239-253.

Simunovic, J., Swartzel, K. R. and Adles, E. 2004. Method and system for conservative evaluation, validation and monitoring of thermal processing. U.S. Patent No. 6,776,523. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Singh, R.P., Heldman, D.R. 2001. Introduction to Food Engineering. 3rd edition. Academic Press, London, U.K.

Stoforos, N.G., Merson, R.L. 1991. Measurement of heat transfer coefficients in rotating liquid/particulate systems. Biotechnology Progress. Vol. 7: 267-271.

Swartzel, K.R., Simunovic, J. 1999. Method and system for residence time measurement of simulated food particles in continuous thermal food processing and simulated particles for use in same. U.S. Patent Application No. 5,932,813. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2000. Method for conservatively evaluating continuous thermal treatment process for a particulate-containing food product stream. U.S. Patent No. 6,015,231. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2003. Plurality of particles made of a detectable magnetic implant and a carrier in combination with a plurality of magnetic field sensors. United States Patent 6,536,947. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Swartzel, K.R., Simunovic, J. 2004. System for measuring residence time for a particulate containing food product. U.S. Patent No. 6,766,699. U.S. Patent and Trademark Office, U.S. Department of Commerce, Alexandria, VA.

Tessner, W.A., Farkas, B.E., and Sandeep, K.P. 2001. Use of ablation to determine the convective heat transfer coefficient in two-phase flow. Journal of Food Process Engineering. Vol. 24: 315-330.

Tobback, P., Hendrickx, M.E., Weng, Z., Maesmans, G.J., and De Cordt, S.V. 1992. The use of immobilized enzymes as time temperature indicator system in thermal processing. In Advances in Food Engineering (editors: R.P. Singh and M.A. Wirakartakusumah). CRC Press Inc., Boca Raton, FL. pp. 561-574.

Toledo, R.T. 1991. Fundamentals of food engineering. 2nd edition. Chapman and Hall, New York, NY.

Toledo, R.T., Chang, S.Y. 1990. Advantages of aseptic processing of fruits and vegetables. Food Technology. Vol. 44: 72-78.

Zareifard, M.R., Ramaswamy, H.S. 1999. A calorimetric approach for evaluation of fluid-to-particle heat transfer coefficient under tube-flow conditions. Lebensmittel-Wissenschaft und Technologie. Vol. 32: 495-502.

Zitoun, K.B., Sastry, S.K. 1994a. Convective heat transfer coefficient for cubic particles in continuous tube flow using the moving thermocouple method. Journal of Food Process Engineering. Vol. 17: 229-241.

Zitoun, K.B., Sastry, S.K. 1994b. Determination of convective heat transfer between fluid and cubic particles in continuous tube flow using noninvasive experimental techniques. Journal of Food Process Engineering. Vol. 17: 209-228.

Zuritz, C.A., McCoy, S., and Sastry, S. 1987. Convective heat transfer coefficients for non-Newtonian flow past food-shaped particulates. ASAE Paper No. 87-6538, ASAE, St Joseph, MI.

Table 4.1 Measured F_0 values in Data Acquisition Program files compared with the calculated F_0 using temperature data file from Data Acquisition Program

Sample of the temperature and F_0 output file data			Calculated values using temperature output file data	
Time, t in s	Temperature, T in °C	F_0 in s	L(t)	$(F_0)_t$ in s
1085.66556	122.53641	112.00231	1.39200	112.00231
1085.76552	122.53641	112.14146	1.39200	112.14146
1087.26492	122.63412	114.22863	1.42368	114.22863
1087.36488	122.63412	114.37095	1.42368	114.37095
1087.46484	122.63412	114.51326	1.42368	114.51326

Table 4.2 Thermophysical properties of polymers and food materials*

Material	Density (kg/m^3)	Thermal conductivity (W/m-K)	Specific heat (J/kg-K)	Thermal diffusivity (m^2/s)
Polypropylene	910	0.13	2343	6.0972e-08
Polymethylpentene	833	0.17	1968	1.0370e-07
Carrot	1040	0.606	3864	1.5080e-07
Potato	1090	0.554	3517	1.4451e-07

*Complied from Abdelrahim *et. al.*, 1997; Palaniappan and Sizer, 1997; Singh and Heldman, 2001; Toledo, 1991

Table 4.3 Process variable inputs to CPD for particle design generation

Initial temperature of particle	20 °C
Ambient temperature	140 °C
Heat transfer coefficient	1000 W/m²K
Desired F_0	3 min
Target particle density	1000 kg/m³

Table 4.4 Wall thickness and implant weight results for different combinations of food material and polymers

Food material	Polymer	Wall thickness (mm)	Implant weight (g)
Carrot	Polypropylene	1.27	.934
Carrot	Polymethylpentene	2.54	.632
Potato	Polypropylene	1.27	.934
Potato	Polymethylpentene	2.54	.632

Table 4.5 Food materials and there physical dimensions used for testing conservative behaviors (all other food materials had dimensions that of ½ “ cube)

Food Material	Physical dimensions
Okra (*Hibiscus esculentus*)	diameter = 0.837 ”, height = 0.2 “
Lima bean (*Phaseolus limensis*)	height = 0.432 “, length = 0.679 “, width = 0.22 “
Green/String bean (*Phaseolus vulgaris*)	height = 0.286 “, length = 0.461 “, width = 0.207 “
Brown bean (*Phaseolus vulgaris*)	height = 0.522 “, length = 0.818 “, width = 0.201 “
Green pepper (*Capsicum annuum*)	height = 0.275 “, length = width = 0.5 “
Cluster bean (*Cyamopsis tetragonolobus*)	pod of thickness = 0.275 “, length = width = 0.5 “

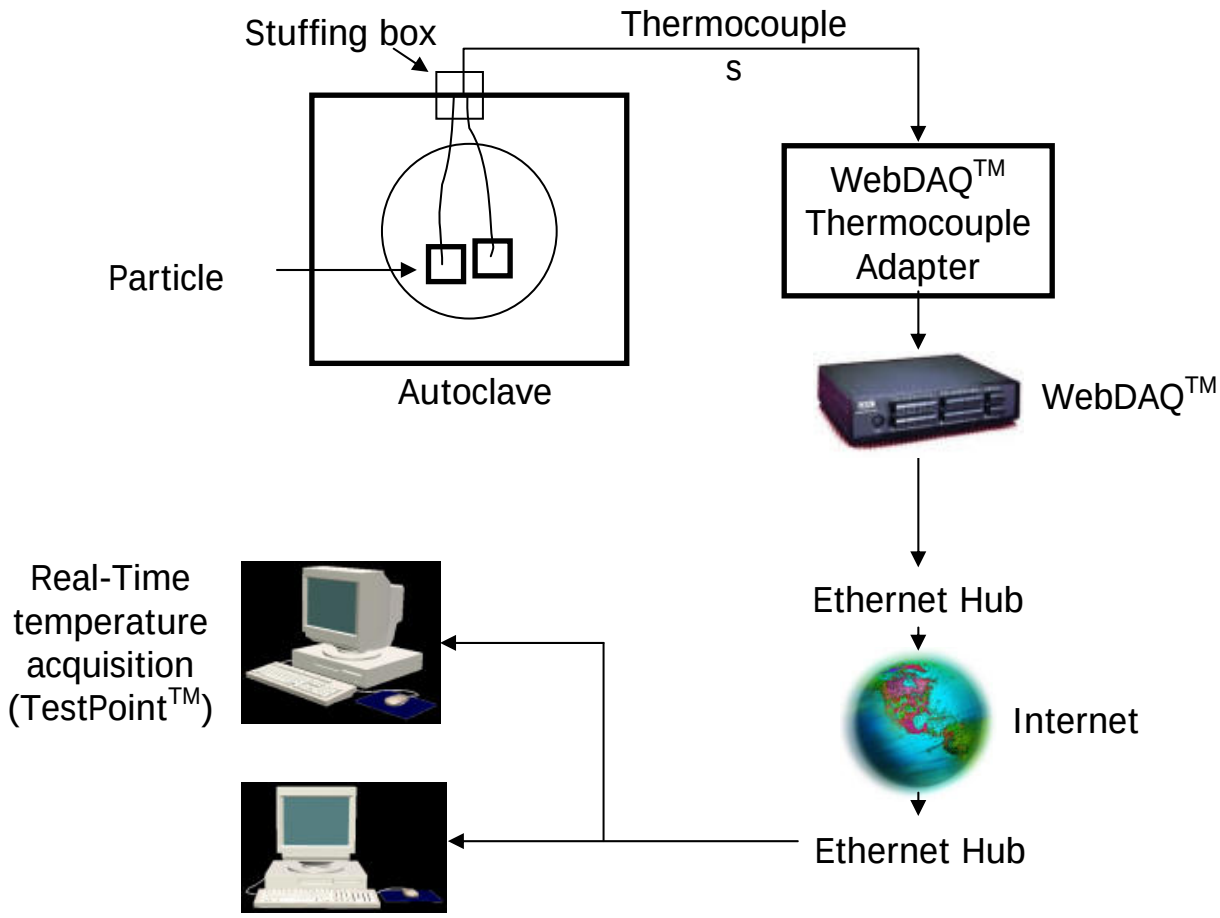
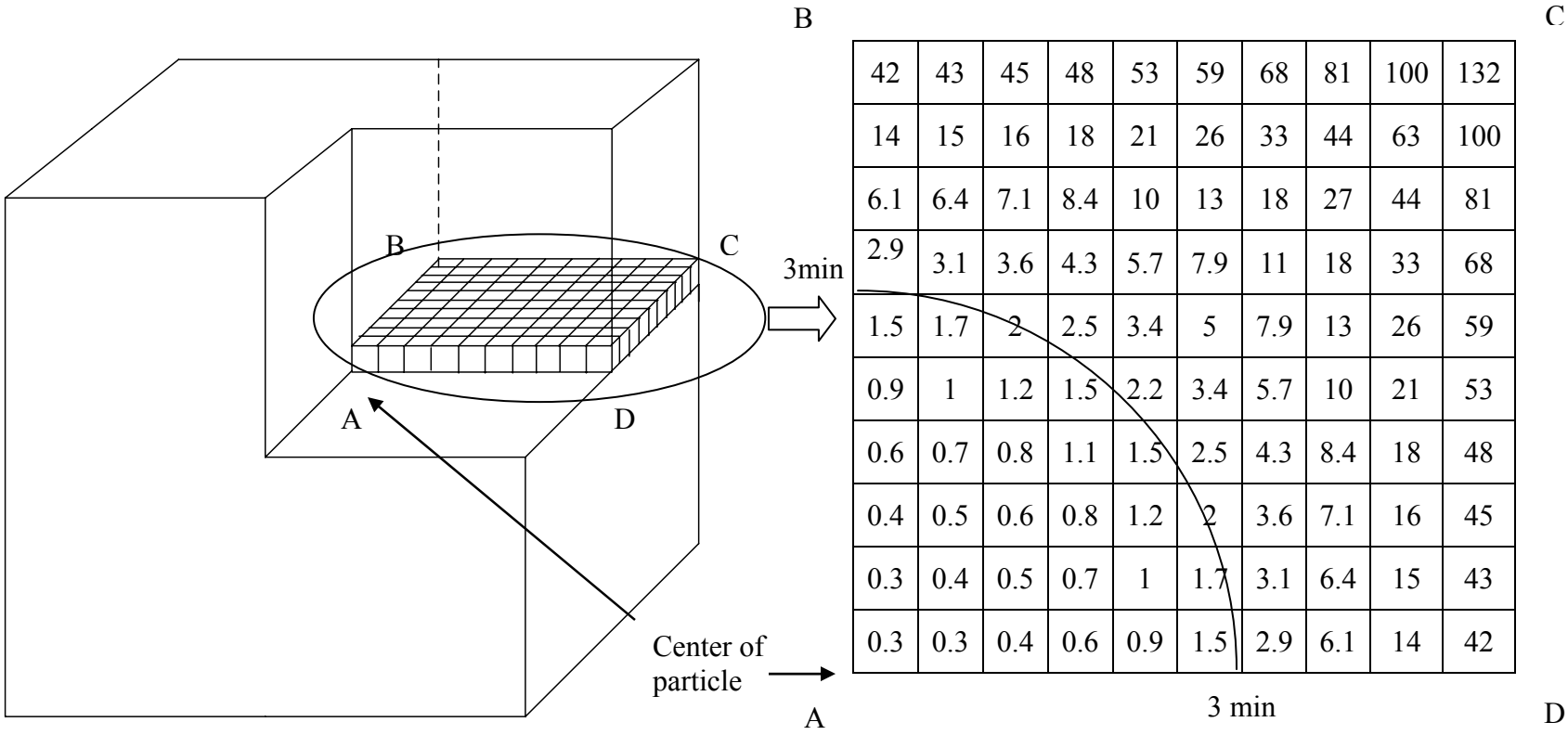


Figure 4.1 Complete experimental setup to acquire real-time time-temperature history and plot temperature and F_0 graphs on the monitor (at run-time)



42	43	45	48	53	59	68	81	100	132
14	15	16	18	21	26	33	44	63	100
6.1	6.4	7.1	8.4	10	13	18	27	44	81
2.9	3.1	3.6	4.3	5.7	7.9	11	18	33	68
1.5	1.7	2	2.5	3.4	5	7.9	13	26	59
0.9	1	1.2	1.5	2.2	3.4	5.7	10	21	53
0.6	0.7	0.8	1.1	1.5	2.5	4.3	8.4	18	48
0.4	0.5	0.6	0.8	1.2	2	3.6	7.1	16	45
0.3	0.4	0.5	0.7	1	1.7	3.1	6.4	15	43
0.3	0.3	0.4	0.6	0.9	1.5	2.9	6.1	14	42

Figure 4.2 F_0 map showing the quarter cube of a particle and the F_0 values at different points inside the cube

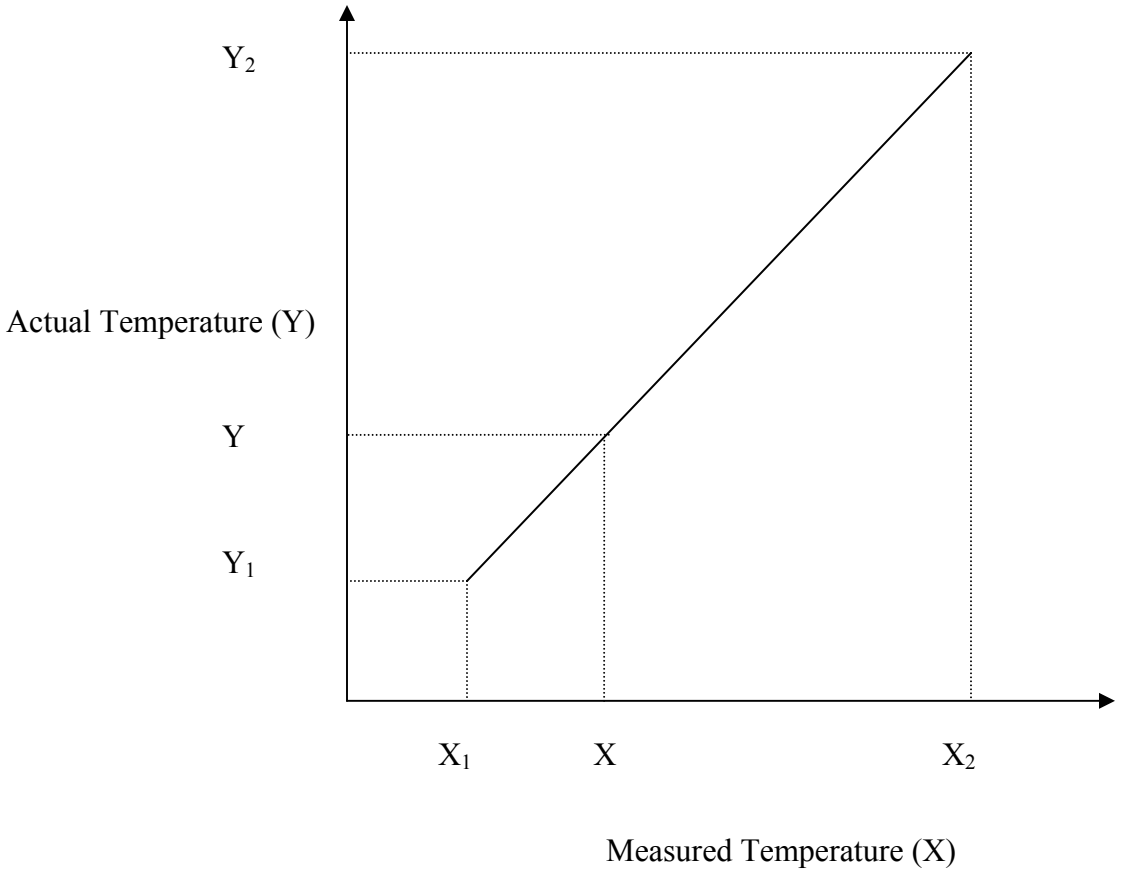


Figure 4.3 Linear relation drawn for determining the actual temperature of a thermocouple (Y) at a measured temperature (X) using the calibration data (Y_1 and Y_2) at ice cold (X_1) and boiling point (X_2)

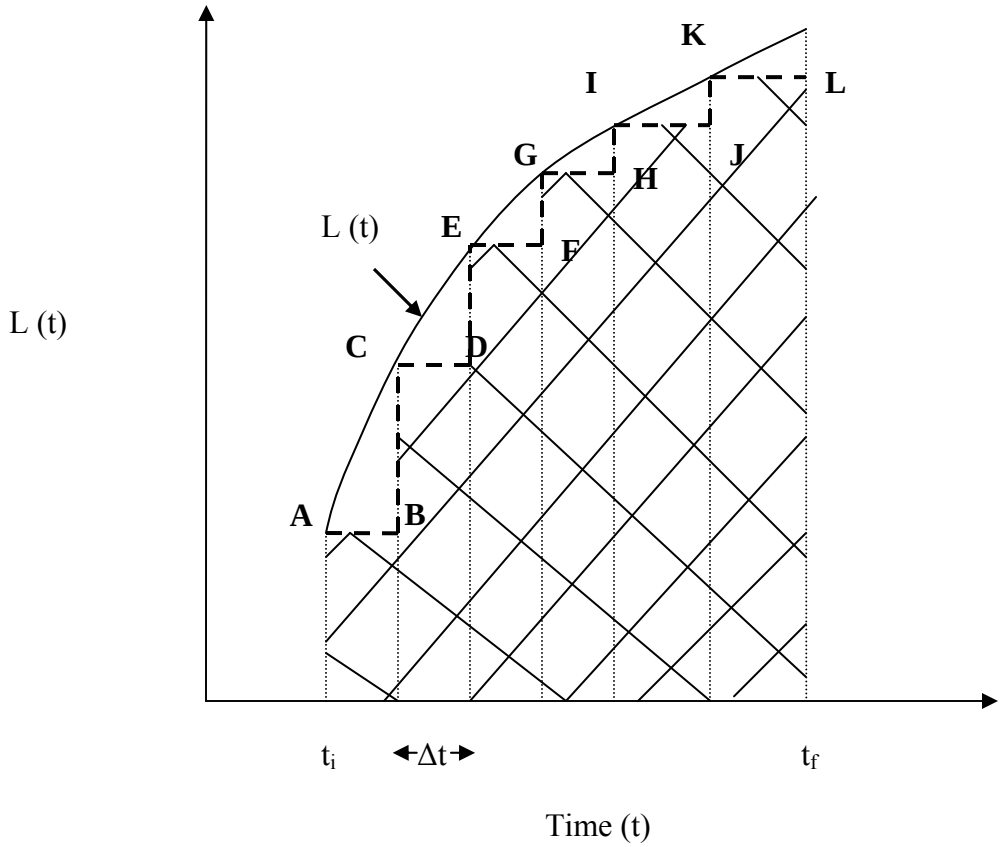


Figure 4.4 Shaded area under the stepwise point function ABCD… which was used by Data Acquisition Program for F_0 value calculations

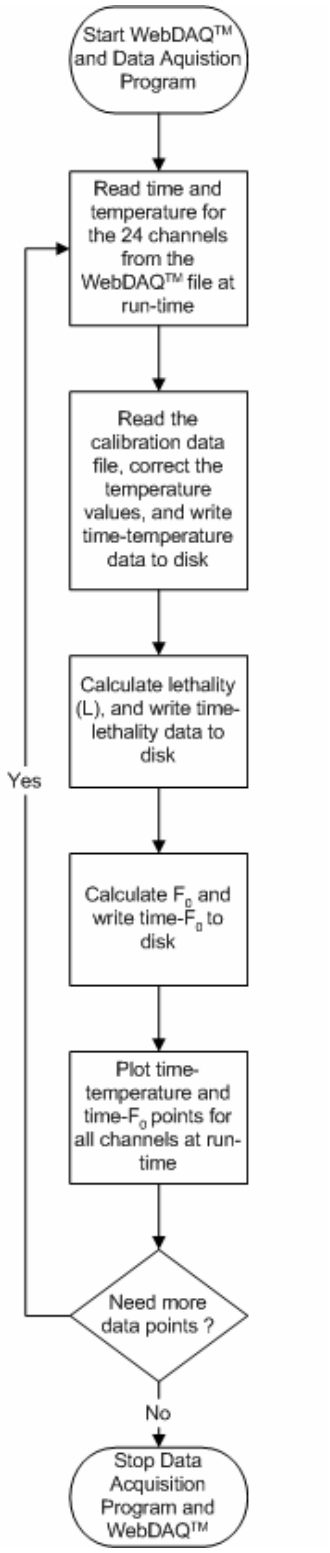


Figure 4.5 Flow diagram showing a brief logic for Data Acquisition Program

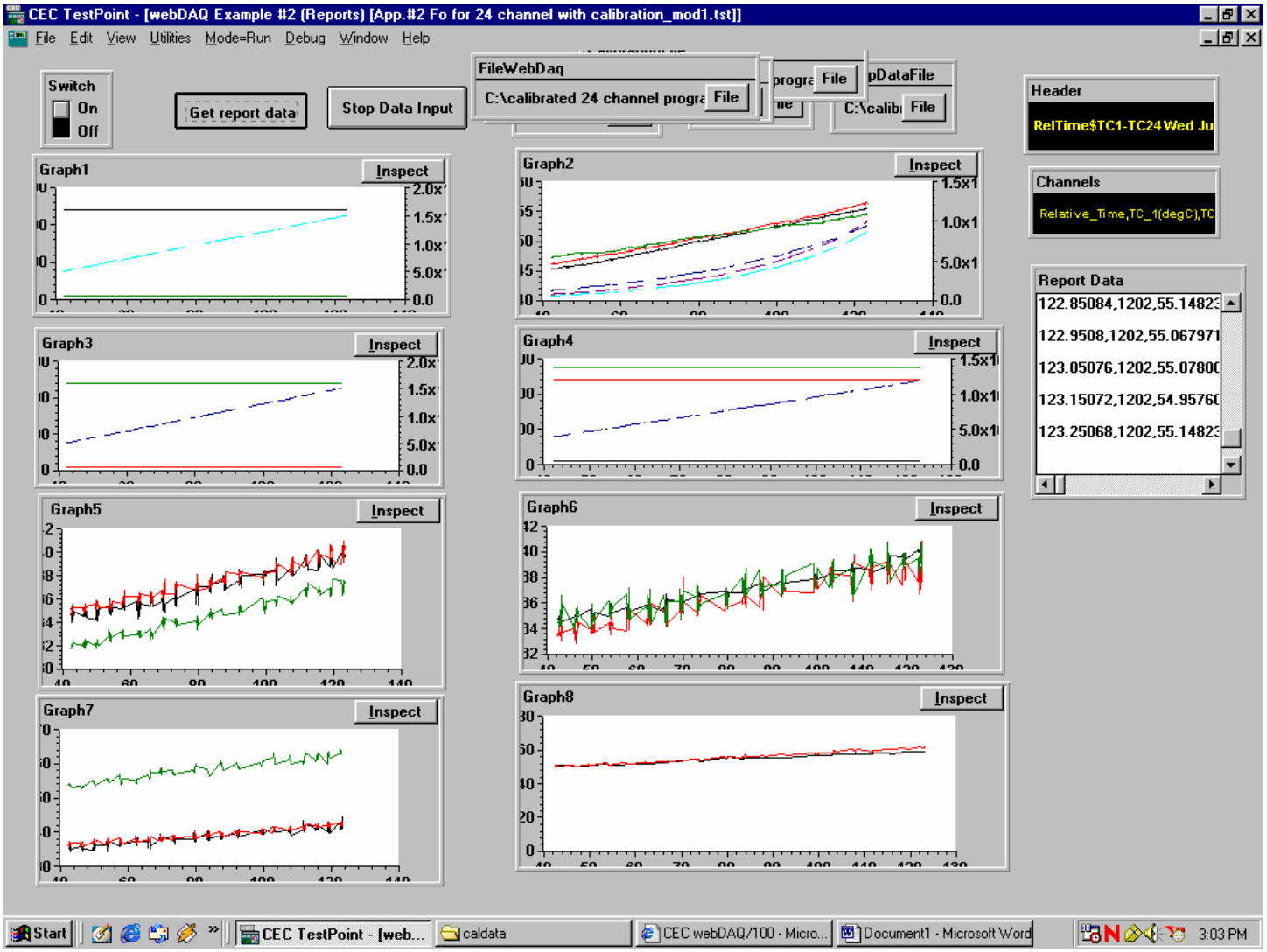


Figure 4.6 A Screen shot of the Data Aquistion Program showing the real-time acquired temperature and F_0 values with their graphs

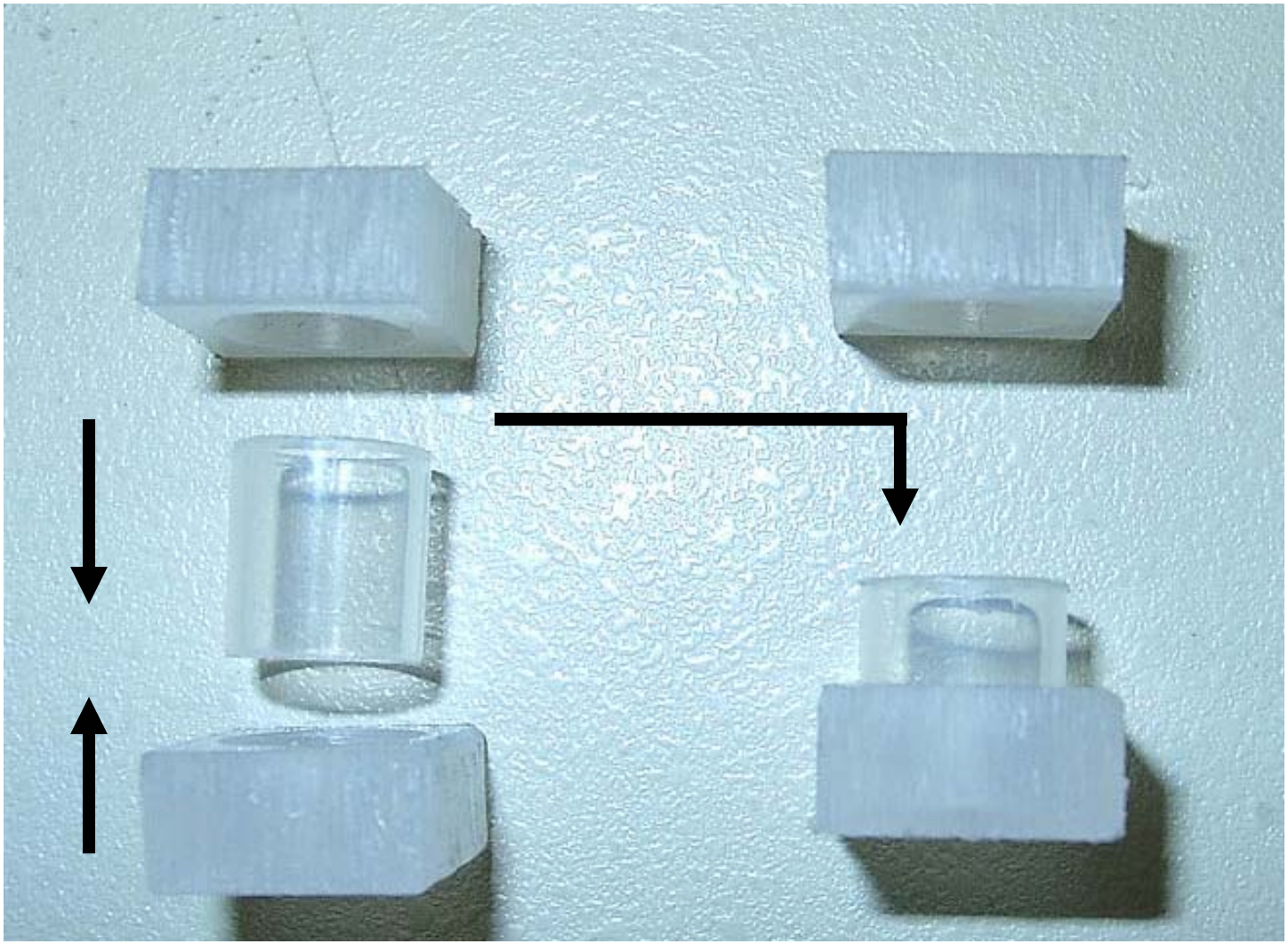


Figure 4.7 A simulated food particle made of PP, showing the two half of the cube (with cavity) and the cylindrical tube, which are joined to complete the particle

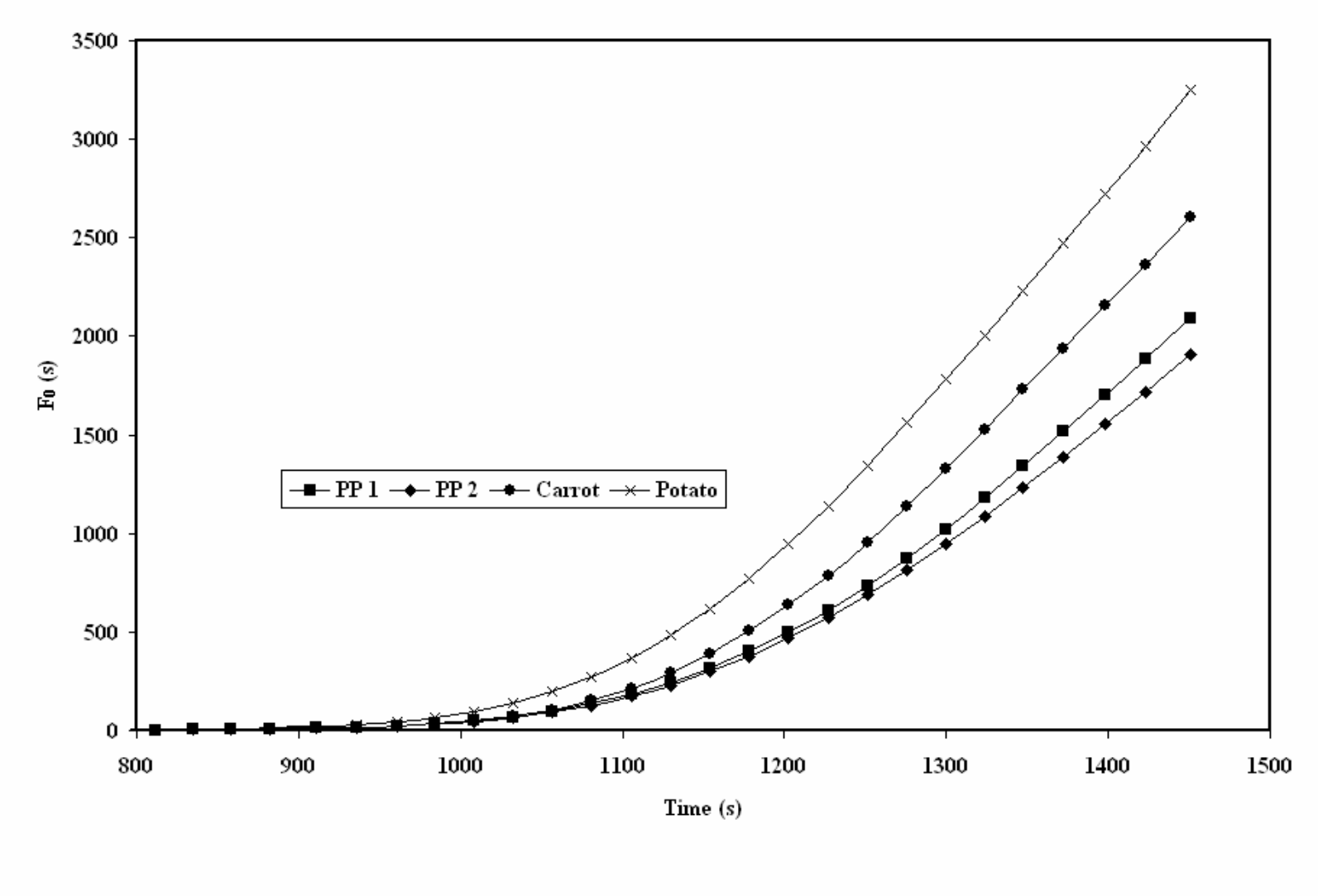


Figure 4.8 F_0 values for carrot and potato with PP under high pressure and temperature conditions

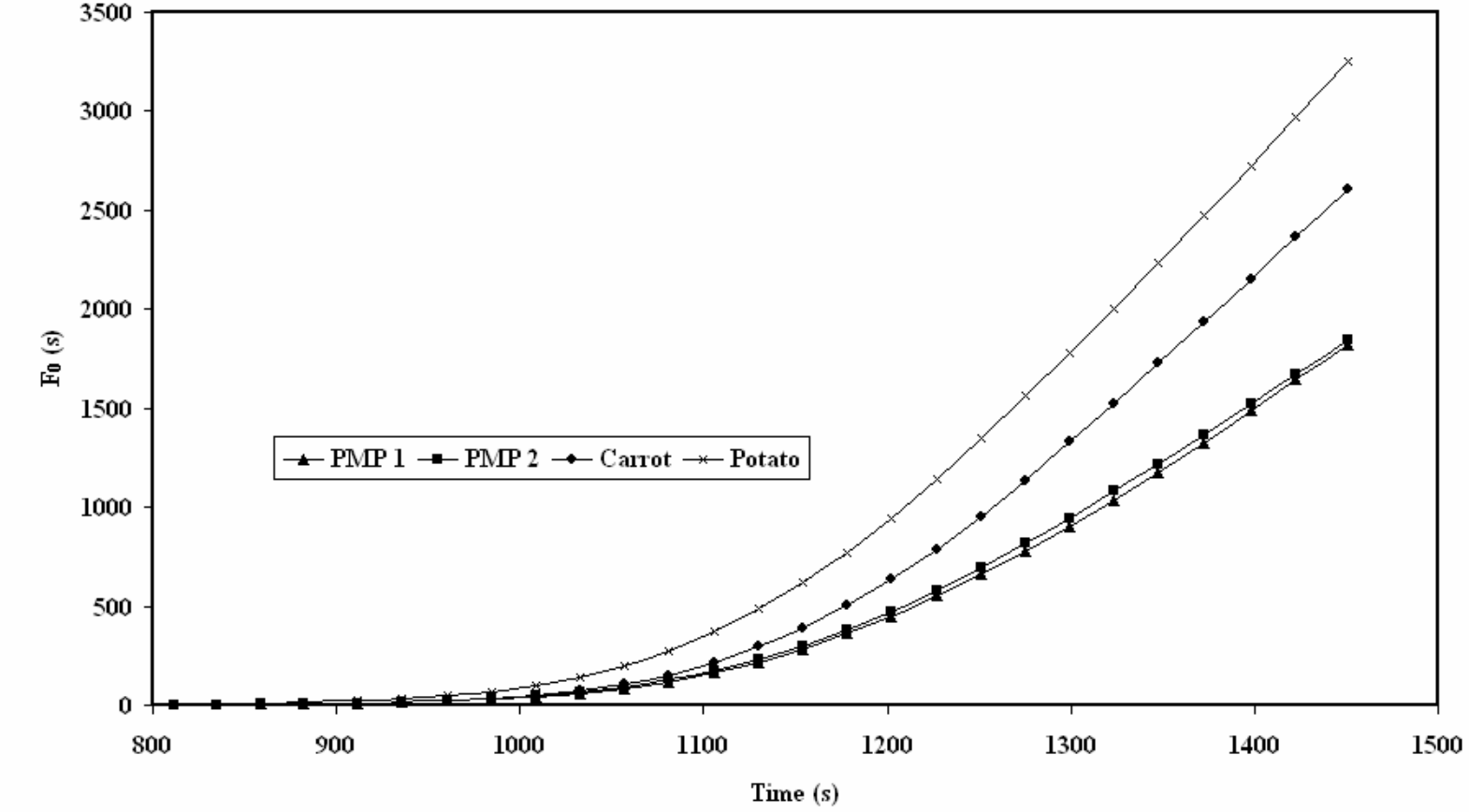


Figure 4.9 F_0 values for carrot and potato with PMP under high pressure and temperature conditions

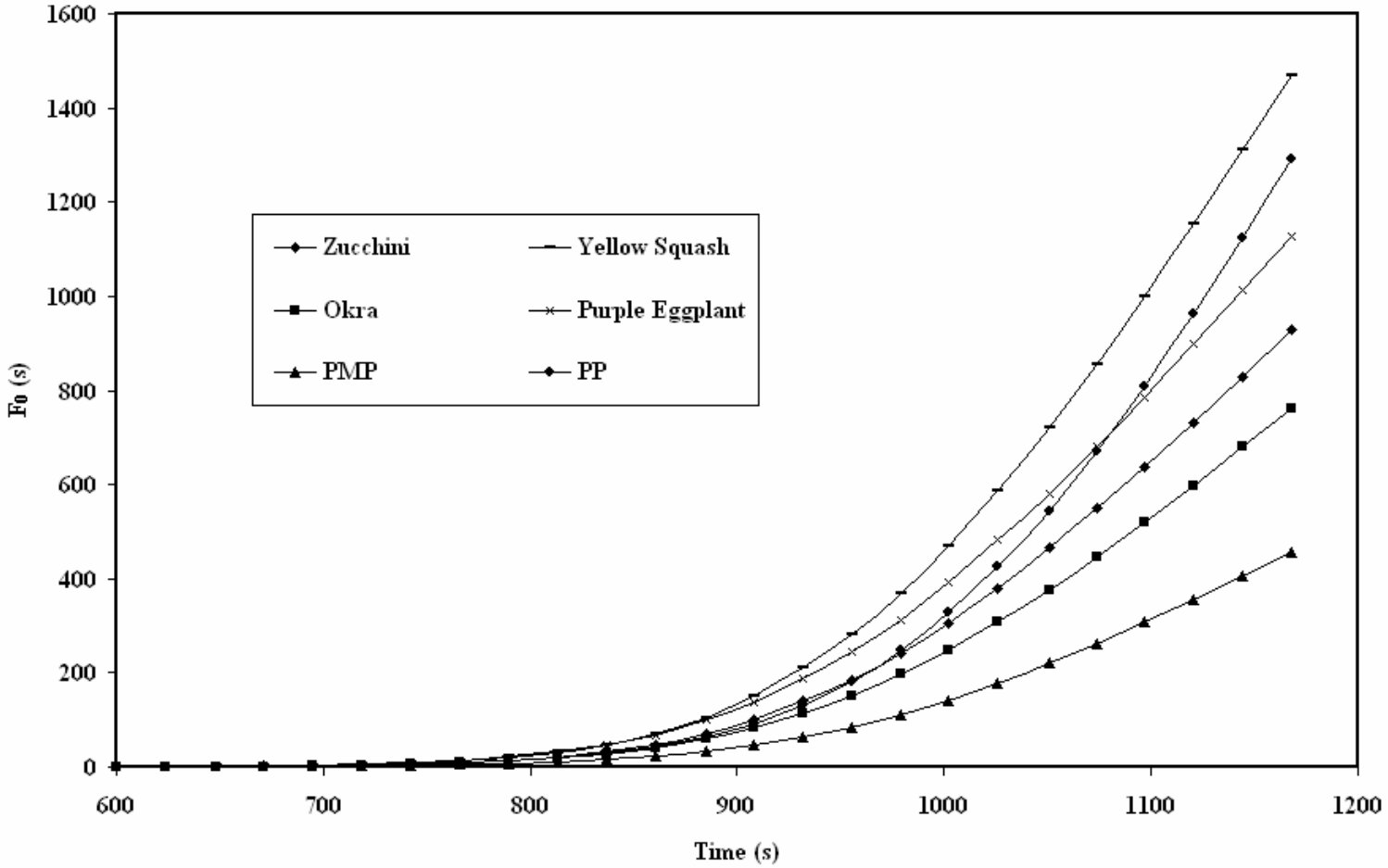


Figure 4.10 F_0 value comparisons for different foods with respect to PP and PMP

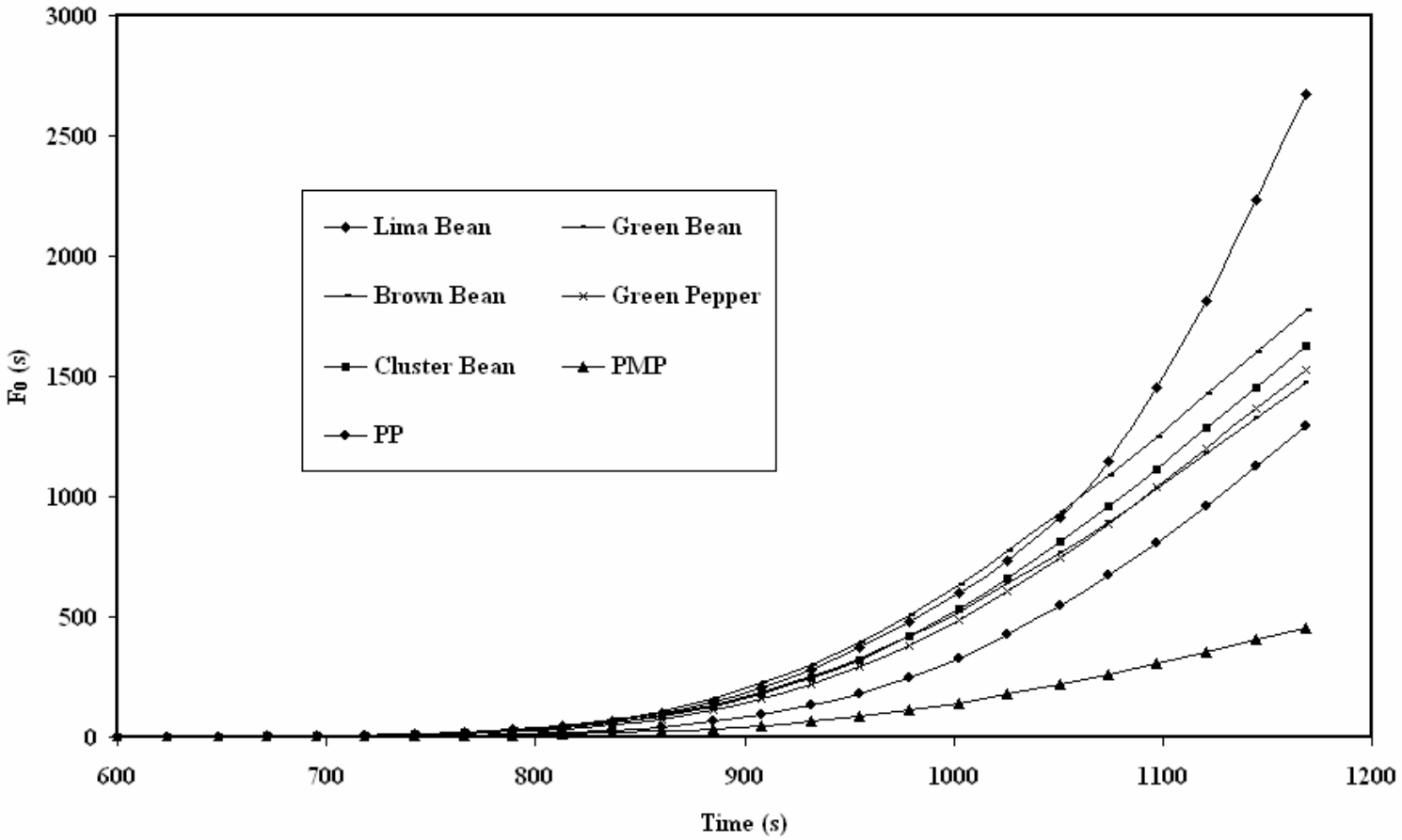


Figure 4.11 F_0 value comparisons for different foods with respect to PP and PMP

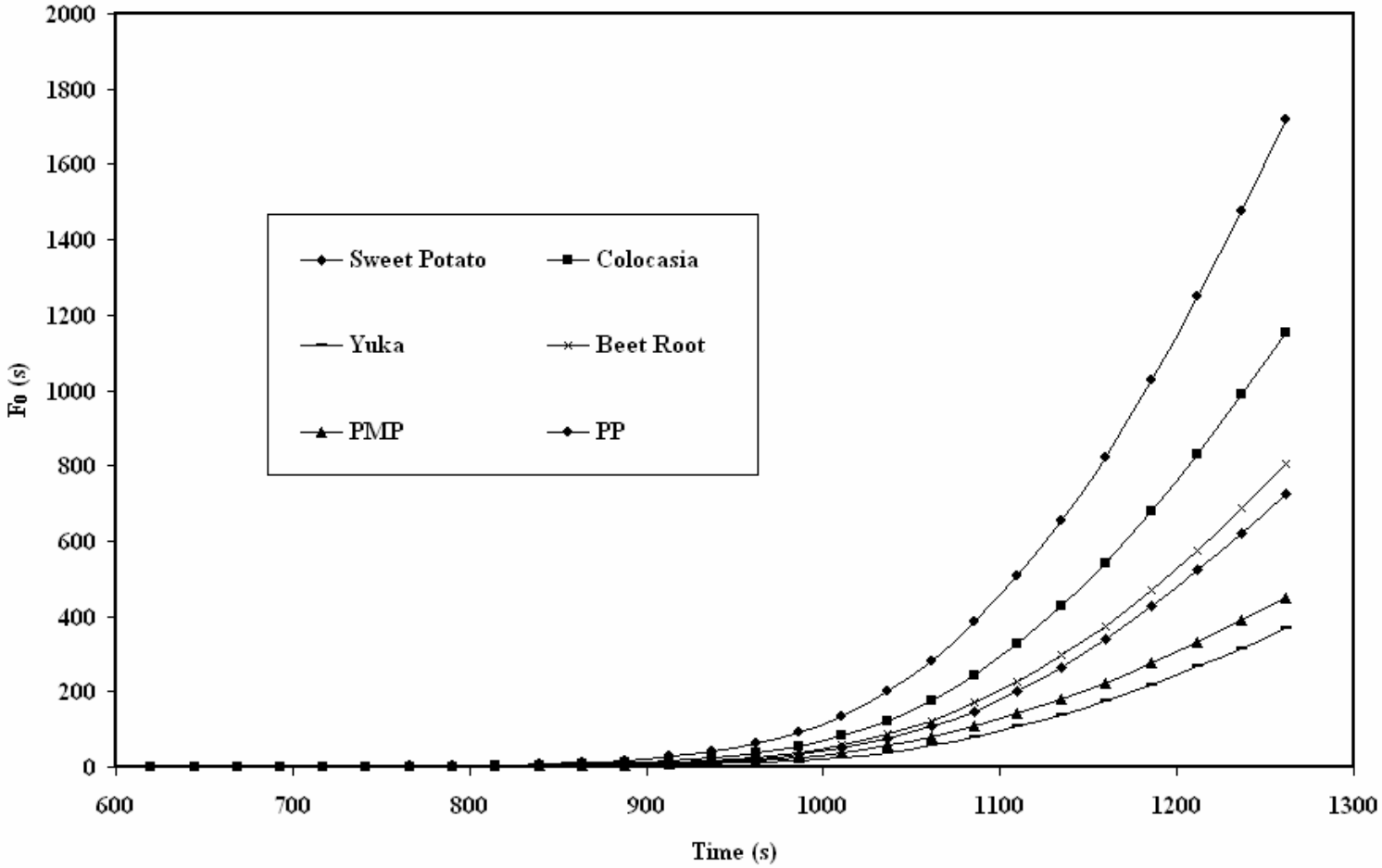


Figure 4.12 F_0 value comparisons for different foods with respect to PP and PMP

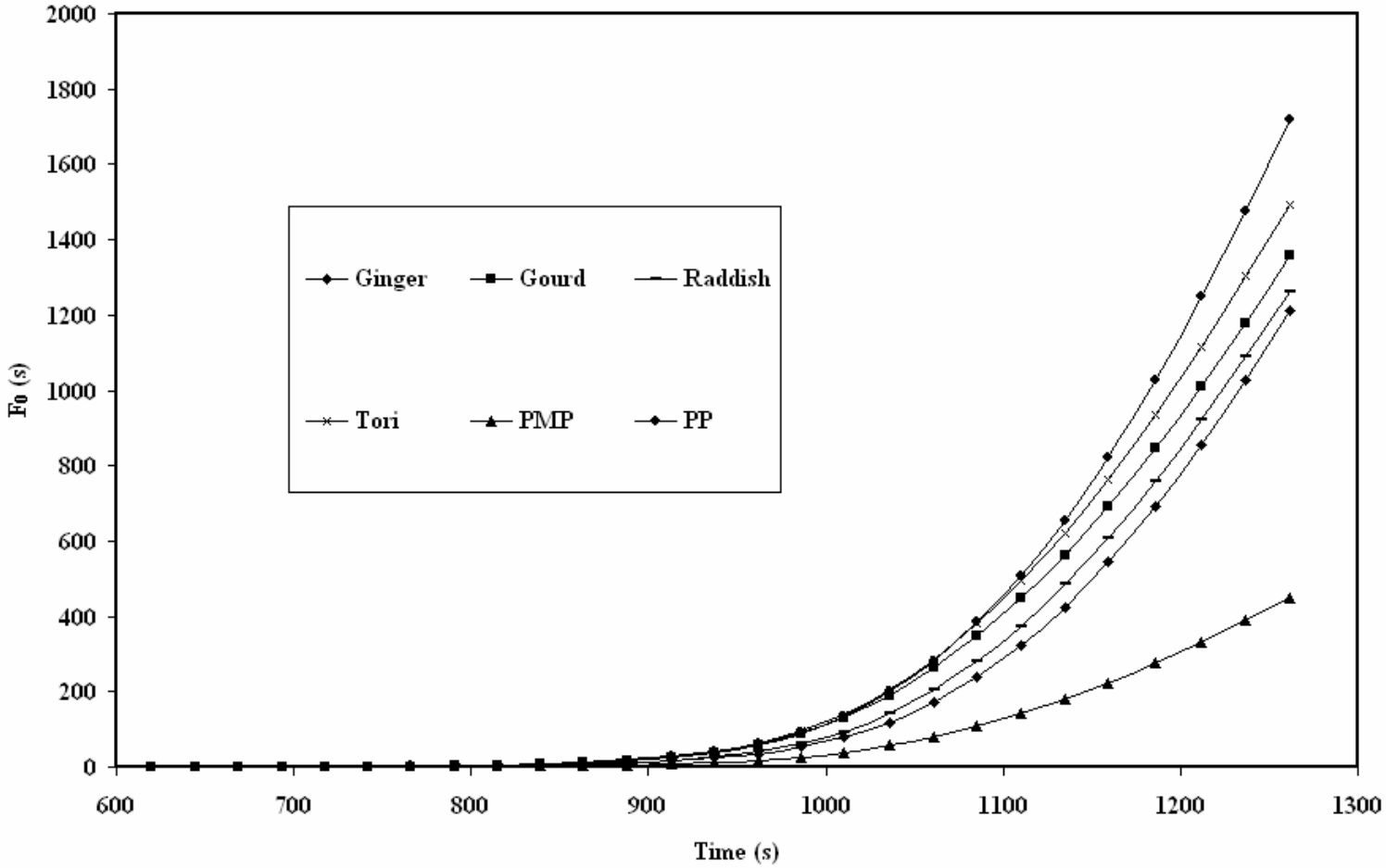


Figure 4.13 F_0 value comparisons for different foods with respect to PP and PMP

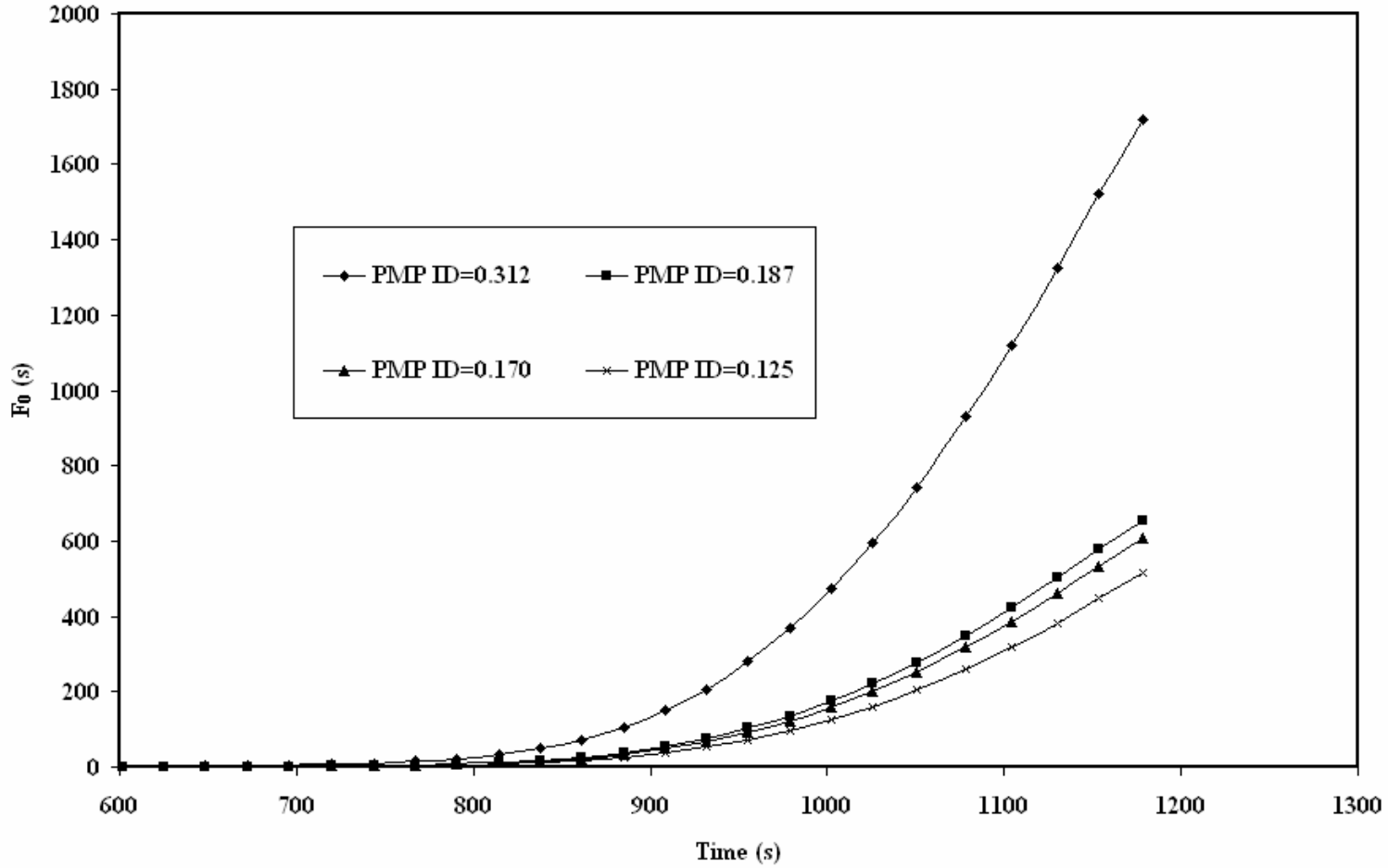


Figure 4.14 F_0 values for PMP particles of 1 mm wall thickness with different inner diameter (ID=inner diameter of insert in inches)

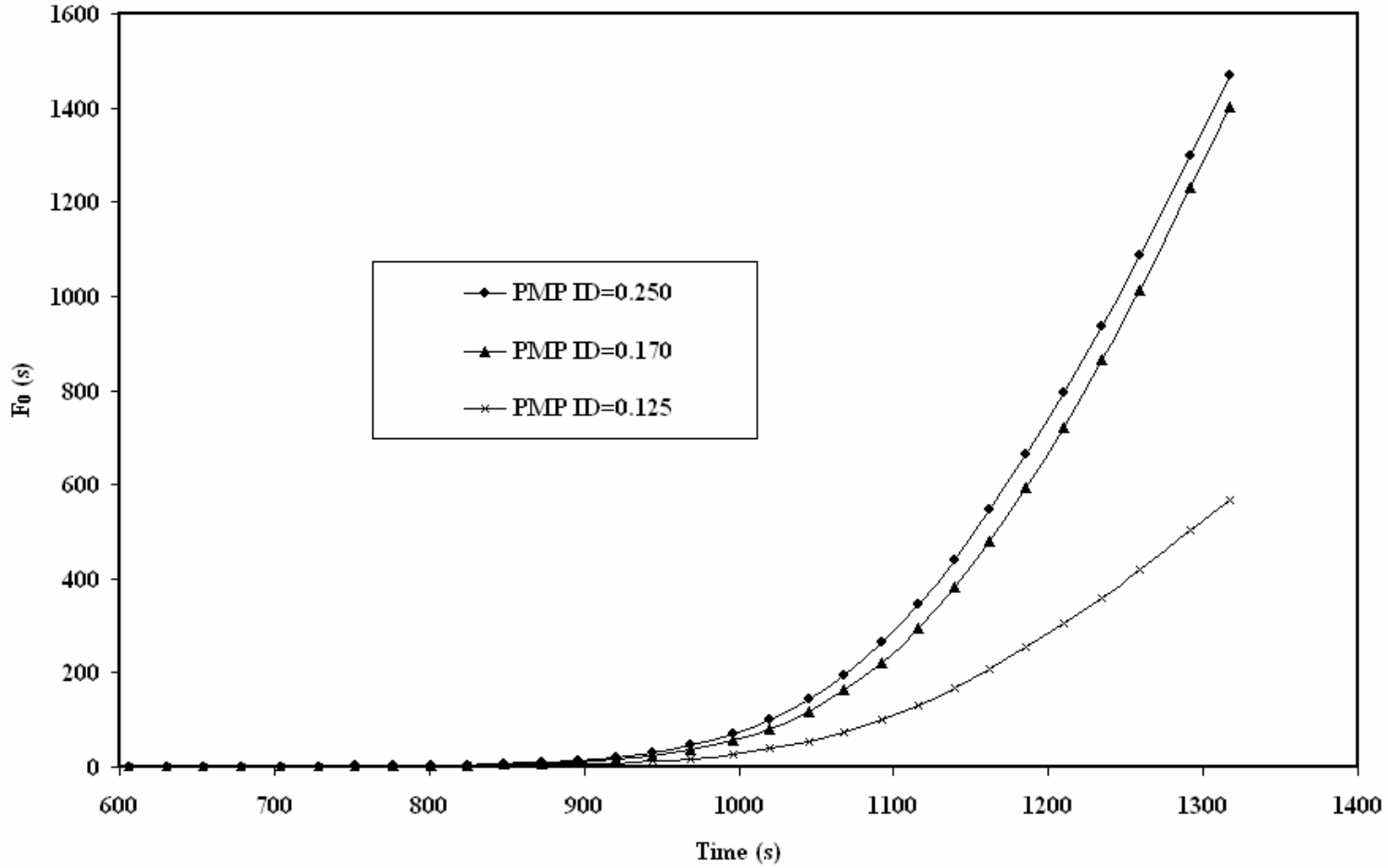


Figure 4.15 F_0 values for PMP particles of 2 mm wall thickness with different inner diameter (ID=inner diameter of insert in inches)

Chapter 5

CONCLUDING REMARKS

A method for fabrication and validation of implant-carrier simulated particles with conservative thermal properties for monitoring and validation of continuous thermal processing of multiphase food products has been developed and implemented. Developed tools and methods could be used by food processors and equipment providers to collect process validation data needed for process safety documentation and filing with regulatory agencies as well as comparison of various products, processes and equipment properties.

The results demonstrate the procedure and feasibility of implementing a conservative carrier particle design for process monitoring and validation. To use these particles for validation of multiphase aseptic processes we need to be confident that they will provide a conservative thermal protection when used for validation of any aseptic multiphase process under similar process variables. For this the fabricated particles were experimentally validated and the experimental data was analyzed using Microsoft® Excel (Microsoft® Corporation, Redmond, WA).

The developed Data Acquisition Program can be used by a user to calculate F_0 as per the requirement for safety (microbial destruction) or quality (nutrient loss) calculations. It incorporates the corrections to the measured temperature readings at run

time and also calculates the F_0 values, displaying them on the monitor screen. In addition, the temperature data file could also be used as standalone to calculate F_0 or other parameters with tools like Microsoft® Excel (Microsoft® Corporation, Redmond, WA). In-addition a method to compare conservative thermal behavior for different food materials along with a method to change the thermal properties to adjust the conservative behavior was shown. This would allow a food processor to employ these designed particles and choose a right configuration to validate an aseptic process for a multiphase food containing different food materials.

Chapter 6

RECOMMENDATIONS FOR FUTURE WORK

Present study develops methods and design parameters that would be required for fabrication and testing of simulated food particles for validation of an aseptic processing of multiphase foods. Results show that particles designed in this study were conservative to the ½ inch cube carrot and potato while it was not conservative to some other food materials like yucca. Some recommendations for future improving the methods and tools developed in this study are presented below:

More research needs to be done on the materials that could be used for fabrication of the simulated food particles. Their manufacturing should be economical and flexible to produce particles of different wall thickness and cavity size, which would help processors validate the products when various foods particles are involved in the product. In-addition more research needs to be conducted in improving particle design to prevent leakages of fluids and steam into the particle from the gap in-between the two half of the cube.

More experimental testing for conservative behavior of the simulated food particles when compared to some other real food materials – which were not tested in this study, is required. Experiments with magnetic implants in these simulated food particles would be the next step for developing the methodology for validation of aseptic processing of multiphase foods. These carriers would be required to carry the magnetic

implants, which would be capable of sending a signal out to the sensors attached along a tube, in which the food needs to be processed. This would be used to record the time-temperature histories of these particles along the tube length to calculate the lethality for aseptic processing.

Adjusting particle insulation properties for changing the thermal behavior of the simulated food particles would another study that might be conducted. This would save time and money for a processor in redesigning the particles if the constituents of the product changes, as each new food material must be tested and compared for its compared to these simulated food material.