

**INDICATORS OF BACTERIAL LETHALITY IN SEMI- RIGID
PLASTIC FOOD CONTAINERS DURING THERMAL
PROCESSING**

by

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Abstract

Thermocouple methods which have been used traditionally for gathering time-temperature data for agitated retort processes have some physical limitations in that thermocouples located in the centres of containers cannot accurately simulate free-moving particles. For this reason alternative approaches have been taken to measure, understand and model such thermal processes, and this was the purpose of the research carried out in these studies.

Thermal diffusivity was determined for a potato/alginate gel in order to determine the thermal characteristics of the gel and thereby ascertain its ability to function as a model food particle. Thermal diffusivity was found to average $2.048 \times 10^{-7} \text{ m}^2/\text{s}$, which approximates values reported in the literature for potatoes (ranging from $1.64 \times 10^{-7} \text{ m}^2/\text{s}$ to $1.93 \times 10^{-7} \text{ m}^2/\text{s}$). Visualisation based on video recording of 307 diameter plastic OMNI® bowls containing four food-simulating particles were conducted in a Lagarde steam/air retort simulator to evaluate the effect of rotation on particle movement. The speed of rotation was 2.8 rpm and particles were found to remain in contact with the outer edge of the container near its lowest point, thus confirming that the principal force acting on the particles is gravitational and not centrifugal. As well, a thin-wire (flexible) thermocouple anchored in the centre of a spherical particle was evaluated as a method of gathering time-temperature data while simulating the movement of free particles. The tethered particle was found to be constrained to rotation in an arc from the top inner surface of the container to the bottom inner surface.

Thermal processing studies were carried out during agitated processes in order to investigate heating of 307 diameter plastic OMNI® bowls within a production-scale Lagarde steam/air retort at various rotational speeds (0, 5, 10, and 15 rpm). Since the goal was to understand processes under production conditions, and since evidence of radial positional effects had been described for

the Lagarde steam/air retort simulator, the degree of positional effect was investigated. Bowls positioned at the axial centre and the outside edge heated faster than bowls positioned in the intermediate portion of the retort basket. The zone of slower heating was thought to be due to the annular disc in the door which served to increase heating medium velocity down the outside edges of the retort vessel and assist in its subsequent drawing back towards the fan through the centre. Experiments were then repeated in the Lagarde retort simulator at 0, 5, 10, 15, 20 and 25 rpm to determine whether equivalent patterns were observed. Studies in the Lagarde were carried out with rigid thermocouples and flexible thermocouples with their sensing junctions in potato/alginate gel particles to determine if the flexible thermocouple gave a better model of the temperature history within a freely moving particle in random motion during heating. Positional effects were determined under static and rotational modes. Flexible thermocouples were found to yield lower values for j_h , f_h and t_B than the rigid thermocouple.

A further stage of this research was aimed at assessing the effects of rotational thermal processes on populations of microorganisms. Suspensions of *Bacillus stearothermophilus* ATCC 7953 were placed into DSC pans, hermetically sealed and incorporated into the centres of potato/alginate particles (18 mm in diameter). Five such particles were placed into a 307 diameter OMNI® bowl container and processed in the Lagarde steam/air retort simulator. Positional effects were determined as well as rotational effects at 0, 15 and 30 rpm. Studies were done with particles immobilised on rigid thermocouples positioned at the centres of containers as well as on flexible probes (to simulate a flexible thermocouple). Within each small volume of the DSC pans, integrated sterilisation (IS) values were determined, based on spore recovery from free particles, particles with centrally located thermocouples and flexible thermocouples, and the liquid portion of the product. With flexibly anchored and freely moving particles there was close agreement for IS values, while for the rigidly held particles and the liquid contents of the container there was close

agreement for IS values.

A final study made use of the thermal indicator concept with *Clostridium pasteurianum*, *Bacillus coagulans*, and lysozyme to evaluate pasteurisation of tomato-based sauces compared to traditional temperature history methods. Three container sizes were used (3.8 L and 1.9 L plastic jugs and a 750 mL glass jar). Measured lethalties were found to be greater than calculated lethalties for all containers.

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Chapter 1

INTRODUCTION

"...It shall be eaten the same day we offer it, and on the morrow: and if aught remain until the third day, it shall be burnt with fire. And if it be eaten at all on the third day, it is an abomination; it shall not be accepted:..."

Leviticus 19:6 - Third Book of Moses

In food manufacturing, the main objective is to formulate and preserve products that are microbiologically safe and of high sensory quality, in the most cost effective manner. During the manufacturing process, many operations require heating or cooling of the product. Increased rates of cooling generally result in improved sensory and nutritional quality, lower bacterial growth and higher yields. For safety in cooking and heating processes, a minimum time-temperature treatment that provides commercial sterility should be followed by rapid cooling to minimise the growth of surviving organisms. For any operation to be economical, it should enable high volume throughputs (i.e., short cooking and cooling cycles) per unit of capital/labour and incur low weight losses, all without compromising the safety and quality requirements.

As a result, the food industry has recognised that research and technology are important factors in the modernisation of food processing techniques. Arising from long research, new insights have

served as the foundation for advanced technological developments. This has presented the equipment industry with challenges for innovation in order to successfully implement more advanced technology. These interrelations are the reason for today's sophisticated technology in the food industry.

In keeping with this development of technology in the food industry, there is an increased need for understanding the mechanisms of the processes that are in use. Understanding implies the ability to predict the results of processes.

The objectives of safe, cost effective, high quality food products are achieved through processes that involve heating, cooling or both; in other words, heat transfer in one form or another. A good knowledge of what happens during heat transfer is vital. This involves gaining a good physical, theoretical and practical understanding of heat transfer, coupled with an ability to predict what happens during thermal processing. From this flows modelling and prediction, which lead to improved process design as well as an ability to trouble-shoot thermal processing problems.

One of the principal areas in which data are needed in order to safely determine thermal processes is that of rotational or agitated retorting. In order to evaluate these processes, a thorough understanding of the heat transfer properties of the retorting medium, the package and the food contained in the package are needed. Movement of containers in an agitating retort makes it difficult to obtain direct measurement of food temperatures during commercial thermal processing.

At the present time, there are well-established protocols for establishing microbiologically safe thermal processes in static heating modes. However, the setting of thermal processes in rotary modes becomes more complex since forced convection will occur within the container.

In multi-component formulated foods, factors such as sauce viscosity, particulate size and quantity, etc., need to be investigated to determine their effect on the rate of heat transfer during the

sterilisation process. In addition, the introduction of mixing dictates that as well as individual effects, the effects of interactions among these parameters, for example, the combined effect of rpm, container position and particulate size must be quantified and understood. This understanding then allows one to identify the worst case conditions with respect to the rate of heating. It is these worst case conditions that must be used to set the scheduled process for the production cycle. By identifying the worst case conditions, it is possible to ensure that all products receive the minimum thermal process considered to be microbiologically safe without occurrence of severe over-processing.

Another area of concern in the field of thermal processing is the use of new forms of packaging; for example, the introduction of a "plastic can" which is a semi-rigid, two-piece, bowl-shaped container with a double-seamed metal end, for example the 307 OMNI® bowl. The designation of 307 refers to the diameter of the container in inches. The first digit represents the diameter in whole inches and the next two digits are the remainder of the diameter in 16th of an inch. Thus 307 is 3 and 7/16th inches. The advantages of this package are: 1) microwaveability (the special shape improves uniformity of heating in a microwave oven); 2) portion-control (single serving); 3) the easy-to-open co-extruded structure containing ethylene vinylalcohol (EVOH) gives long shelf life due to O₂ barrier properties; 4) the structural plastic (polypropylene) resists high temperatures, so it can be thermally processed to stabilise the contents for ambient storage; and 5) the product/package combination is convenient to the consumer.

Products suited to this package include soups and stews, pasta meals, etc., which may undergo conduction, convection or mixed-mode heating during thermal processing. Some are best processed in agitated cooks. Accordingly, there is a need to know the mechanisms of heating and cooling of these varied products in this new container shape, which must be processed

in overpressure media because the package body is comprised of thermoplastic polymers. Since plastics soften, weaken and may deform (i.e., expand due to expanding headspace gases), overpressure is needed to maintain package shape and thus protect against loss of hermetic integrity. Challenging problems exist in understanding heat transfer from the heating medium (steam/air, water immersion, or water spray) to the package and from the package throughout the container contents, especially to the least-lethal or cold-point in the food.

The objectives of this study were to gain a deeper understanding of end-over-end agitated processes of foods in microwaveable plastic containers, as well as applications to pasteurisation processes by means of:

- 1) Testing the hypothesis that patterns of particulate movement under agitation in these containers (307 OMNI® bowls) behave under gravitational forces and not centrifugal.
- 2) Using microprocessor control of retort processes to modify Dickerson's method (Dickerson, 1965) of determining thermal diffusivity to evaluate potato/alginate gel spheres for suitability of thermal characteristics for use as model food particles in this study.
- 3) Testing the hypothesis that positional effects observed in the Lagarde simulator also exist in the production Lagarde.
- 4) Testing the hypothesis that time-temperature data as well as time-temperature indicator systems collected for flexible thermocouples show heating more like free moving food particles than do traditional rigid thermocouples in determining effective thermal processes.
- 5) Extending the hypothesis that the time-temperature indicator concept can be applied to both pasteurisation and sterilisation processes, thus increasing the range of applications.

CHAPTER 2

LITERATURE REVIEW

2.1 Rotational Processing:

The work published by numerous scientists and technologists has spread the knowledge that the major quality characteristics of canned foods (appearance, colour, flavour, texture and nutritional content) could be better maintained using higher temperatures but shorter times than with conventional sterilisation processes (Ball, 1938; Clifcorn, 1948). In 1950, Clifcorn and co-workers drew attention to new principles of rotary sterilisation. Heat penetration to the cold spot could be achieved much faster if the container with a liquid or semi-liquid product and a normal headspace was agitated by end-over-end rotation of the sealed container during processing. As well, agitation processes would allow higher temperatures to be used. Sensory characteristics were considerably improved over those in conventional processed foods produced under static conditions.

Clifcorn et al., (1950) stated that in liquid or semi-liquid products processed in a static retort, heating was mainly by convection. The presence of particulates slowed the heating of the product and heating within particles occurred by conduction. He further observed that since many semi-liquid products heated by both convection and conduction, and transfer of heat to the cold spot resulted in a long process time. Since there was little movement of the product along the can wall,

that portion of the product was overcooked. Agitation allows all parts of the product to approach the temperature of the sterilising environment much faster. Thus:

- a) For viscous products or heat sensitive products, higher retort temperatures such as 126, 132, and 138°C for relatively short times could decrease the problem of overcooking and scorching.
- b) Rapid high temperature-short time methods for sterilising products preserved the colour, flavour and texture as well as the nutritive value of the foods.
- c) Greater flexibility in sterilisation times and temperatures would be available for tailoring the sterilisation process to fit various products in several container sizes, thus allowing processes with the same sterilisation effect (F-value), to be selected on the basis of the degree of cooking (C-value).
- d) Many of the more viscous, semi-liquid or heat sensitive products could be sterilised in larger containers without overcooking.

Clifcorn's work was decisive for the improvement of the quality of thermally processed foods and his method was patented in the United States. However, a picture of the laboratory rotation autoclave used in his experiments, shows that his insights were gained through laboratory experimentation and needed verification and development in an industrial setting. At the time this form of verification was not possible for the lack of a commercial sized end-over-end rotary retort, capable of operating at higher rotary speeds.

Independently of the research in the United States, the idea of a rotary retort which would keep cans in agitation throughout the sterilisation and cooling process, also surfaced in Germany. The basic idea was two-fold, accelerate heat penetration and prevent scorching of the product. The engineer Hermann Stock in Neumunster developed his first rotary machine in the form of a double

vessel, horizontal, full water immersion retort, with overriding pressure, featuring a top water storage vessel and a pump that circulated the water in the bottom processing vessel.

From a commercial standpoint, these developments resulted in a shorter cycle time for products passing through the retort. Thus, capacity in manufacturing locations could be increased in addition to potential improvements in product quality.

There was already a well established protocol (Ball, 1923) for establishing microbiologically safe thermal processes in static mode. However, the determination of thermal processes in rotary mode is more complex since mixing will occur within the container and different containers may experience different heating environments. The traditional robust method of evaluating all the parameters that may affect the rate of heating under these circumstances would require a full factorial experiment, where all possible factor-level combinations are individually investigated (Table 2.1). This example could result in a total of $2^7 = 128$ trials being required to confidently identify the worst case conditions (Haine, 1992). Obviously this is not practical as it would need to be repeated within each operating company under their own unique conditions. This would serve to severely restrict the use of rotary retorts (Haine, 1992). One way to overcome this limitation is through the use of a fractional factorial experimental design. This one form of statistical approach to fractional factorial experimentation is now more commonly referred to as The Taguchi Method (Taguchi and Yokoyama, 1994) which provides a powerful diagnostic approach to designed experimentation and optimisation of product, process and manufacturing designs. The aim of this approach is to identify and quantify the product and process factors which have an effect on the position of the experimental mean value for the selected quality characteristic (for example F_0 = minutes, where F_0 is the number of minutes at a reference temperature or 121°C required to destroy a specified number of organisms whose z value is 10C°; z is a measure of an organism's sensitivity

Table 2.1 To assess all possible interactions at 4 different levels of rpm will require 128 experiments to identify the worst case conditions.

Critical Control Factors - " Experimental Levels"

Critical Control Factor	Level 1	Level 2	Level 3	Level 4
A: Base Viscosity (% Starch)	3.5	4.5	4.5	4.5
B: Retort Temperature (°C)	115	123	123	123
C: Piece Quantity (% Fill Wt.)	20	60	60	60
D: Piece Size (mm)	207	253	253	253
E: Fill Weight (Grams)	207	253	253	253
F: Speed of Rotation (rpm)	0	5	10	20

Adapted from Haine (1992).

to a change in temperature) and the variability about the mean. Having identified these factors, the aim is to move the mean to a desired (target) value and then decrease the variability about the mean value, hence optimising the product and/or process. In this study, Taguchi methods were not used as the number of variables under consideration did not warrant a full factorial experimental design.

Attempts to assess some of the factors such as rotational speed, radius, headspace, particle size and product viscosity have been undertaken (Abbatemarco and Ramaswamy, 1994; Knap, 1994; Ramaswamy et al, 1995; Sablani and Ramaswamy, 1996; Ramaswamy and Sablani, 1997; Sablani and Ramaswamy, 1997. However, Britt (1993) made note that when the position of the headspace bubble together with the angle and radius of rotation for containers on the diagonal through a basket was considered, every container in the plane has the potential to heat and cool differently during a particular process.

2.2 Heat Transfer in Packaged Food:

To start with a simplistic definition, heat is the kinetic energy of the chaotic agitation of molecules making up matter. The level of this kinetic energy determines the temperature of the material, and this in turn, determines whether the material will gain heat from or transfer heat to its surroundings. Since heat is a form of energy, it must obey the law of the conservation of energy; the mathematical form of this conservation law yields the fundamental equations which govern heat transfer.

Heat can be transported within, and can enter or leave a food material by four physical mechanisms:

- 1) Conduction,
- 2) Convection,
- 3) Radiation,
- 4) Phase Change

Conduction heating occurs when thermal energy is transferred from one molecule or atom to an adjacent molecule or atom without gross change in the relative positions of the molecules or atoms. Molecular movement is limited to oscillation about a fixed position, allowing any given molecule to make contact with only its immediate neighbours (Lund, 1975). Therefore conduction requires good contact between the food material and the heat transfer surface for effective external delivery or removal of heat. In most practical systems, excluding plate chillers and band cookers, this is difficult to achieve and conduction therefore plays only a minor role in external heat transfer. However, conduction does play a major role in internal heat transfer within food materials, particularly in the case of solid materials.

The term convection is a potential source of confusion when it comes to discussing heat transfer mechanisms because it is used in two quite different contexts, although it refers to the same physical mechanisms. Specifically, convection refers to the transport of heat by fluid flow. Generally speaking, it is a more efficient transport mechanisms than conduction. In heat transfer by convection, the molecules move about, resulting in mixing of warmer and cooler portions of the same material (Lund, 1975). Convection is thus restricted to flow of heat in fluids, either gases, liquids or suspensions. Clearly, in the case of liquid foods, convection can play a major transport role in the internal transport of heat; a good example of this form of heat transport is the UHT processing of soups and milk. However, the more common use of the term convection applies to those cooking and cooling operations in which heat is transferred between the surface of the food

and the fluid adjacent to it (Tung et al., 1989). Although it is not immediately obvious, this form of heat transport ultimately derives from the fluid flow within the thermal boundary layer near the surface. In most cooking and cooling operations, this surface convective heat transfer is the major heat transfer mechanism, particularly for external heat delivery and removal.

In most operations, radiant heat transfer, which requires a large temperature difference between the surface of the food and the heat transfer surface, plays a minor role. Radiant heat transfer is the primary source of energy in the grilling of small objects and is often used to improve surface appearance. An increasingly utilised application of radiation (i.e., transport of energy by electromagnetic radiation) in the food industry today is the use of microwaves for heating purposes.

Evaporation and condensation cause heat losses and gains which are often overlooked in many conventional cooking and cooling operations. Indeed, in some processes, they constitute the major heat transfer mechanism involved.

Products intended to be stable at ambient temperature have to be rendered biologically safe throughout the distribution chain; this is achieved through the process of commercial sterilisation. For low acid foods this requires that the temperature of every part of the foodstuff reaches approximately 120°C for a least three to five minutes or the equivalent (Fryer, 1992). The degree of sterilisation can be quantified by the so-called F_0 value. This value is defined in terms of the time-temperature history experienced by the product. Since 120°C is well above the boiling point of water, the heating must be done under pressure. Consequently, the sterilisation of many food products, and in particular, products containing solid components, is carried out in pressure retorts.

The sterilisation of liquid products in containers can be achieved relatively quickly and uniformly, since fluid convection is the dominant internal heat transfer mechanism involved. However, if the product contains solid components, the speed at which sterilisation can be achieved

is restricted by how fast heat is transferred within the solid components, and this is governed by thermal conduction. In this case, sterilisation is only achieved when the thermal centre of the largest solid food component (for some products, this may correspond to the entire product) reaches the required temperature for the required time. Consequently, the heating time is determined by how quickly this thermal centre heats. This heating time can be calculated using computational models for virtually any solid food product, providing the thermal properties, configuration and heating conditions are known.

Another important aspect in retort processing is designing heating cycles which achieve the microbiological safety requirements at the thermal centre without over-processing other parts of the product. This quality question is fundamentally dependent on the heat transfer within the product during processing and the kinetics for both microbial and quality attribute destruction.

2.2.1 Conduction Heat Transfer:

As already noted, temperature is the driving force for flow of heat. The larger the temperature difference over a region, the greater will be the heat flow. Heat flow is also proportional to the area perpendicular to the direction of flow. If heat flow through a unit area is occurring at a certain rate, then heat flow through an area twice as large will obviously be twice as great. Finally, rate of heat flow decreases as the length of path for a given temperature difference increases (Watson and Harper, 1987). These conditions are for a steady state system (in which temperatures do not change with time) and are illustrated in Figure 2.1.

In Figure 2.1, the two planes are separated by a distance dx . Over this distance, temperature changes by an amount dT . The rate of heat flow follows Fourier's law:

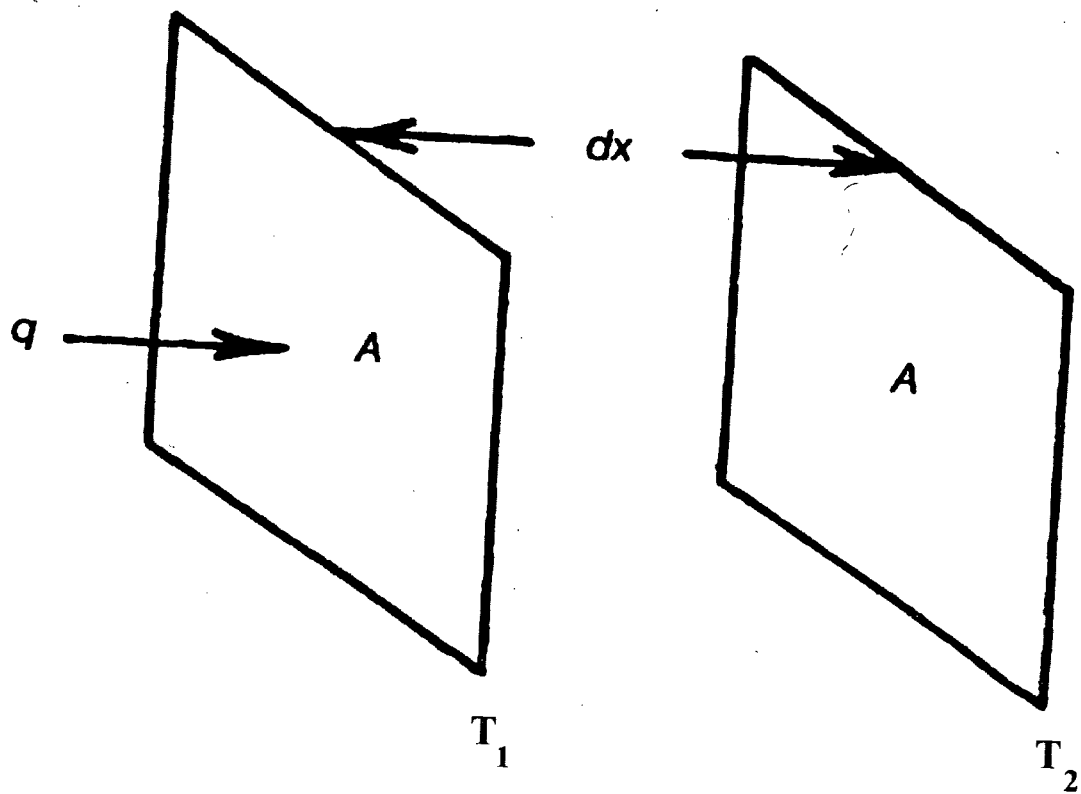


Figure 2.1. Conventions for conductive heat flow (Watson and Harper, 1987).

$$q = -k A (dT/dx) \quad (2.1)$$

with q expressed in SI units as watts, A in m^2 , x in m , and T in $^{\circ}C$ or K . The factor k , is thermal conductivity and is a property of the substance, with units of $W m/m^2 K$. The term dT represents the difference in temperature (denoted T_1 and T_2) between two points, x_1 and x_2 , separated by a distance of x (designated dx). Equation 2.1 applies to conduction of heat in the x direction at a point within a volume of some substance. For a complete mathematical description, similar equations for the other two directions would be included in three-dimensional heat flows, and integrated over the entire volume. The primary interest is in steady-state heat conduction, meaning that q is constant. Thus heat can be considered to be analogous to the fluid in a fluid-flow system. Therefore, just as the mass flow rate of fluid is constant throughout a steady-state system, so must the heat flow rate be constant at every point. Keeping these restrictions in mind, Equation 2.1 can be re-arranged as

$$(q/A) dx = -k dT \quad (2.2)$$

If area A is constant over the length of the heat flow path, and the thermal conductivity can be considered to be constant, equation 2.2 integrates to give

$$(q/A)\Delta x = k (T_1 - T_2) \quad (2.3)$$

Equation 2.3 is the usual integrated form of Fourier's law for unidirectional steady-state heat conduction over a path of constant cross-sectional area.

2.2.2 Concept of Resistance:

The study of natural phenomena produces many instances in which some kind of driving force or potential difference results in a flow of some quantity. Fluids flow as a result of a pressure driving force. Electricity or electric charge flows under the action of an electrical potential, or voltage difference. The driving force in each system is an intensive property of that system. Whatever flows is an extensive quantity, that is, it behaves like a quantity of some substance that can be collected and measured. Obviously heat cannot be measured in the way that fluids can. However, heat flowing through a system must be accounted for in total quantity, just as in a flow system. This is done by making a heat balance around a system in the same manner in which a material balance is made. In systems of the type under discussion, rate is found to be proportional to the driving force. As such, it is common practice to express the proportionality constant as the inverse of resistance.

The flow of electric current is equal to the voltage difference divided by the resistance. This produces the relationship known as Ohm's law. A slight rearrangement of equation 2.3 yields the integrated form of Fourier's law:

$$q = \frac{(T_1 - T_2)}{\Delta x/kA} = \frac{(T_1 - T_2)}{R} \quad (2.4)$$

where thermal resistance R is equal to $\Delta x/kA$, with Δx = to change in distance (m), k equal to thermal conductivity and A equal to area (m^2). This form of Fourier's law provides no new information, but it does simplify many calculations. Since it is mathematically identical to Ohm's

law for electric current flow, many results that have been obtained from electrical techniques can be used directly. This analogy between heat flow and electricity provides a very powerful experimental tool. Apparatus consisting of equivalent electrical components can be used, electrical measurements made, and the results converted to heat flow quantities. There is an additional advantage: electrical measurements can be made much more easily and with greater accuracy than thermal measurements. However, in the case of heat transfer studies, experiments often use thermocouples to determine thermal quantities such as thermal diffusivity and to do heat penetration analyses.

2.2.3 Heat Transfer in Retort Processing:

Saturated vapour condensing on a surface may be considered to spread to form a liquid film over the surface; alternatively the condensate may take the form of individual microscopic droplets which release heat to the condensing surface and then are quickly swept away to allow further condensation of small droplets (Tung et al., 1989). In retort applications, the former description is generally considered to be the more appropriate. A liquid film obviously imposes an additional resistance to heat flow, and heat transfer coefficients are smaller for film-type than for drop-wise condensation. The condition of the heat transfer surface can be expected to have a pronounced effect on the mechanism of steam condensation. Thus, the presence of a soil deposit on the outside of a container may alter the type of condensation as well as create a resistance (Watson and Harper, 1987). In addition, burn-on on the inside surfaces of a container, or the presence of any non-condensable gas in the vapour, can cause a marked decrease in surface heat transfer coefficients.

Due to these variables which cannot be controlled in actual practice, it is not possible to write a

reliable equation to predict condensation heat transfer coefficients. Fortunately, the heat transfer resistance of a condensing vapour is usually small compared to other resistances in series with it, and the uncertainty will not be important.

The principal mechanisms of heat transfer involved in retort processing are convection and conduction. Convection is the means by which heat is transferred from the retort heating medium to the package which contains the product, and as such, provides the boundary conditions for heat transfer. The convective surface heat flux (q) is given by:

$$q = h(T_e - T_s) \quad (2.5)$$

where h is the surface heat transfer coefficient and T_e and T_s are the media and surface temperatures, respectively. For retorts, the value of the heat transfer coefficient is in excess of 15,000 W/m² °C for pure steam (Coulson and Richardson, 1977). For steam/air mixtures, the values can range from 1300 to 11,000 W/m² °C for steam contents ranging from 50.3% to 98.2% respectively (Ramaswamy et al. 1983).

Conduction is the major heat transfer mechanism within solid food components, and as such, provides the heat transfer equation; the conduction heat flux is given by:

$$q = k(T_o - T_c) \quad (2.6)$$

where k is the thermal conductivity, which for most solid foods is of the order of 0.5 W/m °C (for a unit area of 1 m²), and T_o and T_c are the outer edge and centre temperatures of the food material. Physically, what happens during the heating process can be described in simple terms. Initially,

because of the large temperature difference between the heating medium and the product, the surface heats up very quickly and there is a large mismatch between the surface convective heat flux and the interior conductive heat flux. In particular, in the initial stages, the conductive heat flux at the thermal centre is negligible. Consequently, there is a finite time delay before the thermal centre responds to the heating of the surface. This type of behaviour in which the heating rate of the thermal centre is slow compared to that of the surface is called conduction limited heat transfer, and it is a fundamental characteristic of retort processing.

2.2.4 Convective Heat Transfer:

The particle-liquid heat transfer coefficient is a crucial and largely unknown factor in determining the temperature distribution in conventional heating. Data on heat transfer coefficients in food process operations are scarce.

Most processing applications of convection heat transfer are concerned with steady-state transfer between a fluid and a solid surface. Steady-state transfer exists when the temperature at any point does not change with time, i.e., the system is in equilibrium. There are however many important situations in which conditions of steady-state do not prevail. The problems that arise from these situations fall into the category of transient heat transfer or unsteady-state heat transfer. Essentially any time a processing operation is started, there will be conditions of transience in which temperatures continue to change, often throughout the duration of that process. An important transient heating food application is in the thermal processing of foods in individual containers, that is to say, in the retort sterilisation of packaged foods. However for the initial discussion of heat transfer, the steady-state case will be considered.

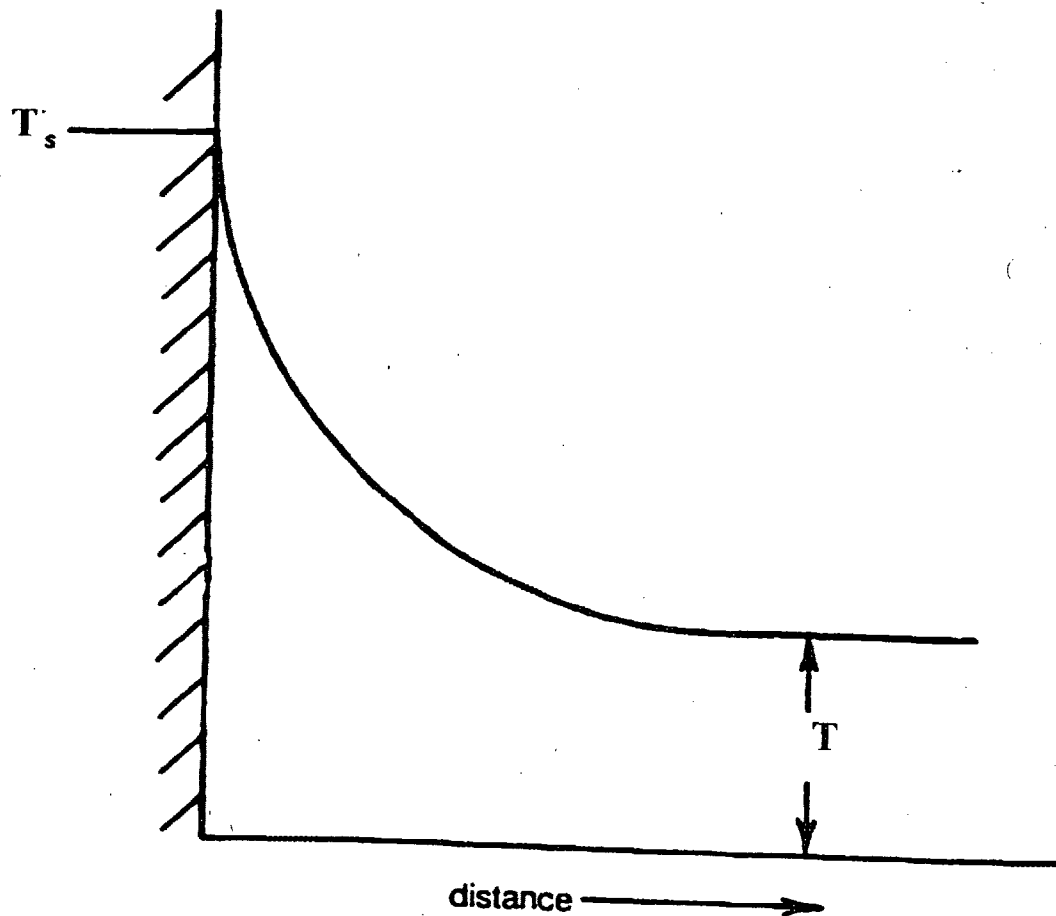


Figure 2.2. Temperature profile in a fluid moving past a surface at temperature T_s (Watson and Harper, 1987).

Figure 2.2 illustrates the situation that must be considered in order to develop the basic convection relationship. In this relationship, a fluid at temperature T is moving past a surface at a higher temperature T_s . The flow may be either natural or forced convection, streamline or turbulent. The curve in Figure 2.2 represents the temperature profile corresponding to a given position on the surface. Under steady-state conditions, this profile does not change with time, but may vary from one position to another along the surface (Watson and Harper, 1987).

At the surface of a solid, velocity of a fluid in contact with the surface is essentially zero, while immediately adjacent to the surface, there is a thin layer in which the velocity is low enough that flow is streamline, regardless of the nature of flow in the mainstream. Heat transfer across this thin, essentially stagnant layer must be by conduction. Since this layer however has no sharply defined thickness, the calculation of heat transfer by a simple application of conduction equations cannot be done. Moving farther away from the surface, the surrounding fluid is believed to increase in velocity, and consequently, the resistance to heat transfer drops.

Figure 2.2 thus shows a sharp increase across the stagnant streamline layer, where thermal resistance is high. Farther from the surface, where convection is more effective and the thermal resistance lower, the temperature gradient is decreased. The profile would have the same shape but be reversed for a fluid temperature higher than the surface temperature. This profile is seen to be very similar to the velocity profile in turbulent flow, which also changes rapidly near the surface and more gradually farther out in the stream.

Newton's law of heating states that the rate of heat flow per unit surface area is proportional to the difference between the surface and fluid temperatures

$$q = h A (T_s - T) \quad (2.7)$$

The proportionality constant h represents a thermal conductance of the fluid film across which the temperature change (and velocity change) takes place. This is commonly called the film coefficient of heat transfer, or simply the heat transfer coefficient. In streamline flow, there are no eddies that bring about bulk mixing of portions of the fluid at different temperatures, and molecular motion accounts for all heat flow. Combining relationships for basic fluid flow and heat conduction, yields an approximation of the heat transfer coefficient, and Equation 2.7 must be considered to represent an empirical law with values of h based on experimental measurements.

This does not pose an insurmountable or awkward a problem as might be assumed. Comparison of Equations 2.3 and 2.7 shows that h has the units of k divided by distance, W/m^2K . Convective heat transfer is frequently expressed in the more convenient form of a temperature difference driving force divided by a resistance, $1/hA$.

In returning to the concept of a stagnant surface layer containing all the resistance to heat transfer and a turbulent bulk where the mixing effect of the eddies result in negligible heat transfer resistance, the value of h is given by the thermal conductivity of the fluid divided by the thickness of the hypothetical stagnant layer. Conversely, the thickness of the stagnant layer is given by the ratio of an experimentally determined h to the thermal conductivity. It is important to remember however, that the stagnant film is purely hypothetical and that there is no definite boundary separating the surface layer from the turbulent bulk.

One of the most common applications of heat transfer in food processing consists of heat flow from a hot fluid, through a solid wall, to a cooler fluid on the other side (one may consider the condensation of steam on a can as fulfilling the first condition). This creates the situation of a series of resistances (Figure 2.3). The relationship for resistances in series is quite general and applies regardless of the nature of the individual resistances:

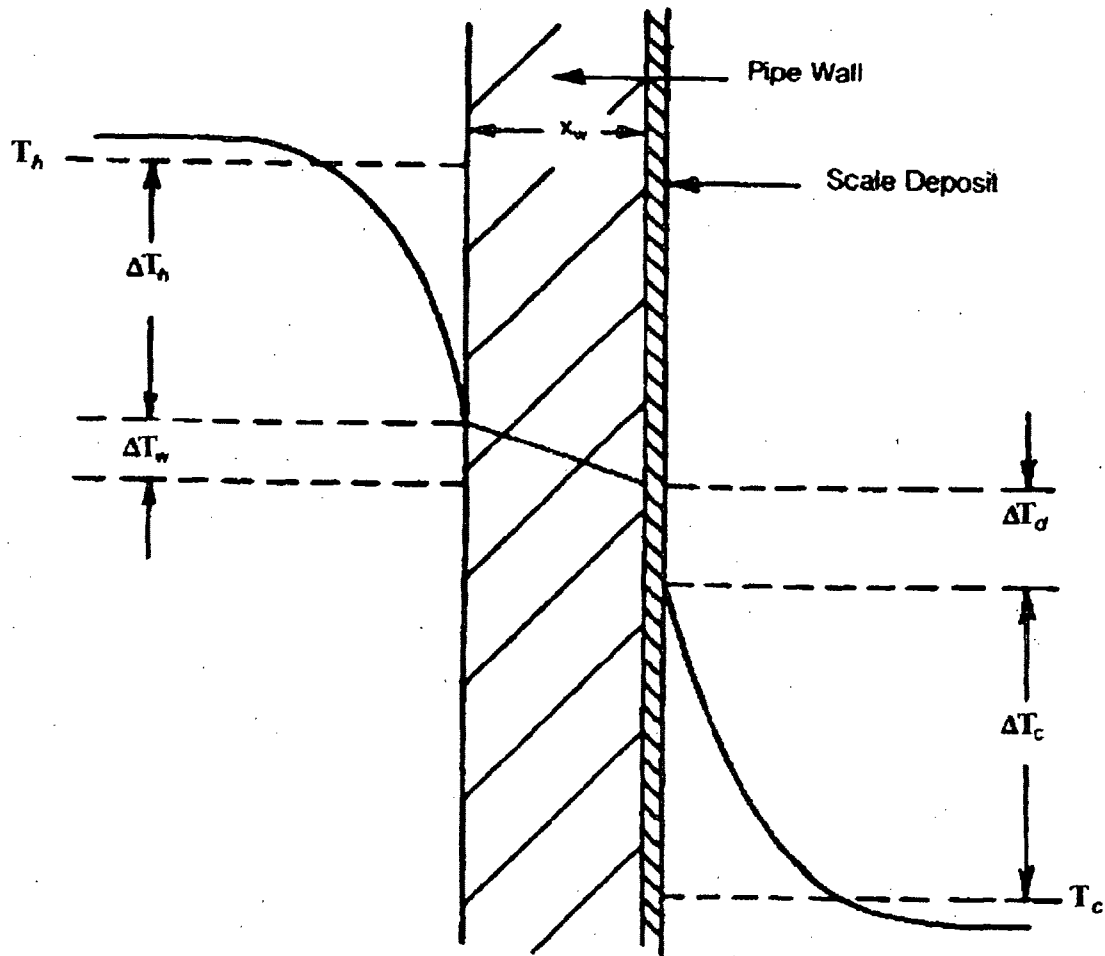


Figure 2.3. Temperature profile from a hot liquid at T_h to a lower fluid temperature at T_c , across a wall (Watson and Harper, 1987). T_c = temperature of bulk fluid inside the wall, ΔT_c = temperature gradient from the inner wall surface into the bulk fluid, T_h = temperature of heating medium, ΔT_h = temperature gradient from the bulk heating medium to the outer surface of the wall, ΔT_d = temperature gradient across a deposit layer on the inside of the wall, ΔT_w = temperature gradient across the wall, x_w = wall thickness.

$$R_t = R_h + R_w + R_c \quad (2.8)$$

or

$$1/U = 1/h_h + x_w/k_w + 1/h_c \quad (2.9)$$

where areas are based on some unit size. The quantity U in equation 2.9 is known as the overall heat transfer coefficient. If the individual coefficients (or resistances) are known, U is easily calculated. Experimental heat transfer studies frequently involve calculation of U under controlled conditions and the calculation of individual coefficients from these. With the value of the product UA being determined, the value of U depends on the area that is used with it. Thus,

$$q = UA(T_h - T_c) = U_c A_c (T_h - T_c) = U_h A_h (T_h - T_c) = U_w A_w (T_h - T_c) \quad (2.10)$$

The above serves well for a situation of streamline flow, which can be handled by an exact calculation, and turbulent flow, which can be handled by a single empirical correlation. However, heat transfer concerns itself with a much wider variety of types of flow and flow system geometries, the differences between which are substantial. In order to take care of such a diversity of situations, it is to be expected that many different empirical correlations are needed and that these should be expressed in terms of dimensionless ratios (Sablani et al., 1997). Table 2.2 lists the more commonly used ratios.

The Reynolds number (Re) characterises flow in a forced convection system. In natural convection, the Reynolds number is not applicable and the Grashof number (Gr) takes its place, involving the coefficient of volumetric expansion β , and the temperature difference as the source of

Table 2.2. Dimensionless ratios employed in convective heat transfer (from Watson and Harper, 1987).

Name	Symbol	Group
Reynolds	Re	$DV\rho/\mu$
Prandtl	Pr	$C_p\mu/k$
Nusselt	Nu	hD/k
Stanton	$St = Nu/Pr Re$	$h/C_p V$
Grashof	Gr	$L^3\rho^2g\beta\Delta T/\mu^2$
Graetz	$Gz = (Re*Pr(L/D))/4$	mC_p/kL
Biot	Bi	$hL/k = UL/k$
Fourier	Fo	$(k/\rho C_p)(\Delta T/r_o^2); (k/\rho C_p)(\Delta T/L_o^2)$

Summary of Abbreviations:

- g = gravity
 D = diameter, m
 V = velocity, m/s
 ρ = density
 μ = viscosity
 C_p = heat capacity at constant pressure, J/kg K
 k = thermal conductivity, W m/m² K
 h = conductance film coefficient of heat transfer, W /m² K
 L = length, m
 β = coefficient of thermal expansion
 ΔT = temperature difference between a hot stream and a cold stream
 m = mass
 r_o = radius of a cylinder or sphere, m
 U = overall heat transfer coefficient (W /m² K

that convection. The volumetric expansion coefficient is defined by the relation

$$\Delta v/v = \beta \Delta T \quad (2.11)$$

where Δv is the change in volume resulting from a small temperature change ΔT . For a gas obeying the ideal gas law, it can be shown that β is equal to the reciprocal of the absolute temperature $1/T$.

The Prandtl number (Pr) is made up entirely of fluid properties and forms an essential part of all general convective heat transfer correlations. The Nusselt (Nu) and Stanton (St) numbers are ratios that include h as a factor, and correlations for forced convection can be written using either the Nusselt or Stanton number. Since velocity is one of the factors in the Stanton number, it cannot be applied to natural convection. The Graetz (Gz) number is a group that enters into correlations for streamline flow.

When faced with the problem of evaluating h for a particular situation, the appropriate correlation must be chosen. Each of these equations do not represent a new theoretical principle, they are all based on the principles discussed earlier and their use is similar. It becomes simply a matter of selecting the appropriate equation for the particular situation.

In most models of sterilisers used commercially, the basic correlation for heat transfer used is:

$$Nu = 2 + a Re^{\alpha} Pr^{\beta} \quad (2.12)$$

where Re is the Reynolds number for the particle based on slip velocity and 2 is the conduction limit. The term a is a constant and is equal to 0.60 for Re between 1 and 70,000 and Pr

between 0.6 and 400 (Heldman and Singh, 1993). In many models, the value $Nu = 2$ is used without attempting to consider flow. Work is necessary to understand the particle-liquid heat transfer coefficient and thus to increase the accuracy of predictions of process sterility and quality (Fryer, 1992).

It has been known for 50 years (Fryer, 1992) that heat transfer between fluid and a surface is dependent on the viscosity of the fluid bulk, that is, its resistance to flow. At the wall:

$$Nu/Nu_0 = (\mu_b/\mu_w)^z \quad (2.13)$$

where Nu_0 is the expected heat transfer coefficient in the absence of viscosity variation, and μ_b and μ_w are the bulk and wall viscosities, respectively. Work at Bath University (Field et al., 1992) showed:

- 1) that an expression similar to the above (Equation 2.13) can be derived from first principles with $z = 0.27$.
- 2) high temperature driving forces during heating or cooling do not always increase the heat transferred; there is a maximal heat transfer flux resulting from the decrease in h with decreasing wall temperature, i.e., due to the increased fluid viscosity near the wall.

2.2.5 Unsteady-state Heat Transfer:

As mentioned above, there are many important situations in which conditions of steady-state heat transfer do not prevail. The problems that arise from these situations fall into the category of transient heat transfer or unsteady-state heat transfer. That phase of heating when the temperature

is changing with time is referred to as unsteady-state (or transient) heating. During this phase, temperature is a function of both time and location (Heldman and Singh, 1993). In thermal processing, this unsteady-state phase dominates the entire process (Heldman and Singh, 1993).

In the basic approach to this problem, an object of initially uniform temperature is suddenly placed in an environment of a different temperature. One wishes to determine the temperature of the object as a function of time. An important point to note is that the mathematical solution to the problem proceeds in the same fashion, regardless of whether the process is one of heating or cooling. The solution for the cooling process is the reverse of the solution for the heating process. Thus, a slight rearrangement of the integrated form of Fourier's law (Equation 2.7) will give

$$q = \frac{(T_1 - T_2)}{\Delta x/kA} = \frac{(T_1 - T_2)}{R} \quad (2.13)$$

where the thermal resistance R equals $\Delta x/kA$. This provides an extremely powerful experimental tool.

In heat flow there exists a convection resistance between the surface of the object and the surrounding medium and an internal resistance within the object. Thus, there will be a temperature difference between the medium and the surface of the object and there will also be a temperature difference within the object which will vary continuously from the surface to the centre.

In the case of liquid food in a can, if there is sufficient agitation to provide food mixing, the temperature throughout the interior will be more uniform, except for a thin film adjacent to the surface. In this case, the external resistance is a series consisting of the outside resistance, the resistance of the container wall, and the inside film resistance. To determine the influence of each

of these resistance, the Biot number (or modulus), designated as Bi , has been developed (Table 2.2). Thus, a very low value of the Biot modulus means that internal-conduction resistance is negligible in comparison to surface-convection resistance. This in turn implies that the temperature will be nearly uniform throughout (Loncin and Merson, 1981; Heldman and Singh, 1981).

In considering a fluid with no mixing, or a solid, the assumption of uniform heating applies. However, as heating is initiated and progresses, temperatures near the surface will change more rapidly than those toward the centre. Thus, while the product may start off at a uniform initial temperature, since there are no convective mixing forces occurring, temperature remains a function of distance from the surface. Also, since the thermal conductivity of food materials is much lower than that of metals and the heat flow is from the outside to the centre, the outer edges of the product are exposed to higher temperatures for a much longer time than the centre of the product.

The general solution to this problem must therefore give temperature as a function of both time and position. Thus, the analysis of the transient heating or cooling processes which take place in the interim period before equilibrium is established must be modified to take the change in internal energy of the body into account, and the boundary conditions must be adjusted to match the physical situation which is apparent in the unsteady-state heat transfer problem.

The objective of any heat transfer analysis is to predict heat flow or the temperature which results from a certain heat flow (Holman, 1976). In most instances the general case of two-dimensional steady-state flow is considered. Therefore, if a steady state exists and one assumes constant thermal conductivity, then the Laplace equation applies

$$\frac{d^2T}{dx^2} + \frac{d^2T}{dy^2} = 0 \quad (2.14)$$

and the temperature in a two-dimensional body will rise as a function of the two independent space co-ordinates x and y .

The solution to Equation 2.14 may be obtained by analytical, numerical or graphical techniques. In considering the choice of solution technique, one has to remember that even though an immense number of analytical solutions for conduction and convection heat transfer problems have accumulated in the literature (Lenz and Lund, 1977; Naveh et al., 1984; Datta and Teixeira, 1987; Pham, 1987; Tan and Ling, 1988; Richard et al., 1991), there are many practical conditions where the geometry or boundary conditions are such that an analytical solution either has not been obtained or involves such a complex series solution that numerical evaluation becomes extremely difficult. For these solutions, one turns to the techniques of finite-difference or finite-element analysis to yield the results.

2.2.6 Particle Heat Transfer Coefficients:

The methodology for analysis of particle heat transfer coefficients in a flowing liquid medium involves the traditional approach of analysis of fluid and particle centre temperatures (Weng et al., 1992). Most authors (Chandarana et al., 1989a; Chang and Toledo, 1989) have used an immobilised particle in a cage and pumped fluid at a predetermined velocity over the particle in order to generate time-temperature profiles. The difficulty is that real particle dynamics in a processing system are not fully represented. Sastry et al. (1989) used a hollow aluminum sphere with a thermocouple attached to it. This allowed for movement in a tube with the same velocity as a free particle, yielding an experimental set-up close to real processing conditions. However, free rotation of the particle was limited by the attached thermocouple. Heppel (1985) immobilised

spores of *Bacillus stearothermophilus* in alginate beads (diameter 3.1 mm) and estimated the fluid-particle heat transfer coefficient from spore survivor data. This approach did not disturb the movement of the food particle.

Weng et al. (1992) used a time-temperature integrator based on an immobilised particle containing horseradish peroxidase in conjunction with mathematical modelling to determine the liquid-particle heat transfer coefficient. Sablani and Ramaswamy (1996) also calculated liquid-particle heat transfer coefficients based on one or more measured temperature histories inside a heat-conducting body and used this as a basis for examining the effects of retort temperature, rotational speed, radius of rotation and can headspace on the associated heat transfer coefficients.

2.3 Thermal Indicators:

Thermal indicators in one fashion or another have been integral in the determination of adequate thermal processes in order to ensure commercial sterility. This is necessary to assess whether the objectives of the thermal process have been achieved. The tests may be for surviving micro-organisms, active nutrients, or quality factors such as colour, texture or flavour. Additional work has focussed on using thermal indicators (also referred to as time-temperature-indicators) to determine thermal properties such as liquid particle heat transfer coefficients (Weng et al., 1992).

A majority of the reactions occurring in foods obey well-established kinetics. Thermal destruction of micro organisms, most nutrients, quality factors (texture, colour, and flavour) and enzymes generally obey first-order reaction kinetics (Lund, 1975). The destruction rate of these components is dependent on the concentration of the component, a relationship that is frequently referred to as a "logarithmic order of inactivation or destruction."

The evaluation of commercial sterility in foods containing discrete particles was the focus of recent thermal processing studies (Stoforos and Merson, 1990; 1992), including both aseptic and agitated-cook retort processes (Chandarana et al., 1989b; Sastry et al., 1989). One of the unsolved problems in these types of processing systems is that it is practically impossible to measure time-temperature profiles at the slowest heating zone within a moving particle without disturbing its movement. This is analogous to the Heisenberg Uncertainty Principle discussed in quantum physics (Allen and Keefer, 1974). This renders mathematical approaches for lethality calculations (i.e., General Method (Patashnick, 1953), Ball's formula method (Ball and Olson, 1957), and other routines) inaccurate. In order to overcome this shortcoming, knowledge of the convective heat transfer coefficient between the liquid and the particle is critical to adequately simulate the heating rate of the particle centre. This parameter is the most difficult to measure (Heldman, 1989).

Earlier researchers in the field assumed an infinite heat-transfer coefficient at the particle-liquid interface (deRuyter and Brunet, 1973; Manson and Cullen, 1974). Dignan et al., (1989), later observed that predicted lethalties for the particle determined by this assumption resulted in an overestimation of actual lethalties delivered to the particle.

2.3.1 Microbial Thermal Indicators:

Bacterial spores are more heat resistant than vegetative cells and spores of different species exhibit a wide range of heat resistance (Gombas, 1983). For thermal process calculations of low-acid foods (pH greater than 4.6), the heat process is based on the resistance of spores. The thermal indicator traditionally used in determining commercial sterility in these food products is *Clostridium botulinum*, which has a D value of ≤ 0.21 minutes at 250°F and a z value of 17.8°F°

(Lund, 1975).

The time required to reduce the microbial population by 90% is called the "decimal reduction time" or D value (Katzin et al., 1943). However, food products do not heat or cool instantaneously. Instead they pass through a time-dependent temperature treatment. Therefore, the inactivation rate at several lethal temperatures must be known. The temperature dependence of the D value is characterised by the z value. The significance of z to the relative effects of temperature on reaction rates can be demonstrated as follows. A small z value such as 10F° indicates that the destruction time decreases by a factor of 10 for a 10F° temperature increase. A large z value such as 50F° indicates that the temperature must increase 50F° to reduce the destruction time by a factor of 10. Therefore, reactions that have a small z value are highly temperature dependent, and reactions exhibiting a large z value are less so (Lund, 1975).

A number of factors affect the thermal resistance of microorganisms. These can be categorised as three main types: inherent resistance, environmental influences active during growth and formation of cells or spores, and environmental influences active during the time of heating cells or spores.

The inherent resistance is demonstrated by the fact that different strains of the same species prepared under identical conditions exhibit widely differing degrees of resistance (Gombas, 1983). Spores or cells exhibiting the greatest resistance are believed to contain proteins with the greatest stability (Lund, 1975). Factors affecting heat resistance that are present during sporulation and growth are: temperature, ionic environment, lipids, organic compounds other than lipids, and age or phase of growth of the culture. The third group of factors, those environmental influences that are active during the heat treatment include: pH and buffer components, ionic environment, water activity, and medium composition (Lund, 1975). Thus, the thermal resistance of an organism is

dependent on many factors and each must be considered and reported.

Pflug et al. (1980) reported that the biological indicator units as used in their study in conjunction with a count reduction procedure, could be used effectively to determine the sterilising value delivered to cans of food processed in agitating retorts. The advantage of this type of system was that a large number of F-value measurements could be made in the same piece of equipment in the same general time period. Some caveats were presented by Pflug et al. (1980), notably that errors may be present in the biological indicator unit system due to its indirect determination of F-values. The errors described were of two types: a) errors present in the thermocouple temperature measuring system used to calibrate the biological indicators, and b) all of the additional variation due to the biological measuring system (as listed above) and its sensitivity to many uncontrolled environmental factors. An example is found when pH is considered.

Pflug et al. (1985) demonstrated the effect on F of pH on spores during heating of *Clostridium botulinum* spores. F is the thermal death time required to decrease the bacterial population by a multiple of the D value (Lund, 1975)). This differs from F_0 , which is the process lethality equivalent to time in minutes at 250°F assuming a z value of 18°F. The data showed that a decrease in pH from 6.0 to 4.6 resulted in a corresponding decrease in $F_{250^\circ\text{F}, z=18^\circ\text{F}}$, from 3.0 min to 1.2 min, respectively.

2.3.2 Enzymatic Thermal Indicators:

If the greatest concern in achieving a "safe" food product is that of microbiological heat resistance, then the thermal indicator of choice would be microbial in nature as this would give the truest reflection of actual sterilisation being carried out. In other words, the ultimate verification of

the effectiveness of a process is to test the actual organism being targeted under the specific process conditions of interest. However, this poses a number of problems. First of all, work involving *C. botulinum* must be carried out using specified biosafety conditions -- well outside the capabilities of the laboratories of the majority of food companies. Secondly, the possibility of contamination of commercial packs with spores from test packs in the plant environment also precludes using *C. botulinum* for determining adequate processes in the processing plant environment.

Similar to microbiological thermal indicators, the objective of thermal processes of enzymes is generally inactivation. Like those of micro-organisms, the thermal stabilities of enzymes are dependent on many factors.

The dependence of enzyme inactivation on temperature can also be expressed in the same way as used for microorganisms. For most enzymes, the D and z values are the same order of magnitude as those for microorganisms (Lund, 1975; Ramaswamy et al., 1995; Makki and Durance, 1996; Ramaswamy et al., 1996). In addition, the selection of the enzyme is of importance since some have been shown to have approximately first order inactivation kinetics while many others do not yield a straight line on a semi-log plot - this is especially true of most enzymes which may involve one substrate but two or three complexes (Lehninger, 1977).

In thermal process calculations based on enzymes, the most heat resistant enzyme capable of altering product quality during storage is selected as the basis of the process. The two enzymes generally selected as the basis for these calculations are catalase or peroxidase (Lund, 1975). Hendrickx et al. (1992) and Weng et al. (1992) demonstrated that lethalties read from the use of horseradish peroxidase as a bio-indicator coincided well with lethalties integrated from physically measured temperature profiles.

2.3.3 Nutrient and Quality Factor Thermal Indicators:

Many of the same factors that accelerate or inhibit microbial destruction or enzyme inactivation also affect nutrient destruction. Harris and von Loesecke (1960) reviewed the effects of heat processing on nutrients and reported that the rates of nutrient destruction were affected by numerous variables. Abbatemarco and Ramaswamy (1994; 1995) evaluated the effects of rotational processing on nutrient retention in steam/air retorts.

The z values for nutrient and quality factor destruction are larger than for microorganisms or heat-labile enzymes (Lund, 1975). D values for nutrients and quality factors are generally 100 - 1000 times greater than those for microorganisms (Lund, 1975). For example, the D value for *C. botulinum* at 250°F is 0.1 to 0.2 min (Lund, 1975) (depending on the factors previously noted), whereas the D value for thiamine, at the same temperature is of the order of 38-380 minutes (Hallstrom et al. , 1988). Mulley et al. (1975a, b) used thiamine as an index of sterility in canning for pea puree, beef puree and peas-in-brine where thiamine-HCl was blended at the rate of 0.16 mg per gram of product. Berry et al. (1990) used thermally labile methylmethionine sulfonium salt (MMS) in model food cubes and characterised thermal destruction of MMS by the amount of heat treatment and by diffusion and shrinkage of the cubes. Abbatemarco and Ramaswamy (1994) determined that percentage reduction in both ascorbic acid and thiamine in buffered aqueous solutions did not differ significantly when processed in the rotational mode (10 and 20 rpm) versus static mode.

2.4 Heat Penetration in Packaged Foods:

The measurement of the temperature history at the slowest heating point in a can of food as it is being thermally processed is known as heat penetration testing. These tests are required in order to establish process schedules necessary to prevent spoilage of canned foods by bacterial action (Alstrand and Ecklund, 1952; IFTPS, 1995).

Some of the earliest heat penetration studies used maximal reading thermometers placed inside cans (Kochs and Weinhausen, 1907). Later developments included the use of thermocouples for heat penetration measurements in 1907 and a report by Biting and Biting (1917) on a study of variations in temperature within a retort. The development of some of the basic equations describing the rate of heat penetration in canned foods has been credited to Thompson (1919), who conducted extensive tests using thermocouples. Bigelow et al. (1920) described the design and use of thermocouples for heat penetration measurements and discussed many of the factors relative to process determination. Benjamin (1938) made a basic improvement to thermocouple design which consisted of a connector type thermocouple which could be disconnected from the lead wire. Ecklund (1949) further improved the design such that the thermocouple did not project from the side of the can, thus allowing the container to pass through commercial production lines and container-sealing equipment. He also described complete specialised heat penetration equipment based on this type of thermocouple, which has become the predominant standard in North America.

Of interest for the present study, is the use of a semi-rigid plastic container, the OMNI® bowl (American National Can, Barrington, IL). Determination of thermal process values in plastic containers, because of the complex shape of the container, requires careful consideration. The geometric centre, for example, may not be the slowest heating point. This may result from the

shape of the container, the relative difference in heat transfer rates between metal and plastic container components (Bhowmik and Shin, 1991; Lu et al. 1991), the gaseous conditions within the headspace and the degree of container distortion experienced during processing. OMNI® bowls are unlike metal cans in that they may soften at temperatures experienced during retort processing. Pressure differences under these circumstances will promote container deformation and consequently pressure control is required within the retort vessel in order to minimise stress on the container. This overpressure is generally required for processing low-acid foods in which scheduled temperatures and pressures are independently controlled. The majority of these retorts rely on overpressure being achieved by the introduction of air. In all overpressure systems, it is vital that the heat transfer medium -- either steam/air mixtures, water immersion or water sprays -- be agitated adequately to ensure uniformity of temperature distribution within the vessel.

CHAPTER 3

THERMAL DIFFUSIVITY OF POTATO/ALGINATE GELS

3.1 Introduction:

Properties such as thermal conductivity, specific heat, and density are important in the analysis and design of food processes and processing equipment. In situations where transient or unsteady-state heat transfer occurs, a property called thermal diffusivity is very useful. Thermal diffusivity is the property which reflects the ability of a material to change temperature and is of significance in determining how fast heat diffuses through a material. This thermophysical parameter is defined as a ratio of three different properties and is expressed as follows:

$$\text{thermal diffusivity } (\alpha) = \frac{\text{(thermal conductivity, } k\text{)}}{\text{(density, } \rho\text{) (specific heat, } C_p\text{)}} \quad (3.1)$$

Thermal diffusivity is affected by both the water content and temperature of the material, as well as composition and porosity (Singh, 1982; Tung et al., 1989; Sandberg, 1991, Sandberg et al., 1994). As a result, since water content and temperature of a food product may change considerably, one can expect a variable thermal diffusivity within a given process. Furthermore, since many food products are non-homogeneous, the thermal diffusivity may vary from one

location to another within the same product.

The thermal diffusivity of a material effectively assumes that natural thermal convection does not occur. Therefore conductivity and not convection is the main heat movement into a material of constant specific heat. Attempts to determine thermal diffusivity can be done through heat transfer experiments to measure each of the properties in Equation 3.1. This requires considerable time and elaborate instrumentation. Thus, several researchers have developed techniques to determine thermophysical properties, such as thermal diffusivity, in foods. Dickerson (1965) developed a technique using a cylindrical tube. Sweat et al. (1973) measured thermal conductivity of chicken meat using a thermal conductivity probe. Hayakawa and Bakal (1973) used a rectangular cell to determine the thermal diffusivity values of several foods.

Since their first introduction by Ball (1923), the values of f_h and j_h have been used widely to predict food temperature by many researchers. These values represent the slope index and intercept coefficient respectively for a linear portion of the time-temperature curve of a heat penetration test for a food material. This has been the main area of interest with researchers using these values to determine, mathematically, the thermal diffusivity (Pflug et al., 1965; Bhowmik and Hayakawa, 1979; Uno and Hayakawa, 1980). The f_h parameter provides less variation than the use of the j_h which can cause considerable variation in the value of α (Bhowmik and Hayakawa, 1979). However, these approaches are more indirect than the technique of Dickerson (1965) which gives a more direct determination of thermal diffusivity for a food material.

In summary, four approaches are commonly used in determining thermal diffusivity: 1) least squares estimation; 2) use of heat penetration curves; 3) use of time-temperature charts; 4) use of analytical solutions (Singh, 1982). Most of these techniques have a time-temperature relationship basis for which the data are gathered during heating (unsteady or quasi-steady state) of known

configurations (slab or cylinder). An estimate of average diffusivity value over a range of temperature is achieved. Two other techniques are the probe technique (Nix et al., 1967; subsequently modified by Choi and Okos (1983) to study temperature dependence; and the peak-time method of Harmathy, 1971.

Mathematical models for food sterilisation and for deterioration of food quality can be valuable tools in process design and optimisation. Thus, the accurate prediction of temperature history in packaged foods during heating and cooling is of considerable importance for establishing safe and efficient thermal processes (Tung et al., 1989).

Thermal diffusivity, particulate size and shape are the three main factors that govern the heating rate of food in situations of high heat transfer (i.e., retorting). Thermal diffusivity reflects the ability of a substance to change temperature when heated or cooled. As mentioned earlier during the discussion of heat transfer, thermal diffusivity, coupled with the package form (i.e., cylinder, spherical, etc.) and the boundary conditions, make it possible to calculate the temperature history in a variety of process conditions. This makes thermal diffusivity of key interest in thermal processing determinations.

Alginate gels themselves are of considerable interest for their potential to serve as model food particles (Dallyn et al., 1977; Brown et al., 1984; Sastry et al., 1988; Knap, 1994). The ability to form a gel around a biological thermal indicator, for example, or to manipulate some of the parameters discussed above (moisture content, size, etc.) to simulate various sizes and types of food particulate in aseptic processes for example may be valuable attributes and overcome difficulties in manipulating actual food materials.

3.2 Materials and Methods:

Thermal diffusivity measurements were performed by a modification of the method of Dickerson (1965). This method involved the measuring of temperatures at the centre (T_c) and surface (T_s) of a food material packed in the cylindrical thermal diffusivity cell, which was then heated at a constant rate (B). The thermal diffusivity was calculated from the difference in temperature ($T_s - T_c$) after an initial temperature lag. Thermal diffusivity was assumed to be constant over narrow temperature ranges which were computed using Equation 3.1 (Dickerson, 1965; Tung et al., 1989)

$$\alpha = B R^2 / 4 (T_s - T_c) \quad (3.1)$$

In this study, the equipment layout was modified in that a constant-rate heating water bath was not used. Since the studies related to a Lagarde steam-air retort, advantage was made of the microprocessor controls (PAL 21, J. Lagarde, Montelimar, FRA) which apply a ramped heating rate to reach a final processing temperature. By considering the temperature differential from start to finish (60 to 118°C) a slope could be calculated and this was entered into the microprocessor which controlled the heating rate. The process was one of a long come-up with no holding time involved. Thus, in this way, thermal diffusivity tests could be carried out while a constant heating rate was maintained. This satisfied the requirement that $\delta T / \delta t$ was constant. In addition, since the rate of heat transfer through the alginate/potato gel is slow relative to the tube, the gel experiences the temperature as if the gel was directly exposed to the steam. Dickerson (1965) had worked this into the theory and subsequent calculations.

3.2.1 Potato/Alginate Gel Preparation:

Alginate was prepared by mixing 300 g of whole russet potatoes with 400 mL of distilled water and putting them together into a 401 x 400 (4 and 1/16th inches in diameter by 4 inches in height) three-piece metal can. The can was then hermetically sealed using an Angeles 60L closing machine (Angeles Corp., Anaheim, CA). The cans were then retorted for 45 minutes at 124°C. Cans were then cooled, opened and the contents were pureed for 5 minutes at room temperature using an Oster Classic 8 Blender (Sunbeam Corporation (Canada) Ltd., Hamilton, ON) on its highest speed setting. Sodium alginate (Grindsted Sobalg FD 170 Sodium Alginate, Solbalg S.A., Landerneau, FRA) was added to a level of 2.5% w/w. This mixture was blended in a Hamilton-Beach milkshake blender (Hamilton Beach, Div. of Scoville Mfg. Co., Racine, WI), and the mixture was placed to a depth of 1 cm in a 211 x 300 can (2 and 11/16th inches in diameter by 3 inches in height) as a mould, and frozen overnight (12 hours). The following day, the potato/alginate cylinders (at -18°C) were separated from the mould, still in a frozen state, and placed into a 4% w/w calcium chloride solution (Anachemia AC-1952P calcium chloride, Anachemia, Toronto, ON) for two hours at ambient temperature. This was followed by curing and thawing of the model food particles in sterile deionized water for at least 24 h at 4°C.

The thermal diffusivity tube (Figure 3.1) was constructed as per Dickerson (1965) and Sandberg (1991). The tube consisted of a cylindrical container of chromium-plated brass with an internal diameter of 0.0478 m and an internal void length (when the end caps are in place) of 0.2286 m. The tube was chromium-plated brass due to the combined requirements of rigidity and high thermal conductivity. The plating also offered excellent food-particle release characteristics without impairing heat transfer as well as offering protection from the corrosive effect of acidic

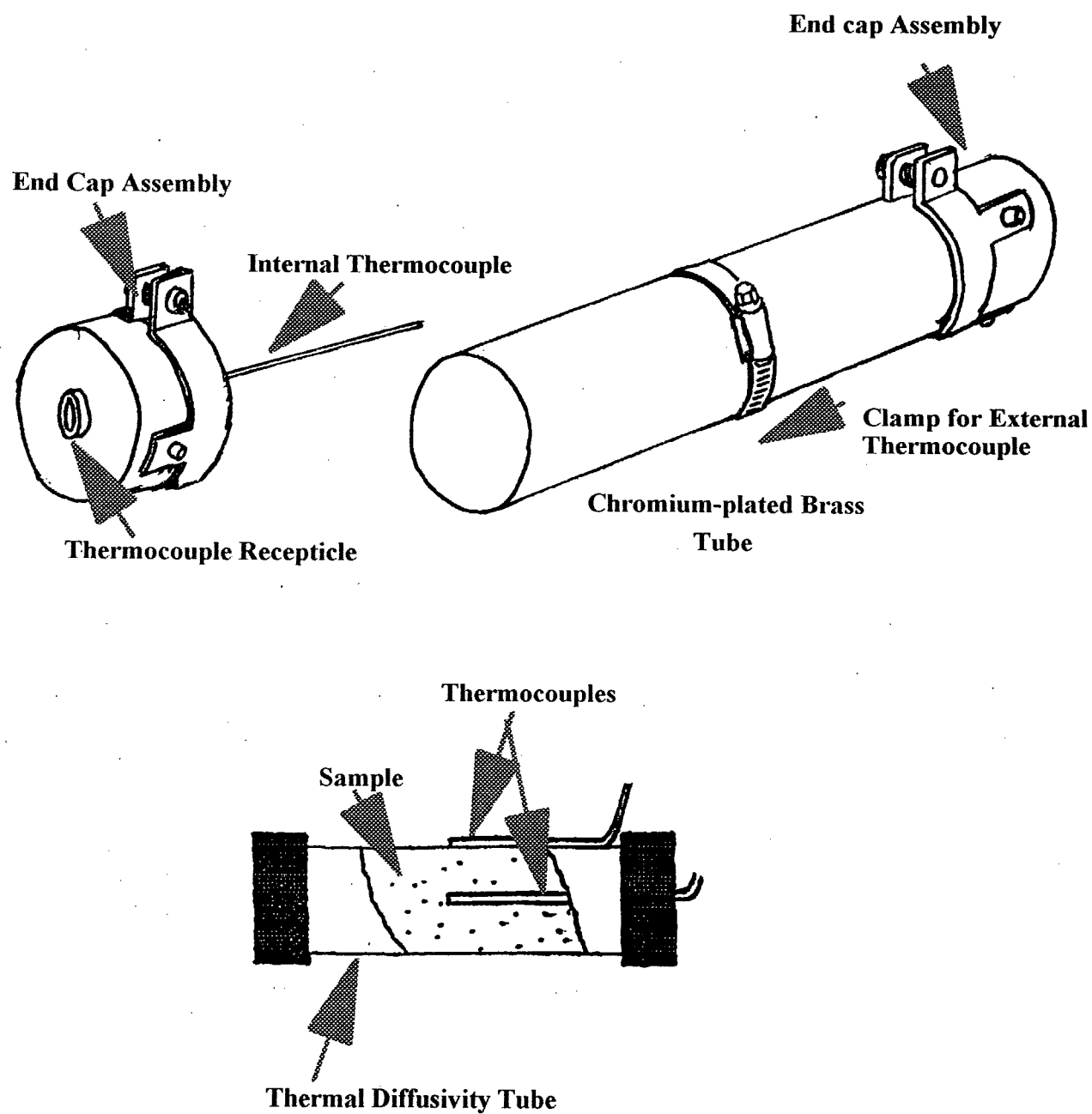


Figure 3.1. Assembly of thermal diffusivity tube.

foods (Dickerson, 1965). The end caps were constructed of Delrin plastic, which has a thermal diffusivity approximating that of food materials. A rubber "O-ring" was used to provide a hermetic seal on the internal ends of the caps and a clamp was attached to maintain the end caps in place during retorting. On one end cap, a hole was drilled and tapped to accommodate a thermocouple receptacle (Ecklund-Harrison Technologies, Fort Meyers, FL). A thermocouple (Ecklund-Harrison Technologies, Fort Meyers, FL) sufficient to reach the internal geometric centre of the tube when the end cap was in place was then inserted into the receptacle. Three thermocouples were clamped to the outer surface of the tube corresponding to the internal geometric centre of the internal void. The external thermocouples monitor the temperature of the sample at radius R (corresponding to the outside radius of the tube), while the central thermocouple monitors the temperature at the centre of the sample. Temperature readings were taken every 20 s.

Following gelation of the potato/alginate discs, the thermal diffusivity tube was used as a cookie-cutter to size the plug needed for the thermal diffusivity studies. Layers were built up in this way until the tube was 100% filled. The assembly procedure consisted of installing the bottom end cap and filling the void space with the potato/alginate gel. The cap with the thermocouple receptacle was placed on top and then the type T thermocouple was inserted to its full immersion depth to ensure that proper longitudinal and radial positioning was achieved.

Following the filling of the tube with the potato/alginate gel, the tube was then capped and the cell inserted into the retort. The thermocouple leads (teflon coated) were connected to a Calwest Model 32 datalogger (Calwest Technology, Canyon Country, CA). Data were stored on an IBM 720C Thinkpad portable computer (IBM Corp., Boca Raton, FL). The computer software used to collect, store and analyse the data was the CALSoft for Windows program from TechniCAL Inc. (New Orleans, LA). Further data analysis was conducted using Lotus 1-2-3

software (Lotus Development Corporation, Cambridge, MA).

Centre and surface temperature measurements were gathered at 20 second intervals as the cell was heated in the retort at a constant rate. Thermal diffusivity was then calculated for each measurement using Equation (3.1). Temperature data were smoothed using the least squares method for linear regression (Tung et al., 1989). This involved the regression of surface temperatures on time over the entire period to obtain the overall heating rate (which corresponded with that pre-determined by the Lagarde microprocessor) as well as localised regression of temperature on time over a narrow range of temperature (20s) consisting of a set of data pairs to derive the value of $T_s - T_c$. In all 241 data pairs were used to determine the thermal diffusivity over the temperature range previously noted. Smoothing was required as the heating of the Lagarde retort relies on alarm settings at the upper and lower limits of a 2°C range (see Appendix 2 for partial representation of raw data). Thus although steam flow was not constant, the overall effect of the microprocessor was to ensure a constant average heating rate.

3.3 Results and Discussion:

Dependence of thermal diffusivity on temperature has been well documented in the literature (Tung et al., 1989; Sandberg, 1991). The calculation used by Dickerson (1965) yielded an average thermal diffusivity across the environmental temperature range. The average thermal diffusivity for the potato/alginate gel used in this study was calculated as $2.08 \times 10^{-7} \text{ m}^2/\text{s}$ (Table 3.1). This compares favourably with the representative value of $1.7 \times 10^{-7} \text{ m}^2/\text{s}$ for food materials.

Rao, et al. (1975) reported thermal diffusivity values of $1.84 \times 10^{-7} \text{ m}^2/\text{s}$ for russet potatoes. Brown et al., (1984) found a mean thermal diffusivity value of $1.64 \times 10^{-7} \text{ m}^2/\text{s}$ with a standard

Table 3.1. Temperature dependence of thermal diffusivity of potato/alginate gel spheres.
 (Values are the average of 3 repetitions, coefficients of variation appear in brackets, temperatures are at the middle of the estimation range).

Temperature (°C)	Thermal Diffusivity ($\times 10^{-7} \text{ m}^2/\text{s}$)
60	2.16 (0.0)
65	2.13 (0.1)
70	2.11 (0.1)
75	2.10 (0.3)
80	2.07 (0.5)
85	2.06 (0.5)
90	2.04 (0.7)
95	2.02 (0.8)
100	2.00 (1.1)
105	1.98 (0.9)
110	1.96 (1.1)
115	1.95 (1.3)

deviation of $1.62 \times 10^{-8} \text{ m}^2/\text{s}$ for a potato/alginate blend similar to that used in this study. Sarikaya and Ozilgen (1991) found a thermal diffusivity value of $1.93 \times 10^{-7} \text{ m}^2/\text{s}$ for potatoes used in their study. Tung et al. (1989) noted that differences in moisture content and formulation had profound effects on thermal diffusivity for a given type of food and this may account for some of the variation in the literature values. However, the potato/alginate gel diffusivity values in this study suggest this material is a suitable material for a model food particle. In addition, no physical changes were noted to the alginate/potato gel. Table 3.1 shows the change in thermal diffusivity with increasing temperature. Tung et al. (1989) demonstrated the temperature dependence of thermal diffusivity and concluded that greater accuracy in the calculation of heat transfer processes which are based on thermal properties as input data (finite difference techniques for example) could be achieved if thermal diffusivity was considered as a function of temperature, rather than a constant value. Sandberg (1991) and Tung et al. (1989) have both stressed the importance of utilising this temperature dependence in the calculation of heat transfer processes which are based on thermal properties as input data for finite difference and other numerical techniques. The consideration of thermal diffusivity as a function of temperature could result in greater modelling accuracy. With the use of microprocessor-based retort control systems, the measurement of thermal diffusivity values across retort temperature ranges was quite simple. In addition, the ease of taking the measurements and the straightforward nature of data handling makes it possible to determine thermal diffusivities for specific products, thus enabling experimenters to rely less on approximate values from literature.

3.4 Conclusions:

With the advent of microprocessors to control retort processes and the use of programmable logic controllers (PLC), finer manipulations of temperature fluctuations can be achieved. In addition, ramping and holding at various stages in a thermal process are possible. With this in mind, the creation of a constant heating rate is easily achieved. This combined with the thermal diffusivity tube as outlined by Dickerson (1965) and Sandberg (1991) leads to a methodology whereby thermal diffusivity values can be determined for any food material with as little effort as is now commonly used for determining thermal processes. The method of data collection outlined by Dickerson (1965) coupled with the stepwise regression technique of Tung et al. (1989) create a powerful method whereby some of the thermal parameters required for modelling can be ascertained. In this study, the concern was to verify that the potato/alginate gel maintained its thermal characteristics at commercial sterilisation temperatures and as such modelling considerations were not a concern.

This ease of measurement and determination of thermal diffusivity values allows for modelling of food materials that may have changing thermal properties upon heating. One obvious example comes from mis-processes (a situation where a commercial sterilisation process designed achieve a certain specified lethality for a specified microorganism fails to do so) which may occur during the course of retort processes. If re-processing of a product is required, being able to determine the changed thermal diffusivity of a food material resulting from changed viscosity, for example, may lead to an ability to determine the degree of heat treatment required during the subsequent process and thus further enhance the food safety aspects.

Finally, the thermal diffusivity values determined for the potato/alginate gel show it to be

suitable as a material for model food particles. Sastry et al., (1988), discussed the importance of the thermal characteristics of model food particles for use in work with biological thermal indicators. Thermal diffusivity summarises these thermal characteristics and combined with the analysis technique discussed here to determine the suitability of potato/alginate gels as model food particles, also points to a method of evaluating the suitability of materials to serve as food modelling substrates.

CHAPTER 4

ROTATIONAL/POSITIONAL EFFECTS

4.1 Introduction:

In order to establish a safe and efficient thermal process, accurate prediction of the temperature history in packaged foods during heating and cooling is of considerable importance. World-wide, there has been a movement by the low-acid canned food industry from still retorts towards agitating or continuous agitating retorts for convection-heating products (Naveh et al., 1984). This has been ascribed to two reasons: 1) the growing demand for improved product quality and decreased nutrient losses and 2) the increase in institutional food service which requires larger containers. Induced convection results in a higher heat transfer rate (i.e., shorter process time) which provides one of the reasons for the trend from still retorts.

A number of difficulties arise when considering the required process time. One difficulty is in considering the type of convective forces that act inside the container. If a thermocouple is positioned traditionally at the geometric centre, and particles are moved to the outside of the container due to centrifugal forces, the traditional method of analysis may yield an over-estimation of process time as the fluid will be obeying convective forces, but the actual particle will reside in very close proximity to the wall. Britt (1993) noted that consideration of the

initial position of the headspace bubble together with the angle and radius of rotation showed that every container in the plane of the basket is located differently and thereby has the potential to heat and cool differently during a particular process.

The amount of heat exposure required for sterilisation is dependent upon five steps in the process of transfer of heat from the heating medium to the particulate as follows: (1) convective heat transfer from the environment to the container wall, (2) conduction through the container surface, (3) convection through the liquid phase, (4) convection and conduction from the liquid phase to the surface of the particle, and (5) conduction to the particle's centre (Heppel, 1985).

This can also be extended to placement of containers in end-over-end rotated baskets whereby containers located at the outside edge may be over-processed relative to products located at the geometric centre of the load. This may be due to increasing centrifugal forces acting from the centre to the outside edge and the increased forced convection effects thereon. In practice, however, gravitational forces always exceed centrifugal forces in all currently known end-over-end rotational retorts (based on size and rpm) (Britt, 1993).

This portion of the thesis considers observations on the movement of particles inside a container as well as the effects of container position in a retort basket.

4.2 Materials and Methods:

4.2.1 Visualisation of Particle Movement During Rotation:

For this study, a 307 OMNI® bowl was hermetically sealed on an Angeles40L closing machine. The designation of 307 is broken down as the first digit being the diameter in whole

inches. The second and third digits are the remainder of the diameter in 16^{ths} of an inch. Therefore 307 would be a diameter of 3 and 7/16 inches. The container was then cut transversely with an industrial bandsaw. Four alginate spheres, colour coded in order to differentiate them, were then placed inside the OMNI® bowl. This was deemed to be a sufficient number to show general patterns of particle movement, although commercial practices would involve greater numbers. A 5 mm thick clear plexiglas rectangle was then placed over the open container and sealed in place with silicone rubber (Dow Corning Inc., Midland, MI) (Figure 4.1). Since the containers were not be heated, and the medium used inside the container was water, there was no need to consider temperature gradients or resulting viscosity changes. All studies were done at ambient temperature.

Three bowls were placed into a bracket, (110 mm centre to centre). The bracket was then fixed diagonally to the outside of the retort basket (basket size: 300 (H) x 300 (W) x 600 mm (L)) of a Lagarde simulator retort (J. Lagarde, Montelimar, FRA), with the centre of the first bowl at the radial centre of basket. This was done to position the container at the outermost limit of the retort basket. The door was left open (Figure 4.2) and the retort was rotated at a speed of 2.8 rpm (the top speed allowed with the door open) and videotaped with a Samsung model SCX 800 videocamera (Samsung Electronics America, Inc., Ridgefield Park, NJ). The recorded tape was then played back and the pattern of sphere movement was evaluated.

4.2.2 Potato/Alginate Gel Particle Preparation:

The potato/alginate gel was prepared according to the method in Chapter 3, section 3.2.2. This mixture was then placed into a rubber mould for the generation of spheres of 18 mm

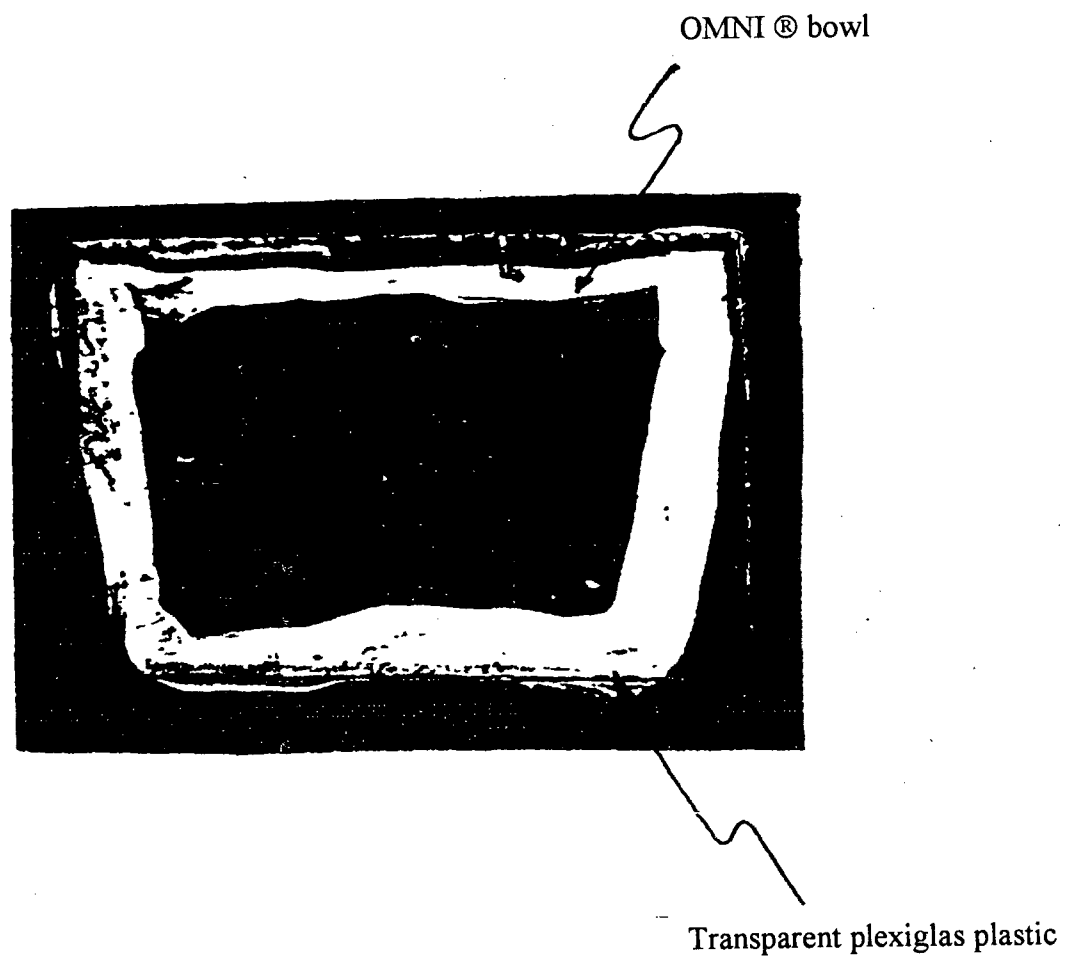


Figure 4.1 Structure of container used for visual recording of free particle movement in a 307 OMNI® bowl containing water inside the Lagarde simulator retort at 2.8 rpm.

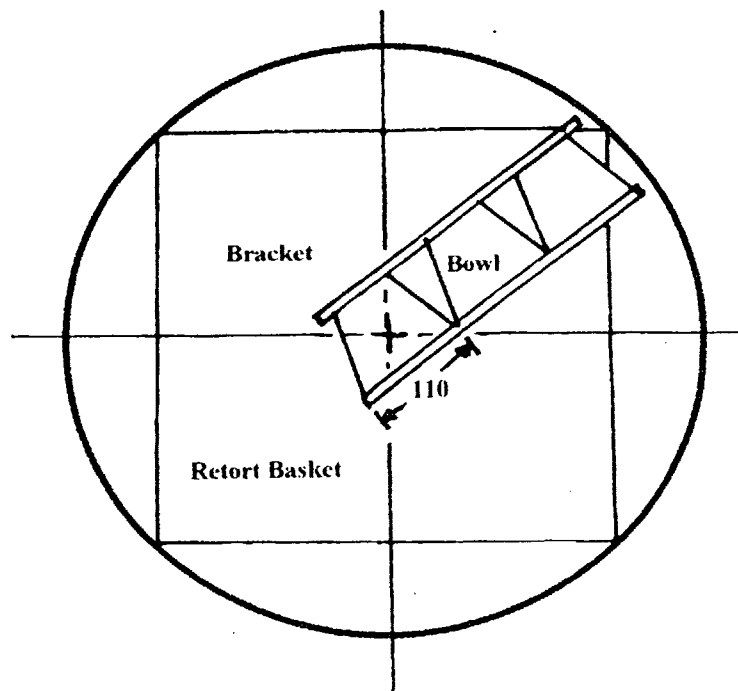


Figure 4.2. Orientation of bowls for videotape study of particle movement in Lagarde lab simulator retort at 2.8 rpm.

diameter and frozen overnight (12 hours). The following day, the frozen spheres were separated from the mould and placed into a 4% w/w anhydrous calcium chloride solution (Anachemia AC-1952P calcium chloride, Anachemia, Toronto, ON) for 2 hours at ambient temperature. This was followed by curing and thawing of the particles in sterile deionized water for at least 24 h at 4°C. The spheres were then spray-painted either yellow, silver, blue (using an enamel paint (General Motors of Canada Limited, Oshawa, ON)) or left uncoloured for the video study.

4.2.3 Positional Effects On Heating During Rotation:

Thermocouples were located at the geometric centre of ten 307 diameter OMNI bowls (American National Can, Chicago, IL). The containers were filled with water, taken to a minimal headspace, and hermetically sealed on an Angeles 40L steam-flow closing machine (Angeles Corp., Anaheim, CA). The containers were then placed in a 5-car Lagarde Steam-Air retort (J. Lagarde, Montelimar, FRA) (basket size 800 mm x 800 mm) and processed at 118°C for 45 minutes with various levels of rotation speed (0, 5, 10, 15 rpm, respectively). A ballast load of approximately 14,400 211 x 105 cans filled with water was used to fill the retort. Centre-point thermocouples were attached to a 32 channel slip-circuit assembly (Eklund-Harrison Technologies, Fort Meyers, FL) and from the slip-circuit assembly to a Calwest Model 32 datalogger (Calwest Technologies, Canyon Country, CA), connected in turn to an IBM Model 720C Thinkpad computer (IBM, Boca Raton, FL) equipped with the CALsoft for Windows software program for acquisition and analysis of heat penetration data (TechniCAL, New Orleans, LA).

Containers were processed until a lethality of $F_0 = 5.0$ minutes was achieved in the slowest-heating container. At this point, the cooling phase was initiated. Data were plotted and analysed

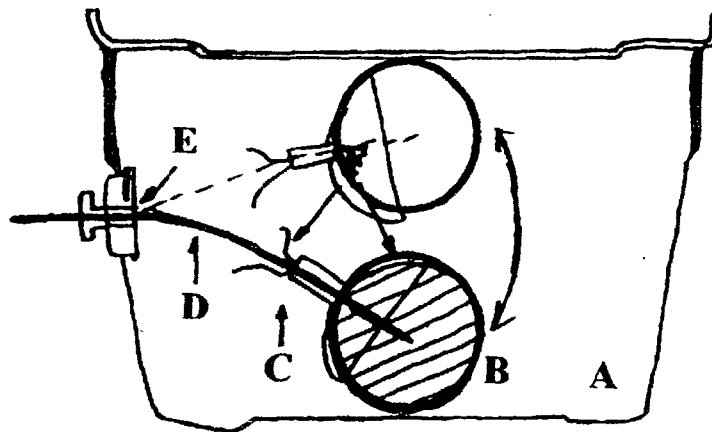


Figure 4.3. Structure and use of a flexible thermocouple mounted in a plastic container to monitor the centre temperature of a potato/alginate gel sphere. Legend: A) 307 OMNI® bowl, B) potato/alginate gel sphere, C) rubber tubing, D) thermocouple, E) mounting receptacle, F) dental floss.

via Ball's equation (Ball and Olsen, 1957).

The study was repeated with a flexible thermocouple (Figure 4.3) in an effort to more closely estimate the movement of a free particle, while still allowing for time-temperature measurements. The particle was free to move in an arc such that when the bowl was inverted, the sphere moved from the top to the bottom (as indicated by the dotted arc and sphere in Figure 4.3).

4.3 Results and Discussion:

4.3.1 Particle Motion During Rotation:

A review of the rotational pattern showed that the four particles moved about the outside edge of the bowl and never traversed the centre section (Figure 4.4). In other words, the particles kept in contact with the inner surface of the container.

Thus use of the traditional thermocouple with a particle located at the end, corresponding to the geometric centre would likely result in an under-estimation of required process time. Knap and Durance (1999) indicated that the practice of impaling a particle on a rigid thermocouple located at the geometric centre would lead to an under-estimation of the required process time versus a "free- ranging" particle. A particle located at the geometric centre achieves a greater lethality than a freely moving particle.

Studies by Anantheswaran and Rao (1985b) with 303 x 406 cans containing 0.75% guar gum suspensions showed that heating of the liquid closer to the container wall was faster than at the geometric centre. This was observed for 6.2 rpm. They explained that for highly viscous pseudoplastic liquids, the core moves like a plug. The movement of the headspace bubble and the

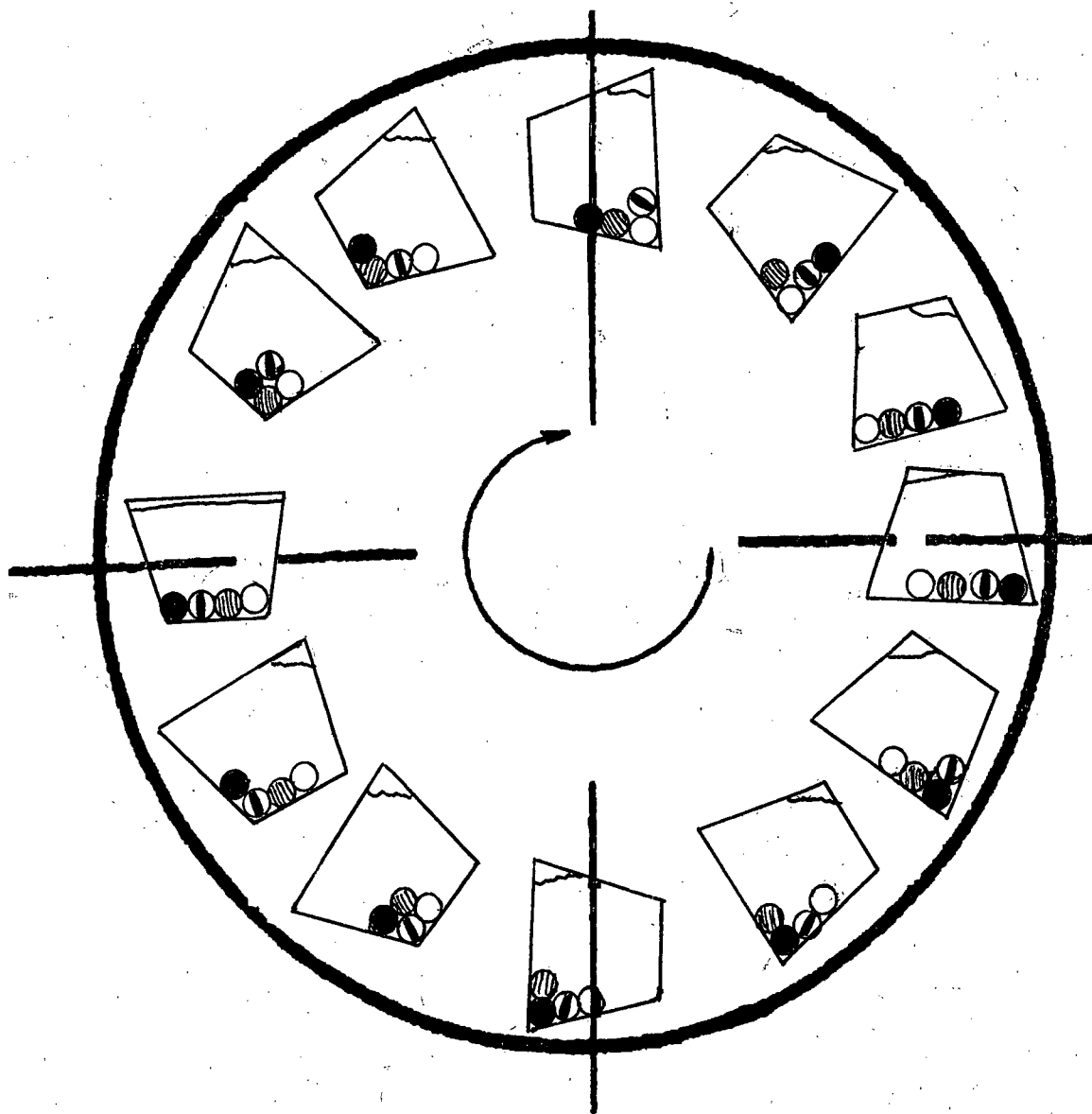


Figure 4.4 Movement of free particles at 2.8 rpm.

resulting agitation is confined to the wall of the can. End-over-end rotation of the can at low speeds does not, therefore, bring about any agitation of the core.

Further work by Anantheswaran and Rao (1985a) showed that the temperature at the centre of a can for water and sucrose solutions exhibited oscillations about an increasing average temperature. The frequency of oscillation increased with the speed of rotation. Further, the amplitude of the oscillation was directly proportional to the viscosity of the product and it decreased with the speed of rotation from a maximum at the beginning of the heating cycle. The amplitude also decreased as the temperature within the liquid increased. Further, the oscillations disappeared at temperatures close to the retort temperature. They put forward the theory that a highly viscous fluid moved through the container as an undisrupted mass which was not dispersed by container agitation so that heat transfer by conduction was the predominant factor in the process.

This presents a conundrum which may be explained by the fact that the Knap (1994) study as well as this one used water as the liquid phase (low viscosity). In the case of the visual observations made in this study, the rotational speed was relatively low (2.8 rpm) versus the speeds used in the Knap (1994) study (5 r.p.m. and greater). Thus it may hold, for low rotation speeds for both Newtonian and non-Newtonian liquids, that end-over-end rotation of the can at low speeds does not bring about any agitation of the core. This would suggest that there is a minimum threshold that must be crossed in order to maximise the movement of the headspace bubble such that agitation of the can's contents is brought about. Britt (1993) indicated that a thick viscous product may exhibit heating by conduction as the dominant form of heat transfer. Berry et al. (1985) also indicated in rotational studies with ravioli in brine that situating the ravioli in the geometric centre of the can on the end of a rigid thermocouple created a situation whereby the ravioli-thermocouple acted as a paddle. This influenced the agitation as well as the heating rate.

Although the combined effect on heating was not known, it indicated a substantial decrease in F_0 delivered at the centre of the ravioli compared with that measured in the brine.

The present study was repeated with a flexible thermocouple in an effort to more closely estimate the movement of the particle – even though the particle attached to the flexible thermocouple could not reach the corners of the container - while still allowing for time-temperature measurements as classically done. The particle was free to move in an arc such that when the bowl was inverted, the sphere moved from the top to the bottom (Figure 4.3). A thin gauge thermocouple wire, constructed of teflon coated 40-gauge wire (copper/constantan), with the ends soldered together, were located at the geometric centre of one 307 diameter OMNI® bowl (American National Can). The wire was fed through the container body using a flexible pouch connector (Figure 4.3). A small rubber washer was inserted and a tight connection was obtained by adjusting the tightness of the outer nut. Inside the container, the potato/alginate gel sphere (18 mm diameter) was connected to the thermocouple with a small rubber washer and a loop of dental floss. The sphere was impaled on the thermocouple wire to its geometric centre. A loop of dental floss was passed through the sphere and a small black washer used to apply tension in order to trap the sphere. The containers was filled with water and taken to a minimal headspace and the plexiglass siliconed in place.

4.3.2 Positional Effects of Containers Undergoing Rotation:

There appear to be regions within the retort where there are restrictions to the steam/air circulation. This supports the findings of Tung et al. (1989) who reported there was a large coefficient of variation for observed heating rate throughout the test car when using flow-restricting

package supporting racks in each of the four positions in a four-car retort. Their observed coefficients of variation were 19.2, 22.0, 19.1 and 2.63% respectively.

In this study, the test results (Table 4.1) were concentrated solely in the fifth car (of a five-car retort) which had been shown previously (Sandberg, 1990, unpublished data) to be the slowest heating zone. The general observation was that while some areas may seem to heat faster than others, ANOVA of the j_h and f_h values showed no significant differences to be evident.

4.4 Conclusions:

The use of visual observations alone in attempting to understand heating patterns is only of preliminary value in understanding the processes of heat transfer occurring in a container undergoing rotational processing. Factors mentioned earlier with respect to container configuration, product continuous phase, viscosity, particulate size, rotational speed, etc. all play a role in determining heating patterns. Further testing will be done to verify if the flexible thermocouple exhibits a time- temperature difference from the rigid thermocouple and subsequently if it is effective as a model of the free particles.

With respect to positional effects in the production Lagarde retort, while there are zones where the heating seems to be lower than the majority of the volume of the retort vessel, the positional effects are not of a significant nature, although the importance of establishing a thorough heat distribution pattern prior to the setting of a process of commercial sterilisation still cannot be ignored.

Table 4.1. Lagarde rotational study results for various thermal parameters in terms of Ball's process time and heating rates versus rpm and position (results are average of a minimum of three repetitions, coefficients of variation (%) are shown in brackets).

Thermal Parameter	rpm	Bowl Position				
		Centre 0 mm	282 mm	564 mm	847 mm	Outside 1131 mm
j_h	0	0.52 (0.0)	1.55 (11.0)	1.93 (17.0)	1.71 (7.6)	0.97 (23.7)
	5	0.96 (89.5)	2.48 (26.6)	2.30 (29.1)	1.57 (37.6)	0.58 (91.4)
	10	0.64 (26.6)	0.67 (7.5)	2.50 (32.8)	1.76 (27.3)	0.68 (17.6)
	15	2.05 (7.8)	3.33 (2.4)	3.82 (3.1)	2.76 (8.7)	0.56 (64.3)
f_h (min)	0	2.62 (3.8)	4.94 (6.9)	3.84 (5.2)	4.17 (2.6)	4.51 (4.0)
	5	3.57 (3.4)	3.69 (1.4)	4.14 (4.1)	4.64 (4.7)	3.54 (25.4)
	10	3.96 (2.5)	4.01 (1.5)	4.00 (2.8)	3.78 (4.5)	4.66 (6.7)
	15	3.93 (2.3)	3.22 (5.9)	4.06 (0.3)	3.68 (7.3)	3.33 (14.1)
* t_B (min)	0	16.12 (0.4)	20.85 (0.6)	19.27 (0.4)	19.83 (0.3)	19.41 (0.4)
	5	17.92 (0.5)	19.55 (0.5)	19.72 (0.2)	20.05 (0.2)	17.06 (0.4)
	10	17.79 (0.2)	18.62 (0.3)	19.48 (0.6)	20.08 (0.5)	16.82 (0.3)
	15	19.76 (0.3)	15.74 (0.2)	18.77 (1.1)	19.80 (0.3)	16.19 (0.7)
j_c	0	0.85 (2.4)	0.97 (17.5)	0.90 (4.4)	0.92 (32.6)	0.83 (0.0)
	5	1.08 (9.3)	0.90 (14.4)	0.92 (25.0)	0.94 (4.3)	0.72 (9.7)
	10	0.96 (9.3)	1.08 (21.3)	1.02 (1.0)	1.01 (5.0)	0.91 (9.9)
	15	1.07 (12.2)	1.64 (6.7)	0.87 (30.0)	1.07 (10.3)	0.68 (5.9)

*Note: assumptions were made that:

- 1) 40% of lag time contributes to lethality.
- 2) every point received an integrated lethal effect as great as that received at the cold spot.

CHAPTER 5:

RIGID VERSUS FLEXIBLE THERMOCOUPLES

5.1 Introduction:

The modelling of changes in the thermal properties of food products has brought about an increased demand for more accurate data. While the processes occurring in static retorts are better understood, the addition of rotational or forced agitation processes have added much more complexity as factors such as product viscosity, container headspace and rotation speed become much more important.

Stoforos and Merson (1990) studied the motion of spherical particles in axially rotating cans and characterised the relative particle-to-fluid velocity under idealised conditions of particle motion in rotating cans. Results of their flow visualisation study also revealed that particle motion was much more complicated than idealised in mathematical analyses. Ramaswamy and Sablani (1996) also found variations in particle motion which related to density of the particle as well as medium viscosity in the product container and rotation speed in the retort vessel.

The purpose of this portion of the study was to ascertain the suitability of the flexible thermocouple versus the traditional rigid thermocouple for gathering temperature history data at the centrepoin^ts of spherical particles suspended in packages filled with water.

5.2 Materials and Methods:

5.2.1 Rigid Thermocouple Heat Penetration Studies:

Needle-type thermocouples (44.5 mm in length) (Eklund-Harrison Technologies, Fort Meyers, FL) were located at the geometric centre of six 307 diameter OMNI® containers (American National Can, Chicago, IL). An 18mm potato/alginate gel sphere was then placed on the end of the thermocouple (Figure 5.1a). The six containers plus a sufficient number to provide a ballast load, were filled with water and taken to a minimal headspace and hermetically sealed on an Angeles 40L steam-flow closing machine. The containers were then placed in a lab-sized Lagarde Simulateur Rotatif 0600 1 panier (in english: basket) steam-air (96% steam/4% air) retort (JR Lagarde Inc., Montelinar, France) and processed at 118°C for 45 minutes with various rotational speeds (0, 5, 10, 15, 20 and 25 rpm). Three containers were located on a diagonal, in duplicate, 110 mm apart on the axial line. Centrepoint thermocouples were attached to a Calwest Model 32 datalogger, connected in turn to an IBM Model 720C Thinkpad computer equipped with the Calsoft for Windows software program for acquisition and analysis of heat penetration data.

Containers were processed to a lethality of $F_0 = 5.0$ min. At this point, the cooling phase was initiated. The data were plotted and analysed via the method of Ball (Ball and Olson, 1957).

5.2.2 Flexible Thermocouple Heat Penetration Studies:

Containers were prepared and located as for the rigid thermocouple heat penetration studies (Section 5.2.1). Thermocouples, constructed of 40-gauge wire (copper/constantan), with the ends

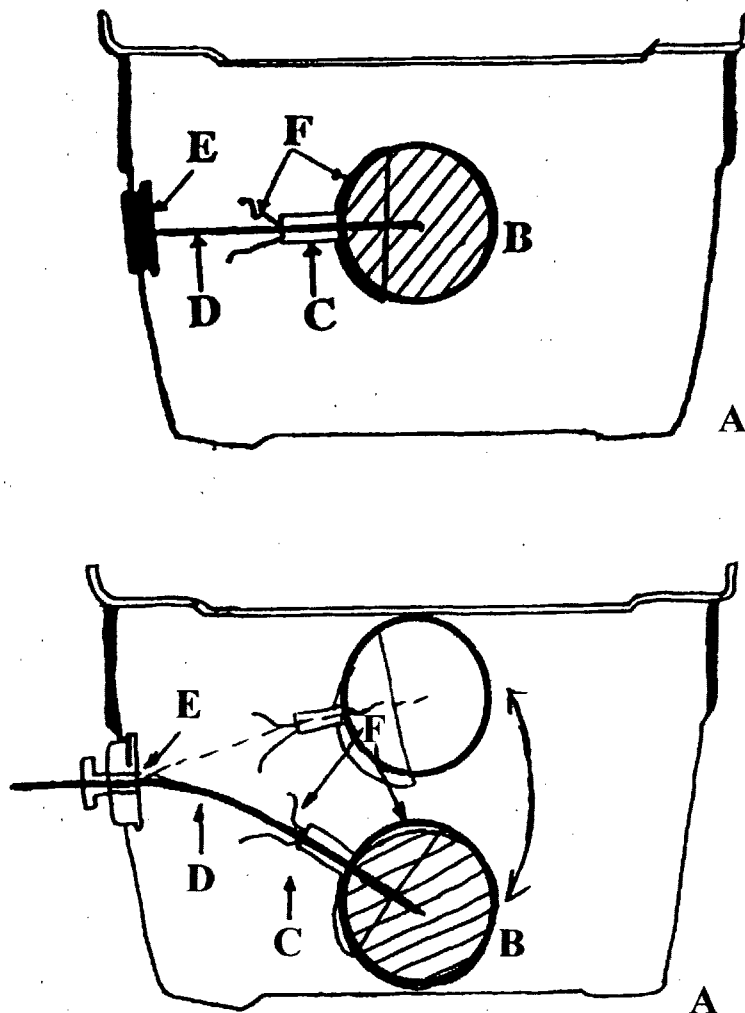


Figure 5.1 Rigid versus flexible thermocouples. Legend: A) 307 OMNI® bowl, B) potato/alginate sphere, C) rubber tubing, D) thermocouple, E) mounting receptacle, F) dental floss.

soldered together, were located at the geometric centre of six 307 diameter OMNI® bowls (American National Can). Wires were fed through the container body using a flexible pouch connector (Figure 5.1 a). A small rubber washer was inserted and a tight connection was obtained by adjusting the tightness of the outer nut. Inside the container, the potato/alginate gel sphere (18 mm diameter) was connected to the thermocouple with a small rubber washer and a loop of dental floss. The sphere was impaled on the thermocouple wire to its geometric centre. A loop of dental floss was passed through the sphere and a small black washer used to apply tension in order to trap the sphere (Figure 5.1 b). Containers were filled with water and taken to a minimal headspace (2 mm or less) and hermetically sealed on an Angeles 40L steam-flow closing machine. The sealed containers were placed in the small Lagarde Steam-Air (simulator) with a ballast load and processed for approximately 45 minutes at 118°C with various rotation speeds (0, 5, 10, 15, 20, and 25 rpm). The containers were located on a diagonal, 110 mm apart on the radial line. Centrepont thermocouples were attached to a Calwest Model 32 datalogger, connected in turn to an IBM Model 720C Thinkpad computer, equipped with the CALsoft for Windows software program for acquisition and analysis of heat penetration data.

Containers were processed at 118°C until a minimum lethality of $F_0 = 5.0$ min. was achieved in all of the test containers. At this point, the cooling phase was initiated. No correction was done for the effect of the thermocouple (Ecklund, 1956) as only 40 gauge wire and Delrin plastic fittings (Eklund-Harrison Technologies, Fort Meyers, FL) through the container wall were used. The data were plotted and analysed via the method of Ball (Ball and Olson, 1957).

5.3 Results and Discussion:

Rigid heat penetration results gathered from use of rigid thermocouples showed the same heating profile as observed in the production-size Lagarde retort. Results for the rigid thermocouples (Table 5.1) indicated the same pattern previously observed in that both j_h and f_h decreased as rotation speed increased. Process time (t_B) to achieve a target $F_0 = 5.0$ min also decreased. This trend, however, changed at 25 rpm where process time increased (Table 5.1).

A similar pattern was observed for the results achieved with the flexible thermocouple (Figure 5.1b). Again, as rotation speed increased, in general both j_h and f_h decreased. There was also a decreasing trend in process time (t_B) to achieve a target $F_0 = 5.0$ min (Table 5.2).

Analysis of Variance (95% confidence interval) (Table 5.3) indicated that for j_h and f_h , whether rigid or flexible thermocouples were considered, the effect of both rpm and radius was statistically significant as well as the interactions. However, when t_B was considered, the effect of rpm and radius were significant for both flexible and rigid thermocouples but the interaction of the flexible thermocouple and radius and rpm was not. Similarly, the interaction of the rigid thermocouple and radius and rpm was also not significant.

5.4 Conclusions:

The main conclusion, based on f_h , is that the flexible thermocouple gives a different temperature history of the particle than the temperature history measured for the rigid thermocouple. This may be of importance as products manufactured in this type of container are typically "high-end" or of greater value to the consumer. Therefore, minimisation of degradation

Table 5.1 Process parameters for rotational process in Lagarde simulator (rigid thermocouple) (results are the average of a minimum of 4 replicates, coefficients of variation are in brackets).

Parameter	rpm	Thermocouple Location		
		Centre 0 mm	Middle 115 mm	Outside 175 mm
j_h	0	3.35 (12.5)	1.52 (31.6)	1.86 (13.4)
	5	2.27 (11.5)	1.14 (29.1)	1.48 (17.6)
	10	1.54 (13.6)	0.73 (71.2)	0.84 (45.2)
	15	2.06 (22.8)	1.85 (7.6)	1.64 (9.2)
	20	1.40 (35.7)	0.72 (26.4)	0.50 (40.0)
	25	1.92 (26.6)	1.12 (17.9)	1.54 (9.7)
f_h	0	4.93 (3.5)	8.28 (2.5)	9.80 (0.7)
	5	4.75 (11.6)	7.98 (2.8)	9.45 (1.4)
	10	4.56 (7.7)	7.66 (4.3)	9.06 (4.1)
	15	4.37 (5.0)	4.36 (6.2)	3.37 (8.0)
	20	4.19 (2.2)	6.70 (3.1)	5.64 (3.0)
	25	3.59 (7.8)	5.11 (4.9)	4.54 (4.6)
t_B	0	23.1 (0.4)	25.8 (0.3)	25.6 (0.3)
	5	22.5 (0.5)	24.5 (0.3)	24.3 (0.3)
	10	22.0 (0.4)	24.3 (0.3)	24.1 (0.4)
	15	22.2 (0.6)	22.0 (0.3)	20.0 (0.4)
	20	21.6 (0.5)	24.8 (0.2)	18.7 (0.4)
	25	25.4 (0.4)	26.0 (0.3)	23.0 (0.4)

Table 5.2 Process parameters for rotational process in Lagarde simulator (flexible thermocouple) (results are average of a minimum 4 repetitions, coefficients of variation are in brackets).

Parameter	rpm	Thermocouple Location		
		Centre 0 mm	Middle 115 mm	Outside 175 mm
J_h	0	1.97 (21.3)	0.81 (57.1)	0.74 (63.5)
	5	2.77 (15.2)	1.48 (17.6)	1.22 (21.3)
	10	3.03 (7.7)	2.79 (7.5)	5.99 (1.2)
	15	1.32 (50.8)	3.59 (10.0)	2.03 (31.0)
	20	0.85 (62.4)	2.28 (29.0)	1.54 (35.7)
	25	0.89 (28.1)	1.16 (40.5)	0.54 (88.9)
f_h	0	4.13 (3.2)	7.85 (3.2)	6.63 (3.8)
	5	2.84 (4.6)	2.47 (6.5)	4.38 (3.7)
	10	2.77 (16.3)	3.51 (2.6)	2.97 (4.7)
	15	4.68 (1.3)	3.09 (2.3)	4.56 (1.3)
	20	1.75 (3.4)	4.17 (7.4)	3.25 (3.4)
	25	4.01 (1.5)	5.53 (0.5)	5.64 (17.6)
t_B	0	21.6 (0.3)	22.5 (0.4)	20.2 (0.5)
	5	20.1 (0.1)	22.0 (0.2)	21.0 (0.2)
	10	21.0 (0.6)	21.7 (0.1)	20.9 (0.2)
	15	20.2 (0.2)	20.3 (0.2)	19.8 (0.8)
	20	19.7 (0.4)	21.5 (0.4)	19.2 (0.3)
	25	20.7 (0.2)	23.6 (0.3)	20.7 (0.4)

Table 5.3. Analysis of variance (ANOVA) overall for rigid (R) versus flexible thermocouple (F) data presented in Tables 5.1 and 5.2. (Rad = radius).

Dependent Variable	Source	Sum-of-Squares	df	Mean-Square	F-Ratio	P
j_h	R or F	1.846	1	1.846	21.526	0.000
	rpm	9.585	4	2.396	27.945	0.000
	Rad	2.382	2	1.191	13.891	0.000
	R or F x rpm	9.082	4	2.271	26.479	0.000
	R or F x Rad	0.897	2	0.439	5.123	0.009
	rpm x Rad	5.325	8	0.666	7.762	0.000
	R or F x rpm x Rad	2.421	8	0.303	3.529	0.002
	Error	5.145	60	0.086		
f_h	R or F	72.686	1	72.686	162.776	0.000
	rpm	35.718	4	8.930	19.997	0.000
	Rad	40.199	2	20.099	45.011	0.000
	R or F x rpm	53.661	4	13.415	30.043	0.000
	R or F x Rad	10.559	2	5.279	11.823	0.000
	rpm x Rad	23.646	8	2.956	6.619	0.000
	R or F x rpm x Rad	18.679	8	2.335	5.229	0.000
	Error	26.792	60	0.447		
t_B	R or F	54.242	1	54.242	14.922	0.000
	rpm	51.079	4	12.770	3.513	0.012
	Rad	61.659	2	30.829	8.481	0.001
	R or F x rpm	31.921	4	7.980	2.195	0.080
	R or F x Rad	6.367	2	3.184	0.876	0.422
	rpm x Rad	47.458	8	5.932	1.632	0.135
	R or F x rpm x Rad	24.073	8	3.009	0.828	0.582
	Error	218.097	60	3.635		

to various quality or nutritional attributes becomes very important. The results of the statistical analysis indicate that there is an effect of rotational speed on the thermal parameters calculated, however, it appears to be overshadowed by the effect of position as evidenced in the large Lagarde retort. Britt (1993) commented on the effect of orientation with the resulting differing angles and radii of rotation.

One interesting observation concerns the values of f_h observed for the data collected for the flexible thermocouple. It is interesting to note that those values were lower than for the rigid thermocouple. One possible explanation is that, as Berry et al. (1985) mentioned in their rotational studies, the ravioli under rotation acted as a paddle and served to influence the agitation as well as the heating rate. In this scenario, a similar event may have happened. With the Newtonian system present in this study, the actions of a relatively heavy particle on the end of a light-gauge wire would be similar to the paddle-structure envisioned by Berry et al. (1985). In fact, under increasing rpm and the gravitational forces, the effects on heat transfer in this experiment would be even more pronounced. A system of greater viscosity may not show this pattern.

Parchomchuk (1977) studied the effects of rotational speed, viscosity and headspace on processing times and reported decreased process times to 40 rpm, relatively constant process times to 80 rpm then increasing process times to 144 rpm. These were consistent with observations of Clifcorn et al. (1950) with respect to gravitational and centrifugal forces and their influence on the fluid within a container. It should be noted however that there are no commercial processes that utilise these rotational speeds and as such at lower rotational speeds, gravitational forces will have a greater influence than centrifugal forces (Britt, 1993).

The use of the flexible thermocouple to monitor centre temperatures of spheres in water-filled containers showed overall lower values for the thermal parameters of j_h , f_h and t_B . This suggests

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that the forces being exerted on the container - both centrifugal and gravitational - also acted on the thermocouple. Therefore rotational speed cannot be considered entirely in isolation. On the other hand the flexible thermocouple does provide evidence of shorter and perhaps more realistic process times than the rigid thermocouple. Following from this, the next step would be to employ a thermal indicator and to again compare the rigid versus flexible thermocouple.

CHAPTER 6:

THERMAL INDICATORS: RETORT PROCESSES

6.1 Introduction:

Movement of containers in an agitating retort makes it difficult to measure temperatures of food materials directly during the commercial application of the thermal process (Berry et al., 1986). Yawger (1978) developed a microbial method of process determination through the spore count reduction method and this is frequently used to establish or verify a thermal process.

Pflug et al. (1980) observed that in comparing F_0 values calculated from time-temperature data with F_0 values determined by biological indicator units, a greater degree of accuracy could be expected from data calculated from time-temperature data than from biological indicators. The rationale given was that time-temperature data measurements were being made directly. The measurements were made indirectly with the biological indicator units. Thus, the biological indicator system exhibits the errors that might be present in the thermocouple temperature-measuring system which is used to calibrate the biological indicators, plus all of the additional variation which may be due to the biological measuring system and its sensitivity to a great number of uncontrolled environmental factors as outlined in the introduction.

Houtzer and Hill (1977) reported, in a comparison of the two sterilisation values made for food products, values of integrated sterilisation (IS) that were nearly 5 min. higher than F_0 . Berry et al., (1986) indicated a difference of 2 min. between IS and F_0 . Generally IS has been found to be

greater than F_0 for the same thermal process.

However, increased industry interest in agitated processes to reduce process times and thus improve quality and decrease energy consumption as well as interest in the aseptic processing of particulate foods has led to increased interest in process design to ensure commercial sterility of the end product. Since particles suspended in carrier fluids experience significant thermal lags within heat exchangers, the sizing of holding tubes becomes critical in aseptic processing (Sastry, 1986). A useful approach to the design of aseptic processes is through the use of mathematical models, nevertheless, it is important to verify thermal processes by microbiological testing.

In order to accomplish this objective, Sastry et al., 1988, identified a number of characteristics that an indicator must possess in order to offer reliable microbiological testing. The indicator must contain heat resistant bacterial spores that experience temperature histories similar to those in the cold spots of real particles. Therefore, according to Sastry et al., 1988, the bioindicator should be in the form of a particle, possessing at least the following as necessary and/or desirable characteristics: (1) large size (about 2.54 cm), containing the immobilized bacterial spores throughout the interior, and especially at the slowest heating zones; (2) geometry, thermal properties and responses similar to real food particles; (3) easy recovery and visual distinguishability from real particles; (4) retention of spores through all process steps, without leakage; (5) shelf-stability, permitting easy reconstitution and use whenever desired – this is more desirable rather than necessary; and (6) physical durability to be able to withstand process stresses without disintegration.

Sastry et al., 1988, reported that their alginate gel particles were sufficient to offer negligible spore leakage, had similar thermal response characteristics to real particles of similar product and showed expected decreasing population trends with increasing process time. They concluded

that the bioindicator had potential as a useful indicator of particle sterility although they stated more extensive microbiology was necessary for further validation.

In this study, such a particle system for agitated retort processes was used to compare spore count reductions for rigid particles (as impaled on a thermocouple), a flexible thermocouple to simulate a free-moving particle and lastly as free particles in a container. One further difference from the study presented in Chapter 5 concerns the addition of six particles to the container along with the one impaled on a thermocouple. The purpose being to see if the effect observed in Chapter 5 was still evidenced. Thermal diffusivity values determined earlier indicate that the potato/alginate gel serves as a suitable model for a food particle.

6.2 Materials and Methods:

6.2.1 Bacterial Spore Preparation and Enumeration:

Populations of *Bacillus stearothermophilus* ATCC 7953 spores were produced using a modified method of Kim and Naylor (1966) with subsequent modifications by Knap and Durance (1999). Pre-sporulation inoculum was grown in Tryptone Yeast Glucose Broth (TYG broth) with the following composition: 1% tryptone (Difco, Franklin Lakes, NJ), 0.5% yeast extract (Difco, Nepean, ON), 0.5% glucose (Fisher), 0.2% K₂HPO₄ (Fisher). The sporulation medium, Tryptone Yeast Agar (TYG agar) contained: 0.8% nutrient broth (Difco), 0.4% yeast extract (Difco), 10 ppm MnCl₂•4H₂O (Fisher), 3.0% agar (Difco). Medium pH was adjusted to 7.2 ± 0.1 prior to sterilisation. Cooled sporulation media was poured into disposable petri dishes (Fisher), allowed to solidify over a period of 45 min in a laminar flow cabinet and stored at 4°C prior to use. On the

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basis of initial experiments, moisture retention in the medium was improved at incubation temperature (55°C) with the use of 3% agar in the sporulation medium rather than the recommended 2%. This also facilitated washing the spores off the agar surface.

Preparation of pre-sporulation inoculum was divided into two parts. Freeze dried spores were initially reconstituted by adding TYG broth (0.3 to 0.4 mL) to the vial and allowing a few minutes for spore rehydration. The entire contents of the vial were transferred to a 20 x 150 mm test tube containing 5 mL of TYG broth and mixed well. Approximately 5 mL of the rehydrated spore culture was transferred into a 125 mL Erlenmeyer flask containing 15 mL of TYG broth and incubated in a single-speed agitated water bath (Blue M) at 55°C for 12-24 h. This step served to rejuvenate the culture and increase the microbial numbers. A number of TYG agar slants were inoculated with the culture at this point and incubated at 55°C for 48 h and were subsequently stored at 4°C to serve as back-up cultures.

In the second part of the production, the pre-sporulation inoculum used 10mL of culture from the first part above. This was transferred into a 2000 mL Erlenmeyer flask containing 150 mL of fresh TYG broth which was incubated with agitation in a single-speed agitated water bath (Blue M) at 55°C for an additional 16-18 h. The final step involved inoculation of sporulation agar (TYG) plates with approximately 1 mL of pre-sporulation suspension followed by incubation at 55 °C (Blue M, Gravity Convection Bacteriological Incubator) for 8-10 h in the upright position to allow for inoculum absorption by the agar. Plates were inverted to reduce further dehydration and incubated for an additional 36-38 h.

At the end of the incubation period (≥ 48 h), the presence of sporulation was checked by microscopic examination of glass slides with stained (Shaeffer-Fulton method) spore preparations. Surface growth from petri plates was then washed off with ice cold 0.1 M phosphate buffer (pH

6.5) into sterilised 250 mL Nalgene bottles. Spores were concentrated by centrifugation (Sorvall, RC2-B centrifuge) at $5000 \times g$ for 15 min at 4°C . Supernatant liquid was discarded, the spore sediment was re-suspended in an equal amount of buffer, and then re-centrifuged. The washed spore pellets were transferred to a 500 mL Erlenmeyer flask by re-suspending them in 200 mL of 0.1 M phosphate buffer containing lysozyme (Sigma, 100 ug/mL), and were incubated with agitation for 1 h (Freeherry et al., 1987). The addition of lysozyme served to rupture remaining vegetative cells allowing spore release. The spore stock solution was then, as previously described, centrifuged and washed 5 more times with equal volumes of ice cold buffer. In the final step, spore pellets were pooled and re-suspended in 50 mL of buffer. The spore stock solution was then subdivided into 10 mL aliquots and stored in screw-top test tubes at 4°C until needed. Initial enumeration indicated that the stock spore suspension contained 6.5×10^7 cfu/mL.

6.2.2 Spore Enumeration

Throughout the TDT study and thermal process validation experiments, viable spores were enumerated using the Hydrophobic Grid Membrane (HGM) filtration method (QA Life Sciences, Inc., San Diego, CA). Aliquots (1 mL) of appropriate spore dilutions were filtered through ISO-grid membranes, with the filters subsequently incubated for 48 ± 4 h at 55°C on TSA agar (BBL) in the lids down position. Colonies of *Bacillus stearothermophilus* ATCC 7953 grown on ISO-grid membranes on TSA medium were lightly transparent in appearance and thus difficult to enumerate. To improve colony visibility, Fast Green FCF (Fisher) dye was incorporated into TSA at a level of 0.25 g/L (Entis and Bolesczuk, 1986). For all dilutions, sterile 0.1% peptone (Difco) water was employed. Initial (zero time) counts of spore suspensions, were obtained from appropriate

dilutions of 1 mL spore aliquots which were heat shocked (5 min @ 100°C) in 1 mL glass capillary tubes, sealed as per Stern and Proctor, 1954.

6.2.3 Spore Thermal Resistance

The capillary tube method (Stern and Proctor, 1954) was employed to determine the thermal resistance of a *Bacillus stearothermophilus* ATCC 7953 spore population suspended in 0.1 M phosphate buffer (pH 6.5). Glass capillary tubes (Fisher) were sterilised in dry heat for 180 min at 121°C prior to use. Disposable 1.0 mL syringes fitted with 25 gauge needles were used to fill each tube (100 mm length, 1.9 mm O.D.) with 0.1 mL of spore suspension. Filled capillary tubes were sealed with an oxygen/natural gas flame and stored at 4°C prior to use.

Tubes were heated in a constant temperature oil bath (Blue M, Magic Whirl), with temperature monitored by a digital thermometer, to $119.5^{\circ}\text{C} \pm 0.2^{\circ}\text{C}$. Tubes were removed at 5 min intervals to a maximum of 30 min. For each interval, 3 individual capillary tubes were evaluated. Heating times were corrected with a come-up time of 15 s reported for the capillary tubes (Brown et al., 1984). At the end of each heating time, capillary tubes were removed from the oil bath and placed in an ice water bath to arrest further thermal inactivation. Prior to spore enumeration, capillary tubes were individually cleaned of the remaining heating oil by washing their external surfaces with detergent solution (liquid soap and water) followed by 70% alcohol. Spore aliquots were removed from capillary tubes by snipping one end of the tube with a pair of needle-nosed pliers, placing the capillary tube into a dilution test tube, then breaking off the other end to allow the contents to flow out. To ensure complete spore removal from the capillaries, each tube was

flushed with 1.0 mL of diluent (0.1% peptone) introduced by a 1.0 mL disposable syringe fitted with a 25 gauge needle. Viable spore counts were obtained using the HGM filtration method, as previously described. From surviving spore counts, $D_{121.1}$ and z values for the spore crop were determined.

6.2.4 Potato/Alginate Gel Particle Preparation:

Potato/Alginate gel spheres were prepared as outlined in Chapter 3.

6.2.5 Retort Treatment of Thermal Indicators (Rigid Thermocouple, Flexible Thermocouple and Free Particles):

Evaluation of the effect on heat transfer of immobilisation of a food particle on a rigid or flexible thermocouple was done using the method of Knap (1994) as modified from Ramaswamy and Ghazala (1990). Bacterial spore suspension was encapsulated in hermetic anodised aluminum DSC pans (I.D. 6.6 mm) (Westech Industries Ltd., Calgary, AB) at a volume of 0.02 mL per pan, and suspended in alginate particles as prepared in Chapter 3. Thermocouples were located at the geometric centre of six 307 diameter OMNI® bowls. Six additional particles, also containing DSC pans with bacterial spore suspension were placed into the bowl. Thermocouples were constructed of 40 gauge wire (copper/constantan), with the ends soldered together. The wires were fed through the container body using a flexible pouch connector (Eklund-Harrison Technologies, Ft Meyers, FL) (Figure 6.1). A small rubber washer required for the flexible pouch connector (Eklund-Harrison Technologies) was inserted and a tight connection was obtained by adjusting the tightness of the outer nut. Inside the container, the alginate gel sphere was connected to the thermocouple

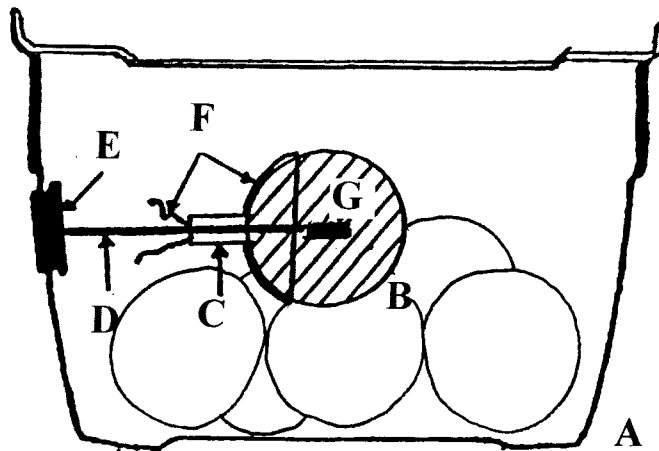


Figure 6.1 Particle and thermocouple structure for thermal indicator studies – rigid thermocouple.

Legend: A) 307 OMNI® bowl, B) potato/alginate gel particle, C) rubber tubing, D) thermocouple, E) mounting receptacle, F) dental floss, G) DSC pan.

with a small rubber washer (2 mm diameter, 3 mm length) and a loop of dental floss. The sphere was impaled to its geometric centre on the thermocouple wire. A loop of dental floss was passed through the sphere and a small rubber washer used to apply tension in order to trap the sphere (Figure 6.2). The OMNI® containers were filled with water and taken to a minimal headspace (2 mm or less) and hermetically sealed with an Angeles 40L steam-flow closing machine (Angeles Corp., Anaheim, CA). The filled, sealed containers were then placed in a small (simulator) Lagarde Steam-Air retort and processed at 118°C for 45 minutes with various rotation speeds (0, 15, and 30 rpm, respectively). The containers were located at radial positions on a diagonal at 0, 115 and 175 mm on the axial line. Following processing, thermal indicator samples were recovered and assayed for residual activity as outlined above.

"Free" spheres with thermal indicator pans, were prepared as above, however, they were allowed to circulate freely, in the water, in the container, again at varying rotation speeds (0, 15 and 30 rpm). The residual activity of the thermal indicators was then determined as outlined above.

6.2.6 Spore Count Reduction Method:

The spore count reduction method of Yawger (1978) was used to determine the integrated sterilisation value (IS) of the DSC pan in the model food particles and the surrounding fluid following the thermal process. This concept is based on the following equation:

$$IS = D_r (\log I - \log S) \quad (6.1)$$

Where IS = integrated sterilising value of the DSC pan

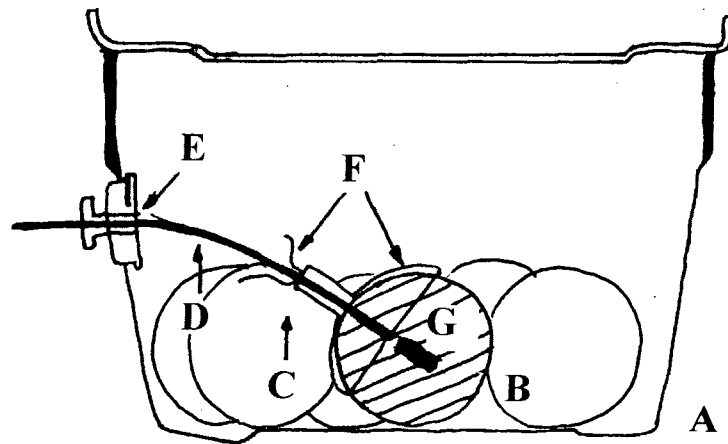


Figure 6.2 Particle and thermocouple structure for thermal indicator studies - flexible thermocouple. Legend: A) 307 OMNI® bowl, B) potato/alginate gel particle, C) rubber tubing, D) thermocouple, E) mounting receptacle, F) dental floss, G)DSC pan.

- D_r = 90% spore reduction time at 121.1°C
 I = initial count
 S = survivor count

For the purpose of this study, the IS value for the particle was modified in that the sterilising value was not integrated over the entire volume of the particle, but only of the DSC pan. One must still account for the volume of the pan (0.02 mL) as this volume prevents the DSC pan from being a centrepoin measurement of lethality. Thus for the pan, the IS was designated IS_{pan} .

6.3 Results and Discussion:

From the survivor curve, the decimal reduction value for *Bacillus stearothermophilus* ATCC 7953 at 121.1°C was determined to be 2.5 min. This was within the range previously reported in the literature by Stumbo (1973). Lund (1975) noted that D values are dependent on the strain chosen as well as inferring that pH was a factor.

The results shown in Table 6.1 indicate that the integrated sterilisation (IS_{pan}) values of the freely moving particles were quite consistent over position, whether the container was located at the geometric centre of the basket (0 mm) or at the outermost edge (175 mm). There was a large change in IS going from 0 to 15 rpm but a smaller change (within two standard deviations) in going from 15 to 30 rpm. Tables 6.2 and 6.3 summarise the statistical determinations.

For the liquid portion, the IS showed a relatively small positional effect, however there was a more pronounced rpm effect. It also appears that the IS increased at the centre position up to 30 rpm. In the other positions (115 mm and 175 mm), the IS appeared to level off at 15 rpm and remain somewhat constant (within one standard deviation) at 15 and 30 rpm.

Table 6.1 Position, rpm and integrated sterilisation values (IS_{pan}) for free, rigid and flexible thermocouples and the integrated sterilisation value (IS) for the liquid portion. Coefficients of variation are given in brackets, values within a column with different superscripts are significantly different ($p < 0.05$). 0 mm is the centre of the retort basket, 175 mm represents the outside corner on the diagonal.

RPM	Position (mm)	IS_{pan} - Free Particle (min)		IS_{pan} - Rigid Thermocouple Particle (min)		IS_{pan} - Flexible Thermocouple Particle (min)		IS - Liquid (min)	
0	0	4.82 ^a	(0.021)	5.38 ^b	(0.016)	4.82 ^a	(0.012)	5.22 ^a	(0.005)
	115	4.84 ^a	(0.013)	5.13 ^a	(0.008)	4.88 ^a	(0.012)	5.22 ^a	(0.005)
	175	4.82 ^a	(0.012)	5.36 ^b	(0.015)	4.84 ^a	(0.013)	5.25 ^a	(0.006)
15	0	5.21 ^c	(0.012)	5.79 ^c	(0.004)	5.28 ^{bc}	(0.005)	5.65 ^b	(0.005)
	115	5.17 ^b	(0.002)	5.80 ^c	(0.004)	5.30 ^{bc}	(0.003)	5.76 ^c	(0.005)
	175	5.23 ^c	(0.001)	5.87 ^c	(0.004)	5.33 ^c	(0.003)	5.86 ^d	(0.011)
30	0	5.17 ^b	(0.011)	5.41 ^b	(0.008)	5.18 ^b	(0.006)	5.79 ^c	(0.005)
	115	5.15 ^b	(0.001)	5.43 ^b	(0.009)	5.21 ^b	(0.002)	5.79 ^c	(0.004)
	175	5.16 ^b	(0.001)	5.50 ^b	(0.014)	5.27 ^c	(0.011)	5.90 ^d	(0.005)

Table 6.2. Analysis of variance (ANOVA) overall for data presented in Table 6.1. IS_{pan} and IS position are designated T, RAD = radius and RPM = rpm, df = degrees of freedom.

	df	F	P
T	3	927.20	0.0000
RAD	2	18.20	0.0000
RPM	2	1012.00	0.0000
T X RAD	6	5.23	0.0016
T X RPM	6	50.27	0.0000
RAD X RPM	4	2.88	0.0280
T X RAD X RPM	12	3.38	0.0006
ERROR	72		

Table 6.3 Comparison of mean IS between IS positions (IS refers to both IS_{pan} and IS)

IS Position	P	Result
Free versus Rigid	0.0001	Significant
Free versus Flexible	0.77	Not Significant
Free versus Liquid	0.0001	Significant
Rigid versus Flexible	0.0001	Significant
Rigid versus Liquid	0.522	Not Significant
Flexible versus Liquid	0.0001	Significant

The IS_{pan} values for the rigid and flexible thermocouple both followed the previously mentioned pattern of increasing in value from 0 rpm to 15 rpm and then decreasing from 15 to 30 rpm. The difference is that the IS_{pan} for the rigid thermocouple more closely followed the IS for the liquid, while the IS_{pan} for the flexible thermocouple more closely followed that of the free particles. Of interest is that the initial process time used was calculated for an $F_0 = 5$ min. Table 6.1 shows that the IS_{pan} values for the rigid particle are very close to that value.

Pflug et al. (1980) noted that due to the natural variation found in biological systems, thermocouple studies were more reproducible. However, sterilisation of food in a container is a biological problem. In a sense, the thermobacteriological approach to the problem brings us closer to the reality we wish to study. This again points to a need to continue this work to enhance the prediction of sterilisation and pasteurisation processes. Biological indicators are an additional tool to complement rather than supplant the use of thermocouples and traditional time-temperature methods for process determination work.

Houtzer and Hill (1977) found IS values to be nearly 5 min greater than F_0 for the same thermal process for 303 x 406 cans of whole-kernel corn and 1.9 min greater for cut green beans. Berry and Bradshaw, (1980), found IS values to be as much as 6.9 min higher in condensed cream of celery soup and 6.5 min higher in the brine for mushrooms (Berry and Bradshaw, 1982). Spore count reduction was used to predict thermal lag in ravioli heated in brine in an agitating retort at 123.9°C (Berry et al., 1985) and IS was found to exceed F_0 by approximately 5 min.

Spore leakage has been one factor ascribed to these observed data. Also, the z -value is of importance. Finally, Berry and Bradshaw (1986) observed that IS values exceeded F_0 values at retort temperatures above 121.1°C, which was consistent with investigations cited above. This also appears to be consistent with this study as the temperature in the retort vessel was below 121.1°C.

The use, however, of DSC pans to contain the spores means that leakage is not of concern. Also, improvements in spore enumeration through the use of the hydrophobic grid mean that what may have been interpreted as spore destruction may in fact have been spores that were viable but not recovered. Berry and Bradshaw (1986) mention a number of sources of error including the consequence of sampling at different locations in a container when less than the entire contents are used. With the technique presented here, the entire contents of the DSC pan are recovered.

6.4 Conclusions:

There are three main conclusions to be drawn from this portion of the study. The first is that there is strong evidence for the use of thermal indicators in thermal studies, particularly if one needs to consider inoculated pack studies as part of the process determination/verification procedure. The second conclusion is that the flexible thermocouple appeared to provide a more accurate picture of the sterilisation treatment received by a particle moving at random in a forced convection environment. The third conclusion arises from a comparison of the data generated in Chapter 5 versus the data presented here. The difference in heating is noted in that Chapter 5 where the particle was considered in isolation showed more rapid heating of the flexible thermocouple versus the rigid. In the data presented here, the opposite is true. This suggests that the presence of the additional particles is responsible for the difference in heating observed.

The IS_{pan} values shown in Table 6.1 show that the flexible thermocouple values were more closely aligned with those for the free particle while the IS_{pan} values for the rigid thermocouple were more closely aligned with the IS values for the liquid portion. If one was concerned about setting a process based on achieving a certain sterilisation value for the particulates as well as the

bulk of the food product, reliance on a food particle placed on the rigid thermocouple suggests that lethality requirements for free particles may not be met adequately. Further work is warranted not only with other species of bacteria, *C. botulinum* for example, but additional environments inside the DSC pan as well, for other factors such as pH, ionic strength, etc.

CHAPTER 7:

THERMAL INDICATORS - PASTEURISATION

7.1 Introduction:

Verification of processes in non-static retorting conditions as well as pasteurisation processes with traditional thermocouples can be technically difficult. For example, process verifications in hydrostatic retorts may involve hundreds of metres of thermocouple wire being fed into the retort. The same may be said for tunnel pasteurisers in which tunnel lengths of 20 metres are not uncommon. Kimball and Heylinger (1990) noted that in all continuous retorts and some batch agitating retorts, the same difficulty arises for carrying out temperature distribution studies as well. This has been overcome by the use of radio telemetry or self-contained microprocessor based units to record time-temperature information.

One limitation of radio-telemetry measurements concerns the possibility of data corruption. During the Second World War, Motorola Corporation developed a technology known as spread-spectrum. The purpose was to transmit information on a range of frequencies that enemy forces could not intercept and involved using a range of frequencies from 902 to 928 MHz. However, even with this technology, due to its range of 902-928 MHz, information transmission can be corrupted by stray FM inputs. Microprocessor based units due to their size, can also upset

convection patterns of heating or act as a heat sink themselves.

In this section of the study, thermal indicators were evaluated to avoid shortcomings noted above. The choice of thermal indicators is of importance however as the D and z value of the indicators must be chosen such that they are of the same order of magnitude as the target organism(s). *Clostridium pasteurianum* in the pH range of 4.1 to 4.6 and at 93°C has D and z values of 1.7 min and 8.33°C° respectively (Unilever, 1997). *Bacillus coagulans*, in the pH range of 4.3 to 4.6 at 90.0°C, has a D value of 3.2 min and a z value of 9.5°C° (Sandoval et al., 1992). In addition to the microbial thermal indicators, an enzyme was chosen.

Lysozyme (muramidase; EC 3.2.1.17) has stimulated considerable interest as a natural food preservative. It has been shown to be effective in preserving a variety of foods such as fresh fruits and vegetables, tofu bean curds, meats, seafood and wines, for which many Japanese patents have been granted (Cunningham et al., 1991). Lysozyme is also attractive for use as a thermal indicator in pasteurised and heat-sterilised food products due to its relatively high thermal stability. Lysozyme, at a pH of 6.24, at 93°C has a D value of 13.8 min and a z value of 18.2°C° (Makki and Durance, 1996).

7.2 Materials and Methods:

7.2.1 Preparation of *Bacillus coagulans* Cultures:

Cultures of *Bacillus coagulans* (ATCC 8038) were prepared as per Sandoval et al. (1992) on tomato juice agar (TJA) (Difco, Franklin Lakes, NJ) with the pH adjusted to 5.0 for maintenance of the strain. Sporulation was induced on the selected strain by incubating on TJA at pH 5.0 for 3

days at 45°C (Stumbo, 1973). The spores were collected when a level of about 90% sporulation was observed by microscope. The spore suspensions were prepared by standard methods as described by the National Canners Association Research Laboratories (1968). For this study, the pH was adjusted to 4.3 with citric acid to simulate the pH environment of the product and diluted, to a population of 1×10^8 cfu/mL and pipetted into hermetic anodised aluminum DSC pans (I.D. 6.6 mm) (Westech Industries Ltd., Calgary, AB) at a volume of 0.02 mL per pan. DSC pans were affixed to the inside of the containers in positions as shown in Figures 7.1 and 7.2 using silicone. As only a very small amount was used to provide adhesion on the surface adjacent to the internal wall of the container or underside of the lid, thermal effects were assumed to be insignificant.

Following processing, the pans were recovered and crushed open into a test tube containing 0.98 mL of diluent (0.1% peptone). The surviving population was then plated on TYG agar with 0.1 and 0.9 mL of the original 1 mL for the respective organism type. Plates were incubated at 37°C for 24 h and colonies counted (York et al., 1975).

7.2.2 Preparation of *Clostridium pasteurianum* Cultures:

Cultures of *Clostridium pasteurianum* (ATCC 7040) were prepared by growing the organism on Glucose Yeast Agar Extract (GYAE) (DSMZ, Braunschweig, Germany) for 24 h at 37°C under anaerobic conditions. Sporulation was induced on the selected strain by incubating on GYAE at 37°C for 5 days. The spores were collected when a level of about 90% sporulation was observed by microscope. Spores suspensions were prepared by the methods as described by Stumbo (1973). For this study, the pH was adjusted to 4.3 with citric acid to simulate the pH environment of the product in the container, and diluted, to a population of 1×10^8 cfu/mL and pipetted into

hermetic anodised aluminum DSC pans. Following processing, the pans were recovered and enumerated as described above.

7.2.3 Preparation of Lysozyme:

A solution of lysozyme was prepared by dissolving 342 mg of 3x crystallised, dialysed and lyophilised chicken egg white lysozyme (Sigma Chemical co., St. Louis, MO) in 10 mL of 0.066 M KH_2PO_4 buffer at pH 6.24. The solution was pipetted into anodised aluminum DSC pans (I.D. 6.6 mm) (Westech Industries Ltd., Calgary, AB) at a volume of 0.02 mL per pan and then hermetically sealed using a specific crimping tool (Westech Industries Ltd., Calgary, AB). Pans were then located as per Section 7.2.2.

7.2.4 Lysozyme Assay:

Activity of the lysozyme prior to and following pasteurisation was determined using the turbidimetric assay method as outlined in the Sigma Chemical Co. (St. Louis, MO) Bulletin No. 11-77. The procedure was modified slightly. The lysozyme samples and the *Micrococcus* suspension (Sigma Chemical Co.) were equilibrated to 25°C prior to the assay. The *Micrococcus* suspension was prepared as per Bulletin No. 11-77 from Sigma Chemical Co. For each assay, 0.1 mL of the lysozyme sample was added to 2.5 mL of the *Micrococcus* suspension. The assay mixture was quickly but gently mixed by inversion and placed immediately into a 1 cm light path cuvette and into the spectrophotometer. The time lag between the addition of the lysozyme sample and time "zero" was 23 s. Decrease in A_{450} versus the reference buffer solution was recorded at 5 s intervals for 50 s. One unit of activity was defined as the amount of enzyme

producing a change in A_{450} of 0.001 per min under these conditions. Lysozyme samples were assayed in triplicate. For comparison purposes, "lethalities" were determined at the same temperatures as lethalities determined for *Clostridium pasteurianum* and *Bacillus coagulans*.

7.2.5 Pasteurisation Studies:

Pasteurisation studies were carried out in three phases. The first phase relied on the use of thermocouple leads inserted in both a 3.8 L and a 1.9 L plastic jug as per Figure 7.1 and a 750 mL glass container (Figure 7.2). A thermocouple receptacle (Eklund-Harrison Technologies, Fort Meyers, FL) was modified to serve as a stuffing box for the thermocouple leads. This provided a leak-proof seal. For the handle thermocouple in the 3.8 L and 1.9 L plastic jugs, a knot was tied in the thermocouple wire such that the thermocouple could sit in the middle of the handle void. All containers were filled with a commercial spaghetti sauce product (T.J. Lipton, div. of Unilever, Canada Inc.) and lids applied by hand. Thermocouple leads were of sufficient length to travel through the heating section plus the first few minutes of the cooling section of the pasteurisation tunnel (2.46 m wide by 23.7 m long). The hot water section of the pasteuriser was 4.6 m long, there was a 1.54 m long tempering section and the balance of the pasteuriser was devoted to cooling. Ten lateral spray bars in the hot water section provided the heating medium at 93.3°C.

Thermocouples were attached to a Calwest Model 32 datalogger, connected in turn to an IBM Model 720C Thinkpad computer equipped with the CALsoft for Windows software program for acquisition and analysis of heat penetration data. Data were collected at 60 second intervals. Processing stages included: a) infusion heating of the spaghetti sauce to 92-94°C, b) a holding stage of 92-94°C, c) filling was done at 92°C, d) pasteurisation for 10 minutes at 90°C, followed

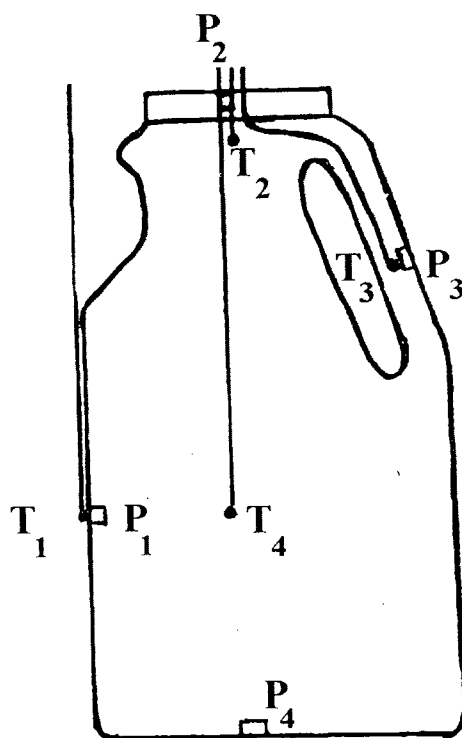


Figure 7.1 Thermocouple junction and DSC pan (thermal indicator) placement for pasteurisation studies – plastic jugs. Letters “P” with numerical subscripts denote DSC pans, letters “T” with numerical subscripts denote thermocouples.

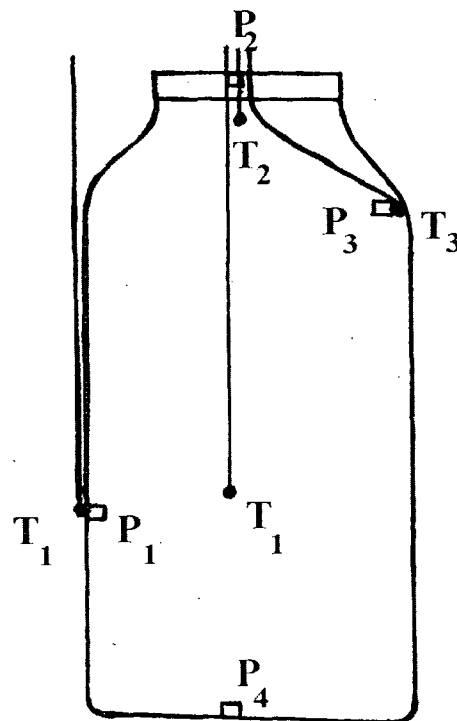


Figure 7.2 Thermocouple junction and DSC pan (thermal indicator) placement for pasteurisation studies – glass jar. Letters “P” with numerical subscripts denote DSC pans, letters “T” with numerical subscripts denote thermocouples.

followed by cooling with water sprays to provide an exit temperature, from the pasteuriser, of 43°C at the centre of the product. 3.8 L and 1.9 L plastic jugs travelled through the pasteuriser at a tunnel belt speed of 0.26 m/min and 750 mL jars travelled at a tunnel belt speed of 0.46 m/min.

The second phase utilised DSC pans containing the bacteria which were the thermal indicators. The same processing conditions described above were utilised. The locations within the containers were the same as for the thermocouple tests with the exception that the pan for the geometric centre was allowed to rest on the bottom of the container. This was done to ascertain if significant lethality was lost on the bottom edge of the container as it passed through the tunnel pasteuriser.

The third phase involved the usage of lysozyme as a thermal indicator. The same processing conditions previously mentioned for the second phase were used. The purpose was to ascertain if thermal indicators other than bacterial gave similar results.

Lethalities for thermal indicators were determined by using the following formula:

$$L = 10^{(T-93.3)/z} \quad (7.1)$$

7.3 Results and Discussion:

Conditions for processing *C. pasteurianum* showed an average population decrease of 10^3 cfu/mL for the lid. For DSC pans located in the product, the average population decrease was on the order of 10^4 to 10^5 cfu/mL. DSC pans located at the bottom of the container showed a reduction of 10^5 . The US Food and Drug Administration (FDA) recommends processes to give a 6-log decrease of spores of *C. pasteurianum* in acid products with a pH range 4.1 of 4.6 (Unilever,

1997). This represents 10 minutes at 93.3 °C, based on a D-value of 1.7 min and a z of 8.33C°.

For the three container types used, lethalties attained in the headspace at the end of the process indicated that a 5 log reduction was not achieved (Table 7.1). The glass container showed more uniform heating, however, it demonstrated the lowest log reduction, while the plastic containers showed more variation. *B. coagulans* spores showed slightly greater heat stability than *C. pasteurianum* spores in the glass jar. D values for *C. pasteurianum* would suggest that it is more heat resistant than *B. coagulans*.

The importance of achieving adequate heating of the headspace in containers undergoing pasteurisation was demonstrated. Inadequate heating may result in conditions such that insufficient lethality is achieved to render the product "safe" from a food safety perspective. Such lethality has traditionally been taken to be 10⁶. This serves to demonstrate that an inoculated pack done in the traditional fashion may indicate that the product achieves an adequate process, but areas that are assumed to be pasteurised by product temperature (such as headspace) may in fact not achieve adequate pasteurisation. One would need to consider bacterial loading in the external environment in the processing area and post-processing that may serve to cause post-process spoilage (i.e. the degree to which preparation of product is separated from filling, labelling, warehousing, etc).

Container configuration also appears to play a role. Both the 3.8L plastic and 750 mL glass jar showed similar patterns of pasteurisation process. The 1.9 L plastic jug, however, showed a more pronounced difference with respect to heating of the lid.

Knock et al. (1959) found a D value of 13.4 min. at 93.3°C and a z value of 16.1 C° for *B. coagulans* in tomato juice. Sandoval et al. (1992) gave D and z values of 3.2 min and 9.5 C° respectively at 90 °C for *B. coagulans* in double concentrated tomato paste. Rodrigo et al. (1990)

Table 7.1 Location versus log reduction data for *C. pasteurianum* and *B. coagulans* in 3.8 L, 1.9 L plastic and 750 mL glass containers. Lysozyme assays were done in 750 mL glass containers. (Data presented are an average of nine measurements [3 DSC pans per position in 3 containers], coefficients of variation are shown in brackets). Starting populations were 2.0×10^6 cfu/pan.

Container	Location	<i>C. pasteurianum</i> log reduction	<i>B. coagulans</i> log reduction	Lysozyme dA ₄₅₀ /min
3.8 L Plastic Jug	Lid	4.17 (0.18)	4.21 (0.09)	
	Handle	4.50 (0.04)	4.22 (0.10)	
	Bottom	4.37 (0.09)	4.40 (0.04)	
1.9 L Plastic Jug	Lid	2.60 (0.04)	3.97 (0.14)	
	Handle	4.23 (0.15)	3.83 (0.12)	
	Bottom	3.77 (0.19)	3.63 (0.04)	
750 mL Glass Jar	Lid	2.83 (0.12)	4.07 (0.05)	0.017 (0.235)
	Shoulder	3.00 (0.06)	3.87 (0.08)	0.018 (0.056)
	Bottom	3.07 (0.14)	3.97 (0.04)	0.020 (0.050)

studied *B. coagulans* ATCC 8038 in crushed tomatoes at pH 4.5 and pH 4.3 and found D values at 90°C of 12.3 and 9.0 min, respectively. There was also a corresponding change in z values of 13.7 and 13.9 C° for pH 4.5 and 4.3, respectively.

Data for lysozyme in the 750 mL glass jar showed a pattern similar to that observed for the bacterial indicators. The 1.9 L and 3.8 L plastic jugs had a plastic cap, with a liner, fitted to the container. The cap, for the glass jar, was metal. Thus the DSC pan fixed to the metal lid of the glass jar would achieve a more severe heat treatment due to the better conducting properties of the metal lid. The treatment that was received in this region would then better approximate that found in the bulk of the product. Residual lysozyme activity was 62% of the control activity for the headspace region while in the DSC pans at the sides and bottom of the product there was a 56% decrease in activity. Due to the higher D and z values for lysozyme versus the bacterial indicators, residual activity was higher, as expected.

Calculation of lethalties for *C. pasteurianum* and *B. coagulans* (Tables 7.2 to 7.4 and 7.5 to 7.7 respectively) showed again that the temperature data provided a pattern similar to the survivor data from the DSC pans. Data presented for *C. pasteurianum* in Table 7.2 showed a similar pattern in that there was less lethality in both the lid and handle regions of the container (Figure 7.1) compared to the bulk of the product. This compares with the log reductions shown in Table 7.1.

The centre lethality for the 3.8 L container (Table 7.2) was 3.8 times greater than that for the lid and handle. The calculated lethality for the lid showed a 5.5 log decrease in numbers while the microbially measured lethality was a 4.17 log decrease. For the handle a 5 log decrease was calculated while the measured was 4.50 log. Finally, for the bottom a 4.37 log decrease was determined but a 20.44 log reduction was calculated.

For the 1.9 L container (Table 7.3), the pattern was similar in that the calculated bottom

Table 7.2 Temperature versus lethality (calculated by equation 7.1) for *C. pasteurianum* in a 3.8 L plastic jug $D_{93.3} = 1.7$ min, $z = 8.33$ C°.

Process Time (min)	Pasteuriser Temp. (°C)	Lid		Handle		Bottom	
		Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)
2	31.3	82.5	0.05	53.2	0.00	95.8	1.99
3	76.0	82.0	0.04	68.5	0.00	95.8	1.98
4	93.7	86.0	0.13	86.2	0.14	96.3	2.30
5	92.2	88.7	0.28	90.0	0.40	97.4	3.13
6	94.9	90.2	0.43	92.3	0.75	97.3	3.06
7	95.9	91.5	0.61	93.7	1.10	97.3	2.99
8	96.7	92.9	0.89	92.9	0.90	97.9	3.57
9	97.4	93.4	1.02	93.8	1.15	97.5	3.15
10	97.3	93.6	1.09	95.0	1.59	97.0	2.80
11	98.2	96.0	2.14	94.6	1.43	98.9	4.75
12	97.9	96.9	2.68	94.2	1.28	99.1	5.02
TOTAL			9.36		8.74		34.74

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Table 7.3 Temperature versus lethality (calculated by equation 7.1) for *C. pasteurianum* in 1.9 L plastic jug, , $D_{93.3} = 1.7$ min, $z = 8.33$ C°.

Process Time (min)	Pasteuriser Temp. (°C)	Lid		Handle		Bottom	
		Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)
2	87.8	85.0	0.10	69.3	0.00	84.5	0.10
3	90.6	86.0	0.13	74.5	0.01	88.6	0.37
4	92.6	87.4	0.19	79.2	0.02	89.8	0.76
5	91.8	98.0	0.23	82.8	0.05	90.3	1.18
6	92.4	89.0	0.30	85.5	0.11	90.8	1.69
7	90.5	89.2	0.33	87.5	0.20	90.7	2.18
8	92.6	90.5	0.46	89.6	0.36	92.7	3.02
9	94.1	91.5	0.61	90.4	0.44	91.0	3.55
10	91.2	90.6	0.48	90.5	0.47	90.8	4.05
11	92.9	90.8	0.50	90.9	0.52	91.2	4.62
12	92.8	90.3	0.44	91.2	0.56	90.9	5.13
TOTAL:			3.77	2.68		26.65	

Table 7.4 Temperature versus lethality (calculated by equation 7.1) for *C. pasteurianum* in a 750 mL glass jar,, $D_{93.3} = 1.7$ min, $z = 8.33$ C°.

Process Time (min)	Pasteuriser Temp. (°C)	Lid		Handle		Bottom	
		Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)
2	63.9	84.0	0.08	96.5	1.43	87.6	0.18
3	98.7	89.1	0.26	96.5	1.41	97.9	1.94
4	97.0	92.3	0.54	96.6	1.45	91.0	0.39
5	100.2	94.0	0.80	96.4	1.37	92.7	0.58
6	100.1	95.1	1.02	96.5	1.43	94.0	0.80
7	102.4	96.5	1.42	97.2	1.67	94.8	0.94
8	104.7	98.0	1.98	97.3	1.70	95.7	1.19
9	107.3	98.8	2.39	97.0	1.59	97.0	1.58
10	108.2	100.1	3.24	97.3	1.68	102.7	5.95
11	109.5	101.0	3.96	97.6	1.80	100.8	3.76
12	113.7	103.6	7.32	98.8	2.40	103.4	6.92
TOTAL:			23.01	17.93		24.23	

Table 7.5 Temperature versus lethality (calculated by equation 7.1) for *B. coagulans* in a 3.8 L plastic jug, $D_{90} = 3.2$ min, $z = 9.5$ C°.

Process Time (min)	Pasteuriser Temp. (°C)	Lid		Handle		Bottom	
		Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)
2	31.3	82.5	0.16	53.2	0.00	95.8	4.06
3	76.0	82.0	0.15	68.5	0.00	95.8	4.05
4	93.7	86.0	0.39	86.2	0.40	96.3	4.62
5	92.2	88.7	0.73	90.0	1.00	97.4	6.06
6	94.9	90.2	1.15	92.3	1.73	97.3	5.92
7	95.9	91.5	1.44	93.7	2.43	97.3	5.81
8	96.7	92.9	2.02	92.9	2.03	97.9	6.80
9	97.4	93.4	2.27	93.8	2.51	97.5	6.08
10	97.3	93.6	2.40	95.0	3.34	97.0	5.48
11	98.2	96.0	4.32	94.6	3.04	98.9	8.73
12	97.9	96.9	5.29	94.2	2.75	99.1	4.11
TOTAL			20.20		19.24		61.72

Table 7.6 Temperature versus lethality (calculated by equation 7.1) for *B. coagulans* in a 1.9 L plastic jug, $D_{90}=3.2$ min, $z=9.5$ C°.

Process Time (min)	Pasteuriser Temp. (°C)	Lid		Handle		Bottom	
		Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)
2	87.8	85.0	0.30	69.3	0.0	84.9	0.29
3	90.6	86.0	0.38	74.5	0.02	88.6	0.71
4	92.6	87.4	0.53	79.2	0.07	89.8	0.96
5	91.8	98.0	6.94	82.8	0.17	90.3	1.07
6	92.4	89.0	0.78	85.5	0.33	90.8	1.22
7	90.5	89.2	0.83	87.5	0.54	90.7	1.19
8	92.6	90.5	1.13	89.6	0.90	92.7	1.92
9	94.1	91.5	1.43	90.4	1.09	91.0	1.28
10	91.2	90.6	1.16	90.5	1.14	90.8	1.22
11	92.9	90.8	1.22	90.9	1.25	91.2	1.35
12	92.8	90.3	1.09	91.2	1.33	90.9	1.24
TOTAL:			15.78		6.87		12.45

Table 7.7 Temperature versus lethality (calculated by equation 7.1) for *B. coagulans* in a 750 mL glass jar, $D_{90} = 3.2$ min, $z = 9.5$ C°.

Process Time (min)	Pasteuriser Temp. (°C)	Lid		Handle		Bottom	
		Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)
2	63.9	84.0	0.23	96.5	4.88	87.6	0.56
3	98.7	89.1	0.80	96.5	4.81	97.9	6.74
4	97.0	92.3	1.76	96.6	4.96	91.0	1.26
5	100.2	94.0	2.66	96.4	4.68	92.7	1.91
6	100.1	95.1	3.43	96.5	4.88	94.0	2.66
7	102.4	96.5	4.87	97.2	5.77	94.8	3.16
8	104.7	98.0	6.90	97.3	5.77	95.7	4.02
9	107.3	98.8	8.40	97.0	5.48	97.0	5.43
10	108.2	100.1	11.57	97.3	5.81	102.7	21.93
11	109.5	101.0	14.28	97.6	6.25	100.8	13.57
12	113.7	103.6	27.28	98.8	8.46	103.4	25.74
TOTAL:			82.17		61.87		86.98

lethality was 8.2 times higher than either the lid or handle lethalties. Measured lethalties for the lid showed a 3 log reduction while a 2.18 log decrease was calculated. For the handle and the bottom of the container the measured lethality was 4.23 and 3.77 log respectively. The calculated lethalties were 1.6 and 15.7 log respectively.

Calculated lethalties of 13.05, 10.6 and 14.3, log reductions for the lid, shoulder and bottom locations of the 750 mL glass container (Table 7.4) were determined but the measured lethalties were 2.83, 3.00 and 3.07 log reductions for all locations. It should be noted however, that 50 cfu/mL was the resolution limit of the assay, so essentially the 750 mL container sustained destruction of the organism to that point of resolution. Higher initial inoculum levels of indicator organisms could be used to confirm this. The other containers differ between measured and actual lethalties by a factor of 10 for the lid and handle and substantially more in the case of the bottom.

The lethality data calculated for *B. coagulans* (Tables 7.4 to 7.7) showed an overall pattern that was similar to that seen for *C. pasteurianum* in Table 7.2. For the 3.8 L jug, the lid and handle calculated lethalties represent a 6 log reduction but are 3.2 times lower than for the bottom of the container. The measured lethalties showed a 4.21, 4.22 and 4.40 log reduction for all three locations.

Calculated lethalties of the 1.9 L jug for the lid and bottom were 4.92 log and 3.88 log, respectively, thus they were in close agreement with the measured lethalties of approximately 4 fold in both cases. For the handle, calculated lethality was 2.15 and measured lethality was again 4 log.

The 750 mL glass jar showed a pattern similar to the 1.9 L jug but the calculated lethalties were all approximately 8 times higher. When compared to the measured lethalties in Table 7.1, it

can be seen that only a 4 fold decrease (4.07, 3.87 and 3.97 for the lid, shoulder and bottom respectively) was measured. This may be due to the lower counting limit of the microbiological method.

It is interesting to note for both test organisms there was greater lethality in the metal lid of the 750 mL glass jar than the plastic lids on the plastic containers. This suggests that the metal lid of the 750 mL glass jar contributed to the pasteurisation effect which developed in the headspace. The plastic lids on the plastic containers may have provided a greater insulating effect. Furthermore, since a low pressure (partial vacuum) is achieved in the application of the lid, there was already less medium available for heat transfer.

Table 7.8 shows calculated "lethalities" for lysozyme. Using the D and z values presented earlier, the data for lid and shoulder indicate decreases of 1.37-log and 1.34-log. The data for the bottom indicate a calculated "lethality" equivalent to a 1.4-log decrease in lysozyme activity. The measured values showed 2.8-log, 2.6-log and 2.3-log decreases in lysozyme activity for the lid, shoulder and bottom positions respectively.

7.4 Conclusions:

From the results of this study, problems associated with strict reliance on product temperature for ensuring pasteurisation within the container headspace have been demonstrated. In order to safely set the required pasteurisation process, some knowledge of the thermal events occurring in the headspace is necessary -- whether it be microbial or temperature measurement in nature. Pflug et al. (1985) studied the effect of pH on populations of *Clostridium botulinum* and demonstrated that in the range of pH 4.6 to 6.0 there was a marked effect on F-value (lethalities).

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Table 7.8 Temperature versus "lethality" for lysozyme in a 750 mL glass jar.

Process Time (min)	Pasteuriser Temp. (°C)	Lid		Handle		Bottom	
		Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)	Temp. (°C)	Lethality (min)
2	63.92	83.95	0.32	96.54	1.57	87.63	0.51
3	98.66	89.08	0.61	96.48	1.55	97.87	1.85
4	96.97	92.34	0.92	96.61	1.58	90.96	0.77
5	100.21	94.04	1.14	96.37	1.53	92.66	0.96
6	100.14	95.08	1.30	96.54	1.57	94.04	1.14
7	102.43	96.53	1.56	97.23	1.71	94.75	1.25
8	104.66	97.97	1.88	97.31	1.73	95.74	1.41
9	107.29	98.78	2.08	97.02	1.66	96.98	1.66
10	108.21	100.1	2.46	97.26	1.71	102.74	3.43
11	109.52	100.97	2.74	97.56	1.78	100.76	2.67
12	113.71	103.64	3.84	98.81	2.07	103.4	3.73
TOTAL:			18.85		18.46		19.38

This study also demonstrated the value of thermal indicators in environments where traditional thermocouple usage may be either cumbersome or impossible to carry out. The results suggested a close agreement between thermal inactivation of bacterial populations and the lethalties calculated for *C. pasteurianum* and *B. coagulans*, based on the time-temperature history of the product in a specific container.

Container configuration may also play a role. Changes in container shape, the presence or absence of a handle, material of construction - especially with closures (i.e., metal versus plastic) - not to mention product composition factors are important. For example, survivor numbers showed lower lethalties were attained in the headspace compared to other locations tested, however, the calculated lethalties from time-temperature measurements in the jug handles indicated less lethtality would be expected there.

Lastly, traditional inoculated pack studies introduce the possibility of contamination of saleable product with the indicator organisms. The DSC pans allow inoculated pack studies to be conducted, but the pan provided a physical barrier such that the bacterial population experienced the same temperature environment as the product without the inherent risks of contaminating product or processing plant environment. The spores could be heated in a defined buffer rather than the less reproducible natural food product.

Finally, use of lysozyme as a heat-labile indicator offers another method of validating pasteurisation processes in addition to traditional inoculated pack studies and time-temperature measurements. While the actual "process lethalties" measured for lysozyme were lower than for either of the bacterial indicators, lysozyme did show its utility for pasteurisation process evaluations. Principally, it is capable of surviving a severe pasteurisation process and although the D and z values are relatively high in comparison to the bacterial indicators, they are useful as a

cumulative measure of product temperature history.

CHAPTER 8:

CONCLUSIONS

The objectives of this study were to gain a better understanding of end-over-end agitated processes for particulate food in microwaveable plastic containers, as well as applications to pasteurisation processes.

The first objective of this study was to observe patterns of particulate movement under agitation in retortable plastic (OMNI) bowls. At 2.8 rpm, it was found that the particles tended to travel around the inner surface of the container, never traversing the centre. The fact that the source of movement in forced convection is the headspace bubble, and that gravitational forces greatly exceed centrifugal forces may be the main factors in this observation.

The second objective was proving a new method for determining thermal diffusivity of food materials. The method used by Dickerson (1965) was enhanced by use of micro-processors in retort applications to achieve a constant heating rate. The one caveat is that the controls – valving, etc. must respond in real-time so that the actual temperatures oscillate closely around the required straight heating curve plus or minus a pre-determined temperature interval. Linear regression can then be used to calculate the actual thermal diffusivity using Dickerson's formula.

The third objective was to evaluate rotational and positional effects on heating responses in the Lagarde retort. There were definite areas of slower heating. In the production model Lagarde this

appeared to be located at the centre of a diagonal line drawn through a retort basket from the central axis to the outside corner of the basket. This may be due to the annular disc found in the door that, in conjunction with the fan serves to circulate the steam/air mix. The implication is that the ring actually restricts air flow somewhat so that the velocity of the heating medium is slower in the area of the basket previously mentioned. Similar effects were found in the Lagarde simulator, however, they did not seem as pronounced due to the smaller size of the rotating basket in the retort.

Objective four was to test whether time-temperature data and thermal indicator results of the flexible thermocouple system gave better results than for the rigid thermocouple system. In the case of time-temperature data, the results showed that the rigid thermocouple led to a greater estimation of process time required for a given target lethality and that the flexible thermocouple gave a shorter process time to achieve an F_0 of 5.0 min. However, when IS_{pan} values were compared, the particles on flexible thermocouples required process times equivalent to the free particle. The rigid thermocouple IS_{pan} values were closer to those calculated for the liquid portion of the test product. The net result is that for IS_{pan} , the values for the rigid thermocouple were higher than for the flexible thermocouple and thus closer to the calculated F_0 of 5.0 min based on time-temperature data. This suggested that the flexible thermocouple would lead to an under-estimation of required process time to achieve an F_0 of 5.0 min. In other words, if a process was to be based on rigid thermocouple IS_{pan} values or time-temperature data, the process would under-estimate the time needed to achieve sufficient lethality in free particulates. On the other hand, usage of the flexible thermocouple IS_{pan} data would lead to over-estimation of the process required for sufficient lethality in the liquid portion. This situation will change, however, when non-Newtonian fluids are considered as well as initial temperature, particle number, etc. This leads back to the initial

discussions presented in the Introduction concerning Taguchi Methods for forming experimental design.

The fifth objective was to examine the use of time-temperature indicator systems in DSC pans in both sterilisation and pasteurisation processes. The bacterial and enzymatic systems used in the pasteurisation process produced patterns of lethality similar to the time-temperature profiles. The indicator system overcame some of the physical restrictions of traditional time-temperature measurements using thermocouples. Two items of note, the D and z values for the enzymatic system should be comparable to the target organism used to set the process in the first case. The experimenter may be able to adjust D and z of the indicator by adjusting the suspension buffer in terms of pH, ionic strength, etc.

In summary, the use of thermal indicators, while not without some difficulties, did provide alternate methods for determining thermal process effectiveness - whether in pasteurisation or sterilisation applications. In addition, the flexible thermocouple appeared to offer a more accurate technique for monitoring of the behaviour of particulates in a forced convection environment such as end-over-end rotation, than did the rigid, centrally mounted thermocouple technique.

Appendix I

NOMENCLATURE

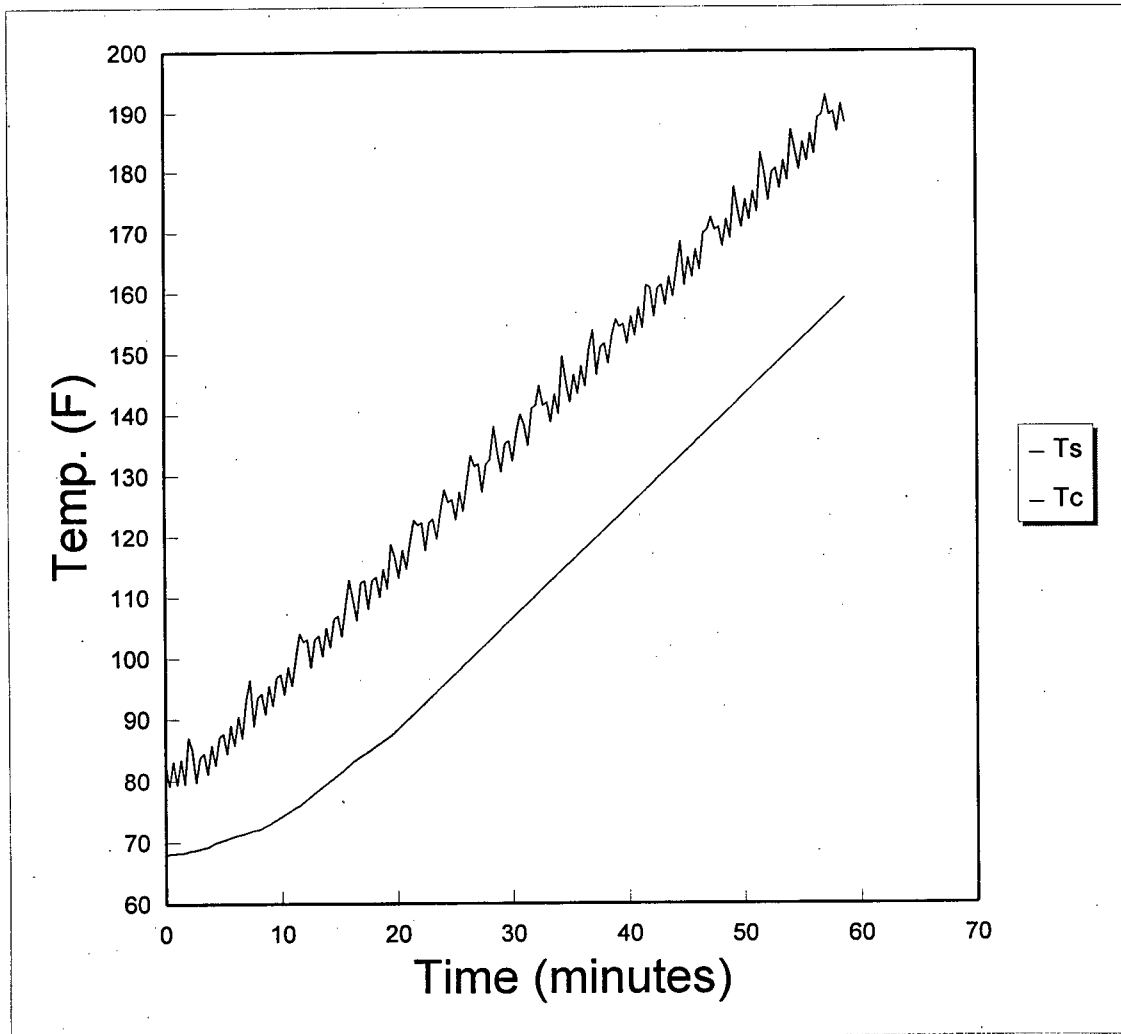
A	Area (m^2)
B	Rate of temperature increase ($^\circ\text{C}/\text{sec.}$)
Bi	Biot number
C_p	Specific heat (constant pressure) ($\text{kJ/kg } ^\circ\text{K}$)
F	The total lethal effect of heat applied at different temperatures expressed as minutes at some reference temperature.
F_o	F at the geometric centre of the container when $T_x = 121.1^\circ\text{C}$ and $z = 10^\circ\text{C}$ (min)
f	The time for the linear section of a heating or cooling curve plotted on semi-log co-ordinates to traverse one log cycle (min)
f_h	Heating rate index, f associated with the heating curve (min)
Gr	Grashoff number
h	Surface heat transfer coefficient ($\text{W}/\text{m}^2 \text{K}^\circ$)
j_h	Lag factor for heating = $(T_r - T_{pih}) / (T_r - T_{ih})$
j_c	Lag factor for cooling = $(T_w - T_{pic}) / (T_w - T_{ic})$
k	Thermal conductivity ($\text{W}/\text{m K}^\circ$)
N_o	Expected heat transfer coefficient in absence of viscosity variation ($\text{W}/\text{m}^2 \text{C}^\circ$)
Nu	Nusselt number
q	Heat flow (watts)
Pr	Prandtl number
P_t	Process time, the time after venting required to achieve a desired F_o (min).
R	radius (m)
R_c	Internal fluid film resistance to heat flow

Re	Reynolds number
R_h	External fluid film resistance to heat flow.
R_w	Conduction heat transfer resistance of container wall.
t	Time (min)
t_B	Thermal process time as calculated by Ball's formula method (Ball and Olson, 1957) (min).
T	Final product temperature ($^{\circ}\text{C}$)
T_c	Centre temperature of food material ($^{\circ}\text{C}$)
T_e	Temperature of heating media (environment) ($^{\circ}\text{C}$)
T_{ic}	Product temperature at the start of the cooling cycle ($^{\circ}\text{C}$)
T_{ih}	Initial product temperature ($^{\circ}\text{C}$)
T_o	Temperature at outside surface of food material ($^{\circ}\text{C}$)
T_{pic}	Pseudo-initial cooling temperature ($^{\circ}\text{C}$)
T_{pih}	Pseudo-initial product temperature ($^{\circ}\text{C}$)
T_x	Retort temperature ($^{\circ}\text{C}$)
T_s	Temperature at outside surface of container ($^{\circ}\text{C}$)
T_w	Temperature of cooling water ($^{\circ}\text{C}$)
U	Overall heat transfer coefficient ($\text{W}/\text{m}^2 \text{ } ^{\circ}\text{C}$)
z	Number of degrees required for the thermal death time curve to traverse one log cycle ($^{\circ}\text{C}$)
α	Thermal diffusivity (m^2/s)
β	Coefficient of expansion for a fluid being heated ($1/^{\circ}\text{C}$)
ρ	Density (g/cm^3)
μ	Viscosity ($\text{Pa}\cdot\text{s}/\text{cm}^2$)

μ_b	Bulk fluid viscosity (Pa•s/cm ²)
μ_w	Fluid viscosity at container wall (Pa•s/cm ²)

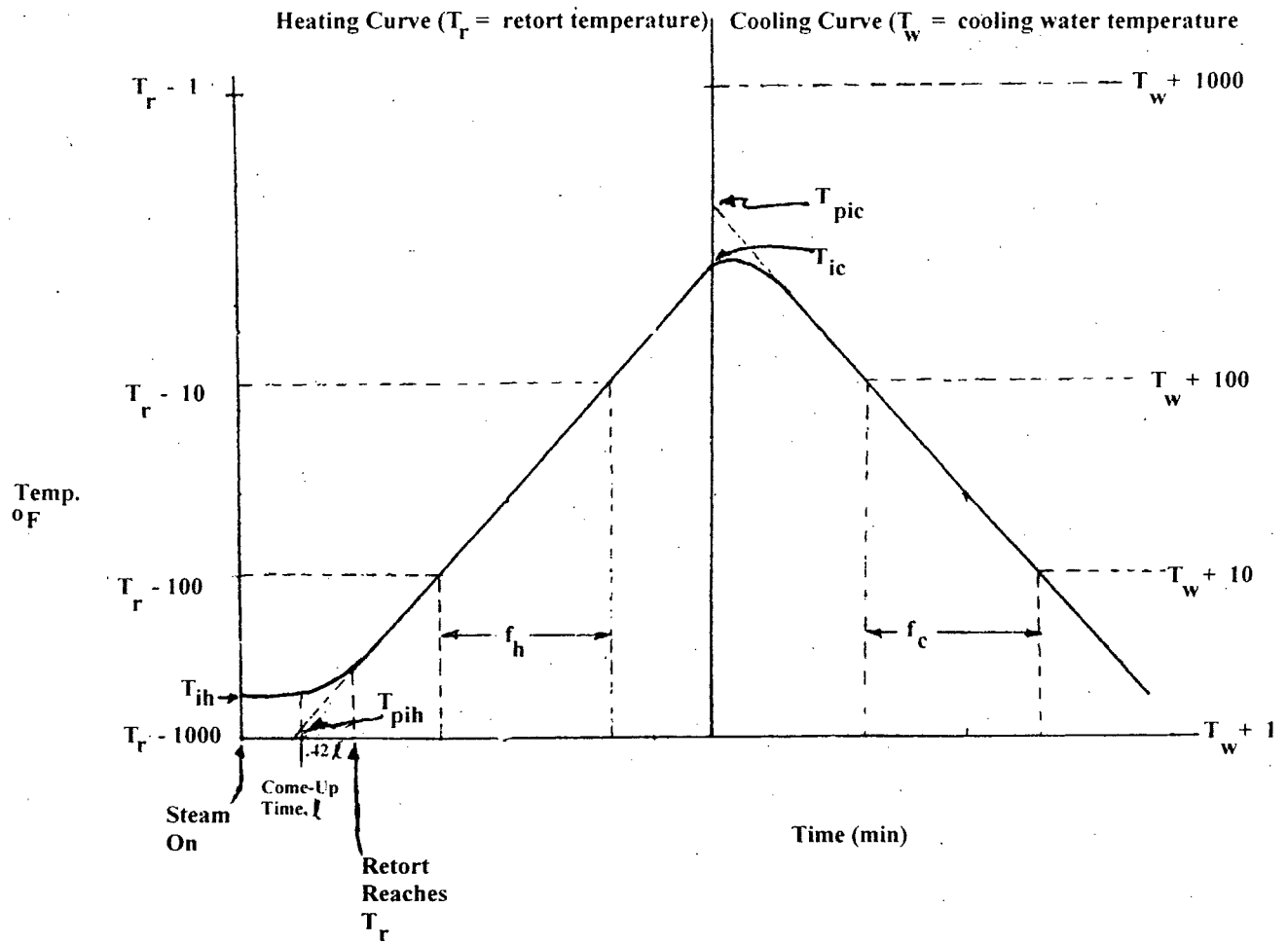
Appendix II

Thermal Diffusivity Graph – Potato/Alginate Gel



Appendix III

Jackson Plot of Heat Penetration Data Showing Derivation of Terms



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