RESIDENCE TIME DISTRIBUTIONS (RTD) OF FOOD PARTICLES AND RHEOLOGICAL PROPERTIES OF CARRIER FLUIDS UNDER ASEPTIC PROCESSING CONDITIONS

by

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DEDICATION

To whom I belong

My Wife Nahid and my Son Mohammed

My Parents

ABSTRACT

Residence time distributions (RTD) of meat and carrot cubes in starch solutions were evaluated in three stages in a commercial pilot scale aseptic processing system. In the first stage, RTD of meat (10, 15 and 20 mm) and carrot (6 &13 mm) cubes were investigated in the scraped surface heat exchanger (SSHE) of the aseptic system using a full factorial design of experiments employing flow rate (10, 15, 20, and 25 kg/min), starch concentration (4, 5 & 6% w/w). In the second stage, RTD experiments were conducted in the holding tube length (4.6, 9.2, 13.8 and 18.4 m) with meat (10 & 20 mm) and carrot (6 &13 mm) cubes, flow rate (10/15 & 20 kg/min) and starch concentration (4 & 6% w/w) at room temperature. All factors significantly ($p < 0.05$) influenced the fastest particle residence time (FPRT, FPNRT), mean particle residence time (MPRT), particle residence time standard deviation (SRTD). In the holding tube, larger particles moved faster than the smaller ones and vice-versa with respect to the SSHE. Finally, RTD studies were performed with the whole aseptic processing system at higher temperatures (80 &100°C) employing carrot cubes (6 & 13 mm), flow rate (15 & 20 kg/min), starch concentration (3 and 5% w/w), and holding tube length (1.5, 17.45 & 26.65 m). All factors significantly ($p < 0.05$) in influenced the RTD parameters. A logistic growth model was used to describe RTD curves of meat and carrot cubes. Three model parameters (an accumulation rate factor, B; a concentration upper limit factor, U and a half-time internal age factor, H) fully described the RTD curve. The model was used to describe the influence of product/process parameters and obtain *E*-type RTD curves. All test factors significantly influenced ($p < 0.05$) the logistic model parameters. Using

multiple regression model parameters were related to the experimental factors ($R^2 > 0.78$).

A bioindicator made from *Bacillus subtilis* spores immobilized in carrot/alginate cubes (12 mm) was used for RTD evaluation in the temperature range (95-120°C) with 15 kg/min flow rate and 1.5 and 26.65 m holding tube length. Complete destruction of bacterial spores was achieved at temperatures ≥ 110°C. The experiments showed that RTD, temperature and holding tube length will determine the extent of survival/destruction of the *Bacillus subtilis* spores.

Rheological properties of gelatinized starch (3-6% w/w) and carboxymethyl cellulose (CMC) solutions (0.5-2.0 % w/w), both widely used as carrier fluids in low acid liquid foods containing particulates, were investigated at aseptic processing temperature range using a high temperature/high pressure viscometry. The high pressure sensor was calibrated with a conventional sensor using viscosity standards of silicone oils. Both temperature and concentration significantly influenced the rheological properties of both CMC and starch solutions. Flow curves were well described by the power law model, while the temperature dependency of the rheological parameters was modelled using a modified Turian approach. Time dependency was evaluated employing a modified Weltmann model. All mentioned models were modified to accommodate concentration.

Finally, the RTD data of food particles and rheological properties of starch were used to develop dimensionless correlations for the maximum velocities of meat and carrot cubes in the SSHE, holding tube and the whole system. Particle velocities were related to particle-to-tube diameter ratio, carrier fluid Froude, Reynolds and Archimedes (for particle also) numbers. Viscosity was very critical for RTD studies at high temperatures.

RESUMÉ

Une étude sur la distribution des temps de résidence (RTD) de cubes de carotte et de viande en solution d'amidon a été réalisée en trois étapes. Dans la première étape, on a étudié la RTD des cubes de viande (10, 15 and 20 mm) et des cubes de carotte (6 et 13 mm) dans un échangeur de chaleur à surface raclée (SSHE) d'un système aseptique en utilisant un plan statistique factoriel complet ayant pour facteurs le débit volumétrique (10, 15, 20 et 25 kg/min) et la concentration d'amidon (4, 5 et 6% w/w) dans le fluide porteur. Dans la deuxième étape, on a déterminé l'effet de la longueur des tubes de retenue (4.6, 9.2, 13.8 et 18.4 m), de la grosseur des particules de viande (10 et 20 mm) et de carotte (6 et 13 mm), du débit volumétrique (10, 15 et 20 kg/min) et de la concentration d'amidon (4 et 6% w/w) sur la distribution des temps de résidence à la température de la pièce. Il s'est avéré que tous les paramètres d'étude sont significatifs ($p < 0.05$) et ont une influence sur le temps de résidence de la particule la plus rapide (FPRT, FPNRT), sur le temps moyen des particules (MPRT) et sur l'écart type de la distribution des temps de résidence (SRTD). Dans les tubes de retenue, les particules les plus grosses se déplacent plus rapidement que les plus petites particules alors que le phénomène inverse est observé dans l'échangeur de chaleur à surface raclée. Enfin, des travaux sur la distribution des temps de résidence ont été réalisés pour le procédé aseptique complet à haute température (80 et 100°C) en utilisant les cubes de carotte (6 et 13 mm) avec des débits volumétriques variant de 15 à 20 kg/min, une concentration d'amidon de 3 à 5%w/w et des longueurs des tubes de retenue de 1.5, 17.45 et 26.65 m. On a conclu que tous les facteurs sont significatifs ($p < 0.05$) et influencent la distribution

des temps de résidence. De plus, on a utilisé un modèle mathématique de croissance logistique pour décrire le comportement des courbes de RTD des cubes de viande et de carotte. Les trois paramètres du modèle soit: B, le taux d'accumulation; U, la limite supérieure de la concentration; et H, l'âge interne de la demi-vie permettent de modéliser adéquatement les courbes des RTD. Le modèle a été utilisé pour décrire l'influence des paramètres du produit et du procédé et pour obtenir la courbe typique de E. On a constaté que tous les facteurs influencent significativement ($p < 0.05$) les paramètres du modèle logistique. En utilisant une régression multiple, les paramètres du modèle ont été correlés aux paramètres expérimentaux avec un $R^2 > 0.78$.

Aussi, nous avons développé une méthode d'évaluation de la létalité du système de stérilisation à l'aide d'un indicateur biologique fait de spores de *Bacillus subtilis* immobilisées dans des cubes (12 mm) de carotte et d'alginate. Cette méthode a été utilisée pour l'évaluation de la létalité des RTD dans une échelle de température variant de 95°C à 120°C avec un débit volumétrique de 15kg/min pour deux longueurs des tubes de retenue soit 1.5 et 26.65 m. Une destruction complète des spores a été observée à des températures supérieures à 110°C. Les expériences ont montré que la RTD, la température and la longueur des tubes de retenue déterminent l'efficacité de la destruction des spores de *Bacillus subtilis*.

Les propriétés rhéologiques de l'amidon gélatinisé (3-6% w/w) et des solutions de carboxymethyl cellulose (CMC) (0.5-2.0% w/w), ces deux composés étant largement utilisés comme fluide porteur dans les procédés de stérilisation en continu de liquides contenant des particules, ont été mesurées aux températures des systèmes aseptiques en

utilisant un viscosimètre à haute température et à haute pression. La calibration du senseur de haute pression a été réalisée à l'aide d'un senseur conventionnel en utilisant des standards d'huile de silicone. On a trouvé que la température et la concentration influencent significativement les propriétés rhéologiques des solutions d'amidon et de CMC. Le modèle de la loi de puissance décrit adéquatement les courbes rhéologiques. L'approche de Turian modifiée a été utilisée pour décrire la dépendence des paramètres rhéologiques avec la température. Pour évaluer la dépendence par rapport au temps, le modèle de Weltmann modifié a été utilisé. Dans tous ce cas, les modèles ont été modifiés pour tenir compte des effets de la concentration.

Enfin, les résultats des RTD pour les cubes d'aliments et les propriétés rhéologiques de l'amidon ont été utilisées pour développer des correlations sans dimension pour la vitesse maximum des cubes de carotte et de viande dans l'échangeur de chaleur à surface raclée, les tubes de retenue et le système complet. Les vitesses des particules ont été reliées au rapport du diamètre des particules/diamètre du tube, aux nombres de Froude, Reynolds et Archimedes. On a trouvé que la viscosité était critique pour les RTD à hautes températures.

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NOMENCLATURE

a	Lower asymptote associated with the logistic model (a = 0).
A, B, a, b	Constants
A	Cross-sectional area, m^2
$Ar_{f,p}$	Archimedes numbers for carrier fluid and particle
B	Slope of the linear portion of the curve (particle accumulation rate).
c	Particles concentration, g
C	Carrier fluid concentration, % w/w / Constant
$c(\theta)$	Particles concentration at any normalized time, g
C_o	Total concentration of the particles, g
C_p	Specific heat capacity, kJ/(kg.K)
D, d_e	Pipe and particle equivalent diameters, m
D_e	Axial dispersion coefficient
$E(\theta)$	Equivalent of E(t) in a normalized form.
E(t)	Exit age distribution or RTD, min^{-1}
E_a	Energy of activation for flow, cal/g mole
$F(\theta)$	Cumulative RTD function with normalized time, dimensionless
F(t)	Cumulative RTD function, dimensionless
F_o	Particle centre-point lethality (min)
$Fr_{f,p}$	Froude numbers for carrier fluid and particle
Gr	Grashoff number
$GRe_{f,p}$	Generalized Reynolds numbers for carrier fluid and particle
H	Half concentration internal age (mid point concentration)
h_{fp}	Particle-fluid interfacial heat transfer coefficient, $W/m^{2o}K$
k	Thermal conductivity, W/m°K
L	Holding tube length (m)
m	Consistency coefficient, Pas^n / mass of carrier fluid, kg
M	Total concentration of the particles, g
M_f	Fill mass of the aseptic processing system, kg
m_o, n_o	Turian model constants
m_o, m_c	Casson yield stress, Pa, Casson viscosity, Pas
n	Number of CSTRs in series / flow behaviour index, dimensionless
Nu	Nusselt number
P	Pressure, Pa
Pr	Prandtl number
Q	Mass or volumetric flow rate of the fluid, kg/s or m^3/s
r	Radial co-ordinate, m
R	Tube radius, m / Universal gas constant, 1.987 cal/ (g-mol-K)
Re	Reynolds number
RT	Fluid or particle residence time, min /s
RT_{min}	Fluid or particle minimum residence time, min / s
t	Time, sec / min
T	Fluid or particle temperature at any time (°C)

T_c	Particle centre-point temperature (°C)
T_i	Initial temperature, °C
t_m	Mean residence time, min
T_r	Thermal processing reference Temperature (121.1°C)
T_s	Surface temperature, °C
T_{wall}	Heat exchanger wall temperature, °C
T_∞	Fluid medium temperature, °C
u	average axial velocity, ms^{-1}
U	Upper asymptote (upper limit particle concentration)
u_f, u_p	Carrier fluid and particle velocities (ms^{-1})
u_{max}	Fluid maximum velocity, ms^{-1}
u_{mean}	Fluid average velocity, ms^{-1}
x	A parameter associated with the growth curve equation (x = 1)/ Constant.
x, y, z	Cartesian co-ordinates
z	z-value (C°)
α	Thermal diffusivity, m^2s^{-1}
γ	Shear rate, s^{-1}
Γ	Gamma function
η_{ap}	Apparent viscosity, pas
θ	Normalized time (t/tm), dimensionless
$\rho_{p,f}$	Particle or carrier fluid density, kgm^{-3}
σ	shear stress , Pa
σ^2	Variance, min^2
σ_o	Shear stress constant, Pa
Φ	Function of
∇	Differential operator

Abbreviations

%SS	Percentage contribution to total sum of squares
CMC	Carboxymethyl cellulose
FPNRT	Fastest particle normalized residence time
FPRT	Fastest particle residence time
HNT	Half concentration normalized internal age
HRT	Half concentration internal age
HTST	High temperature short time
MPRT	Mean particle residence time
MS	Mean of sum of squares
RTD	Residence time distribution
SRTD	Standard deviation of RTD
SSHE	Scraped surface heat exchanger
UHT	Ultra high temperature

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CHAPTER I

INTRODUCTION

Aseptic processing is a high temperature/short time (HTST) process for continuous sterilization and packaging of foods offering the potential, at reduced costs, for the production of high quality foods. Aseptic processing, a commercial success for liquid food products, is currently gaining popularity and industrial interest with reference to low acid particulate foods. Aseptic processing consists of four distinct operations carried out simultaneously under a sterile environment using a mechanically synchronized process: 1) sterilization of the product under appropriate quick heating, holding and cooling; 2) sterilization of the packages; 3) aseptic filling of the product into the sterile packages; and 4) sealing of the packages under aseptic conditions (Woodroof, 1990). The following advantages have been recognized for aseptic processing over the traditional thermal process because it offers potential for: (1) HTST operation (2) energy savings (3) higher efficiency (4) faster and more uniform heat transfer (5) reduced variation in process parameters due to the high level of automation employed; and (6) lower production costs (Chandarana *et al.*, 1987a). Adaptation to new package forms, savings in packaging costs and convenience are also regarded as major advantages (Toledo and Chang, 1990). The major advantage envisaged in aseptic processing is the possibility of HTST processing which have been proved to yield better quality retention in processed products. However, there are possible disadvantages as well: (1) capital investment is initially high; (2) cost of training personnel may be substantially high; (3) more complex instrumentation control

required; and (4) destruction of certain heat resistant enzymes may be more difficult (Bernard *et al.*, 1987; Chandarana *et al.*, 1987b).

Aseptic processing technique has been successfully applied to liquid foods (milk and fruit juices) and to acid foods containing discrete particulates (Sastry, 1986; Sastry and Zuritz, 1987). The development and success in this area has brought about interest and attention to the aseptic processing of low-acid heterogeneous liquid foods containing discrete particulates (Heldman, 1989). However, the extension of aseptic processing to these foods has been difficult due to lack of data on critical factors such as interfacial heat transfer coefficient between the liquid and the particle (h_{fp}) as well as the residence time distribution of particles in the holding tube of the aseptic system (Sastry, 1986; Singh, 1987; Berry, 1989).

Experimental time-temperature data at the particle centre as it travels through the heat exchangers and holding tube are difficult to gather. Hence conventional thermal processing calculation methodology cannot be employed for the establishment and enforcement of these processes. Mathematical modelling followed by biological verification has been attempted as a possible alternative. These models require accurate data on the thermo-physical properties of the particles, associated fluid to particle heat transfer coefficient (h_{fp}) as well as residence time distribution (RTD), especially in the holding section of the system. Both h_{fp} and RTD depend on several factors which may also be interdependent: rheological properties, flow rate, temperature, and density of the carrier fluid, shape, density and concentration of the solid particles, as well as holding tube diameter and length.

Residence time distribution (RTD) or internal age distribution is defined as the length of time different elements spend in a particular system under consideration (Danckwerts, 1953). RTD for homogeneous fluids in typical aseptic processing systems has been extensively studied (Chen and Zahradnik, 1967; Heppell, 1985a; Sancho and Rao, 1992). The introduction of discrete particulates into fluid foods make their flow behaviour more complex. Not only are the residence times more difficult to measure, but also the associated variability is large and a function of many more factors (Berry, 1989; Pflug *et al.*, 1990; Ramaswamy *et al.*, 1992). Rheological properties of the carrier fluids have been shown to influence fluid velocity profiles in the holding tube, particle RTD in the heat exchangers and the holding tube, as well as the critical Reynolds number for turbulence considerations (McCoy *et al.*, 1987; Singh and Lee, 1992). RTD patterns of both carrier fluid and particles are expected to be different and specific for the system configurations. While the scraped surface heat exchanger (SSHE) is regarded as a perfectly mixed reactor, the flow in the holding tube is more likely to resemble that of plug flow reactor. Also in a typical aseptic processing system the effects of the SSHE and the holding tube on RTD cannot be individually evaluated, requiring the whole system to be evaluated as an integral unit.

The main objectives of this research were to:

(1) evaluate residence time distribution of food particles in starch solutions in a commercial scraped surface heat exchanger (SSHE);

(2) evaluate residence time distribution of food particles in the holding tube of a commercial pilot scale aseptic processing system;

(3) evaluate residence time distribution of food particles in starch solutions in an assembly of a whole commercial pilot scale aseptic processing system;

(4) conduct a biological evaluation of residence time using mesophilic microbial spores of *Bacillus subtilis* immobilized in carrot-alginate cubes in a commercial aseptic processing system;

(5) evaluate the rheological properties of starch solutions under aseptic processing conditions;

(6) evaluate the rheological properties of carboxymethyl cellulose (CMC) solutions under aseptic processing conditions; and

(7) evaluate flow dynamics of the food particles as influenced by the factors related to the fluid, particle properties and the processing system using dimensionless correlations.

CHAPTER TWO

LITERATURE REVIEW

Historical Perspective

The concept of aseptic processing originated as a measure of solving the problems associated with the conventional "in-container" sterilization of foods such as the low rate of heat penetration to the slowest heating point in the container, the long processing times required to deliver the required lethality, destruction of the nutritional and sensory characteristics of the food, low productivity and high energy costs (Smith *et al.*, 1990). Aseptic processing was initiated in 1927 at the American Can Company Research Department in Maywood, IL under the direction of C. Olin Ball (Mitchell, 1988). The result was the development of the HCF (*heat, cool, fill*) process for which Ball was granted a patent in 1936. The HCF process was targeted at pumpable liquid and semi-liquid foods. In 1938, two HCF units were installed for the commercial production of chocolate-flavoured milk beverage. The product was heated to 300°F in less than 15 seconds, immediately cooled (Ball and Olson, 1957) and filled into sterile cans. The process was not a commercial success because of the associated high cost of the equipment, inflexibility with respect to can size and the frequent blockages of the can-closing machine. Despite these problems, Ball is considered the pioneer of aseptic processing (Mitchell, 1988).

The Avoset process was another development in the field of aseptic processing credited to George Grindrod at the Avoset plant located at San Joaquin Valley of

California (Ball and Olson, 1957). A cream product was introduced under the Avoset label in 1942. This process is unique in that the filling and closing area were treated to eliminate bacteria and further protection was accomplished by ultraviolet (UV) lamps (Mitchell, 1988). The area was also enclosed by a wall with an opening for conveying the finished product. Sterilization was achieved by direct steam injection to a temperature of 260-280°F. The Avoset process is no longer in operation, but nevertheless it was another milestone in the development of aseptic processing.

Real progress in the commercial development of aseptic processing technology of foods began with the invention of the Martin-Dole process in the late 1940s in California by the Dole Engineering Company (Lopez, 1987). The process could be used for the sterilization of any low or high acid fluid. The technical success was not, however, accompanied by the expected high level of exploitation; the reason being the package, a metal can, had nothing to differentiate from the conventional "in-container" sterilized foods (Dennis, 1992). Product heating and cooling was achieved by heat exchangers based on the principles of high temperature/short time sterilization, while the containers were sterilized by superheated steam (Mitchell, 1988). The first commercial Dole system was installed in the early 1950s in California for the production of soups (split pea soup) and sauces (white, cheese and Hollandaise). Another historic development was brought about in the early 1960's by Loelinger and Regez of Switzerland who successfully used hydrogen peroxide (H_2O_2) to sterilize flexible packaging materials used in the manufacture of milk containers (Lopez, 1987). Obtaining better quality products than conventional processing was the main objective of Martin-Dole process, whereas extending the

shelf-life of fresh milk without refrigeration using low cost containers was the goal of Loelinger and Regez.

In the U.S., however, the use of hydrogen peroxide as a package sterilant was approved by the FDA only in February 1981 (Cousin, 1993). This approval provided the opportunity for the use of laminates for consumer-size packages. Currently, there are more than 500 aseptic systems in commercial operation in the U.S. (Nelson, 1993). Now aseptic containers come in many different sizes and shapes, from small packages to large 100,000 gallon bulk storage tanks. The bag-in-box technology has significantly grown in the past few years and its thrust has been in units ranging from 2 to 300 gallon units (Nelson, 1990). Their use and performance by the industry has been quite impressive.

A typical aseptic process involves sterilization of the product by a heat-hold-cool approach followed by filling and sealing into pre-sterilized containers under aseptic conditions. Product heating in aseptic systems can be performed directly (steam infusion and steam injection) or indirectly (plate, tubular or scraped surface heat exchangers) for pasteurization and sterilization of foods, whereas the containers are sterilized by superheated steam and/or H_2O_2 (Harte, 1987; Lopez, 1987; Lund, 1987).

ASEPTIC PROCESSING OF PARTICULATE FOODS

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

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

The real challenge, however, is the establishment of a microbiologically safe process for particulates, without sacrificing the essential quality factors. An appropriate thermal process can be determined if accurate time-temperature data measured at the slowest heating point within the largest and fastest particle flowing through the aseptic system is available. Thermal process establishment for liquid foods undergoing aseptic processing is simple because the liquid temperature could be measured at the point of interest (Rao, 1992). Heat penetration measurements for a food particle travelling through an actual aseptic processing system is almost impossible at the present time (Sastry, 1986; Lee and Singh, 1990), thus rendering the establishment of process times for particulate fluid foods a real challenge. Biological validation is considered as the most reliable routine to address safety issues, but the process is tedious and not entirely reliable (Pflug *et al.*, 1990). In the absence of experimentally determined values of temperature, mathematical modelling is the only alternative for establishing the aseptic process for particulate fluids (Heldman, 1992). Consequently, establishing a process for continuous sterilization of low acid particulate foods requires consideration of the following four aspects (Dignan *et al.*, 1989): (1) identification and selection of the appropriate

sterilization value; (2) development of a conservative model to predict the sterilization value achieved at the slowest heating location in a particulate by the time the particle leaves the holding tube; (3) quantitative microbiological validation of the lethality delivered; and (4) a list of the critical factors and the procedures to be used for controlling these factors. de Ruyter and Brunet (1973), Manson and Cullen (1974), Dail (1985), Heppell (1985b), Sastry (1986), Chandarana *et al.* (1989) and Lee *et al.* (1990) were among several researchers who have tried to address the problem. The most critical factors in mathematical models were identified as particle size and shape, the fluid particle heat transfer coefficient, and the residence time distribution (RTD) in the heat exchangers and the holding tube determined with actual food flowing in a real system (Dignan *et al.*, 1989; Heldman, 1989, 1992; Rao, 1992). In a continuous flow system as in aseptic processing, not all particles of the product remain in the processing equipment for the same time. The particles or parts close to the wall (boundary layer) or in dead space, move or flow much slower than those travelling in the centre of a flow passage (Danckwerts, 1953). Therefore, there is a distribution of residence times through the heat-hold-cool sections of the aseptic system, with small proportions of the total flow spending either significantly less or more than the mean residence time (Burton, 1988).

ASEPTIC PROCESSING SYSTEMS FOR PARTICULATES

Batch Processing Systems

APV Jupiter System

According to Hersom and Shore (1981) the food particles are heated and cooled

using a sequential batch method while the carrier fluid is processed in a conventional continuous heat exchange system. The particles are tumbled in a double cone aseptic processing vessel (DCAPV) which rotates about a central axis, into which steam is passed under pressure. Such a design and operational mode will provide a better heat transfer to the particles. The particles are heated by steam as well as its condensate. The steam in the double cone is replaced by sterile air, while the steam supply is replaced by cooling water. The vessel continues to rotate, cooling its contents consisting of food particles and the processed carrier fluid. At the end of the cooling cycle, the rotation of the vessel is terminated and the fluid is removed through a drain valve. The fluid may be retained for use in a subsequent batch or any other purpose as determined by the final product.

Another similar batch sterilization process was proposed by Sawada and Merson (1986) using a water-fluidized bed system for sterilizing spherical particulates for aseptic filling. The feasibility was analyzed, but using empirical equations for the heat transfer coefficient (h) originally used for air-to-particle heat transfer. No experimental work was carried out in a real processing system for verifications.

Steripart System

Single-flow fraction-specific thermal processing (Single-Flow FSTP) is a new technology developed by Stork of The Netherlands in 1985 (Hermans, 1991). The main objective was to increase the retention time of particulates by specially designed sieves. Particles are sieved from the carrier fluid in the hot hold section of the aseptic system, in order to increase their retention time. Time-temperature profiles of each of the fractions

(solid or liquid) are independently controllable with a high degree of flexibility and accuracy. According to Hermans (1991) and Dennis (1992) Single-Flow FSTP requires one or more of the so-called "Selective Holding Sections (SHS)" that can be defined as continuous time-adjustable temporary separating mechanisms to separate either a particulate fraction from the liquid, or to separate a size-fraction of bigger particulates from smaller ones including the liquid fraction. Two types of SHS are available: (1) Rota-Hold device that consists of a cylindrical vessel and can handle only two fractions. Fork blades are mounted on a central axis that rotate slowly at a controllable speed. The solids, unable to pass the blades, are conveyed around the cylinder until they are released at the exit port. The liquid can flow freely reaching the outlet in a short time. (2) Spiral-Hold consists of a vertically positioned tube shaped vessel, with in-feed at the top and the discharge at the bottom. A central shaft fitted with spirally mounted spokes rotates slowly at an adjustable speed. The inside of the tube also has spokes that are mounted spirally but in contrary pattern to those on the shaft. Moving pockets are formed between the spokes and the wall, carrying particles at a pre-determined rate through the system. By varying the thickness of the spokes, either in steps or gradually, the separating characteristics can be varied for different particle sizes imparting shorter residence times with decreased sizes (Anon, 1989; Dennis, 1992).

Continuous Sterilization of Particulates

Continuous process of sterilization has received much attention because of its success and effectiveness with liquid foods. This explains why only a few researchers

considered batch sterilization for low-acid liquid foods containing particulates. But there are some exceptions to this, as some batch processing systems were introduced for the treatment of liquid-particle suspensions (Jupiter and Steripart processing systems, discussed earlier).

The problem of particle integrity was best addressed using a scraped surface heat exchanger (SSHE) which is a double tube heat exchanger with the addition of a scraping mutator shaft located with the product tube in order to continuously scrape the heat transfer surface and increase the mixing turbulence. The equipment is perhaps ideally suitable for high viscosity products with or without particulates. The history of SSHEs goes back to 1928 when Vogt patented an ice cream freezer (Harrod, 1986). Currently, SSHEs are used for a wide array of food products such as rice porridge, pea soup and puddings. These heat exchangers have the ability to satisfy the demands for efficiency and labour saving processes because they are continuously operated. In a scraped surface heat exchanger, the mutator shaft rotates within the product tube (Figure 2.1). The product passes through an annulus formed between the blade shaft and the heat transfer tube, while the heating or cooling medium flows in a jacket. The unit is insulated to minimize energy loss and protect personnel. The insulation is, in turn, protected by a polished, easily cleanable stainless steel cover. The design may differ according to construction material, mixing performance, mechanical strength, dimensions, mounting (horizontal *vs* vertical) and accessability for cleaning and inspection. The capacity of the heat exchanger is determined by the heat transfer area, while the gap between the shaft and the heat transfer tube dictates the size of the food particles to be processed in the equipment

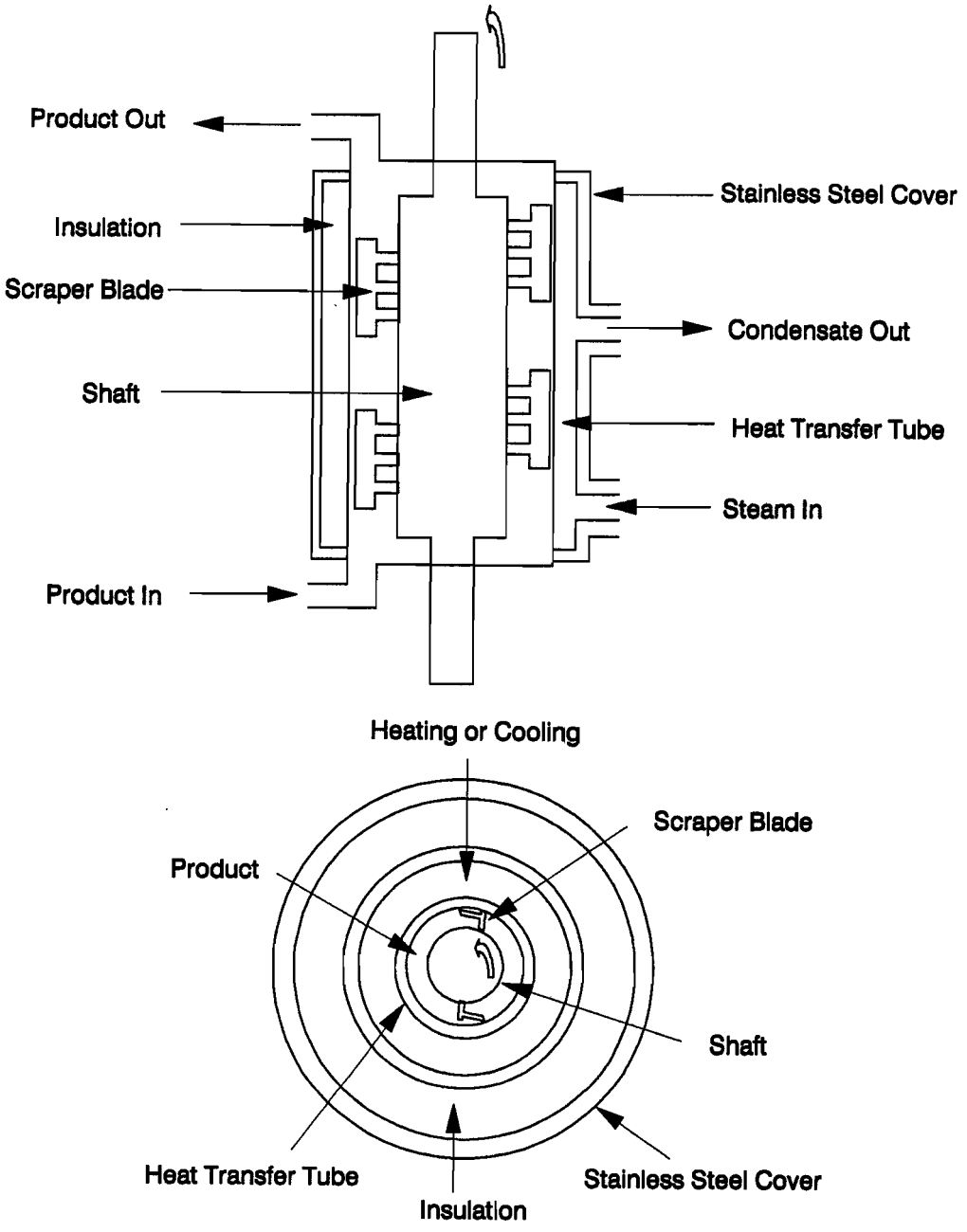

Figure 2.1. Schematic drawing of the scraped surface heat exchanger (SSHE).

(Hallstrom *et al.*, 1988). Information on some of the manufacturers, trade names and sizes of widely used SSHEs are given in Table 2.1.

The flow patterns in SSHEs are complex as they are influenced by: axial flow, length of the equipment, time, radial temperature differences, non-Newtonian fluids and blades (Harrod, 1986). These flow patterns can be divided into different flow regimes: the rotational flow which is characterized by a Taylor number (T_a) and the axial flow characterized by an axial Reynolds number (Re_{ax}). Depending on the magnitude of these numbers the exact type of flow in SSHEs can be identified (laminar, turbulent, vortex, plug flow, wavy vortex, modulated wavy vortex, ...etc.).

Requirements for Aseptic Processing of Particulate Foods

There are stringent regulatory requirements in North America for the commercial application of aseptic processing to low acid liquid foods containing particulates. The major concern has been due to difficulty of obtaining experimental time-temperature data required for predicting the sterilization value delivered by the thermal process. A graphical representation of the time-temperature profiles for *liquids* throughout the aseptic processing system is illustrated in Figure 2.2. With *particulates*, the prediction of these profiles is almost non-existent, although they are required to calculate accumulated process lethality (F_o):

$$F_o = \int_0^t E(t) \cdot 10^{(T_c - T_r)/z} dt \qquad (1)$$

where F_o is the particulate centre-point lethality (min), t is time (min), E (t) is the critical

Table 2.1. Market survey of the different types of scraped surface heat exchangers*.

Manufacturer	Trade name	Heat transfer area (m^2)	Gap (mm)	Diameter (mm)
Alfa Laval	Contherm	0.3-0.9	11-50	152
Cherry Burrell	Termutator	0.3-0.9	6-50	152
	votator			
	Vogt			
APV-Crepaco	Rota Pro	0.2-0.9	6-50	76-152
Schroder	Kombinator	0.1-1.5	5-15	60-336
Gerstenberg Agger	Prefector	0.3-0.8	6-14	105-180
Fran Rica	Fran Rica	1.1-4.7	10-75	305-610
Luwa	Thermalizer	0.3-18.0	----	100-1000
Lehmann	KBF	1.3	15	250

*After Harrod (1986)

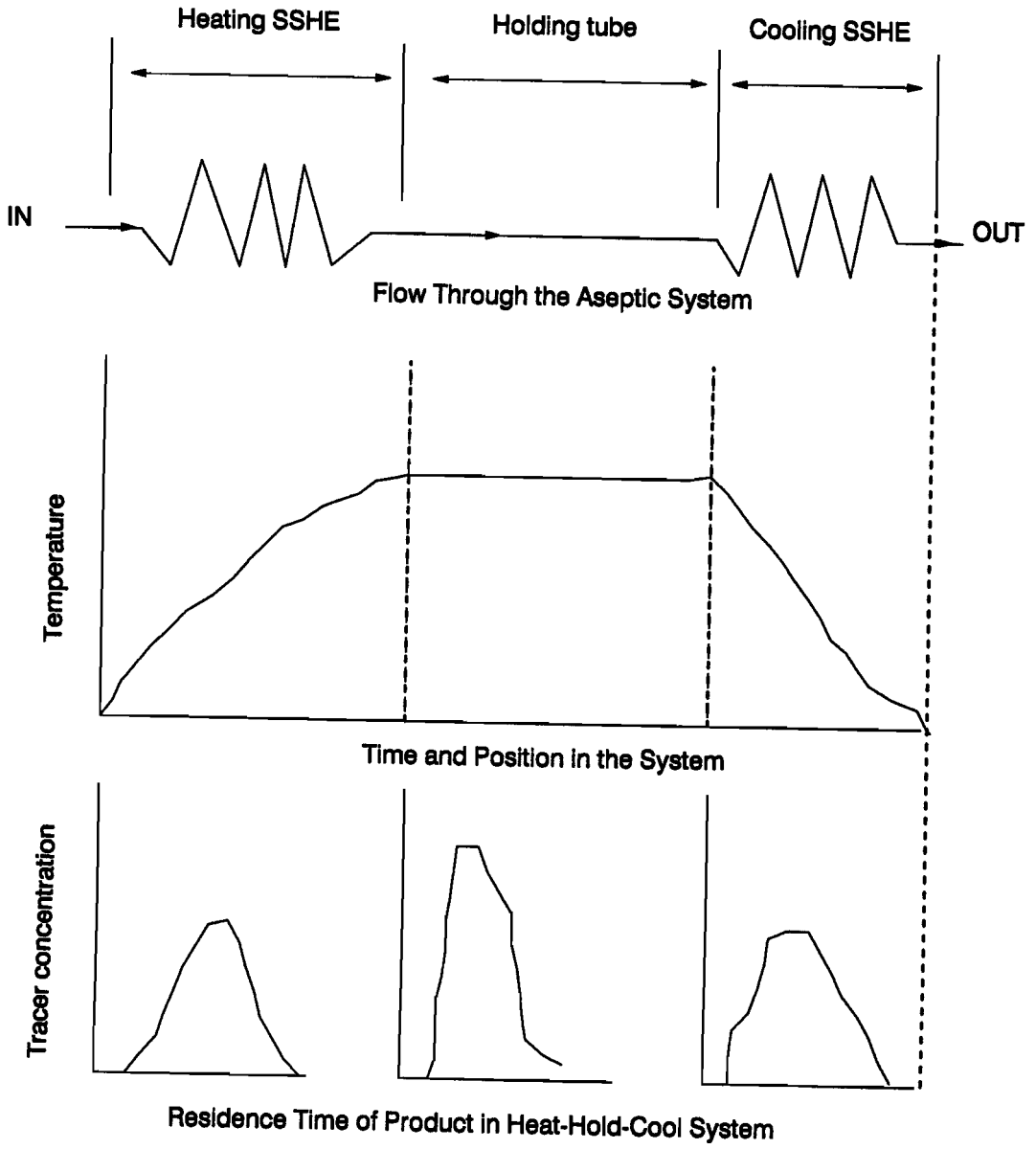

Figure 2.2. **A graphical representation of the time-temperature profiles for liquids in the aseptic processing system.**

particle residence time (fastest/largest), T_c is the particle centre-point temperature (°C), T_r is the reference temperature (°C), and z is the slope of the logarithmic thermal death time (TDT) curve (C°). The residence time of the fastest particle is critical to ensure that target lethality has been acheived. From a quality standpoint, residence time distribution (RTD) plays a prominent role as can be realized by the thermal-time distribution (TTD) obtained by coupling temperature history with RTD (Dutta, 1991). The design of thermal processes for particulates can only be addressed through mathematical modelling and biological validation. Modelling of thermal processing usually involves the solution to the conduction heat transfer involving a solid exposed to a surface convection:

$$\nabla . (k \nabla T) = \rho C_p (\delta T / \delta t) \qquad (2)$$

For constant thermal conductivity, Eqn 2 may be rewritten in scalar three-dimensional form (in Cartesian coordinates):

$$\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} = \frac{1}{\alpha} \frac{\partial T}{\partial T_t} \qquad (3)$$

subject to the time-dependent boundary condition:

$$k \nabla T . n = h_{fp} [T_s - T_\infty(t)] \qquad (4)$$

The use of mathematical models allows maximum flexibility concerning process alternatives and possibilities while minimizing the amount and cost of process testing necessary. A mathematical model is usually used to estimate the minimum residence time required under the specific conditions necessary to produce the desired lethality.

Information on the particle to fluid heat transfer coefficient (h_{fp}), particle size, particle shape and thermo-physical properties of the food particle are required. These are affected by the fluid flow rate, physical properties and dimensions of the holding tube, and the rheological properties of the carrier fluid (Chandarana *et al.*, 1989; Heldman, 1989). The associated heat transfer coefficient is critical, because it determines the time-temperature profile and hence the resulting lethality in the critical food particle. Most experimental measurements of the fluid-particulate heat transfer coefficients in heat exchangers and holding tubes involve stationary particles. The minimum time requirement predetermined by the model has to be coupled with RTD data for the aseptic system. It is necessary to ensure the largest and the fastest travelling particle will have a residence time at least as low as the model predicted time. Residence time distribution (RTD) of food particles is affected by factors related to: the particles themselves (type, shape, size, density and concentration); the carrier fluid (type, density, flow rate, concentration and rheological properties); the aseptic system (configurations, temperature, mutator speed and pumps). Both heat transfer and residence time distribution factors are inter-related especially with reference to the rheological properties of the carrier fluids. The degree of turbulence in an aseptic system is estimated from the dimensionless Reynolds number which has a viscosity term in it ($\{\rho\, u_f\, D\} / \eta_{ap}$). With non-Newtonian fluids, the viscosity is a function of shear rate (flow velocity), and the prediction of residence time distribution becomes more challenging with distorted parabolas being the likely result (Richardson and Selman, 1991).

Mathematical Models For Heat Transfer

Several mathematical models (de Ruyter and Brunet, 1973; Manson and Cullen, 1974; Dail, 1985; Sastry, 1986; Chandarana *et al.*, 1989; Lee *et al.*, 1990; Awuah *et al.*, 1993) have been developed to estimate the temperature at the centre-point of a particulate in a liquid food passing through the heat-hold-cool sections of an aseptic process. The design considerations were based on the largest-fastest moving particle.

The first trial at modelling aseptic processing of liquid foods containing particulates is credited to de Ruyter and Brunet (1973) who analytically solved the partial differential equation (PDE) for heat transfer (Eqn 3) to evaluate heating of a liquid food containing spherical particles in an SSHE based on the heat transfer analytical solution of Carslaw and Jaeger (1959). These authors assumed: an infinite convective heat transfer coefficient at the particle/carrier fluid interface, and the carrier fluid temperature changes linearly during heating, holding, cooling and discharge sections of the aseptic system. Besides, no reference was made to residence time distribution and its effects on process calculations.

The modelling attempts of Manson and Cullen (1974) emphasized the importance of residence time distribution (RTD) on process design for finite cylindrical food particles in SSHE and holding tubes. The underlying assumptions were: an infinite convective heat transfer coefficient at the particle/fluid interface, and the fluid temperature rises linearly during heating. Two possibilities were considered for the holding section: constant liquid temperature or exponential temperature drop; and a negligible heat loss from the surface of the holding tube. The approach using infinite h_{fp} has been generally criticized in most

studies due to possibility of gross overprediction of estimated lethality (Sastry, 1986; Awuah *et al.*, 1993).

Dail (1985) discussed the limitations of Ball's formula method as applied to aseptic processing of liquid foods containing discrete particulates and showed that the equations derived by the analytical solution of the partial differential equations (PDE) for heat transfer into a finite-sized body of regular geometry, may be coupled with Ball's equation to determine the lethality in continuously flowing particulates. The assumption of an infinite fluid to particle heat transfer coefficient will likely result in overprediction of particle temperature. But Dail proposed to compensate for this deficiency by determining an apparent thermal diffusivity for particles of sizes larger than those to be processed.

A more comprehensive approach was proposed by Sastry (1986) by using the Galerkin-Crank-Nicolson algorithm involving three-dimensional finite elements in space and finite differences in time to simulate the continuous aseptic processing of low-acid foods containing spherical particulates. The mathematical model of Sastry involves the determination of fluid temperature profiles over the heat exchanger and the holding tube, and the determination of temperature and lethality of the slowest heating location of an individual representative particle. Unlike de Ruyter and Brunet (1973) and Manson and Cullen (1974), this model assumed a finite heat transfer coefficient (h_{fp}) existing at the interface between the particles and the carrier fluid. For a spherical particle, a Nusselt number of 2 was assumed to represent the situation of conduction heating from a still non-moving carrier fluid.

Further studies by Zuritz *et al.* (1987) and Heppell (1985b) indicated that the

magnitude of the surface heat transfer coefficient had a small finite value (548-1,175 $W/m^2{}^oK$). Thus, there were two problems to be considered in the calculation of the heating rate of particulates in a flowing liquid (Hallstrom *et al.*, 1988): the establishment of the heat transfer coefficient in the system under investigation, and the actual driving force which is the temperature difference in this case. Generally speaking, the analytical solution of the energy equation for a spherical particle in a motionless fluid gives:

$$Nu = 2 \tag{5}$$

This relationship assumes pure conductive heat transfer, since no convection can take place because the temperature difference is infinitely small. Considering the movement of the carrier fluid relative to the particulates, either as a result of a temperature-induced density gradient (natural convection) or as a result of particles not having the same velocity as the bulk of the liquid (forced convection) implies that Eqn 5 is no longer valid. Consequently, for a liquid in a laminar flow state, correlations adopted are of the form (C being a constant):

$$Nu = C.\,Gr^{x}\,Pr^{y} \tag{6}$$

for pure natural convection, and:

$$Nu = C.\,Gr^{x}\,Pr^{y} + 2 \tag{7}$$

for pure forced convection, with x and y in the order of 0.25-0.33. For low velocities, relative to temperature gradients, mixed convection takes place and the following correlation can be developed:

$$Nu = C.\, Gr^{x} Re^{y} Pr^{z} + 2 \qquad (8)$$

Eqn 8 has a theoretical background to it. If the velocity is increased, the influence of natural convection decreases and may be neglected and the exponent of Re & Pr becomes 0.33. For higher particle velocities, the boundary layer becomes unstable as the inertial forces are too large relative to the viscous forces and turbulence is a reality. The exponent of Reynolds number then changes from 0.33 to above 0.5:

$$Nu = 1 + C.\, Gr^{0.58} Pr^{0.33} \qquad Re > 10^{5} \qquad (9)$$

thus heat transfer will only be affected by turbulence. The extent of this influence is mainly dependent on the relative turbulence intensity and the dimensions of the eddies responsible for the heat exchange relative to the size of the particulates. The particulate integrity, however, is more effectively maintained with laminar rather than with turbulent flow in the holding tube.

Another effort to model the convective heat transfer coefficient at the particle/fluid interface in aseptic processing systems was made by Sastry *et al.* (1989). Their experimental procedure involved a specially constructed metal transducer particle with a thermocouple attached to it. Special care was taken to ensure that the particle had the same behaviour as that of a free food particle. The h_{fp} was determined using Newton's law of heating with a Biot number less than 0.1:

$$\frac{m C_p}{A} \ln \left[\frac{(T - T_\infty)}{(T_i - T_\infty)} \right] = -h_{fp}.\, t \qquad (10)$$

and an h_{fp} of up to 2507 W/m^{2o}K was obtained for a flow rate of 10.6 gal/min. However,

more experimentation is needed with fluid foods of non-Newtonian behaviour.

Larkin (1989) argued that the Ball formula method cannot be used to model the time-temperature profiles for simulated aseptic processes due to the existence of a tailing effect in the semilogarithmic heating profile. Ball assumed that the heating profile was completely linear and assigned a value of 1.41 for the initial cooling lag factor (j_c) by marking the temperature difference between the food centre and cooling water. Larkin's modifications were based on the observation that the shape of the curved portion (transition from heating to holding) varied considerably over the difference process conditions making the use of a constant heating or cooling lag factors impractical. The modifications involved by making: (1) the hyperbolic function straight line asymptote to the linear portion of the semilogarithmic plot of the time-temperature data, and (2) the power of the hyperbolic function a variable instead of the equation-specific constant 2.

Chang and Toledo (1989) showed that the heat transfer coefficients (h) between a sweet potato cube and water were 239 and 303 $W/^{2o}K$ at 0 and 0.86 cm/s relative velocity, but in a 35% sucrose solution, an h value of only 146 $W/m^{2o}K$ was obtained. Rheological properties such as the consistency coefficient, flow behaviour index and time dependency (thixotropy) have been shown to have serious implications on the modelling aspects of flow properties of viscous foods (Cheng, 1987; Harrod, 1989a,b,c; Dail and Steffe, 1990a,b). According to Chang and Toledo (1990), a batch process may be more easily implemented than a continuous one, particularly when the particles are relatively large and the particle to fluid ratio is high. Chang and Toledo employed the finite difference modelling technique to determine the heat transfer coefficient (h) and the

thermal diffusivity simultaneously under non-constant heating temperature profiles. An assumption was made that the fluid temperature in the scraped surface heat exchanger increases exponentially as indicated in Eqn 11:

$$T = T_{wall} + (T_1 - T_{wall})\ (e)^{-Ct} \qquad (11)$$

where T fluid temperature at any time ; T_{wall}, the heat exchanger wall temperature; T_1, the initial fluid temperature; t, the come-up-time in the heat exchanger; and C, a constant calculated on the basis of the mean residence time in the heater. This assumption is similar to the assumptions made by Larkin (1989), but is more simplified and straight forward once the value of (C) is computed. Chang and Toledo (1990) concluded that particulate sterilization using high temperature/short time technology is possible using batch sterilization by pumping hot fluid through a bed of solids, since the major factor which prolongs the holding time for sterilization in a holding tube of continuous system was the drop in fluid temperature as heat was absorbed by the particles. Further investigation is required to evaluate this process in terms of energy requirements, nutrient retention, non-Newtonian flow behaviour and the size/shape factors of food materials.

Residence Time Distribution (RTD)

RTD of food particles in the heat exchangers and the holding tube of an aseptic system is an integral part of the thermal process establishment, since the temperature at the slowest heating point of a particle is a function of both time and heating rate (Manson and Cullen, 1974; Taeymans *et al.*, 1986, Lee and Singh, 1991). Residence time distribution [E (t)] is defined as the fraction of material in the outlet stream that has been

in the system for times between t and $t+dt$. RTD can be used as a diagnostic tool to precisely determine and give reasonable predictions of the performance of aseptic systems. RTD of food particles is thus a system parameter that is essential for the final product specifications. RTD is influenced by various inter-related, interdependent factors originating from the food material and the processing system. The food factors may be further subdivided between the carrier fluid and food particles. The fluid factors include: type, concentration, density, flow rate and rheological properties; while the particle factors are: type, size, shape, density and concentration with reference to the carrier fluid. On the other hand, the system factors include: temperature, pumping system, mutator speed and configuration, heat exchanger orientation, holding tube length and diameter. With such a complicated system, the assumptions of either a plug or laminar flow regimes, and that the residence times can be calculated from volume and flow rate for process design purposes would be unacceptable (Janssen, 1994). It is, therefore, necessary to measure the RTD of food particles in the specific aseptic system under consideration.

RTD for homogeneous fluid foods flowing in a typical aseptic system have been extensively studied (Heppell, 1985a). However, introduction of particulates to liquid foods renders the flow conditions to be more complex. While the fastest particle residence time is required for process calculations, the residence time for particle-bulk will determine the extent of quality factor retention.

Only limited experimental data are available on fluid/particle flow in aseptic systems (Sastry and Zuritz, 1987). For aseptic processes of low-acid particulate foods, the critical factors are: the relationship between particle size and the distribution of liquid

velocity within the heat exchanger and the holding tube. The type of heat exchanger, its orientation, as well as the configuration of the holding tube and are also critical factors in relation to the flow characteristics and consequently the residence time distribution (Lalande *et al.*, 1991). Recently, several research groups have investigated the RTD of simulated and real food particles in the SSHE (Taeymans *et al.*, 1985, 1986; Alcairo and Zuritz, 1990; Singh and Lee, 1992; Lee and Singh, 1991, 1993) and the holding tube (McCoy *et al.*, 1987; Nesaratnum and Gaze, 1987; Sastry and Zuritz, 1987; Berry, 1989; Segner *et al.*, 1989; Dutta and Sastry, 1990a, b; Hong *et al.*, 1991; Palmeiri *et al.*, 1992; Ramaswamy *et al.*, 1992). Most of these studies were performed using only one part of the aseptic system (SSHE or holding tube) at below boiling temperatures under atmospheric pressure conditions.

Biological Validation

Microbiological verification of mathematical models is an essential step in thermal process evaluation. Thus, biological indicators are required for validating continuous sterilization processes. A general requirement in biological validation is that the microbiological procedures be carried out quantitatively (Pflug *et al.*, 1990) . Inoculated particles (Sastry *et al.*, 1989; Cerney *et al.*, 1989; 1990), simulated particles (Hunter, 1972; Dallyn *et al.*, 1977; Bean *et al.*, 1979; Ronner, 1989; 1990) and chemical index markers (Mulley *et al.*, 1975; Berry *et al.*, 1989; 1990, Kim and Taub, 1993) have also been used with limited success.

Rheological Properties of Carrier Fluids

Rheological properties determine the velocity profiles in tube flow (Steffe, 1992). Velocity profiles are essential for developing a clear understanding of instrument performance and process calculations. Sizing of the holding tube length in a thermal processing system is one example. Velocity distribution of liquid-particle mixtures within the heat exchangers and the hold tube is critical for heat transfer and fluid flow. Product flow in pipes is considered to be either plug, turbulent or laminar flow. In plug flow, all components flow at the same rate as a uniform mass. Thus, the velocity profile is flat, which rarely occurs. In laminar flow, the velocity profile is parabolic with the fastest particle at the centre theoretically moving at twice the average rate. For turbulent flow, the fastest particle is also at the centre, and the relationship between the fastest particle and the average is assumed to be about 1.25 times the average flow rate. But all these flow regimes are determined through the rheological properties of fluids which are in turn dependent on the processing conditions. Unfortunately, not much is known on the viscosity of fluid foods under aseptic processing conditions, a reason being the difficulty of viscosity measurements under pressurized conditions (Rao, 1992).

RTD of Food Particles in Aseptic Processing Systems

In aseptic processing systems not all fluid and food particles remain for the same time inside the system, meaning that the particles have a distribution of residence times (Rao, 1992). Thus residence time distribution (RTD) can be defined as the length of time that different elements spend in a system. Residence time of the fastest moving particle

in the heating and the holding sections of the aseptic system is critical from a public health standpoint. Of equal importance is the determination of mean and maximum particle residence times (Singh and Lee, 1992). Since the main focus of this study is the RTD of food particles, it will be discussed in some detail with respect to theory, method of determination and modelling.

Theoretical Background of RTD

Residence time distribution can be described by an E or F function through the introduction of tracer particles at the entrance of the system and enumerating them at the exit end (Levespiel, 1972). Typical inputs for the RTD determination are the step, pulse, sinusoidal, ramp and random sequence (Figure 2.3). Although similar information can be obtained with these different techniques, the latter two were not frequently used. The principal reason for employing the step, pulse or sinusoidal inputs in RTD analysis is the convenience and ease of the mathematical analysis. Moreover, it is easier to establish the output response for a given model when these functions are used as inputs. While employing liquid systems, the RTD can be determined by injecting a tracer dye into the system at the entrance and measuring the change in the dye concentration with time at the exit. It is virtually impossible to produce an exact step function experimentally, but it can be reasonably approximated by an input with a fast rise time compared to the process response time. The advantage of the step and impulse inputs is that all the information about the process dynamics is contained in the response to a single input; thus, the experimentation is more economical. The major disadvantage is that the necessary process

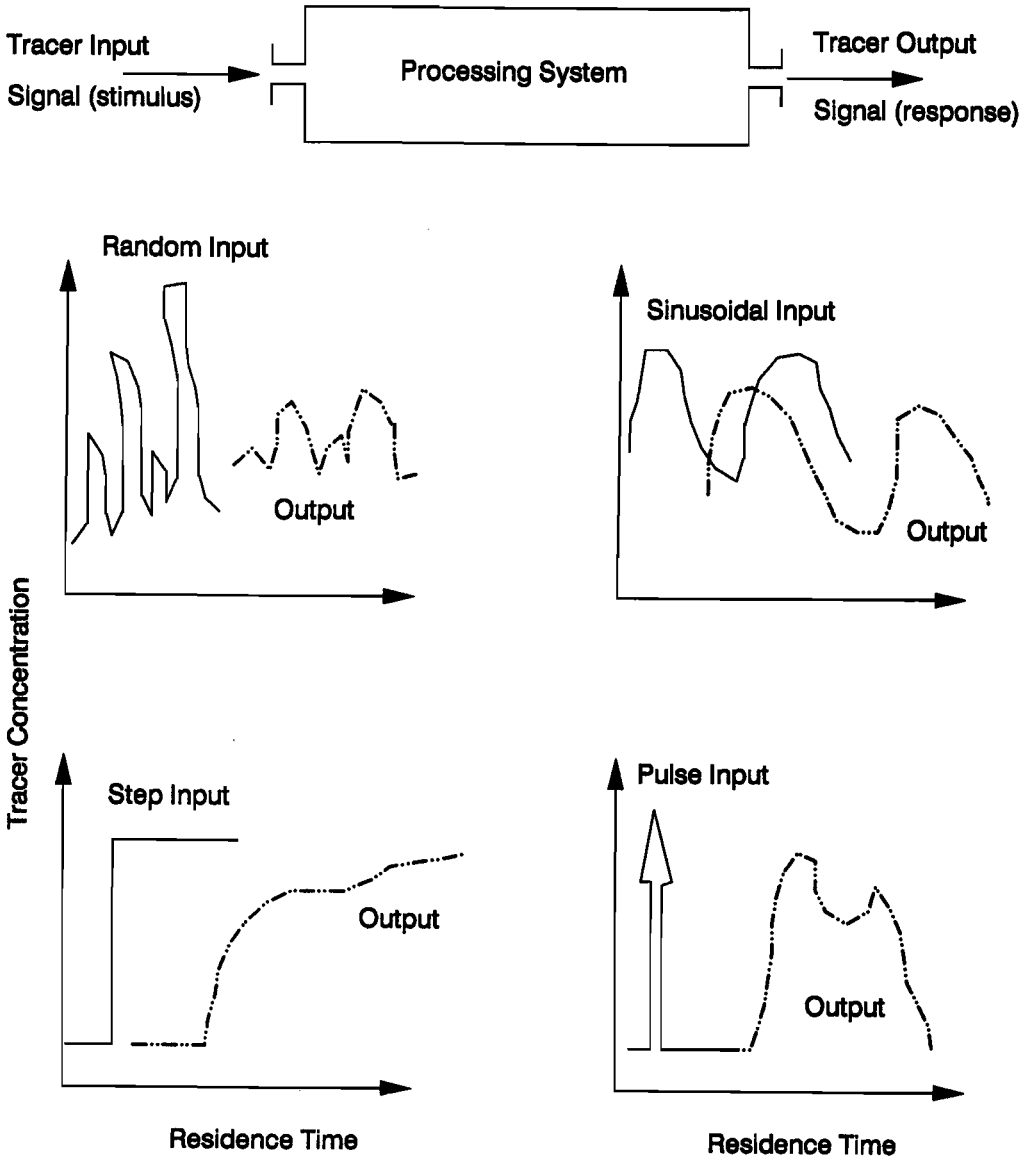

Figure 2.3. **Typical inputs for the RTD determination in continuous processing systems: (the random, sinusoidal, step, and pulse inputs).**

information is packed into a small amount of output response, and if some noise is present, much of the fine details will be obscured especially when the analysis is performed by the cumulative density function (*F*).

The *E* function gives the RTD of the fluid and particles or exit age distribution for any non-ideal flow. Residence time distribution [E (t)] is defined as the fraction of material in the outlet stream that has been in the system for times between *t* and *t+dt*. A typical *E* curve is shown in Figure 2.4. Mathematically, the *E(t)* curve is given by:

$$E(t) = \frac{c(t)}{\int_0^\infty c\,dt} \approx \frac{c(t)}{\sum_0^\infty c\Delta t} \qquad (12)$$

The *F* function is related to the *E* function by measuring the output response when a step change of tracer is introduced. Consequently, the *F* function, which represents the accumulation of particles at the exit with a residence time of t seconds or less (Figure 2.4), is given by:

$$F(t) = \int_0^t E(t)\,dt = \frac{\sum_0^t c\Delta t}{\sum_0^\infty c\Delta t} \qquad (13)$$

The mean residence time which is used to indicate the location of the distribution can be obtained from the following mathematical expression:

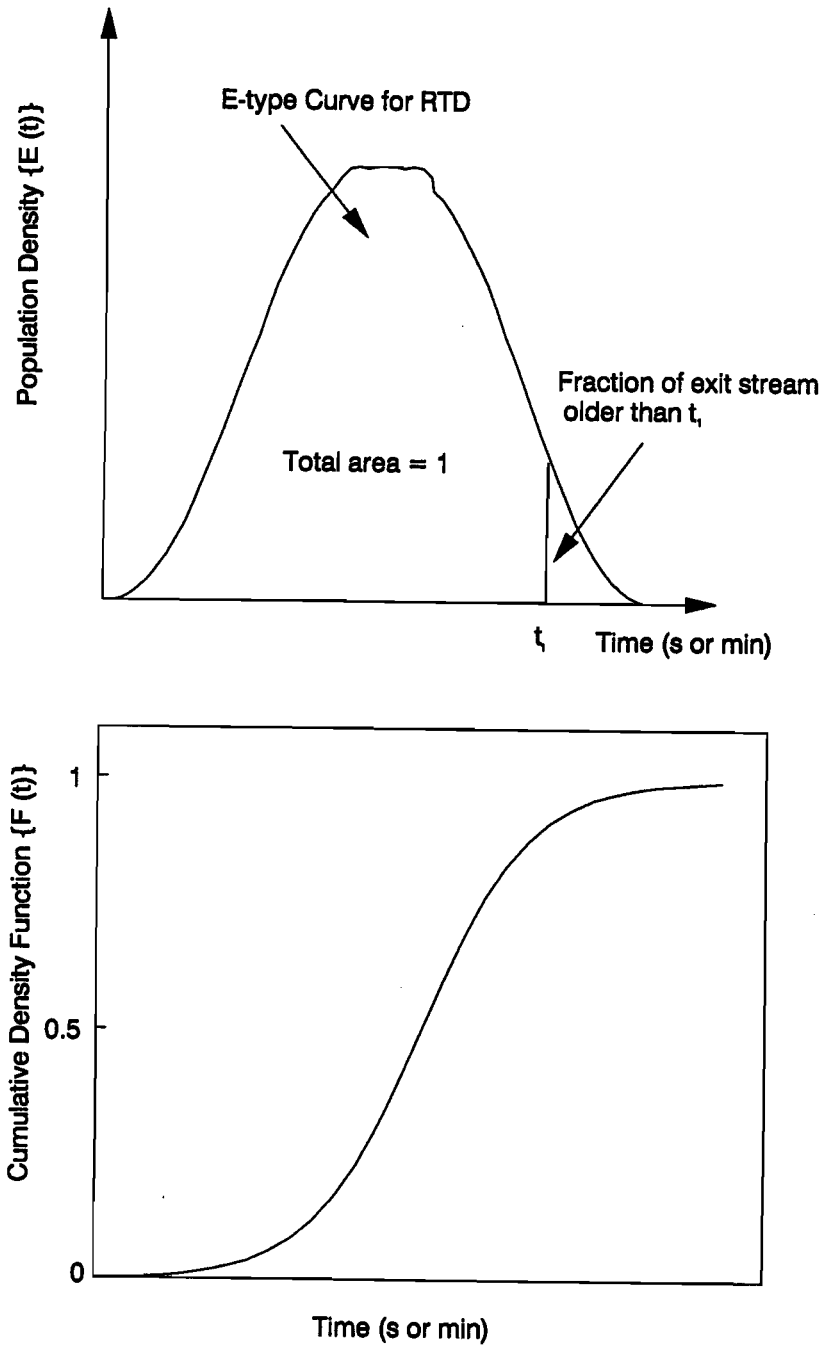

Figure 2.4. Typical *E* and *F* curves for RTD studies in aseptic systems

$$t_m = \int tE(t)\,dt \approx \frac{\sum_0^\infty tc\Delta t}{\sum_0^\infty c\Delta t} \qquad (14)$$

and the variance which is used to measure the spread of the distribution can be calculated by:

$$\sigma^2 = \sum_0^\infty (t - t_m)^2 E(t) \qquad (15)$$

Both the mean and the variance are characteristics of the RTD curves. The mean residence time can also be obtained from the fill mass of the system (M_f) of whole aseptic processing system mass and the mass flow rate (Q):

$$t_m = \frac{M_f}{Q} \qquad (16)$$

This equation requires the satisfaction of two conditions: (1) there must be no change of density of the flowing stream as it passes through the system and, (2) no back-mixing in the system upstream past the feed pump, otherwise the product stream will back-up into the system (Denbigh and Turner, 1984).

It is often useful for comparative purposes to define a dimensionless time variable which is the ratio of the particle (or fluid) residence time (t) to that of the mean residence time of the carrier fluid (t_m):

$$\theta = \frac{t}{t_m} \qquad (17)$$

It is worthwhile mentioning that the theoretical analysis of residence time distributions

(RTD) are usually based on the ideal assumption of either plug flow or perfect mixing conditions expressed by, for plug flow:

$$F(t) = 0; \quad t < t_m \tag{18}$$

$$F(t) = 1; \quad t \geq t_m \tag{19}$$

and for perfect mixing:

$$F(t) = 1 - e^{-t/t_m} \tag{20}$$

The required condition for plug flow which usually takes place in tubular reactors is that: over any cross-section normal to the fluid motion the mass flow rate and the fluid properties are uniform; and there is only negligible diffusion relative to the bulk flow (Denbigh and Turner, 1984). Thus, the velocity profile is distinctively flat, meaning that all the fluid elements have the same residence time.

A perfect flow model usually prevails in continuous stirred tank reactors (CSTR) into which there is a continuous flow of reacting material, in such a way that the fluid inside the reactor is thoroughly mixed and uniform throughout to an extent that the exit stream has the same composition as the mass within the reactor. A fair approximation to perfect mixing can be easily achieved in a CSTR, provided that the fluid phase is not too viscous. The advantages of the CSTR were listed by Denbigh and Turner (1984) as: the simplicity of construction (ease of cleaning the internal surfaces especially when the material has the tendency for deposition), and ease of temperature control. This is quite important, particularly for aseptic processing of liquid particulate foods as fairly uniform heat distribution can be ensured with less fouling problems and efficient heat transfer.

Several studies on RTD in continuous sterilization and pasteurization systems of foods have been reported (Chen and Zahradnik, 1967; Roig *et al.*, 1976; Lin, 1979; Heppell, 1985a; and most recently Sancho and Rao, 1992). These studies, however, were only applicable to liquid foods and do not consider the two-phase flow. RTD for homogeneous liquids can be easily determined by two widely used techniques: the radioactive tracers and dyes. A radioactive technique using MnO_2 was developed and introduced by Wolf and White (1976) to study the RTD in extrusion processing. According to Fichtali (1990), this technique can be utilized in two ways either by directly using labelled radioactive material or by activating the tracer in the extrudate after processing. The major disadvantage with these methods is the requirement for either a scintillation counter and/or a nuclear source (i.e. a Slowpoke reactor) to activate the sample in addition to safety and hazards concerns. However, these methods are accurate in detecting even low concentrations of tracer outputs. Dyes have also been widely employed to evaluate the effects of process variables on RTD. Employing dye technique offers simplicity of analysis and the possibility of visualization allowing easy monitoring of the output. Other approaches such as using soluble salts (KNO_3, $NaNO_3$, NaCl) or metal oxides (ZnO_2) have also been reported, which in turn were subsequently analyzed by conductivity or atomic absorption, respectively (Sancho and Rao, 1992; Lin, 1979).

Solid/liquid two-phase flow have been well discussed with reference to long distance transportation of mineral slurries. These studies mainly dealt with (1) high density solids, (2) Newtonian carrier fluids, (3) small particle-to-pipe diameter, and (4) the predominance of turbulent flow conditions. According to Zandi (1971), in a review

focusing on the transportation of bulky materials, the flow of two-phase systems can be grouped into five categories: homogeneous, heterogeneous, intermediate regime, saltation and capsule flow. Homogeneous flow occurs when fine and light particles are encountered, or when the mean velocity of flow is sufficient to keep a uniform suspension throughout the flow vessel. Heterogeneous flow is characterized by large density gradients in the flow to the extent that the two phases behave like separate streams due to the existence or the presence of course-dense particles. An intermediate flow regime is the direct result of the co-existence of the conditions of both homogeneous and heterogeneous types of flow. Saltation flow occurs when the particles form a bed at the bottom of the flow vessel and proceed in discontinuous jumps. In capsule flow the solids are packed in cylindrical capsules of diameters slightly smaller than the internal pipe dimensions and are transported into series.

None of these flow regimes would exactly describe the flow of liquid/food particle mixtures, but Sastry and Zuritz (1987) argued that the capsule flow is of relevance to aseptic processing especially when particles having sizes of 20 mm and above are to be considered. Very little is known about the two-phase flow under bounded regions such as in aseptic processing systems where the carrier liquid medium is usually non-Newtonian in nature and the particles are heterogeneous in composition and irregular in shape. The co-existence of flow models from the three ideal flow reactors (ideal complete mixer, plug flow system and dead space) in the case of liquid/particle food systems could be a possibility (Hallstrom *et al.*, 1988). This can be easily justified for a typical aseptic processing system, as the scraped surface heat exchangers (SSHEs) represent the ideal

mixer, holding tube is the plug flow system, while the accumulation of particles in some parts of the system can be considered as a promoting factor for dead space formation. However, this claim requires satisfying two conditions: experimental residence time distribution models or functions (verification and validation) and real or intuitive knowledge of the flow patterns (analysis and interpretation). Unfortunately, heterogeneous flow models rely on the assumption that the flow patterns of the two phases differ from each other. The fact that the two phases (solid and liquid) behave differently from each other cannot be over-emphasized, but they cannot be independent of each other as the drag, shear, and gravity forces developed might be contributing to the momentum balance between the two phases (Hallstrom *et al.*, 1988).

RTD Determination of Food Particles

A vast interest has recently been focused on RTD of liquid foods containing particles in aseptic processing systems, however, particles large enough in size and whose densities were close to that of the carrier fluids have not yet been considered for particle RTD research. Most studies were performed using simulated particles such as the polystyrene spheres, alginate beads, and rubber cubes. There have been numerous techniques developed for studying and investigating the RTD of solid/liquid two-phase flow (Yang and Swartzel, 1991; 1992). These methods include visual observation, photography, laser beam, play-back videotaping, radioactive tracer and magnetic response. These different techniques require the use of different experimental set-ups and various types of particles and liquid media.

Visual Observation: The technique of visual observation has been adopted and used by Toda *et al.* (1979) to investigate the transition velocities of commercial spherical and non-spherical glass particles, iron sand, calcite, and basic lead acetate in horizontal solid-liquid two-phase flow, and Kim *et al.* (1986) for sand slurries containing non-spherical silica particles (ρ = 2634 kg/m^3) in order to determine their minimum velocities. These investigations focused on bulk particle flow characteristics in non-food systems. However, McCoy *et al.* (1987) were the first to employ visual observation with the help of a stop-watch to investigate the RT of simulated food particles in a transparent polystyrene tube as influenced by particle size, density, and initial input location of the particle. They have used the "*one particle at a time*" approach for their investigations. Video taping has been used by Berry (1989) to monitor rubber cubes (6, 10 and 13 mm with the specific gravity of 1.08) flowing in carboxymethyl cellulose (CMC) solutions in a fabricated holding tube, and Dutta and Sastry (1990a,b) who used spherical polystyrene particles with a diameter of 0.95 cm (ρ = 1044.5 kg/m^3) suspended in CMC solutions flowing in a holding tube. Using a laboratory fabricated apparatus, Ramaswamy *et al.* (1992) were able to carry out studies on the flow behaviour of different kinds of particles (potato and carrot cubes, polystyrene spheres) suspended in water and starch solutions at elevated temperatures with the help of a video camera and/or visual observation. These studies were useful in determining the behaviour of individual particles and gaining some knowledge and insights on particle-particle and particle-wall interactions.

A photographic method was developed by Ohashi *et al.* (1979) for studying solid/liquid flow of small diameters (321-807 μm) in the density range 1190-1390 kg/m^3. They employed a stroboscope, slit and a camera to determine the instantaneous local velocities of the particles as they underwent an upward flow in vertical tubes. The photographic method, however, is somewhat similar to video taping though it operates in a discontinuous fashion.

Visual observation can be used in a manual mode (i.e. in the absence of an instrument) or instrumentally with the assistance of a high speed photography or a video camera. The obvious disadvantage of the manual visual observation is the subjectivity and vulnerability to human error and fatigue after performing a number of experiments. Moreover, it is difficult to visualize particles in opaque carrier fluids and non-transparent flow vessels, besides the difficulty of monitoring and tracking more than one particle at a time. Instrumental visual observation, on the other hand, offers the advantage of reducing the human error and the possibility of following many particles provided that both the carrier fluid and the tube are transparent. However, video taping is a time-consuming process when the video tape is back-played slowly for analysis, and the flow vessels and the carrier fluid have to be transparent to allow particle tracking.

Visual observation (manual or instrumental) can be used to observe and record particles only from a distance of the transparent observing field. The exact location of particles in the cross-sectional direction may not be possible. As a result, the determination of the particle trajectories may be difficult to interpret and explain especially under rapid flow conditions and non-transparent carrier fluids (Yang and

Swartzel, 1991). Also using transparent tubes of glass and plastic which cannot withstand high pressure/high temperature conditions would limit the maximum allowable temperatures. With some types of glass, high temperatures may be used, but haze can develop and limit the particle visibility.

Laser Beam: The use of a laser beam technique was developed by Ohashi *et al.* (1980) to evaluate the average particle velocities in solid-liquid two-phase flow through vertical and horizontal tubes. The technique has the advantage of convenience and precision in determining particle local velocity and concentration, over the photographic conventional method. However, the optical instruments and laser beam signal processing systems are quite expensive.

Radioactive Tracers: Using radioactive tracers can help to alleviate some of the problems associated with the visual observation methods. Toda *et al.* (1973) used radioactive particles as tracers to investigate particle velocities in solid-liquid two-phase flow through straight pipes and bends. Scintillation probes were used to detect the intensity of gamma-rays of the particles as they passed. The same was performed for the bends as the two scintillation probes were placed at the inlet and outlet of the bend. Particle velocity can be easily estimated by dividing the trajectory length for the particle between the two scintillation probes by the time of residence. The major concern of this technique is the health hazard involved with radioactive materials and their disposal, besides only one particle can be handled at a time.

Magnetic Response: Segner *et al.* (1989) introduced a small piece of magnet into the geometric centre of a turkey cube in a starch-based gravy carrier medium flowing in a holding tube of a commercial aseptic system. The magnets were constructed from a 3 mm thick flexible magnetic fabric with dimensions of 3 x 3 x 6 mm with a weight range of 0.1-0.2 g. A copper coil was wound at the exit and entrance of the holding tube. These coils were wired to an amplifier and connected to a double pen potentiometer. As the turkey cube entered the holding tube, and passed the copper coil, the change in the electromotive force is recorded. The same response is repeated at the other coil and the difference in time between the two responses is taken as the particle residence time. The advantages of this technique are the precision and the possibility of detecting irregular particles. However, it is difficult to determine the location of the particle at the cross-section using this method. Also, the embedded magnets contribute an additional weight to the particle resulting in density changes, besides the limitation of handling only one particle at a time.

Most recently, the above method was modified by Tucker and Richardson (1993). The principle of the method is based on the detection of magnetic particles flowing in a stainless steel pipe using the Hall effect sensors (Bueche, 1986). These sensors are capable of detecting the presence of a magnetic field. The Hall effect sensors are also capable of operation through the pipe walls at a range of up to 40 mm, using pieces of ceramic magnetic material of approximately 5 x 5 x 2 mm. The set-up of these sensors around the holding tube is presented in Figure 2.5. These magnets can be embedded into a variety of food particles like carrots, potato and meat as well as non-food materials

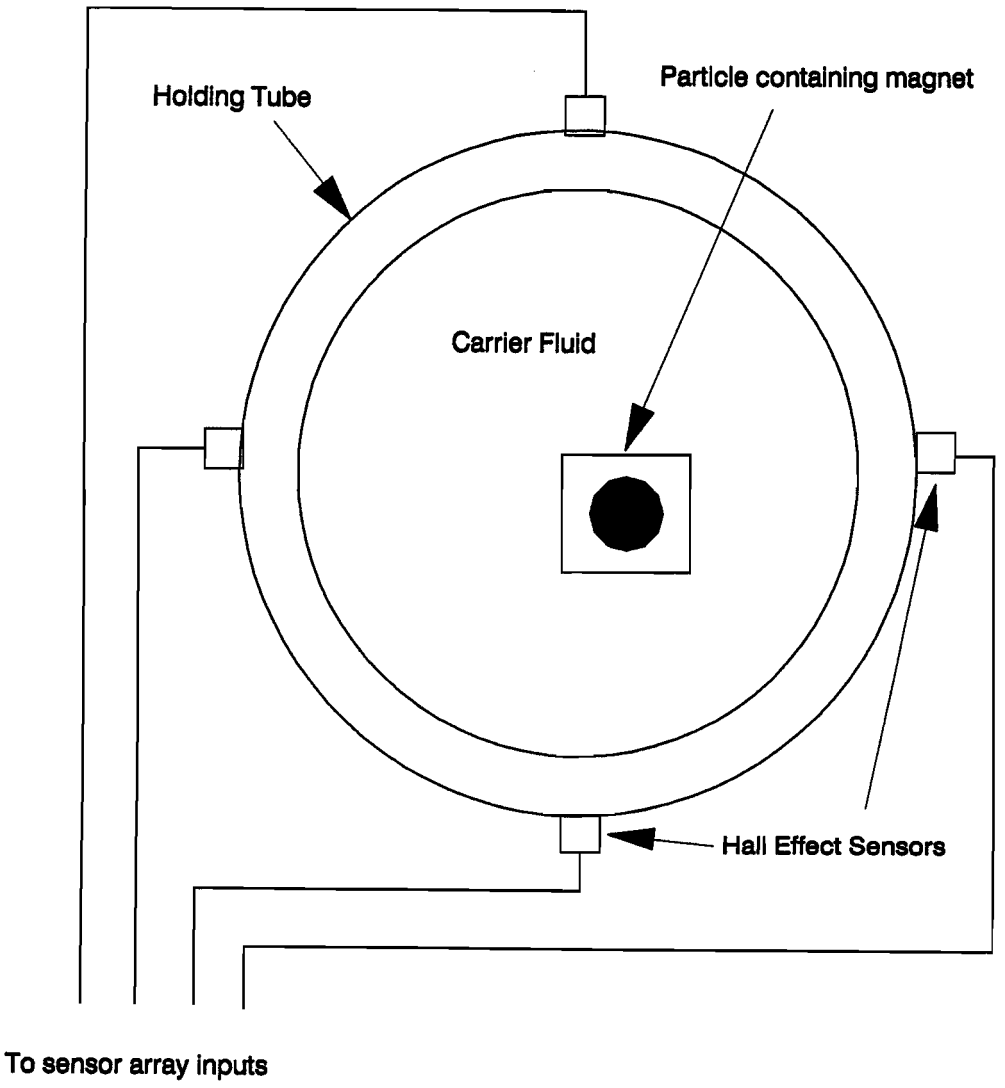

Figure 2.5. The set-up of the Hall effect sensors around the holding tube.

such as adhesive tapes, silicon elastometers and heat shrink sleeving (Tucker and Withers, 1992). The advantage of the Hall effect sensors is that the output varies linearly with the strength of the magnetic field, i.e. the closer the magnet to the sensor; the greater is the output voltage. Consequently, when a number of Hall effect sensors in an array are fitted to the holding tube, the relative output signals obtained from the sensors could be tuned and analyzed to give an indication of the position of the magnetic food particle as it passes through the sensor array. Thus, residence time of this kind of particle can be determined by placing two arrays of the Hall effect sensors, each consisting of four individual sensor devices (Figure 2.5) mounted on the holding tube at a fixed distance apart.

This modified technique has advantages over the original method introduced by Segner *et al.* (1989) for the possibility of determining the position of the particle. Moreover, if a number of sensors were placed at regular intervals or distances along the holding tube or even the heat exchanger; the trajectories of the magnetic food particle can be determined. This can help in understanding the mechanism whereby the particle moves in the holding tube in terms of the relative position of the particle. In addition, the carrier fluid and the holding tube need not be transparent, thus the stainless steel tubes of aseptic systems could be used. This means that RTD studies can be performed at the high temperature conditions of aseptic processing as the Hall effect sensors are not affected by high temperatures. The disadvantage in this case is the difficulty of handling more than one particle at a time, as the introduction of a number of particles will likely result in interference between the different responses of different particles.

Photosensor Methodology: Photoelectric sensors have been successfully used for many decades in security systems, industrial control systems, and home electronic equipment to provide non-touch switches and remote control devices (Yang and Swartzel, 1991). The use of photoelectric sensors to study RTD of food particles was introduced by Yang and Swartzel (1991). Their approach was to use optical beams that can create an "optical-grid" which divided the cross-sectional area of the holding tube into several small regions. The "optical-grid" was created by a horizontal-vertical two dimensional array of photoelectric sensors set up around a transparent glass tube. As the particles passed through the "optical-grid", they blocked the light beam and switched "off" the corresponding receivers. The sensors can be interfaced to a microcomputer based data acquisition system for analyzing the "on" and "off" signal patterns. Thus, the position of the particle can be determined at that specific cross-sectional area. Yang and Swartzel (1991) were able to determine the particle velocity and hence its residence time by the installation of two separate "optical-grid" sensors at a specific distance along the holding tube. Again as with the Hall effect sensor, a number of "optical-grid" can be arranged along the holding tube to determine the mechanism of particle flow in solid/liquid mixtures. Other advantages offered by this method are the determination of particle position in a specific cross-sectional area as well as the particle trajectory by installing a series of "optical-grids"; no view angle is required as in visual observation; no magnets are required as in the magnetic response and the Hall effect sensors. However, this method has some limitations such as the requirement for the use of transparent glass tube and carrier fluid, particles have to be of similar size and shape for easy data processing,

and complications may arise when more than one particle is introduced. The major advantage of the magnetic response, Hall effect and photoelectric sensors is that they are precise, accurate and not prone to the human error factor.

Different techniques and methodologies are now available for the RTD studies of solid-liquid two-phase flow systems for aseptic processing of liquid foods containing particulates. All these methods have their merits and drawbacks, so it may be useful to compare results when the experiments are carried under approximately the same conditions, but the obvious difficulty is that some of these techniques are not universal for all experimental set-ups. Most of these methods are not directly applicable to real aseptic processing situations, however, some of them are promising in the sense that there is a good possibility of adapting them to the real situation of aseptic processing of low acid liquid foods containing particulates.

RTD Studies of Food Particles in Aseptic Systems

RTD in SSHE: Chen and Zahradnik (1967); Trommelen and Beek (1971); Milton and Zahradnik (1973) and Cuevas *et al.* (1982) were among several researchers who investigated the RTD of liquid media in SSHEs. Using 60% sucrose solutions as a tracer, Chen and Zahradnik (1967) reported that increasing the mutator speed resulted in a broadened RTD which was clearly deviating from the ideal plug flow. Depending on the flow rate and mutator speed, the residence times ranged from 0.1 to 2 times the mean residence time. According to Trommelen and Beek (1971) the RTD of glycerol/water mixtures in SSHEs, measured by injecting a pulse of a coloured dye, was not uniform

because of velocity differences, the length of the streamlines variations, vortices dispersion, and the pumping effect created by the scraper blades. Further investigations by Milton and Zahradnilk (1973) with 25% solution of polyethylene glycol (non-Newtonian) in a Votator indicated that increasing the mutator speed or decreasing the flow rate, resulted in broadening the RTD in agreement with the findings of Chen and Zahradnik (1967). The most significant difference was that mean residence of the non-Newtonian fluid decreased with increasing either the flow rate or the mutator speed, indicating that some of the tracer input would exit soon after injection. The studies of Cuevas *et al.* (1982) which were conducted at elevated temperatures (118-128°C) with a heat stable food colour (FD & C Red #3), indicated that the RTD in the SSHE was somewhere between the ideal laminar (maximum velocity is twice the average velocity) plug flow (maximum velocity is equal to the average velocity), somewhat closer to plug flow.

Most recently, there were more studies on the RTD of liquid food containing particulates in the scraped surface heat exchangers (SSHEs). Earlier works of de Ruyter and Brunet (1973), Manson and Cullen (1974), and Dail (1985) showed the RTD of particles within the SSHE to be critical and the fastest moving particle must be used for process calculations to assure public health concern, but they provided no experimental data to support their claims. Taeymans *et al.* (1985) were among the first to perform experimental RTD work on liquid (water) and particles (calcium alginate beads) mixtures. The effects of carrier fluid viscosity, the specific gravity, and particle diameter on the mean residence time were investigated through the axial and rotational Reynolds number

as well as the centrifugal Archimedes number. The mean residence time of the alginate beads increased with increased mutator speed (increased rotational Reynolds number); whereas increased flow rate (increased axial Reynolds number) and solid phase concentration resulted in a decreased mean residence time. Taeymans *et al.* (1986) showed that the experimental mean residence time of the alginate particles was longer than the mean residence time and increasing the rotational speed of the scrapers was responsible for diverting the flow pattern from the ideal plug flow.

The simulations of Sastry (1986) revealed that the effect of the heat exchanger RTD on achieving the target lethality was quite significant, with faster moving particles greatly increasing the required process schedule. Sastry (1986) based this argument on the heat exchanger residence time ratio, HXRTR (ratio of particle residence time to mean residence time within the SSHE) in the range of 0.2 to 2.5. However, wider ranges of residence times have been observed (Taeymans *et al.*, 1985). Alcairo and Zuritz (1990) showed that increased particle diameter, mutator speed, and flow rate decreased the standard deviation and narrowed the residence time distribution, while carrier fluid viscosity has no effect on the RTD curve. Moreover, the minimum and mean particle residence times were not affected by the mutator speed, particle diameter and carrier fluid viscosity. Higher flow rates resulted in mean particle residence times which were 16% shorter than the mean fluid residence time, while the two of them were about equal at lower flow rates.

More recently, Lee and Singh (1991, 1993) presented detailed studies on the RTD of food particles (potatoes) suspended in carboxymethyl cellulose (CMC) solutions and

showed that the orientation of the SSHE (horizontal or vertical) has no significant influence on the minimum particle residence time. However, the mean and the maximum residence times were significantly affected by the SSHE orientation (vertical orientation was normaly associated with longer residence times), probably due to the action of gravity against the particle flow. The minimum and maximum normalized particle residence times were not affected by carrier fluid concentration, flow rate, mutator speed and particle size (Singh and Lee, 1992).

RTD in Holding Tubes: Residence time distributions of liquid foods in aseptic pasteurization and sterilization systems have been widely investigated. Dickerson *et al.* (1968) and Scalzo *et al.* (1969) studied the residence time of milk (80°C) and liquid egg (62°C) using radioactive iodine and concluded that process calculation methodology should be based on the fastest particle velocity in laminar flow in order to ensure proper pasteurization. The importance of RTD in determining the length of the holding tube in aseptic flow systems was emphasized by Rao and Loncin (1974a,b). The correlations of Palmer and Jones (1976) for the fastest particle residence time and the estimation of the holding tube length were influenced by the rheological properties of the liquid medium (Newtonian *vs* non-Newtonian). It was shown that the estimation of the holding tube length based on laminar or turbulent flow conditions would ensure a microbiologically safe design criterion for Newtonian fluids and shear-thinning fluids, but not for dilatant (shear-thickening) fluids (Rao, 1992). Other correlations relating the RTD function and thermal inactivation kinetics of a microorganism population were introduced by Lin

(1979). The investigations of Sancho and Rao (1992) indicated that maximum fluid velocities in a holding tube were less than those estimated in fully developed laminar flow of Newtonian and non-Newtonian fluids in straight tubes. They showed that the magnitude of the Dispersion Number ($DN = D_e/uL$) can be related to the Reynolds number (Re) for the Newtonian fluids and the generalized Reynolds number (GRe) for the non-Newtonian fluids. Rao (1992) recognized these ranges of Re and GRe: (a) 10-100: DN was almost constant, (b) 100-2000: DN increased with Reynolds, and (c) 2000-10000: DN decreased as Reynolds number increased.

Several researchers have investigated the RTD of particles in holding tubes. Manson and Cullen (1974) stated the importance of RTD as a critical parameter for thermal process calculations. Sastry (1986) reported that the minimum holding tube length necessary for commercial target lethality was greatly influenced by the residence time distribution of particles within the holding tube. This was confirmed by Lee *et al.* (1990a,b) who used numerical simulations to evaluate the influence of RTD on the holding tube length, accumulated lethality and minimum process time determinations.

McCoy *et al.* (1987) investigated the flow behaviour of single particles (plastic spheres with densities in the range of 1003-1053 kg/m^3) in a viscous non-Newtonian carrier fluid within a fabricated transparent holding tube as influenced by particle size, carrier fluid viscosity, flow rate and the influence of bends. The following were some of their conclusions: (1) the bend size and the cumulative effect of the bends has no influence on the particle radial position for high viscosity carrier fluid, but the effect was obvious at lower viscosity levels, (2) the influence of the bends was clear at higher flow

rates, (3) the flow behaviour of the small particles was unstable, while the flow was stable for larger particles. It was also reported that the particles were faster than the average fluid velocity by about 85%.

Segner *et al.* (1989) used 12.7 mm turkey cubes containing 3 mm ceramic magnets in a solution of starch/sugar/water pumped around an SSHE. The magnetic particles were detected by two copper coils; one at the entrance and the other at the exit of the holding tube. The fastest particle residence times were much longer than those based on presumed laminar flow for all the flow rates investigated. The variations between residence times (particle to particle) increased as the holding tube length was increased from 6.9 to 58.2 m and the flow rate was decreased from 47.7 to 28.0 L/min.

Berry (1989) evaluated the residence time distribution in a holding tube using plastic cubes (6, 10 and 13 mm) with up to 6% particle concentration. It was shown that the shape of the distribution curve, the mean and the fastest particle velocity were dependent on particle size, concentration and fluid velocity. Furthermore, it was reported that some of the particles moved with the higher velocity fluid near the centre of the pipe, while others moved with the slower fluid along the tube wall. A statistical definition for the fastest particle residence time is the time below which only 1% of the particles would be expected to exit the system, while the residence time of the slowest particle time is taken as the time below which 99% of the particles would be expected to exit.

Dutta and Sastry (1990a, b) used sodium carboxymethyl cellulose (CMC) solutions to investigate the velocity distributions of 0.95 cm polystyrene spheres in tube flow. The density of both phases was maintained in the same range; thus, this study was

representative of a study of non-settling particles. It was shown that the mean normalized particle velocities were significantly influenced by particle Froude number (Fr_p) and non-dimensionalized viscosity (η_{ND}). Viscosity was revealed as the major factor contributing to the variability of velocity distributions and no particles were observed to travel faster than twice the mean fluid velocity under idealistic laminar flow situations.

A study using 5 mm diameter alginate beads flowing in water was reported by Hong *et al.* (1991). The particles were injected into a 35.6 mm internal diameter holding tube and subsequently collected using a rotating basket arrangement on exit from the holding tube. The residence times of the alginate particles was found to be 1.032 times that of the carrier fluid under somewhat turbulent flow conditions (Re = 13,800). Further, the experiments were conducted at room temperature (25°C), though the equipment was designed to investigate RTD at high temperatures (> 100°C).

Yang and Swartzel (1991; 1992) used a photoelectric sensor methodology to study the RTD of 19.1 mm diameter polystyrene spheres in a holding tube (ID = 50.8 mm). Two photoelectric sensors were placed a known distance apart to detect particles as they pass. The study, conducted under turbulent flow conditions (Re = 34,000), indicated that density was a critical factor in influencing the particle RTD and determining the position of particles in the flow stream.

A recent study by Palmeiri *et al.* (1992) made use of 1 cm potato cubes dyed with iodine in a carrier fluid composed of 10% w/w sodium chloride solution at different flow rates and particle concentration (10 to 30% w/w) and carried out at 15°C. The residence time of the fastest moving particle was reported to be unaffected by the particle

concentration, while that of the slowest particle was significantly reduced by increasing the particle concentration. An explanation given was the collisions taking place among the solid particles could accelerate the overall motion of the solid phase along the pipe and reduce the differences of residence time among various particles. The flow regime was turbulent (Re = 10,000) and the result was applicable to particles denser than the carrier fluid.

Ramaswamy *et al.* (1992) investigated the flow behaviour of polypropylene spheres with a diameter of 0.635, 0.953, 1.27 and 1.905 cm and specific gravity of 1.14 as well as potato and carrot cubes with a length of approximately 0.75 and 1.27 cm in a fabricated holding tube in the temperature range 60-98°C. Larger particles were observed to travel faster than the smaller ones. As more of the particle's cross-sectional area becomes occluded for the flow, the particle will face a stream of fluid that has a higher relative velocity. This will result in a higher particle velocity even though the fluid average velocity remains the same. Their results also indicated, in general, that both polypropylene and food particles travelled faster in 3% starch solution than in water at similar flow rates. This was attributed to the densities of particles being higher than that of water with negligible buoyant effects, while with 3% starch, the associated higher viscosity and slightly higher density could attribute buoyant effects that facilitate faster particle velocities.

Recent studies by Tucker and Withers (1992) and Tucker and Richardson (1993) made use of Hall effect sensors to study the RTD of real food particles in a commercial aseptic processing system. It was claimed that this technique can be used at the high

temperatures characteristic of aseptic processing, but the data presented was from room temperature experiments. It is worthwhile noting that the technique gave satisfactory results as compared to other techniques. Most recently, the simulations of Skjoldebrand and Ohlsson (1993a, b) revealed that RTD of food particles in all of the aseptic system components (SSHEs and holding tube) are essential to capture the food quality benefits of aseptic processing and ensure public health safety. Consequently, meaningful velocity distribution studies require the use of food particles of different shapes and sizes flowing through the entire aseptic processing system including heat exchangers and holding tube in order to determine the fastest particle residence time as influenced by various factors (Heldman, 1989; Dignan *et al.*, 1989). It is difficult to draw valid conclusions from studies carried out with simulated particles flowing in simulated fluids under experimental conditions different from those prevailing under commercial aseptic processing conditions.

Modelling of RTD Curves of Food Particles

The conditions in an aseptic processing system for liquid low acid foods containing particulates are neither that of plug flow nor perfect mixing. Non-idealistic conditions like channelling and dead space can be noticed. Channelling flow occurs when a portion of the tracer emerges from the reaction vessel in an unusually short period of time. It takes place when the feed and exit ports of the reactor are close together. Dead space, however, results in some of the feed being caught in the reactor, causing the velocity of flow to decrease. Dead space is also known as the "hold-back" and is common with ideal mixing reactors (Danckwerts, 1953). It usually occurs around the corners,

baffles, elbows, tube connections, or regions with obstruction to flow. This kind of flow may be viewed as the opposite to the channelling type of flow.

Two models frequently used to describe the flow behaviour of fluids under these non-ideal situations are the "*Dispersion*" and the "*Tank-in-Series*" models. The *dispersion* model combines both the plug flow model and Fick's law of diffusion:

$$c(\theta) = \frac{1}{2\sqrt{\pi D_e / uL}} \exp\left[-\frac{(1-\theta)^2}{4(D_e / uL)}\right] \qquad (21)$$

where D_e is the axial dispersion coefficient, u is the average axial velocity, and L is the vessel length. D_e/uL is the reactor Dispersion number (DN), as indicated earlier, varying from zero for plug flow, to infinity for back mix flow. This model is satisfactory only when the flow does not significantly deviate from the plug flow behaviour.

The *Tank-in-Series* model is an alternate approach to the dispersion model for handling minor deviations from plug flow (Levespiel, 1972). In this model the system is considered as a series of *n* "continuously stirred tank reactors" (CSTR). The population density function in this case is:

$$E(\theta) = \frac{n^n \theta^{(n-1)}}{(n-1)!} e^{(-n\theta)} \qquad (22)$$

A perfectly mixed vessel is represented when *n* is equal to unity and if *n* approaches infinity, plug flow conditions are satisfied.

In aseptic processing of particulates, the ideal flow would the plug flow in order to have a product with adequate homogeneity; but heat transfer would be slow due to the lack of radial and axial mixing. Perfectly mixed flow, on the other hand, is characterized

by intensive agitation which promotes the heat transfer processes but broadens the RTD (Lee and Singh, 1992). The flow conditions in the SSHE are between an ideal plug flow and an ideal CSTR (Taeymans *et al.*, 1986). Consequently, intermediate and mixed flow models are required to describe RTD (Singh and Lee, 1992). The situation becomes more complex when involving SSHE and holding tube under real processing conditions. Consequently, alternate attempts have been made to characterize the RTD or flow behaviour of particulates (Taeymans *et al.*, 1986; Berry, 1989; Dutta and Sastry, 1990a, b; Lee and Singh, 1991; Palmeiri *et al.*, 1992). The majority of these studies were attempts to model experimental RTD data obtained from experiments performed with simulated particles at atmospheric pressures and mostly at room temperatures. Taeymans *et al.* (1986) studied the RTD of calcium alginate beads (6 mm diameter) in water in a SSHE and concluded that the flow behaviour was midway between plug flow and perfect mixing flow models. Using plastic cubes of 6 and 13 mm (size: 100 particles), Berry (1989) found that the RTD of larger particles showed a single normal distribution pattern, while small particles (6 mm) appeared to indicate a pattern resulting from the overlapping of more than one normal distribution curve. He argued that this may have been due to some particles flowing through the centre while others were flowing near the walls of the tube. The normal probability function was also used by Alcairo and Zuritz (1990) to describe the RTD of spherical particles suspended in a non-Newtonian carrier fluid of CMC:

$$E(t) = \frac{1}{\sigma(2\pi)^{1/2}} e^{\frac{(-(RT-RT_{mean}))^2}{2\sigma^2}} \qquad for\ -\infty < t < \infty \qquad (23)$$

Palmeiri *et al.* (1992) also observed that carrot cubes suspended in a salt solution and flowing in a holding tube had an RTD that could be considered as the result of the superposition of two or more normal distribution curves.

Dutta and Sastry (1990a,b) described the RTD of spherical polystyrene particles by a log-normal cumulative distribution function (CDF). They pointed out that the theoretical curves were within 95% confidence limits of the experimental data, and that the development of a general distribution model for velocity and residence time distribution is largely dependent on the experimental conditions. Lee and Singh (1991; 1993) showed that the RTD of potato cubes could be characterized by a gamma distribution function in most cases, while normal distribution was good at lower concentration of the carrier fluid combined flow rates and small size particles. The mathematical representation for the gamma distribution function is given by:

$$F(t) = P[t_\infty \leq t] = \int_0^\infty \frac{t^{(c-1)} e^{(-t)}}{\Gamma(c)} \qquad (24)$$

Both the gamma and log-normal distribution functions can be used for describing the RTD of food particles which show some deviation (skewed to the right) from the well known normal distribution. Sigmoid type curves which characterize the growth of living systems can also be used to describe the RTD of food particles in aseptic systems. The logistic growth model can be used to describe the RTD of food particles :

$$c(\theta) = \frac{U}{[1 + e^{-B(\theta - H)}]} \qquad (25)$$

The above equation is also known as *autocatalytic* or the *inverse exponential* curve as used by Nedler (1962), Zwietering *et al.* (1990, 1991) and van Impe (1992) for modelling of bacterial growth curves. The parameters B, U and H associated with this model can be defined to have relevance to the RTD studies: B which is the growth rate can be taken to represent the particle accumulation rate; U, the upper asymptote to indicate the maximum particles concentration and H to represent the 50% internal age as this corresponds to a time when cumulative concentration is one half of the maximum.

Biological Validation For Particulate Foods

Ensuring commercial sterility of continuously processed foods containing particulates cannot be based on conventional thermal process calculations because time/temperature measurement in particulates is not practical as they move within the aseptic system. In absence of the physical means for temperature measurements, biological validation appears to be the only alternative available to evaluate the lethality (F_o) delivered at the centre-point of the largest/fastest moving particle. According to Ronner (1990), the principle of biological validation is to evaluate the thermal process, which has a chosen F value, by using a selected indicator organism which is recovered and quantified. Biological validation can be performed by direct inoculation of free bacterial spores into the product prior to the process or into particulates carried through the process.

Direct inoculation is of minor interest because of its low sensitivity as only a small proportion of the total inoculation level of bacteria is recovered after the process, and secondly liquid products are not a problem in aseptic processing (Ronner, 1990; Pflug *et al.*, 1990). Although particulate inoculation is simple in concept, but it is receiving more attention mainly because the dynamics of particle movement in an actual aseptic processing system are as complex as the heat penetration into the particles (Dignan *et al.*, 1989). Moreover, the use of biological indictors will only give an integrated sterilization value for the process considered.

Biological Indicators

One of the methods for biologically evaluating the bacterial-killing power at the centre slowest heating location is known as the "biological thermocouple system" which has been used in Europe and North America for about 20 years. It is called the biological thermocouple system because the spores in the biological indicator unit are not in contact with the food product. The spores are placed in a carrier, like glass, which is located in the centre of the food particle. The method has several advantages (Cousin, 1993; Hersom and Shore, 1981): 1) the location of the spores is known; 2) all the spores in a bulb are recovered once the bulb is recovered; and 3) all the spores can be located at the geometric centre. Pflug and coworkers (Pflug and Smith, 1977; Pflug *et al.*, 1980; Jones *et al.*, 1980; Pflug, 1982; Pflug and Zechman, 1983) have used the biological thermocouple with some success.

Inoculated Particles

An alternative approach which has been used for many years is the inoculation of the slowest heating location of real food particles with heat-resistant microbial spores (e.g., *Bacillus stearothermophilus*) and the evaluation of their survival on exit of the aseptic system. The presence of calibration data for the purpose of comparison is essential. Dignan *et al.* (1989) and Pflug *et al.* (1990) listed a number of obstacles to be considered when a thermal process for particulates is biologically validated: (1) the conditions affecting spore destruction must mimic the actual processing situation; (2) an effective N_o must be determined in case of a leakage possibility; (3) N_o must be adjusted in all calibrations; (4) particles must maintain their integrity and size during testing; (5) residence time of the inoculated particles must be representative of the fastest particle; (6) the location and distribution of the spores in the particles must be known; and (7) the z-value of the spores must be determined in order to properly use the time-temperature data.

Sastry *et al.* (1988) used an infusion procedure where mushrooms were placed in a spore suspension for 36 min. The choice for using mushrooms was based on the following reasons: (1) availability in large sizes, permitting conservative testing; (2) high porosity of fresh product facilitating infusion of bacterial spores; (3) a well-characterized albeit-irregular geometry; and (4) easy local availability. However, these indicators were not tested in a commercial aseptic system for mechanical sturdiness and durability.

Sastry (1989) summarized the desirable/necessary characteristics for a bioindicator as follows: (1) large size (2.5 cm); (2) presence of heat resistant spores throughout the interior, especially the slowest heating point; (3) geometry and thermal properties must

be similar to real food particles; (4) visual distinguishability from real food particles, to facilitate recovery from the processed product; (5) retention of spores without leakage throughout the process; (6) shelf stability permitting easy reconstitution and use whenever desired; and (7) physical durability to withstand process stresses without disintegration.

Cerney *et al.* (1989; 1991) utilized formulated food particles of egg white ovalbumin and potato alginate cubes to determine the lethality of immobilized *Bacillus subtilis*, *Bacillus stearothermophilus* and *Clostridium sporogenes* after thermal treatment. The spores in particles were reported to be inactivated less rapidly than those heated in phosphate buffer for the same time-temperature combination. The D-values determined were 1.5 to 2-fold higher in particles than in the phosphate buffer. Increased thermo-resistance was observed with fat-containing particles, but this may be reduced by the addition of emulsifiers. Denatured proteins (ovalbumin) and some starches were reported to have the same effect. Low water activity persistent in particles may be implicated, but the real reasons behind this phenomenon are yet undetermined. Ovalbumin particles containing *C. sporogenes* were used to study the distribution of thermal energy by incubating the heated particles at 37°C for 48 h, and then particles were sliced to visualize the black spots where spore survival is indicated by sulphite reduction (Cerney *et al.*, 1989). Similar experiments were performed with potato cubes and peroxidase enzymes activity.

Simulated Particles

The problems associated with the biological validation of continuous aseptic

processes when actual food particulates are inoculated with bacterial spores have led researchers to develop simulated particulates. Hunter (1972) used polymethylmethacrylate to suspend bacterial spores but, in this plastic material the spores are subject to dry heat destruction. The use of alginate gel particles inoculated with spores to validate thermal processes was reported by Dallyn *et al.* (1977). The alginate particles were made by mixing bacterial spores with sterile sodium alginate solution, then drops of the mixture were introduced into a sterile calcium chloride solution to induce gelation. The resulting beads were reported to have an excellent mechanical strength to withstand processing conditions in scraped surface heat exchangers. This approach has been employed by Bean *et al.* (1979) to determine the lethalities achieved in a commercial aseptic system using *B. stearothermophilus* and by Heppell (1985a,b) to determine the fluid-to-particle heat transfer coefficient. However, the particle sizes used in all of these studies were very small (1.6 to 3.2 mm).

Brown *et al.* (1984) immobilized *C. sporogenes* in large (0.8 to 2.4 cm) food alginate particles made from pureed potatoes, peas and meat. The test microorganism had D-values of 0.7 and 0.8 min in potato-alginate and meat-alginate particles, respectively. The z-values for the temperature range of 115-130°C were 12.5 and 12.7 C°, respectively. Experiments with alginate-food particles showed encouraging success for further research. Moreover the alginate-food particles have a long shelf life and are harmless to the immobilized bacterial spores.

A further development was introduced by Ronner (1989; 1990) who constructed a commercially available bioindicator from polyacrylamide gel (PAG) containing *B.*

stearothermophilus, as the alginate-food particles are susceptible to disintegration in contact with chemical environments containing calcium chelators and can shrink during processing causing changes in shape and thermal diffusivity. The PAG particles are colourless, sable and tough, maintain shape, prevent leakage unless mechanically damaged, and have an impermeable outer layer but an internal microporous structure to allow fast heat diffusion. The main disadvantage is the size of these particles (8 mm) may not be a true representative for the validation of aseptic processing of larger particles.

Chemical Index Markers

Biological indicators offer the most direct proof of sterility, but they are rather tedious and their use under varying process parameters (temperature, flow rate holding tube length, particle size, system configuration, etc.) have limited value (Kim and Taub, 1993). Monitoring chemical changes within the food, which involves compounds either indigenous to or added to the food, offers an alternative route for assessing the integrated time-temperature exposure of the food particulate. These are the chemical indicators which have been used for process validation. Such examples include the loss of thiamin (Mulley *et al.*, 1975; Ramaswamy and Ghazala, 1990), peroxidase (Adams *et al.*, 1984; Weng *et al.*, 1992), pantothenic acid (Ham and Lund, 1978), ascorbic acid (Rao *et al.*, 1981a), anthocyanin (Tanchev, 1983) and methylmethionine sulphonium, MMS (Berry *et al.*, 1989; 1990). The major disadvantage with these markers is that their loss is quite small for the same process lethality at high aseptic processing temperatures, because the z-values for typical chemical reactions are much higher than those for thermal destruction

of microbial spores (Lund, 1977; Jelen, 1983; Sadeghi and Swartzel, 1990). A good example of this is the thiamin destruction in beef puree for which the D-value is 244 min and the z-value is 48 F° (Mulley *et al.*, 1975) in comparison with a z-value of 18 F° for *Clostridium botulinum*.

A recent development has been introduced by Kim and Taub (1993). Using the analytical capabilities of anion exclusion chromatographic (AEC) separation and photodiode array (PDA) detection, they were able to discern some compounds formed in foods as a result of heating. Three intrinsic compounds were detected by three-dimensional representation of AEC-PDA. M-1 (2,3-dihydro-3,5-dihydroxy-6-methyl-(4H)-pyran-4-one) was detected in heated chicken meat, vegetables (potato, green bean, pea, carrot) and fruits (apple, orange, pear) under varying time-temperature conditions. M-2 is associated with protein (aseptically processed meat and potato), but no chemical characterization was given as yet. M-3 is 5-hydroxymethylfurfural (HMF) which is well known as a major degradation product of D-fructose (Shaw *et al.*, 1967). M-3 was detected from aseptically processed juice drinks and heated fruits and vegetables. The kinetics of these intrinsic markers were studied and correlated to the process parameters. The concept of thermal process validators is sound, practical and represents a step in the right direction, however more work is required for standardization.

Rheological Properties of Carrier Fluids

Rheological properties of carrier fluids have a strong influence on fluid velocity profiles in tube flow (Steffe, 1992) which are important with respect to the sizing of the

holding tube length in aseptic processing system. Viscometric characterization of liquid foods relies on the basic engineering principle that the pressure drop along a straight length of pipe is related to the wall shear stress, and the volumetric flow rate is related to the shear rate (Tucker, 1993). The wall shear stress is calculated as a function of the pressure drop under laminar flow conditions:

$$\sigma = \frac{\Delta P d}{4 \Delta L} \qquad (26)$$

The wall shear rate for power law fluids is given by:

$$\dot{\gamma} = \left(\frac{Q}{\pi R^3}\right)\left(\frac{3n+1}{n}\right) \qquad (27)$$

Sastry (1989) reported that the minimum holding tube length must provide sufficient residence time to ensure the commercial sterility of the particulate food. According to Lund (1993) the tube length must be based on the minimum residence time (RT_{min}) which, in turn, depends on maximum velocity (u_{max}):

$$L = u_{max} \cdot RT_{min} \qquad (28)$$

The maximum fluid velocity (u_{max}) and mean fluid velocity (u_{mean}) in the tube flow are inter-related depending on the flow behaviour index (n) of the flowing fluid and the flow regime (laminar, transitional or turbulent):

$$u_{max} = \left[\frac{3n+1}{n+1}\right] u_{mean} \qquad (29)$$

For Newtonian fluids (n = 1) under laminar flow conditions (Re < 2100), Eqn 29 is reduced to:

$$u_{max} = 2\,u_{mean} \qquad (30)$$

For a pseudoplastic fluid, however, n < 1 and as a result $u_{max} < 2\ u_{mean}$, whereas for a dilatant fluid, n > 1 and $u_{max} > 2\ u_{mean}$. Velocity profiles are also influenced by the radial distance in the holding tube in addition to the rheological properties (specifically the flow behaviour index, n) as illustrated in Figure 2.6. Mathematically, the velocity of a fluid at any specific radial location in tube flow can be estimated from the following relationship:

$$\frac{u}{u_{mean}} = \left(\frac{3n+1}{n+1}\right)\left[1 - \left(\frac{r}{R}\right)^{(n+1)/n}\right] \qquad (31)$$

Characterization of the flow properties of foods comprising discrete particles is an area of increasing interest to food processors (Tucker, 1993). The rheological properties of the carrier fluid are required for fluid mechanics studies in order to characterize the flow nature of a fluid as the fluid travels through the aseptic processing system. Flow regime specification (laminar, turbulent or transitional) requires the calculation of the Reynolds number which is a measure of the ratio of the inertial force to the force of internal friction (viscosity). Although there was no specific value at which the flow regime changes from laminar to turbulent flow, a value of Re = 2,000-3,000 has been commonly used to signify the transition zone between the two flow regimes. Determination of interfacial heat transfer coefficient between the food particle and the surrounding carrier fluid as well as the relative velocity between the fluid and the particle requires that the rheological properties of the carrier fluid be known (Dail and Steffe, 1990a). Therefore, the design and evaluation of food processing equipment for fluid foods is dependent on flow characteristics of the food product (Bhamidipati and Singh, 1990).

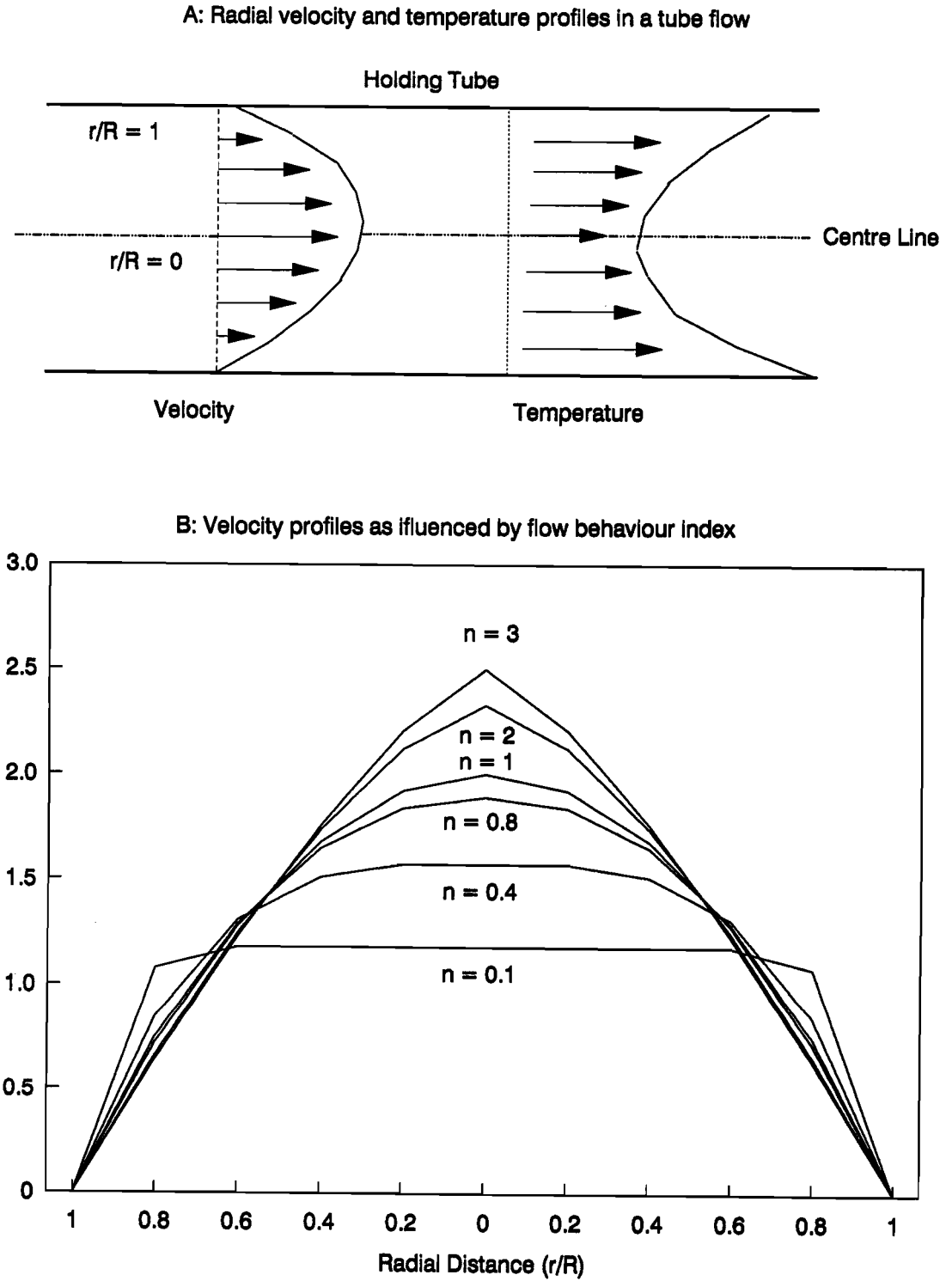

Figure 2.6. **Velocity and temperature profiles in the holding tube as influenced by the radial distance and the rheological properties (flow behaviour index, n) of carrier fluid under laminar flow conditions.**

Most food suspensions are non-Newtonian in nature and are described by a power law model:

$$\sigma = m \dot{\gamma}^n \quad (32)$$

According to Bhamidipati and Singh (1990), there are two limiting cases for laminar flow of pseudoplastic fluids in tubes: (1) for a flow behaviour index of n = 0, a uniform velocity profile exists and (2) for a flow behaviour index of n = 1 (Newtonian fluids), a parabolic velocity profile is obtained. For all intermediate values of n, the velocity profile is between these limiting cases (Figure 2.6).

Viscosity and density are both dependent on process temperature, and therefore a variation in temperature over the path of a flow process will affect the flow behaviour. The rheological parameters (consistency coefficient m and flow behaviour index n) for all non-Newtonian fluids are temperature dependent. For aseptic processing of carrier fluids, it is assumed that the flow regime is laminar and the Reynolds number remained below 2,000, despite the high aseptic processing temperatures (130-150°C) which tend to reduce the fluid apparent viscosity (Tucker, 1992). Only limited information is available on rheological properties of carrier fluids under high temperature/high pressure conditions. Dail and Steffe (1990a, b) provided some rheological data for starch solutions at 121.1-143.3°C using tube viscometry techniques. Only a few studies have been reported on rheological evaluation of carrier fluids containing particulates. These were Bhamidipati and Singh (1990), who used peas in tomato sauce to represent their model system and compared their results to those without peas; and Tucker (1992) who used fresh peas, dried peas and carrots separately suspended in starch based carrier fluids. The conclusion

was that an increase in particle concentration increased the consistency coefficient and decreased the flow behaviour index. This is an indication that the apparent viscosities of carrier fluids increased as particle concentration increased. No temperature effect was investigated.

Evaluation of Rheological Properties: The power law model described by Eqn 32 is the most widely used among rheological models for engineering calculations, especially for pseudoplastic shear-thinning fluids (Rao, 1982). In the presence of a yield stress, the power law takes the form known as the Herschel-Bulkley model:

$$\sigma = \sigma_0 + m_1 \dot{\gamma}^n \qquad (33)$$

The Casson model has also been used for foods, particularly for estimating the yield stress. The two parameters that are frequently discussed are the Casson plastic viscosity (m_c) and the Casson yield stress (m_{oc}):

$$\sigma^{1/2} = (m_{oc})^{1/2} + (m_c \dot{\gamma})^{1/2} \qquad (34)$$

The Bingham plastic model which is a variant of the Newtonian model with a yield stress (σ_o) can also be used:

$$\sigma = \sigma_0 + \eta \dot{\gamma} \qquad (35)$$

The forementioned rheological models are best suited for intermediate shear rate range, which is relevant to this study. Other models such as the Powell-Eyring and Ellis models have been developed to describe the flow behaviour of shear thinning fluids based on a zero shear and an infinite shear viscosity (Rao, 1986).

The time dependant flow behaviour of carrier fluids is generally evaluated by the stress decay under a steady shear over a specified length of time or by measuring the hysteresis loop, the area between the upward and downward flow curves with shear rate varied as a function of time. Weltmann (1943) developed a logarithmic time decay of stress under a constant shear describing the shear thinning behaviour. A modified form of the Weltmann model (Ramaswamy and Basak, 1991) is shown below:

$$\tau = A_w - B_w \log \left(\frac{t}{t_m}\right) \qquad (36)$$

where A_w is the shear stress at an arbitrarily chosen time (t_m) following which the stress decay behaviour is valid, and B_w is the time coefficient of thixotropic breakdown. This model describes the continual stress decay, without reaching equilibrium state even on prolonged shearing. A second model following the first order type relationship was developed by Hahn *et al.* (1959) to include the an equilibrium shear stress:

$$\log (\sigma - \sigma_e) = A_H - B_H t \qquad (37)$$

Temperature has an inverse effect on the apparent viscosity of non-Newtonian fluid foods. An Arrhenius-type equation has been frequently used to describe the effect of temperature on the rheological properties of foods:

$$\eta_{ap} = \eta_0 \exp \left(\frac{-E_a}{RT}\right) \qquad (38)$$

Alternatively, the effect of temperature on both the consistency coefficient (m) and flow behaviour index (n) can be evaluated using the Turian (1964) approach as outlined in Eqns (39) and (40), respectively:

$$\log m = \log m_0 - AT \tag{39}$$

$$n = n_0 + BT \tag{40}$$

The effect of concentration is generally described by either an exponential or a power relationship (Rao, 1986; Fichtali *et al.*, 1993), as given by Eqns (41) and (42), respectively:

$$\eta = \eta_1 \exp(A_1 C) \tag{41}$$

$$\eta = \eta_2 (C)^{A_2} \tag{42}$$

Based on the rheological properties of any specific fluid, multiple regression analysis can be used to generate the parameters required to adequately accommodate the effects of the non-Newtonian flow behaviour, time dependency, temperature and concentration.

CHAPTER III

RESIDENCE TIME DISTRIBUTION (RTD) CHARACTERISTICS OF MEAT AND CARROT CUBES IN STARCH SOLUTIONS IN A VERTICAL SCRAPED SURFACE HEAT EXCHANGERS (SSHE)

ABSTRACT

Residence time distributions (RTD) of meat and carrot cubes were evaluated in the vertical SSHE of a pilot scale aseptic processing system using a full factorial design of experiments employing flow rate (10, 15, 20 and 25 kg/min), particle (meat cubes of size 10, 15 and 20 mm; and carrot cubes of size 6 and 13 mm) and concentration of the carrier fluid (4, 5 and 6% w/w starch) as factors. All factors influenced ($p < 0.05$) the fastest particle residence time (FPRT), mean particle residence time (MPRT), standard deviation of the RTD (SRTD) and their normalized versions. A generalized logistic model showed a good fit for the experimental RTD under all test conditions ($R^2 > 0.95$). Analysis of variance revealed that all test factors affected ($p < 0.05$) the parameters associated with the model: the half concentration internal age (HRT & HNT), particle accumulation rate (B) and upper limit particle concentration (U).

INTRODUCTION

Aseptic processing has been a commercial and technological success for liquid foods. The extension of this technology to low-acid liquid foods containing discrete

particles has been difficult due to lack of data on critical factors such as interfacial heat transfer coefficient between the liquid and the particle (h_{fp}) as well as the residence time distributions (RTD) of the particles in the holding tube of the aseptic system (Sastry, 1986; Yang and Swartzel, 1991; 1992). A detailed review of literature on RTD and carrier fluid rheology was presented in Chapter II. The following highlights the literature relevant to RTD studies described in Chapters III, IV and V.

RTD for homogeneous fluids in typical aseptic processing systems have been extensively studied (Chen and Zahradnik, 1967; Milton and Zahradnik, 1973; Heppell, 1985a; Sancho and Rao, 1992). The importance of RTD of food particles in scraped surface heat exchangers (SSHEs) and holding tubes of aseptic systems have been emphasized by several researchers (de Ruyter and Brunet, 1973; Manson and Cullen, 1974; Sastry, 1986; Taeymans *et al.*, 1986; McCoy *et al.*, 1987; Segner *et al.*, 1989; Alcairo and Zuritz, 1990; Lee *et al.*, 1990a,b; Ramaswamy *et al.*, 1992; Singh and Lee, 1992; Yang and Swartzel, 1992). Rheological properties of the carrier fluids have been shown to influence both fluid and particle residence time distributions in the heat exchangers and the holding tube due to the existence of a velocity profile (Dennis, 1992; Tucker and Withers, 1992). More recently, Yang and Swartzel (1991; 1992) using a photosensor methodology to study the RTD reported that density was also a critical factor in influencing the particle RTD and in determining the position of particles in the flow stream, whereas Dutta and Sastry (1990a, b) emphasized the importance of the viscosity of the carrier fluid in influencing the RTD.

Flow in scraped surface heat exchangers (SSHEs) is influenced by several factors

such as: the length, time-development of rotational and axial flow, radial temperature differences, non-Newtonian flow behaviour and the mutator blades (Harrod, 1986; Dennis, 1992). SSHEs have been used for heating and processing of viscous liquid foods, and recently for the aseptic processing of liquid foods containing particulates because the blades scrape the surface and mix the product resulting in good heat transfer (Taeymans *et al.*, 1986; Hayes, 1988; Alcairo and Zuritz, 1990). RTD for homogeneous fluids in SSHEs have been extensively studied (Milton and Zahradnik, 1973; Heppell, 1985a). One of the earliest studies on RTD of particles in an SSHE (Taeymans *et al.*, 1986) focused on calcium alginate beads (6 mm diameter) in water. The mean particle residence time was found to be more than that of the average fluid residence time, and carrier viscosity, particle specific gravity and particle diameter were found to influence the RTD of the particles. Alcairo and Zuritz (1990) investigated the RTD of single spherical particles suspended in a non-Newtonian fluid in an SSHE, and found that the mean fluid residence time was longer than that of the spherical particles by about 16% at the high flow rates, while they were about equal at low flow rates. More comprehensive studies on the RTD of potato cubes in an SSHE were conducted by Lee and Singh (1991, 1993). Only limited literature is available on RTD of food particles in vertical SSHEs (Lee and Singh, 1993), even though vertically positioned SSHEs are commercially used, have the advantage of self-draining and require minimal floor space and lighter design of shafts that help reducing cost and mechanical load put on the motor (Hayes, 1988),

Particle residence time distribution (RTD) of food particles in the holding tubes of aseptic processing systems is needed to understand their flow behaviour and process

calculations in order to address public health concerns in accordance with the regulatory agencies recommendations (Pflug *et al.*, 1990; Lee and Singh, 1993; Skjoldebrand and Ohlsson, 1993a,b). The relationship between maximum and average fluid velocities is a function of the velocity profile in laminar or turbulent flow situations in tube flow. Laminar flow is the dominant flow regime in food processing systems, therefore it is expected that the fastest particle of the fluid will be moving at no more than twice that of the average fluid velocity (Dignan *et al.*, 1989). Carrier fluid velocity distribution within the holding tube and/or the heat exchanger has to be considered, since each system has its unique flow characteristics that will have a direct effect on the RTD of both the fluid and particles flowing in it (Richardson and Holdsworth, 1989). McCoy *et al.* (1987); Sastry and Zuritz (1987); Berry (1989); Segner *et al.* (1989); Lee *et al.* (1990a,b); Ramaswamy *et al.* (1992); and Grabowski and Ramaswamy (1994) were among several researchers who have realized the importance of the holding tube length as the main factor in process design for aseptic processing of liquid/particles mixtures. Only limited data is available on how the RTD parameters are influenced by varying the length of the holding tube.

Several factors are responsible for variability in RTD studies performed on food particles in holding tubes. Available literature indicate large variations in the test systems used (diameter and length of the holding tube), particles employed (real food particles (carrot, potato) *vs* simulated (plastic or polystyrene), particle properties (size, shape, density) and the characteristics of the carrier fluids (concentration and density). With the exception of the study of Segner *et al.* (1989) who used turkey cubes with small

embedded magnets, there is no reported literature on using meat as a model for studying RTD of food particles. Experimental techniques were also different, such as the photosensor methodology introduced by Yang and Swartzel (1991) and the Hall Effect Sensors employed by Tucker and Withers (1992). RTD data under aseptic processing conditions are limited, as in most of the above studies, simulated particles such as plastic or polystyrene balls or cubes (McCoy *et al.*, 1987; Berry, 1989; Ramaswamy *et al.*, 1992) or vegetable particles like carrots (Chang and Toledo, 1989) and potato (Lee and Singh, 1991; 1993; Palmeiri *et al.*, 1992) have been used with experiments conducted at room temperature. RTD studies on meat particles are limited to perhaps Segner *et al.* (1989) who used turkey cubes with embedded small magnets through an SSHE and a holding tube. Also there is no reported literature on using the whole system for studying RTD of food particles under high temperature conditions.

Some attempts have been made to characterize the flow behaviour of particulates (Taeymans *et al.*, 1986; Berry, 1989; Dutta and Sastry, 1990b; Lee and Singh, 1991; Palmeiri *et al.*, 1992). Taeymans *et al.* (1986) concluded that the RTD curve of particulate fluids was mid way between plug and perfect mixing flow models, while Berry (1989) found that the RTD of large particles showed a single normal distribution, and small particles appeared to indicate a pattern resulting from the overlapping of more than one normal distribution curve. Palmieri *et al.* (1992) also observed that potato cubes in a holding tube showed an RTD which could be considered as the result of the superposition of two or more normal distributions curves, each of which corresponding to a group of particles. Dutta and Sastry (1990b) described the RTD of spherical polystyrene particles

by a log-normal cumulative distribution function (CDF). Lee and Singh (1991) showed the RTD of potato cubes could be characterized by a gamma distribution function in most cases, while normal distribution was good at lower concentration of the carrier fluid combined with low flow rates and small size particles.

The objectives of this phase of study (Chapter III) were to investigate and compare various factors affecting the RTD of meat and carrot cubes in the vertical SSHE of a pilot scale aseptic processing system, to determine the effect of the different factors on the fastest and mean RT as well as standard deviations of particle residence times, and to fit the RTD data to a distribution model.

MATERIALS AND METHODS

Meat Cubes

Beef obtained from a local supermarket (St. Hyacinthe, Quebec), was sliced and cut into 10, 15 and 20 mm cubes. The prepared cubes were kept in a freezer at -5°C until the experimentation time. The particles were then steamed at 100°C for 3 min to coagulate the meat cubes and give them a sturdy texture to prevent particles from sticking to the pump lobes and blades of the SSHE. The density of meat was then determined by a computerized pycnometer (AccuPyc 1330 V1.03, Micrometric, Montreal, PQ).

Carrot Cubes

Carrots obtained from a local farm (St. Hyacinthe, Quebec), were prepared through a dicing machine (Urschel Laboratories, Inc., Valparaiso, IN, USA) with adjustable blades

to obtain cubes of size 6 and 13 mm. The prepared cubes were then washed in water, where the lighter particles and debris were removed using a perforated scoop.

Carrier Fluid

The carrier fluid employed was either a 4, 5 or 6% w/w food grade starch (Therm-flo; National Starch and Chemical Company, Brigdewater, NJ) prepared and pre-gelatinized using the aseptic system by continuously heating to 140°C through the heating SSHE, passing through the holding tube, cooling to 60°C through the cooling SSHE and recirculating to a constant viscosity as determined using a Brookfield viscometer at 60°C (Brookfield Engineering Laboratory Inc., Stuoghton, MA). The viscosity of the carrier fluid was monitored before and after each experimental run to adjust to the predetermined viscosity. The density of the prepared starch solutions was adjusted to 1077 kg/m^3 by adding an appropriate amount of sugar.

SSHE

The test apparatus was a vertical scraped surface heat exchanger (SSHE) (Contherm Division, Alfa Laval Inc., Newburyport, MA) of a pilot scale commercial aseptic processing system. A variable speed positive displacement tri-lobe pump (Albin Pump, Atlanta, GA) was used for feeding the carrier fluid and food particles as regulated by a three-way valve. The layout of the experimental set-up is shown in Figure 3.1, and the dimensions of the SSHE are detailed in Table 3.1.

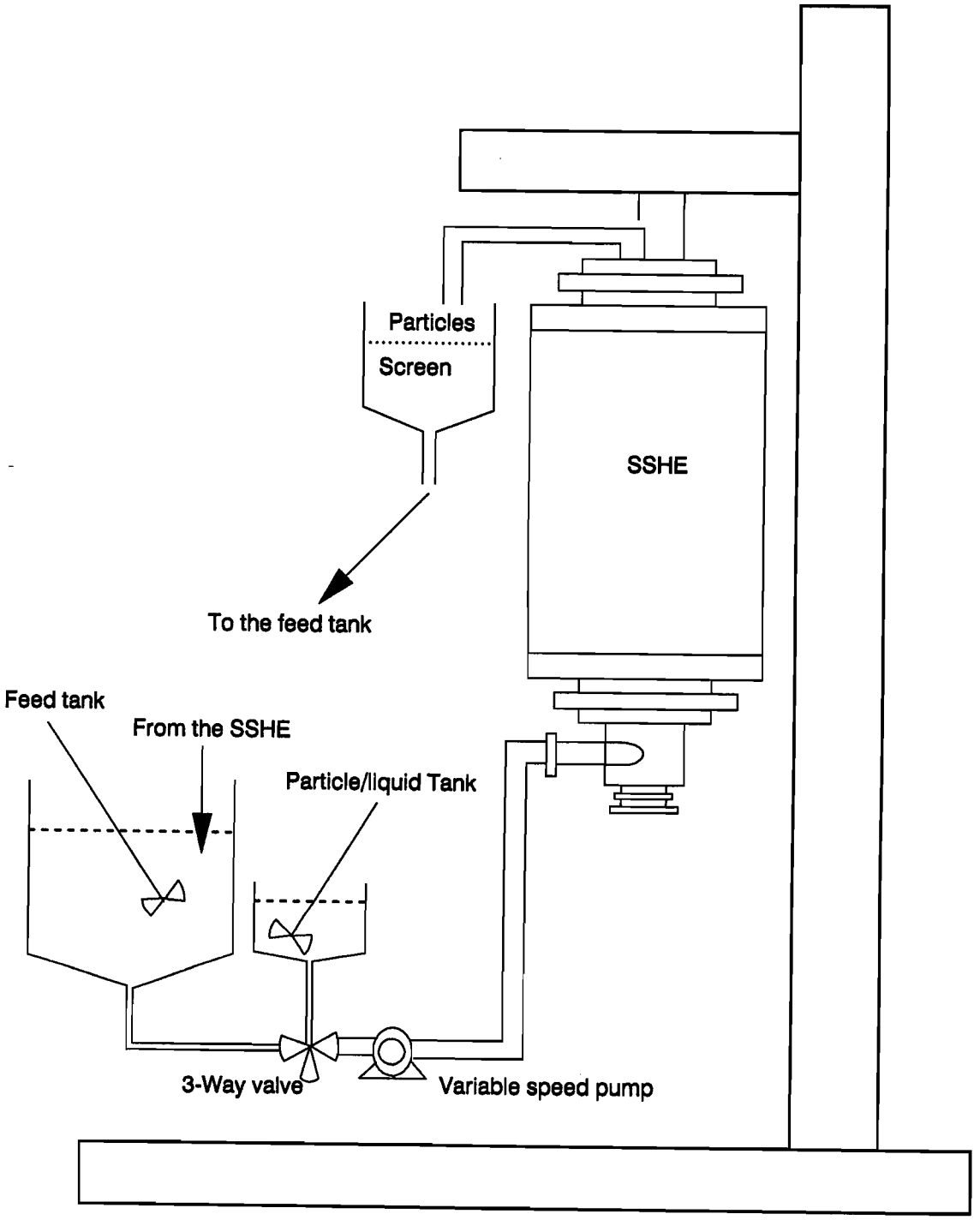

Figure 3.1. **Layout of the experimental set-up used to study the RTD of meat and carrot cubes in starch solutions in a vertical SSHE of a pilot aseptic processing system.**

Table 3.1. Specifications of the aseptic processing system used to study the residence time distribution of meat and carrot cubes.

Parameter	Specification				
Scraped surface heat exchanger					
Scraped surface heat exchanger	1.8 X 0.9 m				
Internal diameter	0.152 m				
Internal shaft diameter	0.076 m				
Length of the heating surface	0.762 m				
Material of construction	Stainless steel				
Dasher speed	150 rpm				
Heating medium	Steam at 150°C				
Cooling medium	Water at 10°C				
Holding Tube					
Total length	26.65 m				
Internal diameter	0.0381 m				
Carrier fluid	Therm-flo® starch				
Gelatinization temperature	140°C				
Concentration	4	5	6		% w/w
Density before adjustment	1011	1019	1026		kg/m^3
Density before adjustment	1077				kg/m^3
Flow rate (meat cubes)	15	20	25		kg/min
Flow rate (carrot cubes)	10	15	20	25	kg/min
Particles	meat		carrot cubes		
Dimensions	10, 15, 20		6, 13		mm
Density	1110		1040		kg/m^3
Concentration	5		5		% w/w

Operation

Usually for RTD studies either step or pulse inputs are used. They involve the instantaneous introduction of a statistically large number of tracer particles into the flow stream. However, this was physically not possible in the system employed as the size of the particles was large. Instead, a slightly modified procedure was employed in the present study as detailed below: 300 g of meat (10, 15 or 20 mm) or carrot (6 and 13 mm) cubes were mixed in the liquid/particles tank with 5.7 kg of the carrier fluid (4%, 5% or 6% w/w) to obtain 6 kg of particle/liquid mixture containing 5% w/w particles in the carrier fluid under study. In a typical run, the system was first equilibrated with the desired carrier fluid and then the particles/liquid mixture was introduced to the system through the three-way valve. After emptying the liquid/particles reservoir, the three-way valve was switched back to take the carrier fluid from the feed tank. A strainer was placed at the exit end to collect the particles while the liquid was returned to the recirculating feed tank. The time interval from the moment the particle/liquid mixture was introduced into the system to when the first particle emerged out of the system was measured and taken to represent residence time of the fastest moving particle. The fluid and particles emerging out were subsequently collected at 20 s intervals and were weighed separately. From this data, the concentration of particles in each stream was determined.

Since this technique is different from the conventional step or pulse input techniques, the following explanation is offered. Addition of liquid particle mixture over the 20-40 s time frame employed in this study was assumed to be a combination of several discrete pulse inputs as shown in Figure 3.2. Each input pulse will have its own

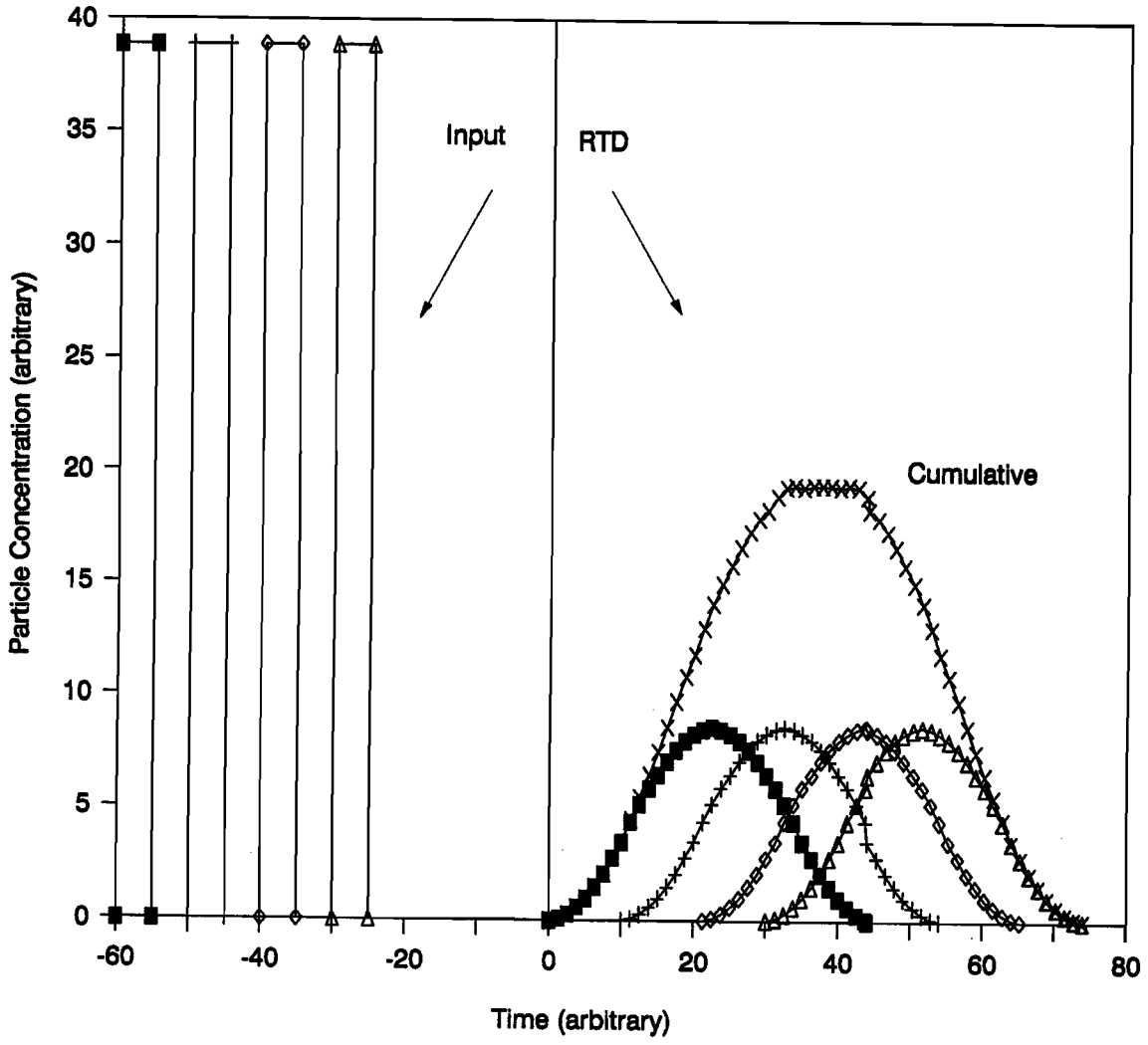

Figure 3.2. Hypothetical pulse inputs for RTD studies.

characteristic RTD and, at low particle concentrations, would give a cumulative RTD as indicated in Figure (3.3) when particle-to-particle interactions (which are ignored in most RTD studies) are not significant. It can be noted that the technique will have no influence on the fastest particle residence time (FPRT) while the mean residence time (peak) will lag the first instantaneous pulse by one half of the duration of input. When adjusted for particle concentration (to match individual cumulative for the discrete input) and for time as above, the cumulative *F*-curve for instantaneous input will almost match that of the discrete combination as shown in Figure 3.3. Residence time distribution data in this study were adjusted for the time required to empty the liquid/particles tank (lasting 20-40 s) by subtracting one half of the particle introduction time from particle residence time. This adjustment was not necessary for FPRT. Details of the technique have been published elsewhere (Abdelrahim *et al.*, 1993a).

Experimental Design

A full factorial design of experiments (Table 3.1) was employed involving three/four levels of flow rate (10, 15, 20 and 25 kg/min) and three levels of starch concentration (4, 5 and 6% w/w). Meat particles were used in three sizes (10, 15 and 20 mm), while only two sizes (6 and 13 mm) were employed with carrot. All the experiments were performed in duplicates at room temperature. Statistical analyses were performed using the General Linear Model Procedure (SAS, SAS Institute Inc., Cary, NC; 1985) to test the influence of different factors associated with the RTD of meat and carrot cubes: (1) the fastest particle residence time (FPRT), (2) mean particle residence time

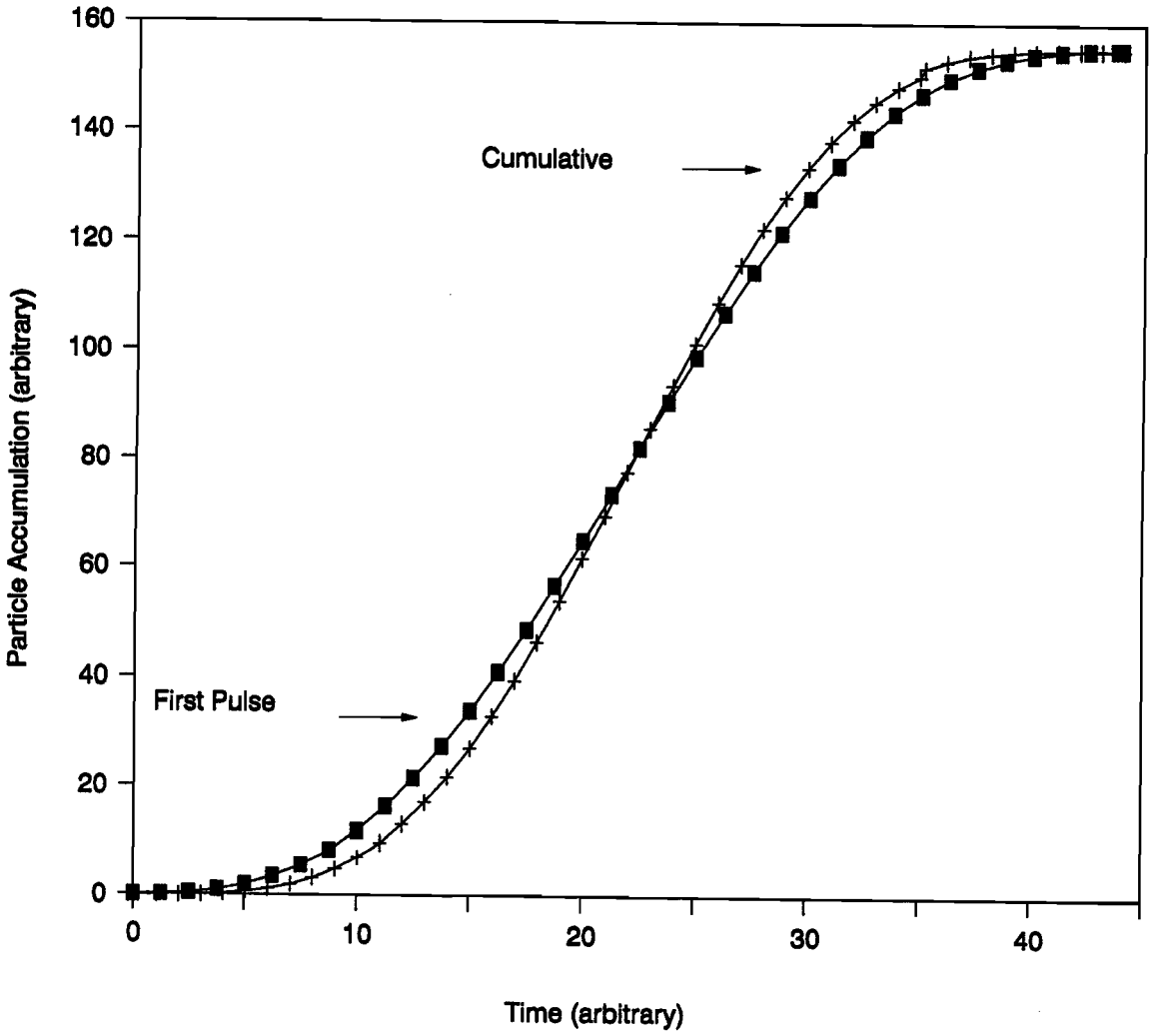

Figure 3.3. Hypothetical cumulative data for RTD studies.

(MPRT) and (3) the standard deviation of residence time distribution (SRTD). FPRT was also normalized by dividing it by the average residence time (t_m) of the fluid to investigate how fastest particle is travelling in relation to the carrier fluid. Average residence time (t_m) was calculated by dividing the fill mass of the system by mass flow rate. Fill mass of the system was obtained by multiplying the fill volume (manufacture's data) by carrier fluid density.

Logistic model for the RTD Data

RTD of food particles showing some deviation from the known normal distribution (skewed to the right) can be described by gamma and log-normal distributions. Sigmoid type logistic curves which characterize the growth of living systems can also be used to describe the RTD of food particles in aseptic systems. In this study the RTD of meat and carrot cubes flowing in starch solutions were described by these curves. The differential equation governing the logistic model was given by Nedler (1962):

$$\frac{dc}{dx} = \frac{B}{x}\left(\frac{c-a}{U}\right)\left(1-\left\{\frac{c-a}{U}\right\}^{x}\right) \qquad (43)$$

with a common solution known as generalized logistic curve:

$$c(\theta) = a + \frac{U}{\left(1+xe^{-B(\theta-H)}\right)^{1/x}} \qquad (44)$$

This equation will only be satisfactory when sufficient data are available for all three sections of the curve: the flat early part, the steep middle part and the flat final part, otherwise one or more of the parameters will become indeterminant(s). In this particular

case, the lower asymptote is set to zero. Consequently, a special case of the logistic model was employed by setting a at zero and x equal to 1:

$$c(\theta) = \frac{U}{(1 + e^{-B(\theta - H)})} \qquad (45)$$

The above equation is also known as *autocatalytic* or the *inverse exponential* curve as used by Zwietering *et al.* (1990, 1991) and van Impe (1992) for modelling of bacterial growth curves. The parameters B, U and H associated with this model can be defined to have relevance to the RTD studies: B which is the growth rate can be taken to represent the particle accumulation rate; U, the upper asymptote to indicate the upper limit particle concentration and H to represent the half concentration internal age as this corresponds to a time when cumulative concentration is one half of the maximum. These are illustrated in Figure 3.4 with the cumulative particle concentration shown as a function of time (both in dimensionless forms). Particle accumulation rate (B) is the first derivative of Eqn (43) with respect to time at the inflection point, with U representing accumulated concentration as time approaching infinity. Eqn (45) is therefore equivalent to the cumulative distribution function (*F-type* distribution, Eqn (13)) and its differential form will give the time-specific particle concentration distribution function (*E-type* distribution) shown earlier as Eqn (12).

Experimental data were fitted to different distribution models (including the logistic) using the Maximum Likelihood Program (MLP, Lawes Agricultural Trust, Rothamsted Experimental Station). MLP is a search procedure aimed at minimizing the sum of squares between the experimental and predicted values. The basic approach was

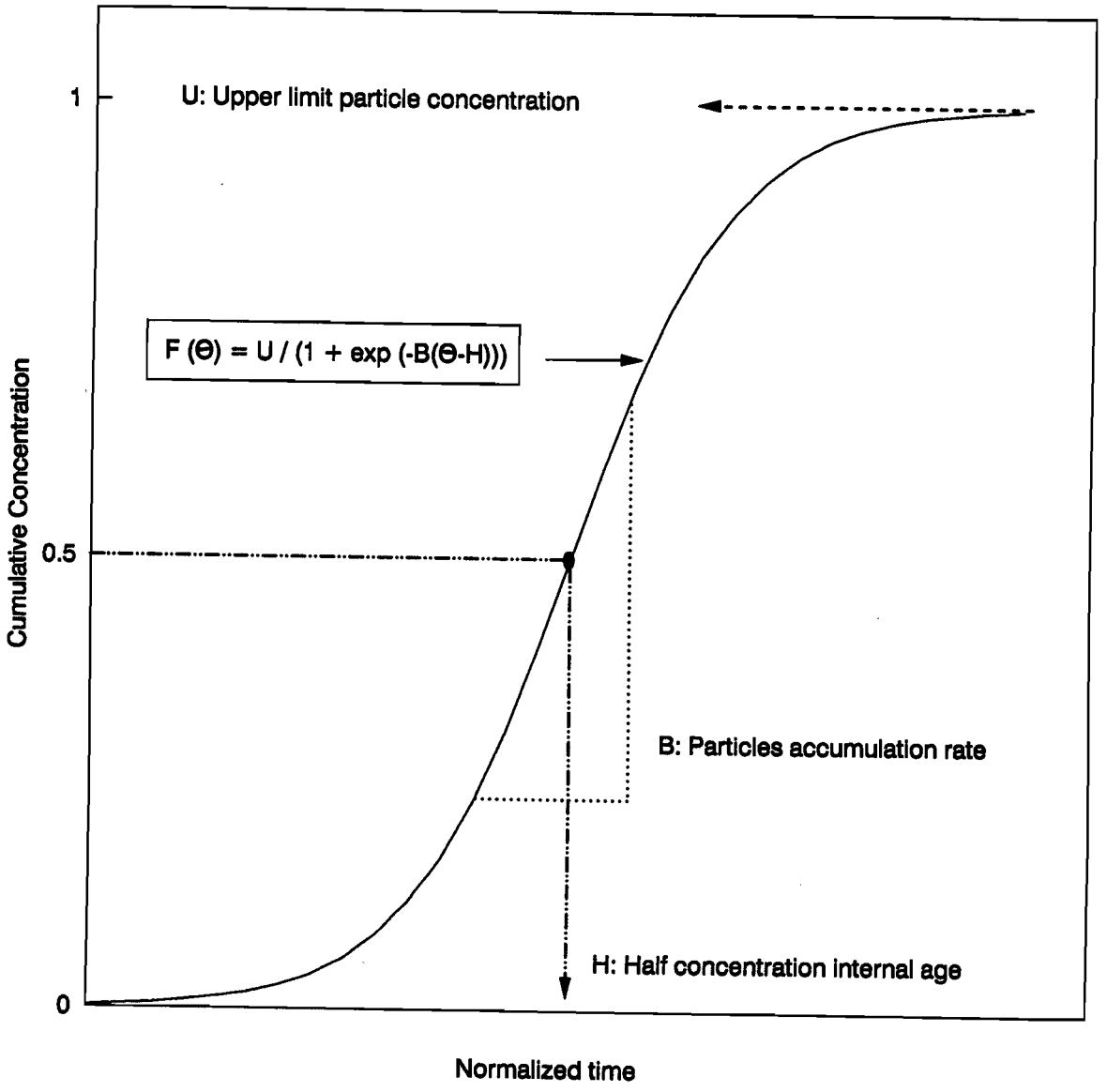

Figure 3.4. Logistic model for fitting the RTD data of meat and carrot cubes.

to fit the logistic curve to the cumulative concentration of the particles with normalized time as the explanatory variable (Abdelrahim *et al.*, 1993b). The starting values were automatically calculated by searching for the steepest part of the curve between four data points (estimate of B) and by taking the equilibrium value as an estimate of U (upper limit of cumulative particle concentration). The logistic model was chosen over the other distribution models, because of its inherent simplicity and good fit (the associated residual sum of squares was the lowest).

Statistical analyses were performed using the General Linear Model Procedure (SAS, SAS Institute Inc., Cary, NC; 1985) to test the effects of different factors on model parameters as obtained from the experimental data of meat and carrot. The parameters were: (1) particle accumulation rate, B (2) observed upper limit of particle concentration, U and (3) the half concentration internal age, H. B and U were independent of the nature of time scale (real or normalized). Since, H depended on the time scale, it was evaluated both on real (HRT) and normalized (HNT) time basis. Predictive equations were developed for the model parameters based on test factors using a multiple regression technique involving significant factors.

RESULTS AND DISCUSSION

RTD of Meat and Carrot Cubes

Figure 3.5 shows the RTD of meat cubes in a vertical SSHE as influenced by particle size, flow rate and starch concentration under the base conditions: 5% w/w starch

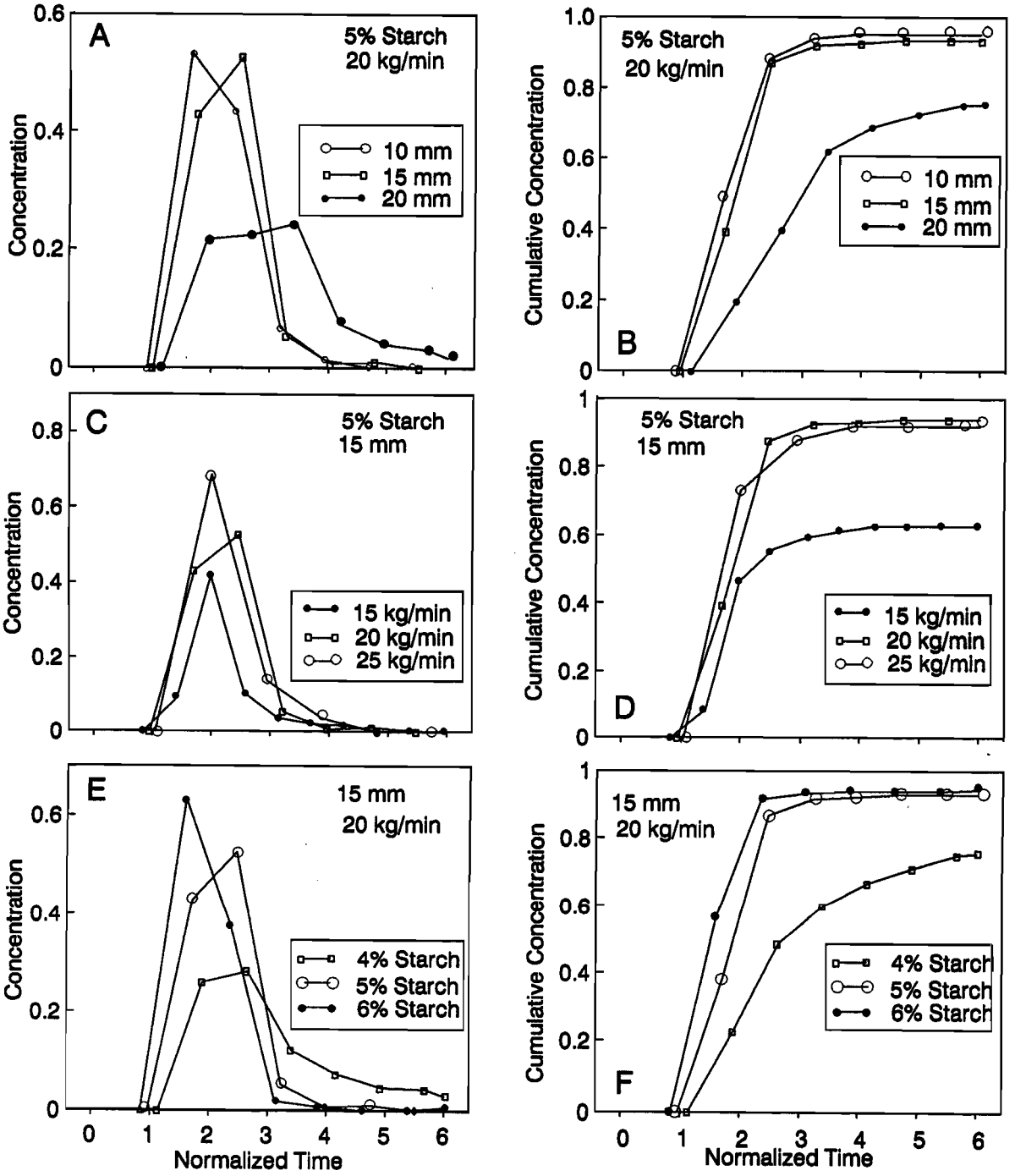

Figure 3.5. **RTD curves (E and F) of meat cubes in a vertical SSHE as influenced by particle size, flow rate and starch concentration under the base conditions of 15 mm particle size, 20 kg/min flow rate and 5% w/w starch concentration.**

concentration, 20 kg/min flow rate and 15 mm particle size. Larger meat particles (20 mm) had longer residence times and a broader distribution curve, probably due to the fact that heavier particles need a greater force for lifting up through the SSHE. Smaller particles (10 and 15 mm) moved faster with a narrower distribution (Figure 3.5, A-B). Movement of particles was also affected by flow rate. Higher flow rates tend to counteract the radial and vortex mixing generated by the shaft and scraper blades, and facilitate easier movement of the meat cubes through the SSHE. Increased flow rates also resulted in a larger amount of particles to exit in a given span of time (Figure 3.5, C-D). Increased starch concentration brought about the same effects as increasing flow rate (Figure 3.5, E-F). Higher starch concentration resulted in reduced residence times and narrower RTD curves. Since the density of all starch preparations of starch were kept at 1077 kg/m^3, this effect could be attributed to the carrier fluid apparent viscosity. It was necessary to set the mutator speed at 150 rpm and increase the starch solutions density to 1077 kg/m^3 by adding sugar to pass meat cubes through the SSHE. Without added sugar, it was not possible to pass meat cubes through the system. Higher speeds of the mutator facilitate faster heat transfer, but at the same time they increase the particle radial flow causing them to retain longer in the SSHE. The trends observed with the carrot cubes (Figure 3.6) were similar, but the effect of flow rate was more pronounced due the fact that carrot sizes were smaller and they were lighter than meat cubes (density of meat and carrot cubes were 1110 and 1040 kg/m^3, respectively).

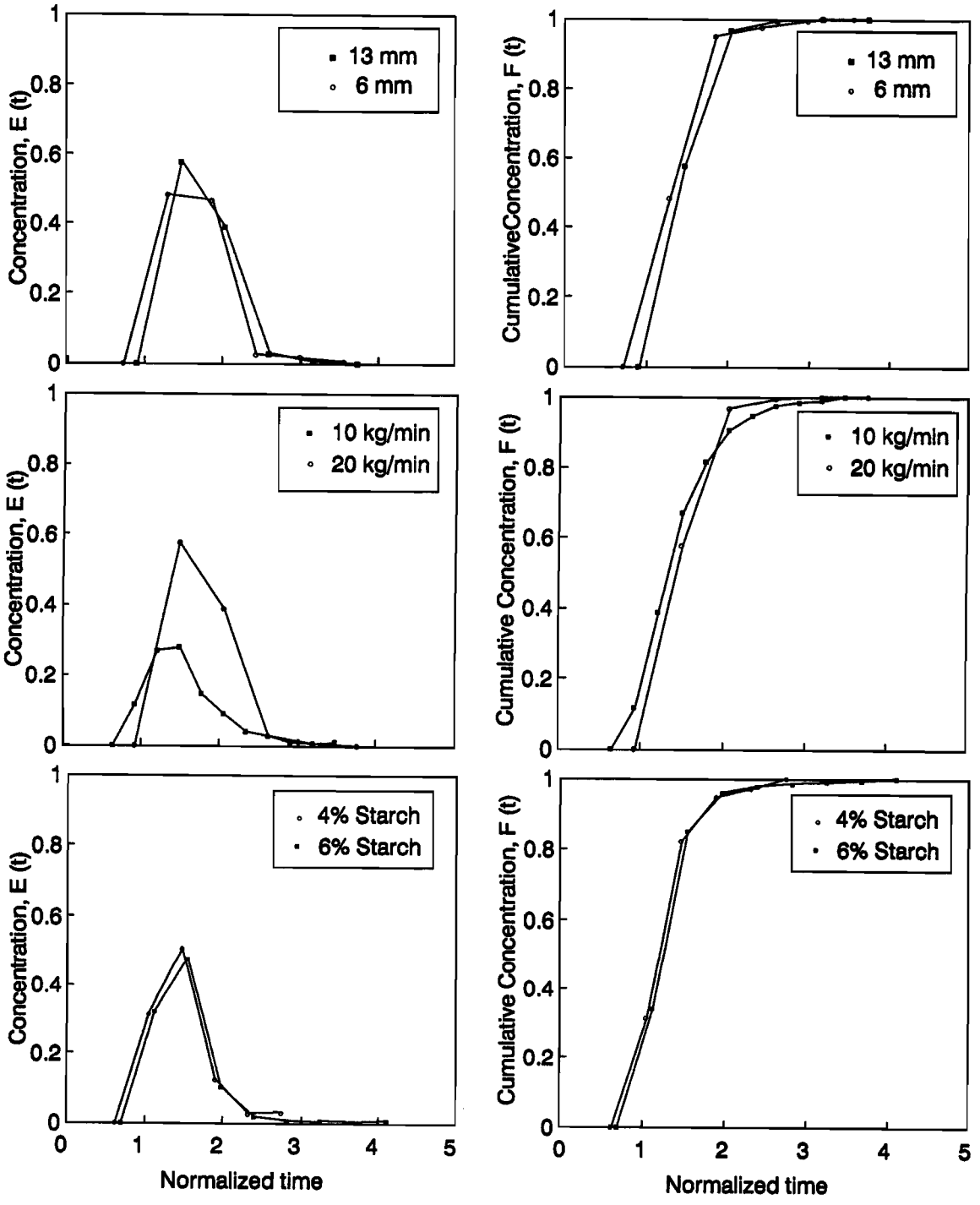

Figure 3.6. **Residence time distribution curves (E and F) of carrot cubes as influenced by the holding tube at conditions of 13 mm particle size, 10 kg/min flow rate and 4% w/w starch concentration.**

Statistical Analysis of the RTD Parameters

Fastest Particle Residence Time (FPRT & FPNRT)

Analysis of variance results for the RTD of meat and carrot cubes in the vertical SSHE are summarized in Table 3.2. All main effects significantly ($p < 0.001$) affected the fastest particle residence times (both FPRT and FPNRT). Flow rate and starch concentration were the major contributors to the variability of the FPRT, whereas for the FPNRT starch concentration and particle size were the major factors. Major interactions were starch concentration-particle size and starch concentration-flow rate with both FPRT and FPNRT. Similar trends were observed with the carrot cubes except that flow rate was the dominant factor for both FPRT and FPNRT contributing 85 and 55% of the total variability, respectively, while starch concentration-flow rate was the major interaction.

Duncan's multiple range tests on FPRT and FPNRT are shown in Table 3.3. FPRT of meat cubes decreased with starch concentration with a significant change between 4 and 5% concentrations, while for carrot FPRT increased with starch concentration. These differences were ascribed to the lower density of carrot cubes as compared to meat cubes, both in relation to the density-enriched starch solutions. The heavier meat cubes appeared to stay longer especially at lower starch concentrations. This obviously was not a barrier for carrot cubes. FPRT of both meat and carrot cubes increased with particle size with the 20 mm meat cube showing a break-away increase from 10 and 15 mm particles. Higher flow rates decreased the FPRT of both meat and carrot cubes. Similar trends were observed with FPNRT of both meat and carrot cubes under all conditions. The overall trend was: meat cubes had longer relative residence times compared to the carrier fluid

Table 3.2. **Analysis of variance of the residence time associated with the fastest particle (FPRT, FPNRT), mean particle (MPRT) and the standard deviation (SRTD) of meat and carrot cubes in a vertically oriented scraped surface heat exchanger.**

	FPRT		FPNRT		MPRT		SRTD	
Source	Meat	Carrot	Meat	Carrot	Meat	Carrot	Meat	Carrot
Main effects	69.2*	92.1	60.1	76.3	73.9	94.5	73.2[a]	91.5[a]
Starch Conc (C)	23.5[a]	5.3[a]	32.8[a]	16.9[a]	34.0[a]	3.0[a]	43.8[a]	2.1[b]
Particle Size (P)	14.2[a]	1.7[a]	23.5[a]	4.3[a]	7.3[a]	0.0[n]	6.1[c]	0.1[n]
Flow Rate (F)	31.5[a]	85.1[a]	3.9[b]	55.1[a]	32.6[a]	91.5[a]	23.2[a]	89.3[a]
Interactions	28.4	6.8	29.7	19.9	15.6	4.9	8.8	6.0
C x P	10.7[a]	0.8[b]	17.5[a]	2.3[b]	3.4[n]	0.9[a]	0.5[n]	0.9[c]
C x F	10.9[a]	3.3[a]	10.0[a]	10.9[a]	9.6[a]	3.5[a]	2.0[n]	3.5[a]
P x F	1.4[n]	0.7[b]	0.4[n]	0.9[n]	1.1[n]	0.4[b]	1.6[n]	1.0[c]
C x P x F	2.4[n]	2.1[a]	1.9[n]	5.8[a]	1.6[n]	0.2[n]	4.6[n]	0.7[n]
Residual	5.4	1.1	10.3	3.8	10.5	0.6	18.1	2.5
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

[a] [b] [c]Significant $p < 0.001$, $p < 0.01$, $p < 0.05$ respectively.
[n]Not significant $p > 0.05$.
*Percentage contribution to the total sum of squares (% SS).

Table 3.3. **Duncan's Multiple range test on the fastest particle and mean particle residence times of meat and carrot cubes in a vertical scraped surface heat exchanger and the associated standard deviation as influenced by the different test factors.**

	FPRT (min)		FPNRT		MPRT (min)		SRTD (min)	
Level	Meat	Carrot	Meat	Carrot	Meat	Carrot	Meat	Carrot
Starch Concentration (% w/w)								
4	0.72^{a}	0.33^{a}	1.49^{a}	0.61^{a}	1.80^{a}	0.81^{a}	0.79^{a}	0.25^{a}
5	0.46^{b}	0.37^{b}	1.00^{b}	0.68^{b}	1.10^{b}	0.84^{b}	0.46^{b}	0.22^{b}
6	0.41^{b}	0.38^{c}	0.90^{b}	0.71^{b}	1.02^{b}	0.91^{b}	0.30^{b}	0.22^{b}
Particle Size (mm)								
6	----	0.35^{a}	----	0.64^{a}	----	0.85^{a}	----	0.23^{a}
13	----	0.37^{b}	----	0.69^{b}	----	0.85^{a}	----	0.23^{a}
10	0.44^{a}	----	0.94^{a}	----	1.16^{a}	----	0.42^{a}	----
15	0.48^{a}	----	1.02^{a}	----	1.23^{a}	----	0.52^{ab}	----
20	0.67^{b}	----	1.44^{b}	----	1.54^{b}	----	0.61^{b}	----
Flow Rate (kg/min)								
10	----	0.49^{a}	----	0.56^{a}	----	1.23^{a}	----	0.38^{a}
15	0.73^{a}	0.38^{b}	1.24^{a}	0.64^{b}	1.76^{a}	0.86^{b}	0.72^{a}	0.23^{b}
20	0.50^{b}	0.32^{c}	1.12^{ab}	0.73^{c}	1.25^{b}	0.71^{c}	0.46^{b}	0.17^{c}
25	0.36^{c}	0.26^{d}	1.03^{b}	0.73^{c}	0.92^{c}	0.61^{d}	0.37^{b}	0.14^{d}

^{abcd}Means within columns within the same parameter having the same letter are not significantly different at $p > 0.05$.

except for 6% starch and 10 mm meat cubes, while carrot cubes had shorter relative residence times under all conditions (Table 3.3).

Mean Particle Residence Time (MPRT)

The mean particle residence time (MPRT) of meat cubes was also significantly influenced ($p < 0.001$) by all main effects (Table 3.2). Starch concentration and flow rate were the dominant factors, while starch concentration-flow rate was the major interaction with meat cubes. Flow rate was the only the major factor contributing the variability (> 90%) of MPRT of carrot cubes, while particle size was non-significant ($p > 0.05$) and interactions contributed only ~5% of total variability. Duncan's multiple range tests revealed that MPRT of meat cubes had a behaviour similar to FPRT: increased with particle size and decreased with starch concentration and flow rate (Table 3.3). Trends were similar with carrot cubes except that MPRT increased with increased starch concentration in a similar fashion to the FPRT.

Standard Deviation of Residence Time (SRTD)

Analysis of variance of standard deviation of the RTD data of meat cubes (SRTD) also showed that all test factors were significant, $p < 0.001$ (Table 3.2). Starch concentration and flow rate were the major factors contributing to variability, while all interactions were insignificant ($p > 0.05$). With carrot cubes flow rate was the major factor (89% of total variability), while particle size was insignificant ($p > 0.05$). Duncan's multiple range tests indicated that SRTD was affected by all different levels of test factors

in a fashion similar to that of the FPRT and MPRT (Table 3.3). SRTD of both meat and carrot cubes decreased with starch concentration and flow rate. Increased particle size resulted in increased SRTD meat cubes, whereas SRTD of carrot cubes was unaffected.

Modelling of the RTD Experimental Data

The generalized logistic model gave an excellent fit for the *F*-type RTD data for meat cubes under all conditions tested ($r^2 > 0.95$) as presented in Figure 3.7. The fit was also good when the model-predicted cumulative RTD data were converted to the time-specific particle concentration data (Figure 3.7, *E*-type curves). Cumulative RTD data of carrot cubes were described equally well by the logistic model (Figure 3.8). Fastest and mean residence times from the model-predicted *E*-type RTD for both meat and carrot cubes showed a general agreement with the experimental values.

Statistical Analysis of the Model Parameters

In order for the model parameters, H, B and U, to be useful they needed to be expressed as a function of process/product related factors. But before a multiple regression technique could be used to develop a predictive model, it was necessary to find out what factors were significant in terms of influencing the model parameters. An analysis of variance was carried out for this purpose.

Half Concentration Residence Time (HRT & HNT): Analysis of variance on parameters associated with generalized logistic model of both meat and carrot cubes is

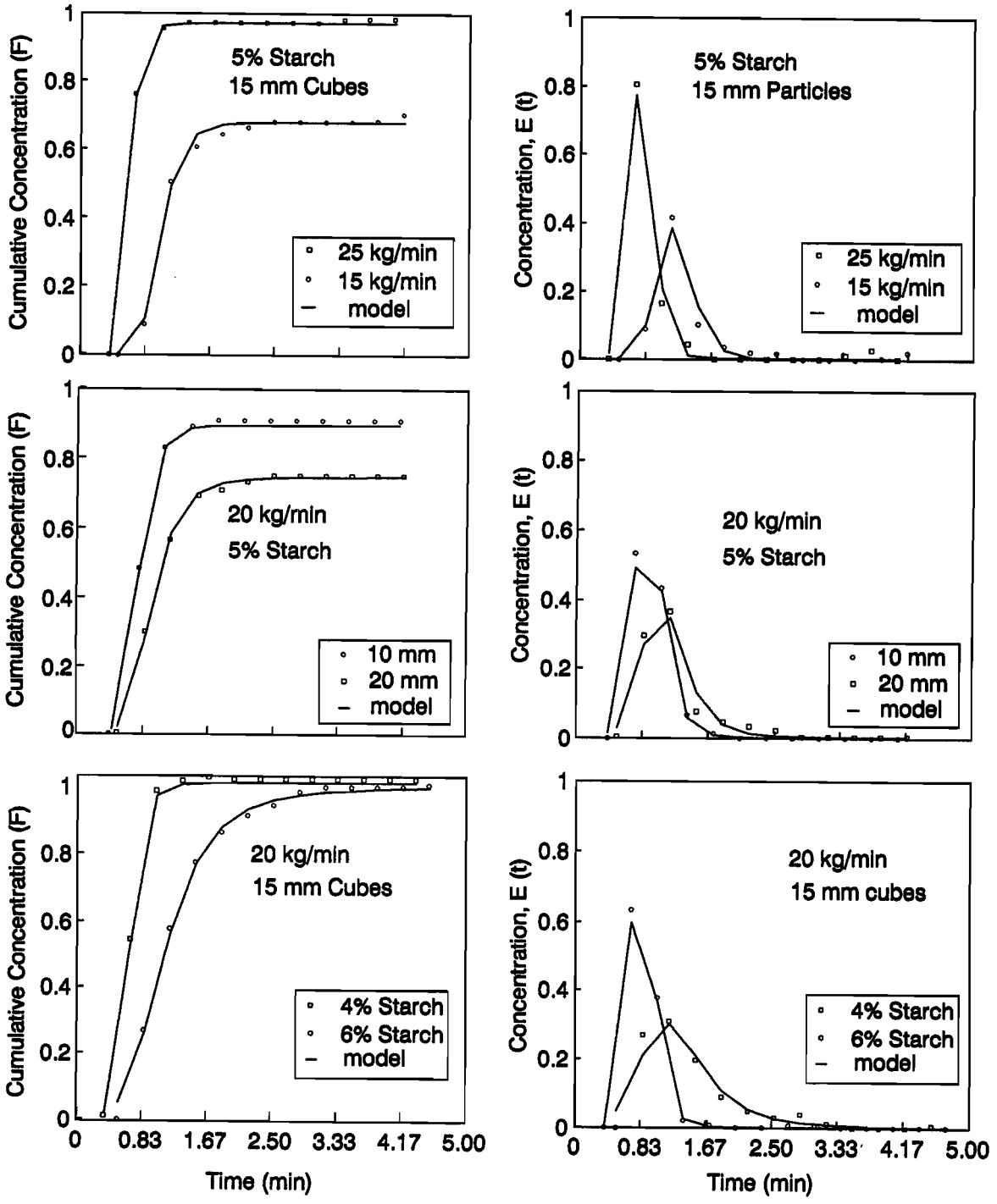

Figure 3.7. **The fit of the generalized logistic model over the experimental RTD data (*F*- and *E*-type curves) of meat cubes in a vertical scraped surface heat exchanger (SSHE) under the base conditions: 20 kg/min flow rate, 15 mm particle size and 5% w/w starch concentration.**

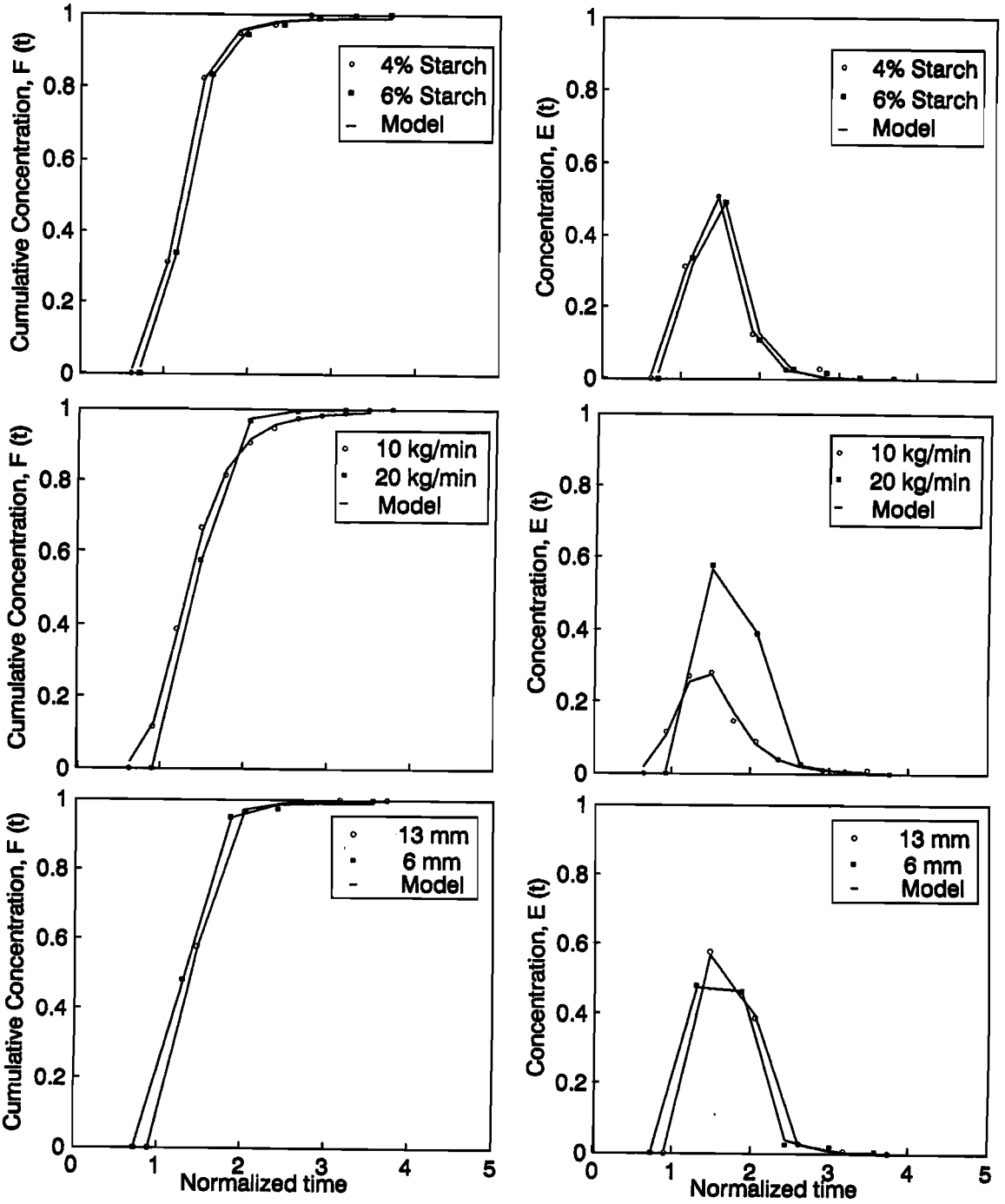

Figure 3.8. **The fit of the generalized logistic model over the experimental RTD data (*F*- and *E*-type curves) of carrot cubes in a vertical scraped surface heat exchanger (SSHE) under the base conditions: 10 kg/min flow rate, 13 mm particle size and 5% w/w starch concentration.**

presented in Table 3.4. Half concentration real and normalized time (HRT and HNT) of meat cubes were significantly ($p < 0.01$) influenced by all main effects (Table 3.4), except flow rate with respect to HNT ($p > 0.05$). Starch concentration was the main source of variation for both HRT and HNT. Only interaction terms involving starch concentration were significant ($p < 0.05$), especially with flow rate (~ 13 and 11% of HRT and HNT variability, respectively). With carrot cubes, flow rate was the dominant factor with ~91 and 40% contribution to variability of HRT and HNT, respectively. Particle size was non-significant ($p > 0.05$) and interactions accounted for 5% of the variability of HRT and ~32% of HNT.

Duncan's multiple range tests (Table 3.5) indicated that HRT and HNT of meat cubes increased with particle size, while they decreased with increased starch concentration and flow rate. However, with carrot cubes both HRT and HNT increased with starch concentration and decreased with flow rate (Table 3.5). It is worthwhile to note that both MPRT (normal distribution) and HRT (logistic model) respond to test factors in a similar way, although they differ in magnitude. This implies that the RTD data somewhat deviated from normality (skewed to the right).

Particle Accumulation Rate (B): Particle accumulation rate (B) of meat was only affected ($p < 0.01$) by starch concentration and flow rate. All other factors and interactions were non-significant ($p > 0.05$). The accumulation of carrot cubes was influenced by all main factors, but flow rate was dominant with a 76.5% contribution to variability. Duncan's multiple range tests showed that B of both meat and carrot cubes

Table 3.4. Analysis of variance of the residence time parameters associated with the logistic model (HRT, HNT, B and U) for meat and carrot cubes flowing in the vertical SSHE.

	HRT[1]		HNT		B		U[2]
Source	Meat	Carrot	Meat	Carrot	Meat	Carrot	Meat
Main Effects	63.2	94.4	50.2	64.2	46.4	87.9	51.8
Starch Conc (C)	30.5^{a}	3.2^{a}	36.1^{a}	24.1^{a}	25.5^{a}	4.4^{b}	28.0^{a}
Particle Size (P)	6.6^{b}	0.0^{n}	9.5^{b}	0.0^{n}	5.2^{n}	7.0^{a}	10.7^{a}
Flow Rate (F)	26.1^{a}	91.2^{a}	4.59^{n}	40.2^{a}	15.7^{b}	76.5^{a}	13.1^{a}
Interactions	28.9	5.1	29.7	31.8	26.1	7.0	37.8
C x P	5.9^{c}	0.8^{a}	7.6^{n}	9.4^{a}	9.2^{n}	0.9^{n}	7.9^{c}
C x F	12.9^{a}	3.8^{a}	11.5^{c}	15.7^{a}	9.1^{n}	2.4^{n}	12.8^{b}
P x F	4.1^{n}	0.3^{c}	4.6^{n}	2.9^{c}	2.3^{n}	0.9^{n}	7.4^{c}
C x P x F	6.0^{c}	0.2^{n}	6.0^{n}	3.7^{b}	5.5^{n}	2.8^{n}	9.7^{c}
Residual	7.9	0.5	20.1	4.0	27.6	5.1	10.4
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0

[1]Percentage contribution to the total sum of squares (%SS).
[2]Not applicable to carrot data, U = 1 under all conditions.
[a] [b] [c]Significant $p < 0.001$, $p < 0.01$, $p < 0.05$; respectively.
[n]Not significant $p > 0.05$.

Table 3.5. Duncan's Multiple range test on the parameters associated with the generalized logistic model: HRT, HNT (half concentration internal age) and B (particle accumulation rate) for meat and carrot cubes in a vertical scraped surface heat exchanger.

	HRT (min)		HNT		B		U
Level	Meat	Carrot	Meat	Carrot	Meat	Carrot	Meat
Starch Concentration (% w/w)							
4	1.54^{a}	0.65^{a}	3.12^{a}	1.18^{a}	9.32^{a}	17.2^{a}	0.78^{a}
5	0.82^{b}	0.69^{b}	1.79^{b}	1.25^{b}	16.4^{b}	17.3^{a}	0.95^{b}
6	0.81^{b}	0.75^{c}	1.76^{b}	1.32^{c}	22.2^{c}	18.9^{b}	0.98^{b}
Particle Size (mm)							
6	----	0.69^{a}	----	1.26^{a}	----	16.8^{a}	----
13	----	0.70^{a}	----	1.25^{a}	----	18.8^{b}	----
10	0.93^{a}	----	1.98^{a}	----	18.0^{a}	----	0.96^{a}
15	0.96^{a}	----	2.01^{a}	----	17.2^{a}	----	0.92^{a}
20	1.28^{b}	----	2.69^{b}	----	12.6^{b}	----	0.83^{b}
Flow Rate (kg/min)							
10	----	1.03^{a}	----	1.17^{a}	----	13.5^{a}	----
15	1.45^{a}	0.70^{b}	2.48^{a}	1.19^{a}	12.1^{a}	16.0^{b}	0.82^{a}
20	1.04^{b}	0.57^{c}	2.27^{ab}	1.29^{b}	14.0^{a}	20.3^{c}	0.92^{b}
25	0.68^{c}	0.48^{d}	1.93^{b}	1.36^{c}	21.7^{b}	21.4^{d}	0.97^{b}

^{abcd}Means with the same superscript within the levels of the same factor are not significantly different at $p > 0.05$.

increased with starch concentration and flow rate, and decreased with particle size (Table 3.5).

Maximum Particle Accumulation (U): Upper limit of particle concentration (U) of meat, though supposed to be a non-variant parameter and equal to unity, was significantly influenced by all of the main factors ($p < 0.001$) as well their interactions ($p < 0.05$) as shown in Table 3.4. Starch concentration was major source of variation among the main effects (~ 28%), whereas starch concentration-flow rate was the major interaction (~ 13%). Complete recovery of the carrot cubes was achieved under all conditions (U = 1.0). This reflects that the type of particle used and its characteristics are important for particle RTD in the aseptic system under consideration. Duncan's multiple range tests indicated that U increased with increased starch concentration and flow rate, while decreased with increased particle size (Table 3.5). Conditions indicating significant deviations in U from unity will result in particle accumulation in the system and may cause subsequent blockage.

Multiple Regression on the Model Parameters

Multiple Regression analysis of the model parameters on selected factors (Table 3.6) showed a good agreement between experimental and calculated parameters for both meat and carrot with a better fit for the latter. The associated determination coefficients varied from 0.62 to 0.76 for meat and 0.76 to 0.83 for carrots. Predicted *vs* experimental data for meat and carrot cubes are shown in Figure 3.9 demonstrating a reasonable fit.

Table 3.6. Multiple regression coefficients associated with the logistic model parameters for carrot and meat cubes in the vertical scraped surface heat exchanger (SSHE).

	Regression Coefficients						
	HRT		HNT		B		U
Variables	Meat	Carrot	Meat	Carrot	Meat	Carrot	Meat
Constant	8.098	0.875	12.14	0.318	-33.31	8.458	0.320
Starch conc. (C)	-1.087	0.075	-1.640	0.119	6.823	-0.098	0.222
Particle size (P)	0.121	0.026	0.230	0.047	2.411	-0.307	-0.115
Flow rate (F)	-0.384	-0.026	-0.579	0.025	-0.158	0.304	0.061
C x P	-0.026	-0.004	-0.054	-0.007	-0.446	0.060	0.012
C x F	-0.056	-0.001	0.089	-0.001	0.214	0.021	-0.015
P x F	0.002	-0.001	0.005	-0.001	-0.024	0.016	0.002
R^2	0.761	0.814	0.723	0.763	0.701	0.826	0.618
Standard Error	0.393	0.096	0.795	0.094	3.062	1.553	0.103

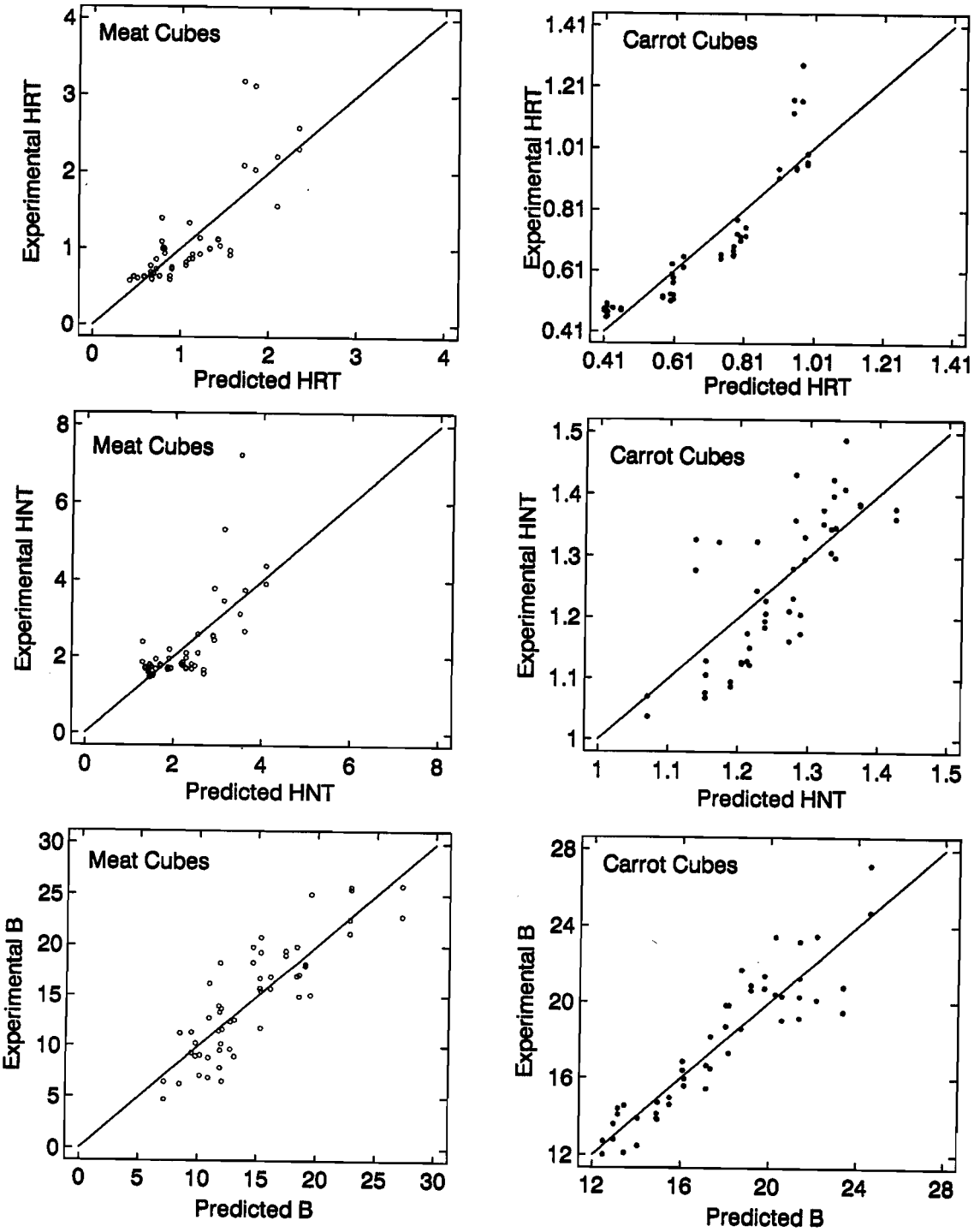

Figure 3.9. **Experimental *vs* calculated HRT, HNT and B for meat and carrot cubes in the vertical SSHE using multiple regression analysis.**

CONCLUSIONS

RTD parameters associated with meat and carrot cubes in the vertical SSHE were influenced by particle size, carrier fluid concentration and flow rate. Residence time of meat particles (FPRT, FPNRT, MPRT, SRTD) decreased with starch concentration and flow rate, while increase with particle size. Similar trends were observed with carrot cubes except for starch concentration effect which resulted in an increase in RTD. Density differences between meat and carrot cubes were considered to be the primary reason for this behaviour. Carrot cubes had a relatively smaller RT than meat cubes.

The logistic model well described the RTD behaviour of both meat and carrot cubes, and the model parameters were responsive to test factors. A predictive model, giving a good fit, was developed for the model parameters based on test factors found to have a significance influence.

CHAPTER IV

RESIDENCE TIME DISTRIBUTIONS OF MEAT AND CARROT CUBES IN STARCH SOLUTIONS IN THE HOLDING TUBE OF AN ASEPTIC PROCESSING SYSTEM

ABSTRACT

Residence time distributions (RTD) of meat and carrot cubes were evaluated in the holding tube of a commercial pilot scale aseptic processing system using a full factorial design of experiments employing holding tube length (4.6, 9.2, 13.8 and 18.4 m), flow rate (10/15 and 20 kg/min), particle (10 and 20 mm meat cubes and 6 and 13 mm carrot cubes) and concentration of the carrier fluid (4 and 6% w/w starch) as factors. All test factors significantly ($p < 0.05$) influenced the fastest particle residence time (FPRT) and its normalized version (FPNRT), the mean particle residence time (MPRT) and standard deviation of RTD (SRTD). Experimental RTD was well described by a generalized logistic model ($R^2 > 0.95$). The model parameters were related to test factors and could be well described using multiple regression analyses of test factors and their interactions.

INTRODUCTION

In the previous chapter, the residence time distributions behaviour of meat and carrot cubes were evaluated in the scraped surface heat exchanger (SSHE). All test factors

studied were shown to influence RTD parameters which was described by logistic model. In aseptic processing systems, generally there will be at least one SSHE for heating and one for cooling together with a set of holding tubes, the length of which depends on the required holding time for a given throughput capacity. Although product heating occurs in the heating SSHE, but FDA proposes to take into credit toward establishing processes only the lethality contributed in the holding section. Hence many of the residence time distribution studies (McCoy *et al.*, 1987; Sastry and Zuritz, 1987; Berry, 1989; Richardson and Holdsworth, 1989; Ramaswamy *et al.*, 1992; Yang and Swartzel, 1992; Tucker and Withers, 1992) have been carried out using only holding tubes.

The objective of this phase of study (Chapter IV) was to evaluate the RTD behaviour of meat and carrot cubes in the holding tube (various lengths) of a commercial pilot scale aseptic processing system, as influenced by various factors (particle size, carrier fluid concentration and flow rate) and to examine the fit of the generalized logistic model to the experimental RTD data.

MATERIALS AND METHODS

Materials

Meat (10 and 20 mm) and carrot (6 and 13 mm) cubes were obtained, stored and prepared as described in Chapter III. The carrier fluid employed was either 4% or 6% w/w food grade starch (Therm-flo; National Starch and Chemical Company, Bridgewater, NJ) prepared as detailed in Chapter III.

Holding Tube

The system used in this study consisted of a series of holding tubes, the length of which could be varied by selecting the number of turns (~4.6 m/turn). Each turn was composed of two identical tubes connected together via a bend. The holding tube was inclined upwards at an angle of 1.19° in accordance with the FDA regulations. A variable speed positive displacement tri-lobe pump (Albin Pump, Atlanta, GA) was used for feeding the system with the carrier fluid and meat/carrot particles as regulated by a three-way valve. The layout of the experimental set-up is shown in Figure 4.1, and the dimensions of the holding tube are listed in Table 4.1. Test runs were carried out as detailed in Chapter III.

Experimental Design

A full factorial design of experiments was employed involving four levels of holding tube length (4.6, 9.2, 13.8 and 18.4 m) and two levels of flow rate (15 and 20 kg/min for meat cubes; 10 and 20 kg/min for carrot cubes), starch concentration (4 and 6% w/w starch) and particle size (10, 20 mm meat and 6, 13 mm carrot cubes). All the experiments were performed in duplicates. Statistical analyses were performed using the General Linear Model (GLM) Procedure (SAS, SAS Institute Inc., Cary, NC; 1985) to test the effects of different factors on the residence time associated with the (1) the fastest particle and its normalized version (FPRT and FPNRT), (2) mean particle (MPRT) and (3) the standard deviation of residence time (SRTD) as detailed in Chapter III. Further statistical analyses, model fitting, influence of test factors on model parameters (half

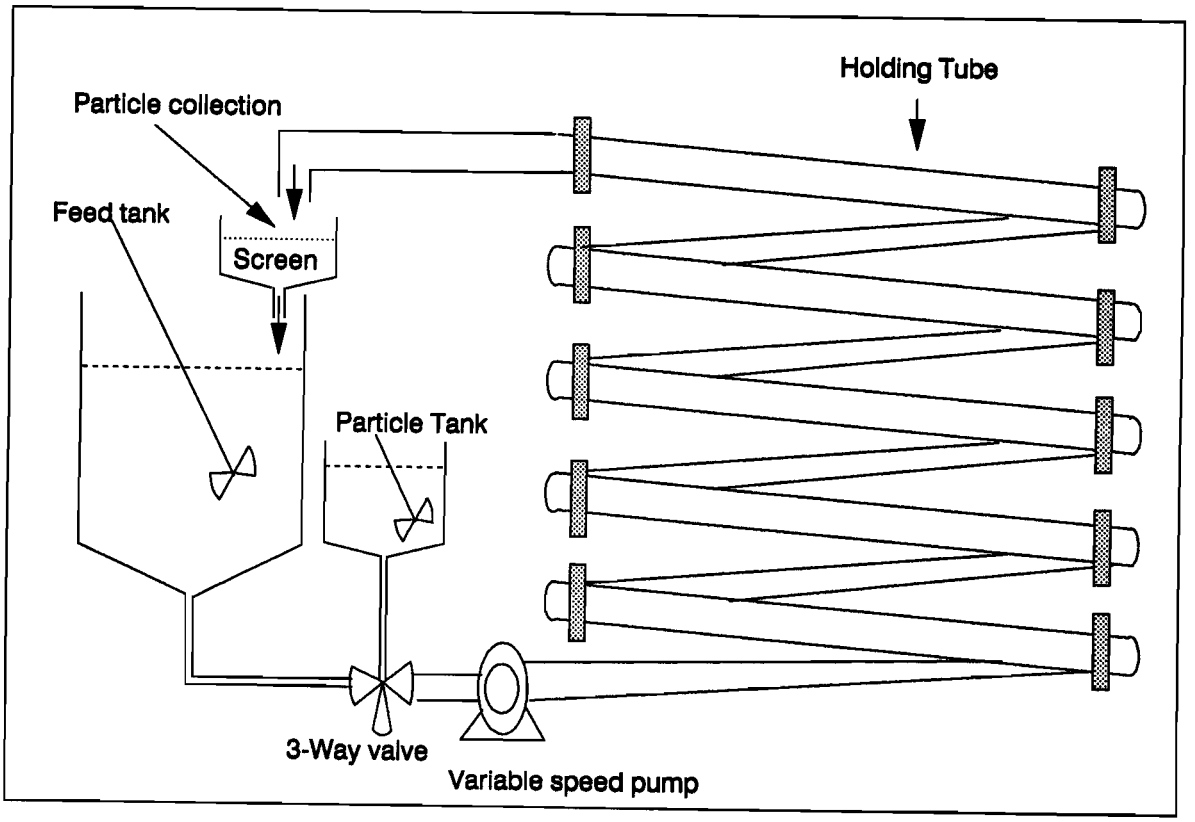

Figure 4.1. **Layout of experimental set-up used to study RTD of meat and carrot cubes in the holding tube of a pilot aseptic processing system.**

Table 4.1. Specifications of the aseptic processing system used to study the residence time distribution of carrot particles.

Parameter	Specification		
Scraped surface heat exchanger			
Scraped surface heat exchanger	1.8 X 0.9 m		
Internal diameter	0.152 m		
Internal shaft diameter	0.076 m		
Length of the heating surface	0.762 m		
Material of construction	Stainless steel		
Dasher speed	300 rpm		
Heating medium	Steam at 150°C		
Cooling medium	Water at 10°C		
Holding Tube			
Total length	26.65 m		
Internal diameter	0.0381 m		
Number of turns	4		
Length of the turn	4.6 m		
Carrier fluid	Therm-flo® starch		
Gelatinization temperature	140		°C
Concentration	4	6	% w/w
Density (before adjustment)	1011	1019	kg/m^3
Adjusted density	1077		kg/m^3
Flow rate (meat cubes)	15	20	kg/min
Flow rate (carrot cubes)	10	20	kg/min
Particles	Meat	Carrot	Cubes
Cube Size	10 20	6 13	mm
Concentration	5	5	% w/w
Density	1110	1040	kg/m^3

concentration internal age, HRT & HNT; particle accumulation rate, B) and multiple regression analyses of model parameters on test factors and their interactions were also carried out as detailed in Chapter III.

RESULTS AND DISCUSSION

RTD of Meat and Carrot Cubes

Representative RTD curves (*E* and *F*-types) for meat (20 mm) and carrot (13 mm) cubes in holding tubes of different lengths (4.6, 9.2, 13.6 and 18.4 turns) are shown in Figure 4.2 under the conditions of 4% w/w carrier fluid concentration, with a flow rate 15 kg/min (with meat cubes) and 10 kg/min (carrot cubes). Increased holding tube length resulted in increased residence times (spreading RTD curve) with a slightly broader spread (Figure 4.2A, B). The *F*-type curves (Figure 4.2C, D) indicated curves of parallel nature with a shift toward generally larger residence time as the holding tube length increased. Further results on the effects of particle size, flow rate and carrier fluid concentration on the RTD of meat and carrot cubes at an intermediate holding tube length of (13.6-18.4 m) are shown in Figures 4.3 and 4.4. The RTD of meat particles of the large size (20 mm) was associated with a slightly longer residence time than the 10 mm cubes (Figure 4.3A). This is in contradiction to the findings of McCoy *et al.* (1987) and Ramaswamy *et al.* (1992), who reported larger particles to travel faster. This may have been caused by the unusually large size particle used in these studies relative the holding tube diameter.

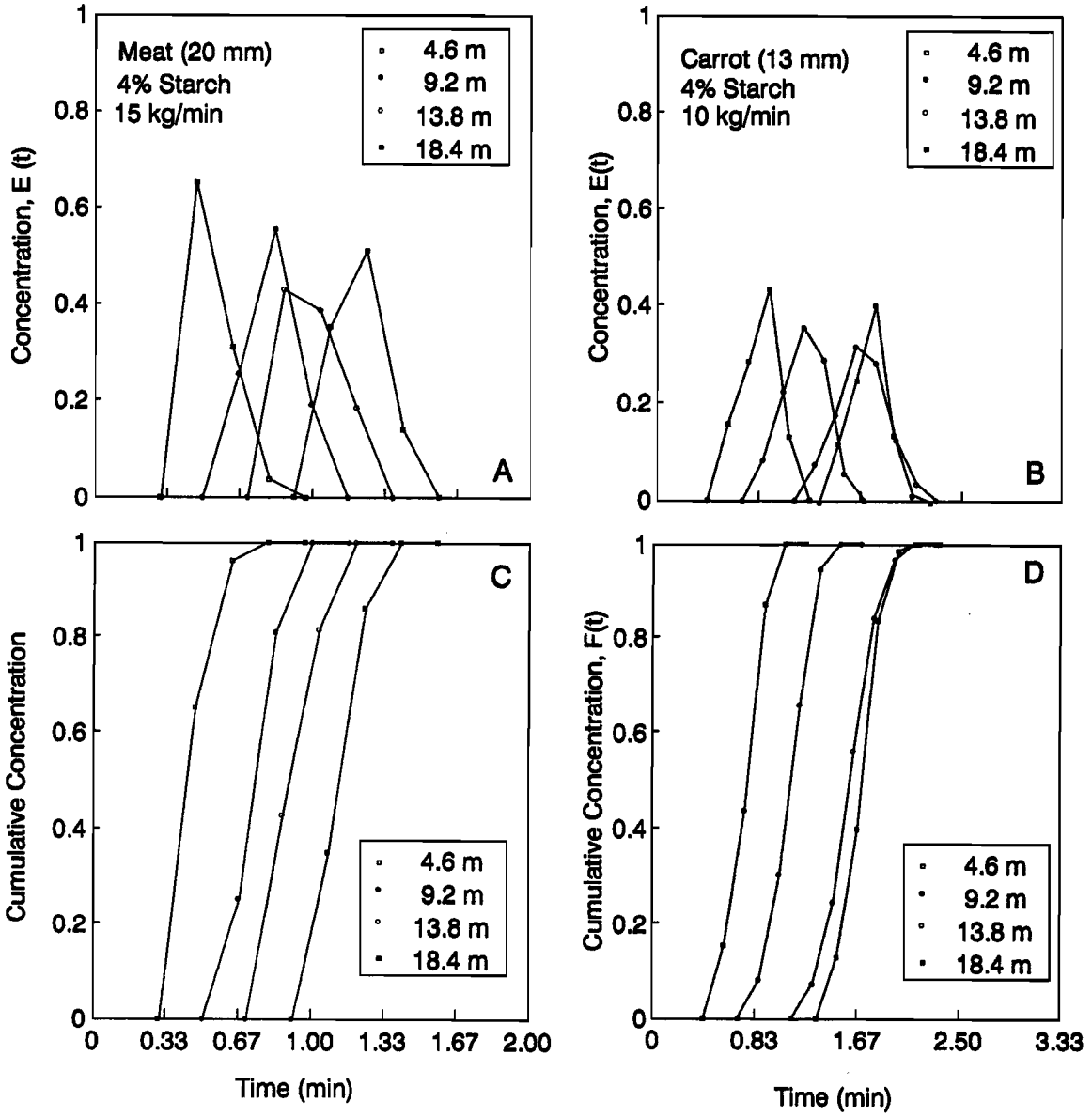

Figure 4.2. **Residence time distribution curves (*E* and *F*) of meat and carrot cubes as influenced by the holding tube at 4% w/w starch concentration (20 and 13 mm particle size; 15 and 10 kg/min flow rate).**

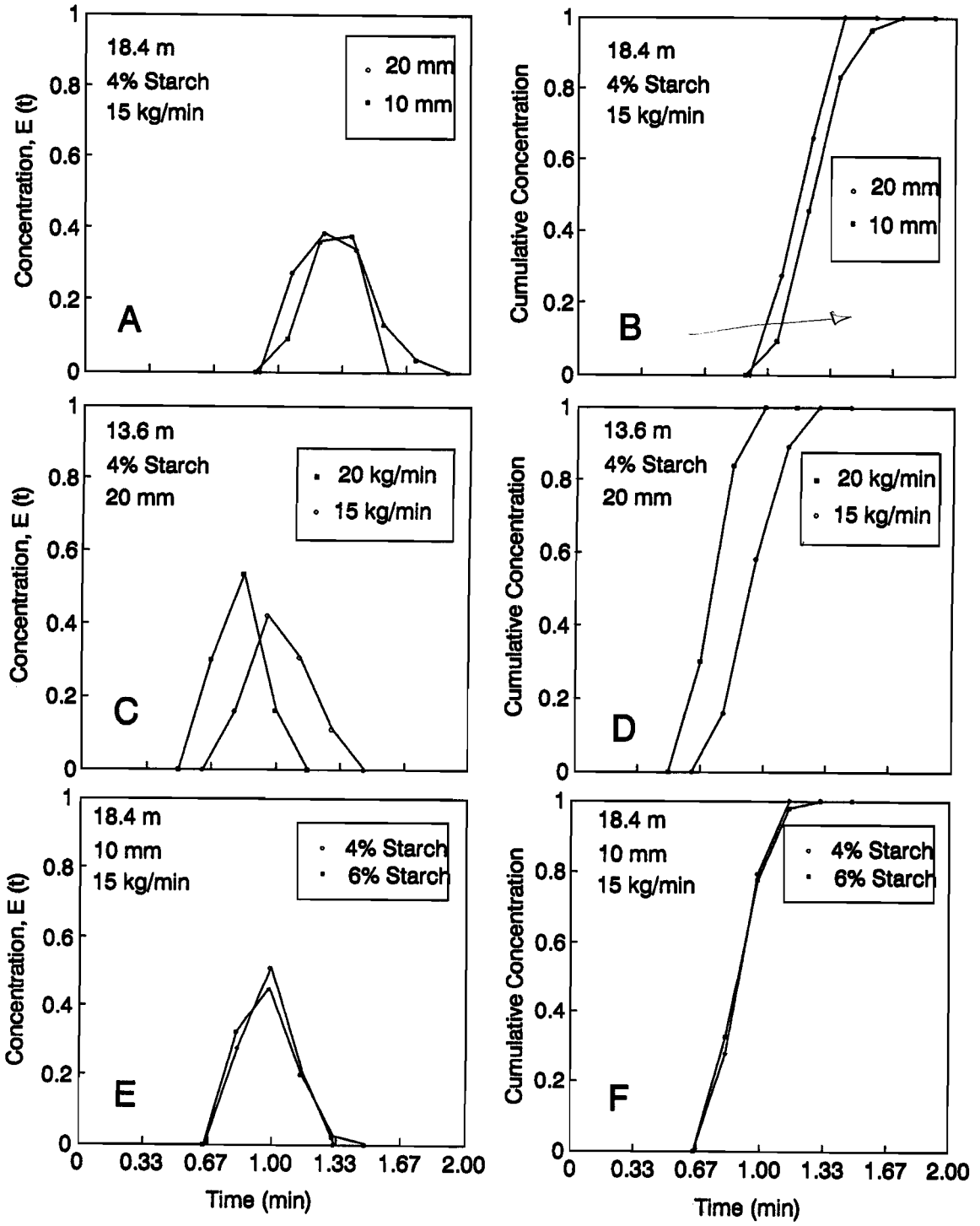

Figure 4.3. **RTD curves (*E* and *F*) of meat cubes in a holding tube as influenced by mass flow rate, particle size and starch concentration under the base conditions of 18.4 m holding tube length, 15 kg/min flow rate, 20 mm particle size and 4% w/w starch concentration.**

The larger meat particle was a cube of 20 mm dimension which would give an equivalent diameter (d_e = volume/surface area) of about 25 mm occupying more than half of the radial cross section. Moreover, meat cubes were heavier than the carrier fluid and they tended to drag at the bottom of the tube as previously noted by Richardson and Holdsworth (1989). Higher flow rates resulted in shorter residence times and a narrower RTD curve (Figure 4.3C, D), while carrier fluid concentration did not appear to have much influence on the RTD of meat cubes (Figure 4.3E, F).

The effect of test factors was more obvious on the RTD of carrot cubes than with meat particles (Figure 4.4 shown for 18.4 m holding tube length at the base conditions: 10 kg/min flow rate, 13 mm particle size and 4% w/w starch concentration). Increased particle size resulted in a sharper curve, shifting to the left. Ramaswamy *et al.* (1992) attributed this effect to the fact that larger particles will cover a larger portion of the cross-sectional area, and consequently will face a stream of fluid that has a higher relative velocity causing the particle to frequently slide and move faster. Similar results were observed at higher flow rates and increased starch concentrations.

Statistical Analysis of the RTD Data

Analysis of variance (Table 4.2) on the fastest particle residence times (FPRT and FPNRT) of meat and carrot cubes showed that all main effects were significant ($p < 0.001$) except for flow rate with FPNRT of meat cubes ($p > 0.05$). Holding tube length constituted the major source of variability for both FPRT and FPNRT with a contribution of about 85 and 39% (meat), ~63 and 29% (carrot); respectively. Starch concentration and

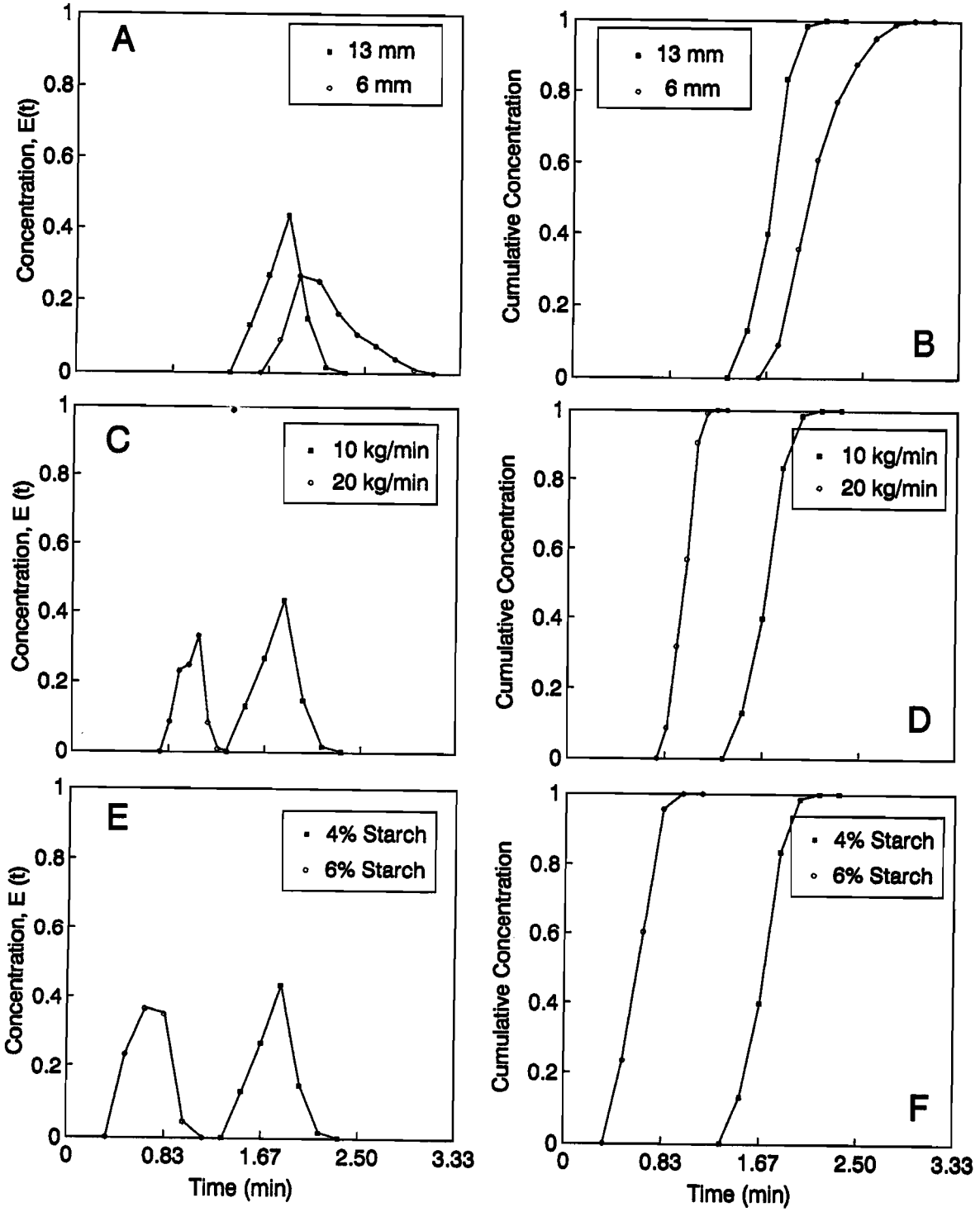

Figure 4.4. **RTD curves (*E* and *F*) of carrot cubes in a holding tube as influenced by mass flow rate, particle size and starch concentration under the base conditions of 18.4 m holding tube length, 10 kg/min flow rate, 13 mm particle size and 4% w/w starch concentration.**

Table 4.2. **Analysis of variance of the residence time associated with the fastest particle (FPRT, FPNRT), mean particle (MPRT) and the standard deviation (SRTD) of RTD curves meat and carrot cubes in a holding tube.**

	FPRT[1]		FPNRT		MPRT		SRTD	
Source	Meat	Carrot	Meat	Carrot	Meat	Carrot	Meat	Carrot
Main Effects	96.3	89.4	71.3	61.1	93.0	90.3	71.6	84.6
Hold Tube (H)	85.1^{a}	63.0^{a}	39.1^{a}	28.5^{a}	77.1^{a}	47.9^{a}	7.7^{a}	7.3^{a}
Starch Conc (C)	0.3^{a}	0.7^{a}	9.4^{a}	12.6^{a}	0.03^{n}	1.0^{b}	11.5^{a}	4.2^{a}
Particle Size (P)	0.9^{a}	0.3^{a}	22.5^{a}	3.3^{b}	0.4^{b}	1.4^{a}	34.8^{a}	12.3^{a}
Flow Rate (F)	10.1^{a}	25.4^{a}	0.4^{n}	16.7^{a}	15.5^{a}	40.0^{a}	17.6^{a}	60.8^{b}
Interactions	3.5	37.2	25.0	37.2	6.7	9.6	17.0	13.9
H x C	0.4^{a}	0.5^{a}	6.5^{a}	2.2^{a}	0.6^{c}	0.6^{b}	4.8^{b}	1.1^{c}
H x P	0.2^{c}	0.4^{b}	6.2^{a}	2.2^{a}	0.6^{b}	0.3^{c}	3.8^{c}	1.7^{b}
C x P	0.0^{n}	0.1^{c}	0.1^{n}	4.7^{a}	0.0^{n}	0.6^{a}	0.2^{n}	2.3^{a}
H x F	2.5^{a}	8.4^{a}	1.4^{n}	6.7^{a}	3.9^{a}	6.7^{a}	1.5^{n}	3.0^{a}
C x F	0.0^{n}	0.01^{n}	0.3^{n}	6.0^{a}	0.0^{n}	0.03^{n}	0.2^{n}	0.2^{n}
P x F	0.0^{n}	0.01^{n}	0.2^{n}	1.0^{c}	0.0^{n}	0.2^{c}	1.3^{n}	2.1^{a}
H x C x P	0.1^{b}	0.2^{a}	3.2^{a}	1.8^{a}	0.6^{a}	0.3^{a}	3.5^{c}	0.8^{b}
H x C x F	0.3^{a}	0.4^{a}	4.9^{a}	5.4^{a}	0.3^{a}	0.4^{a}	0.2^{n}	0.3^{n}
H x P x F	$.0^{n}$	0.2^{a}	0.7^{n}	3.4^{a}	0.6^{a}	0.2^{a}	0.8^{n}	1.3^{a}
C x P x F	0.0^{n}	0.1^{a}	0.2^{n}	3.0^{a}	0.0^{n}	0.01^{n}	0.3^{n}	0.01^{n}
H x C x P x F	0.1^{b}	0.2^{a}	1.3^{c}	0.8^{b}	0.1^{c}	0.4^{n}	0.4^{n}	1.2^{n}
Residual	0.2	0.1	3.7	1.8	0.4	0.11	11.4	1.5
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

[1]Percentage contribution to the total sum of squares (%SS).

$^{a\ b\ c}$Significant $p < 0.001$, $p < 0.01$, $p < 0.05$ respectively.

^{n}Not significant $p > 0.05$

particle size had a significant effect on FPRT & FPNRT, although the magnitude of their contribution to variability was recognizable only with the FPNRT of both meat and carrot cubes. The interactions were generally significant ($p < 0.05$) with the FPRT of meat and carrot cubes contributing ~4 and 11% of the total variability, respectively (Table 4.2). Holding tube length-flow rate interaction was the major contributor to the FPRT of carrot. Interactions were important sources of variability for FPNRT (~25 and 37%) of meat and carrot, respectively. The major interactions for meat cubes were holding tube length-starch concentration and holding tube-particle size; while holding tube-flow rate and starch concentration-flow rate were the majors for carrot cubes.

Mean particle residence time (MPRT) of meat and carrot cubes were significantly influenced ($p < 0.001$) by all test factors, except starch concentration ($p > 0.05$) on meat particles (Table 4.2). Holding tube length and flow rate were the major source of variation for MPRT of meat and carrot cubes. The overall contribution of the interactions to the variability of meat and carrot cubes were ~7 and 10%, respectively, with the holding tube-flow rate being the major interaction (Table 4.2).

Standard deviation of the RTD curves (SRTD) of meat and carrot cubes was also significantly ($p < 0.001$) influenced by all main factors (Table 4.2). Particle size and flow rate were the major factors accounting for about 35 and 18% of the variability of SRTD of meat, respectively. On the other hand, the order of these factors was reversed for SRTD of carrot with ~61 and 12% contribution to the total variability, respectively. From among the interactions only two two-way were significant: holding tube with starch concentration and particle size (~5 and 4%, respectively) and one three-way interaction:

holding tube with starch concentration and particle size (~4%) were significantly contributing to the variability of SRTD of meat (Table 4.2). With SRTD of carrot cubes more interactions were involved with a total of 13.9% of the sum of squares.

Duncan's Multiple Range Tests showed that increasing the holding tube length and particle size increased FPRT and FPNRT of meat cubes (Table 4.3), while increasing starch concentration and flow rate resulted in their decrease. With carrot cubes, increasing the holding tube length and flow rate had the same effects as with meat, but increasing starch concentration and particle size had the opposite effects. A linearly increasing trend was observed with the FPRT of both meat and carrot cubes under all the test factors (starch concentration, particle size, flow rate) as a function of the holding tube length (Figures 4.5). It also was noticed that the FPRT of carrot particles was about 1.5 times lower than meat under most conditions.

The following details are offered to explain the characteristics flow behaviour of meat and carrot cubes: (a) observed association of longer residence times for larger meat particles as compared to smaller: this is contradictory to normal expectations because the larger particles will occupy a larger tube cross-sectional area and, hence will face relatively faster moving fluid bulk, thus acquiring a higher velocity which would result in a lower RT. The meat particles in question were relatively more dense than the carrier fluid. The larger particle can therefore be expected to exert a greater gravitational force in the downward direction perpendicular to the direction of fluid flow. This can translate into a larger wall resistance and hence a longer residence time. Similar results were not observed with carrot cubes because they were lighter than the carrier fluid and the

Table 4.3. Duncan's Multiple range test on the fastest particle (FPRT & FPNRT), mean particle (MPRT) residence times and the associated standard deviation for meat and carrot cubes in a holding tube.

	FPRT (min)		FPNRT		MPRT (min)		SRTD (min)	
Level	Meat	Carrot	Meat	Carrot	Meat	Carrot	Meat	Carrot
Holding Tube Length (m)								
4.6	0.21^{a}	0.30^{a}	0.55^{a}	0.66^{a}	0.50^{a}	0.64^{a}	0.70^{a}	0.92^{a}
9.2	0.40^{b}	0.56^{b}	0.60^{b}	0.71^{b}	0.74^{b}	0.96^{b}	0.69^{a}	1.03^{b}
13.6	0.55^{c}	0.87^{c}	0.62^{c}	0.76^{c}	0.86^{c}	1.26^{c}	0.75^{ab}	1.09^{c}
18.4	0.80^{d}	1.20^{d}	0.67^{d}	0.80^{d}	1.14^{d}	1.61^{d}	0.80^{b}	1.19^{d}
Starch Concentration (% w/w)								
4	0.50^{a}	0.70^{a}	0.63^{a}	0.70^{a}	0.80^{a}	1.07^{a}	0.68^{a}	1.12^{a}
6	0.48^{b}	0.77^{b}	0.59^{b}	0.77^{b}	0.81^{a}	1.17^{b}	0.80^{b}	0.99^{b}
Particle Size (mm)								
6	----	0.75^{a}	----	0.75^{a}	----	1.18^{a}	----	1.19^{a}
13	----	0.71^{b}	----	0.71^{b}	----	1.06^{b}	----	0.93^{b}
10	0.47^{a}	----	0.58^{b}	----	0.82^{a}	----	0.84^{a}	----
20	0.51^{b}	----	0.64^{b}	----	0.79^{b}	---	0.64^{b}	----
Flow Rate (kg/min)								
10	----	0.94^{a}	----	0.69^{a}	----	1.45^{a}	----	1.19^{a}
15	0.56^{a}	----	0.62^{a}	----	0.91^{a}	----	0.81^{a}	----
20	0.41^{b}	0.52^{b}	0.61^{a}	0.77^{b}	0.70^{b}	0.79^{b}	0.67^{b}	0.92^{b}

^{abcd}Means with the same superscript within the levels of the same factor are not significantly different at $p > 0.05$.

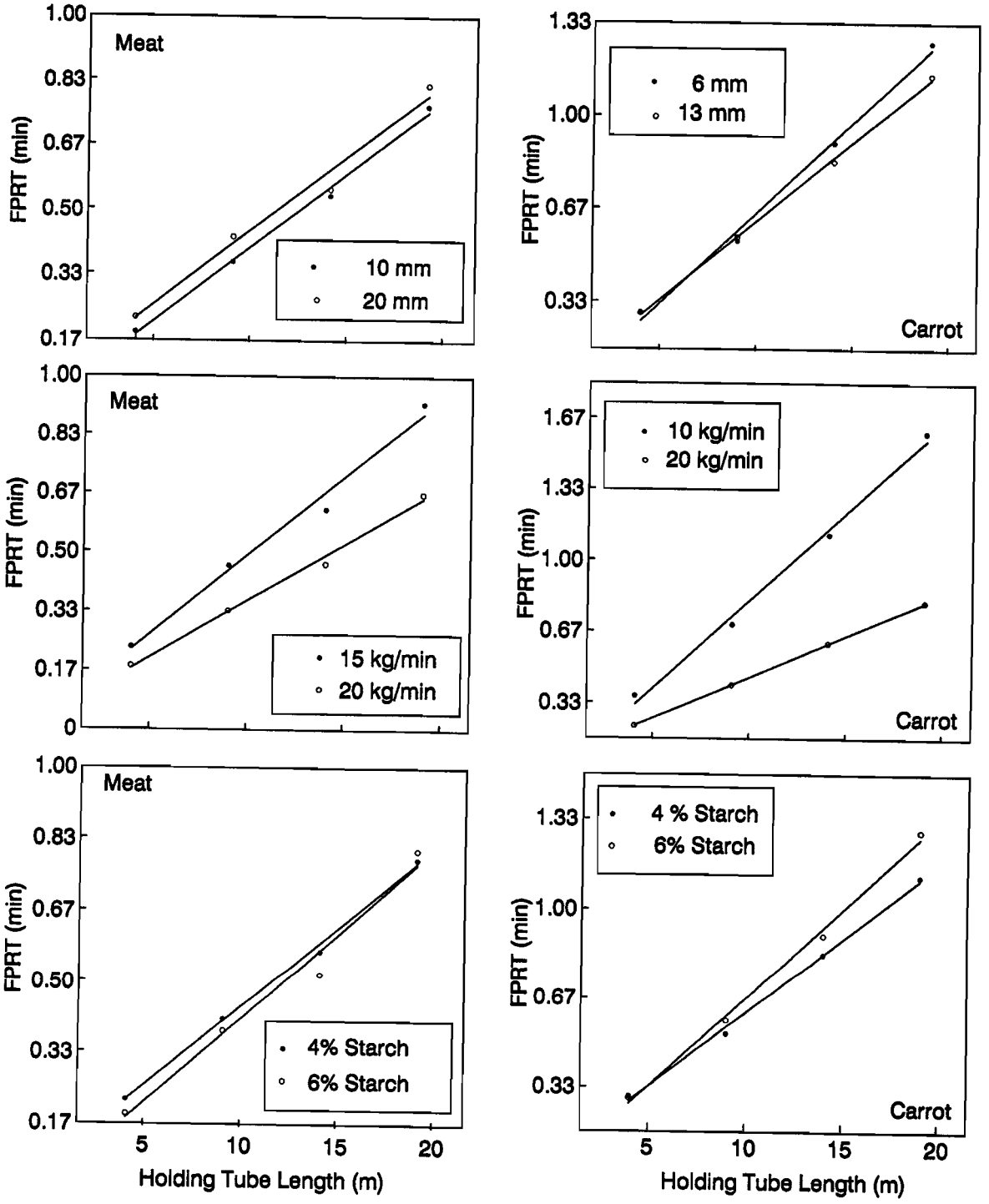

Figure 4.5. **The effect of particle size, flow rate and starch concentration, on the FPRT of meat and carrot cubes as influenced by varying the holding tube length.**

buoyant force acting exactly upwards was probably not as effective as the downward gravitational force; (b) observed longer residence time for carrot cubes as compared to meat particles: this was once again ascribed to the lightness of carrot particles, which was assumed to cause a random as opposed to the streamline movement of meat particles which were heavier. The random movement (requiring a relatively longer path from entrance to exit) would result in the carrot particles to stay longer in the holding tube although the relative velocity could appear at times to be more than for meat particles. This type of behaviour was specially observed at the lower flow rates. As the flow rate increased, the movement of particles would tend to be more orderly and hence the RT's between meat and carrot become more comparable.

MPRT and SRTD of both meat and carrot cubes increased with holding tube length and starch concentration while they decreased with particle size and flow rate (Table 4.3). Again linearly increasing trend was observed with MPRT and SRTD of meat and carrot with the holding tube length.

Modelling of the RTD Experimental Data

The generalized logistic model gave a good fit over the experimental cumulative data, *F*-type curves ($r^2 > 0.95$) of meat and carrot cubes under all experimental conditions as shown in Figures 4.6 & 4.7, respectively for the different conditions listed. The fit was also good when the model-calculated cumulative data was used to generate the time-specific particle concentration data (Figures 4.6 & 4.7, *E*-type curves).

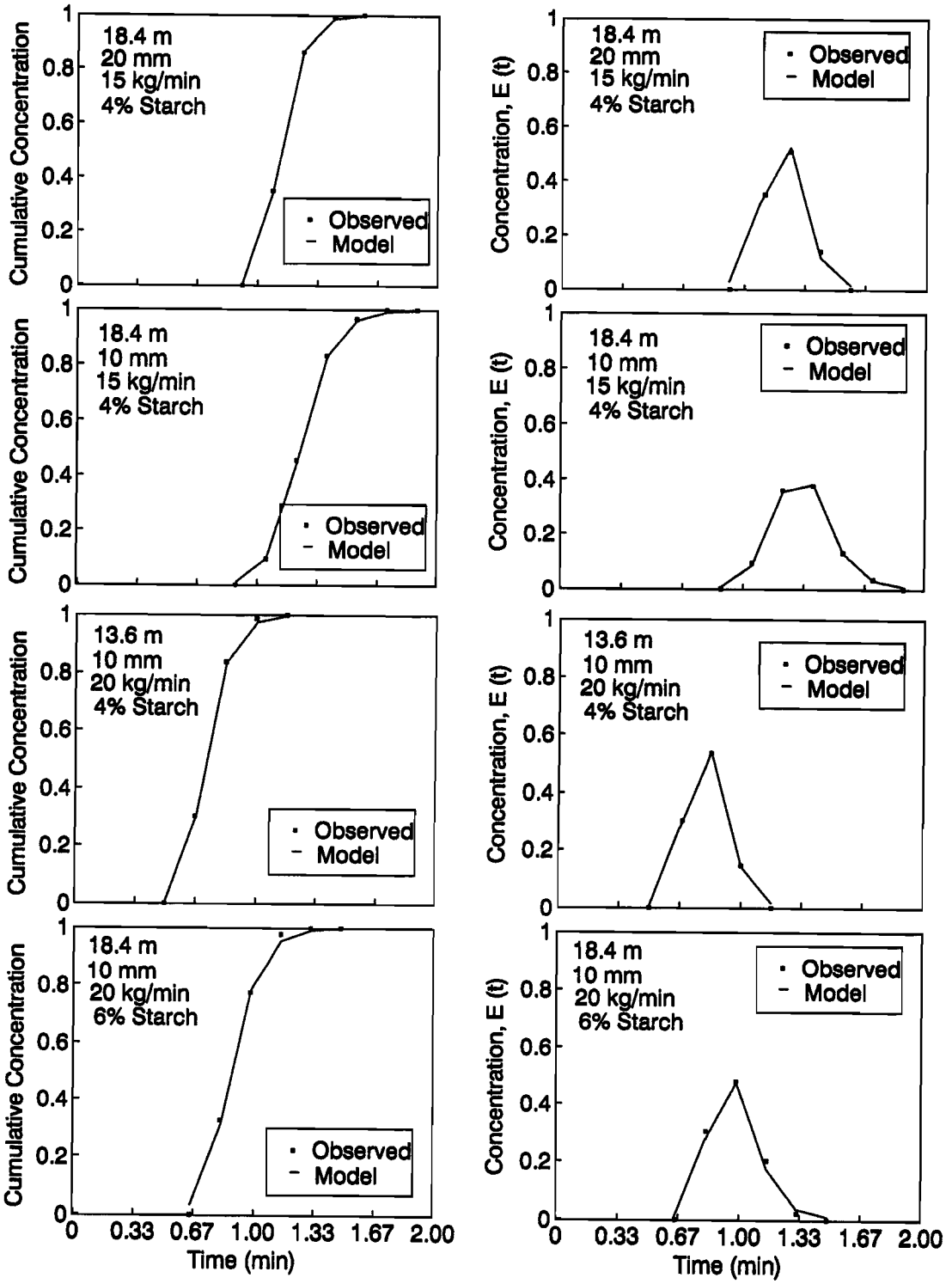

Figure 4.6. **The fit of the generalized logistic model over the experimental RTD data of meat cubes in the holding tube under different conditions.**

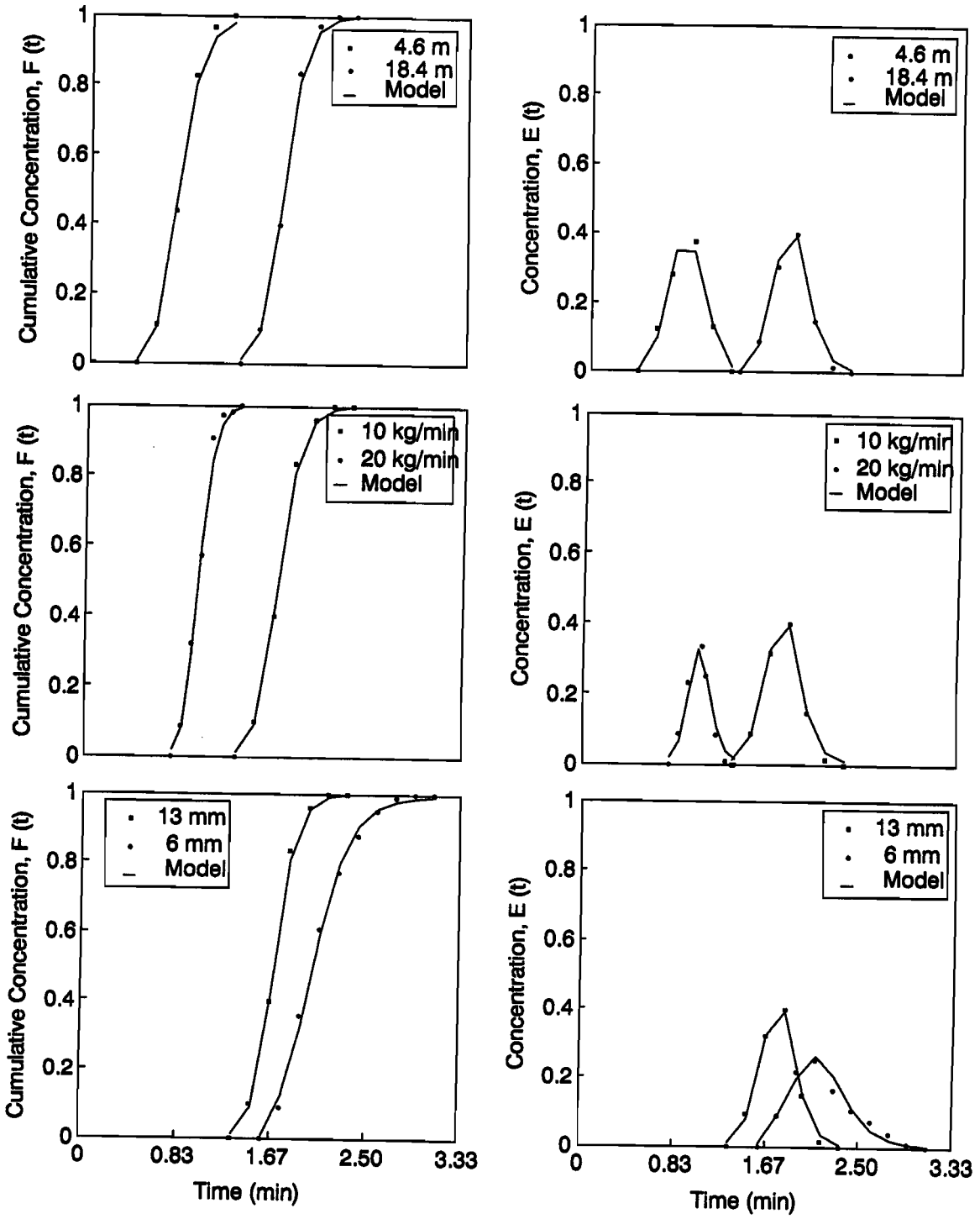

Figure 4.7. **The fit of the generalized logistic model over the experimental RTD data of carrot cubes in the holding tube under different conditions.**

Statistical Analysis of the Logistic Model Parameters

Analysis of variance of the model generated parameters of meat and carrot cubes is presented in Table 4.4. The primary reason for performing analysis of variance was to determine the significant factors to be used for developing predictive equations for logistic model parameters. These empirical relationships will be useful in relating the parameters to the system/carrier fluid/particle characteristics.

Half Concentration Internal Age (HRT and HNT): Both HRT and HNT of meat and carrot were significantly influenced ($p < 0.05$) by all test factors except for the starch concentration (HRT & HNT) and flow rate (HNT) with respect to meat particles. Holding tube was the main factor contributing to the variability of HNT and HRT of both meat (~57-76%) and carrot cubes (~37-49%) as in Table 4.4. The interactions involving holding tube length also significantly affected HRT and HNT of meat cubes, with the holding tube length-flow rate being the major interaction (Table 4.4). More number of interactions were significant ($p < 0.05$) with carrot cubes, particularly with respect to HNT (~37%). The major two-way interaction for HNT was particle size-starch concentration (~8%). also there were two major three-way interactions:holding tube length-starch concentration-particle size and holding tube length-particle size-flow rate.

Duncan's Multiple Range Tests showed that HRT of both meat and carrot cubes increased with holding tube length and starch concentration, and decreased with particle size and flow rate (Table 4.5). HNT, however, decreased with increased holding tube length and particle size, and increased with starch concentration and flow rate. Differences

Table 4.4. **Analysis of variance of the residence time parameters associated with the logistic model (HRT, HNT and B) for meat and carrot cubes flowing in a holding tube.**

Source	HRT (%SS)[1]		HNT (%SS)		B (%SS)	
	Meat	Carrot	Meat	Carrot	Meat	Carrot
Main Effects	92.6	90.1	61.6	61.5	46.28	78.3
Hold Tube (H)	76.6^{a}	49.3^{a}	57.2^{a}	37.1^{a}	17.4^{b}	74.3^{a}
Starch Conc (C)	0.02^{n}	0.54^{a}	0.54^{n}	5.49^{a}	1.40^{n}	0.02^{n}
Particle Size (P)	0.31^{c}	1.16^{a}	3.51^{b}	9.00^{a}	26.2^{a}	2.87^{a}
Flow Rate (F)	15.7^{a}	39.1^{a}	0.32^{n}	9.91^{a}	1.27^{n}	1.09^{b}
Interactions	7.00	9.80	35.0	37.2	25.9	18.5
H x C	0.44^{a}	0.62^{a}	5.77^{a}	2.93^{a}	3.00^{n}	0.74^{n}
H x P	0.65^{a}	0.26^{a}	3.50^{a}	2.15^{a}	1.88^{n}	1.49^{b}
C x P	0.00^{n}	0.70^{a}	0.42^{n}	7.63^{a}	0.08^{n}	0.14^{n}
H x F	4.01^{a}	6.78^{a}	8.13^{a}	5.22^{c}	2.95^{n}	0.24^{n}
C x F	0.07^{n}	0.01^{n}	0.00^{n}	1.43^{a}	0.06^{n}	1.71^{a}
P x F	0.02^{n}	0.17^{a}	0.75^{n}	0.43^{a}	1.62^{n}	0.38^{n}
H x C x P	0.70^{a}	0.25^{a}	6.04^{a}	7.52^{a}	4.48^{n}	3.26^{a}
H x C x F	0.34^{a}	0.46^{a}	5.24^{a}	3.39^{a}	4.56^{n}	0.14^{n}
H x P x F	0.58^{a}	0.20^{a}	4.53^{a}	6.12^{a}	1.79^{n}	8.06^{n}
C x P x F	0.01^{n}	0.00^{n}	0.03^{n}	0.04^{n}	0.05^{n}	0.01^{n}
H x C x P x F	0.18^{b}	0.34^{a}	0.56^{n}	0.31^{n}	5.39^{n}	2.30^{a}
Residual	0.37	0.12	3.56	1.38	27.9	3.22
Total	100.0	100.0	100.0	100.0	100.0	100.0

[1]%SS: Percentage contribution to the total sum of squares.
[a] [b] [c]Significant $p < 0.001$, $p < 0.01$, $p < 0.05$ respectively.
[n]Not significant $p > 0.05$

Table 4.5. Duncan's Multiple range test on the parameters associated with the generalized logistic model: HRT, HNT (half concentration internal age) and B (particle accumulation rate) for meat and carrot cubes in a holding tube.

Source	HRT (min)		HNT		B	
	Meat	Carrot	Meat	Carrot	Meat	Carrot
Holding Tube Length (m)						
4.6	0.40^{a}	0.56^{a}	1.09^{a}	1.24^{a}	19.7^{a}	15.9^{a}
9.2	0.65^{b}	0.89^{b}	1.01^{b}	1.12^{b}	24.5^{ab}	23.0^{b}
13.8	0.78^{c}	1.18^{c}	0.85^{c}	1.03^{c}	31.4^{bc}	29.5^{c}
18.4	1.05^{d}	1.51^{d}	0.89^{d}	1.02^{d}	33.3^{c}	36.1^{d}
Starch Concentration (% w/w)						
4	0.72^{a}	1.00^{a}	0.95^{a}	1.07^{a}	28.7^{a}	26.3^{a}
6	0.72^{a}	1.08^{b}	0.97^{a}	1.14^{b}	25.7^{a}	26.0^{b}
Particle Size (mm)						
6	----	1.10^{a}	----	1.15^{a}	----	24.7^{a}
13	----	0.99^{b}	----	1.06^{b}	----	27.6^{b}
10	0.73^{a}	----	0.98^{a}	----	20.5^{a}	----
20	0.70^{b}	----	0.94^{b}	----	33.9^{b}	----
Flow Rate (kg/min)						
10	----	1.36^{a}	----	1.07^{a}	----	25.2^{a}
15	0.83^{a}	----	0.95^{a}	----	25.7^{a}	----
20	0.61^{b}	0.72^{b}	0.97^{a}	1.15^{b}	28.7^{b}	27.0^{b}

^{abcd}Means with the same superscript within the levels of the same factor are not significantly different at $p > 0.05$.

were observed between HRT (logistic model) and MPRT (normal distribution), although they both indicated similar trends with respect to the influence of test factors. MPRT values were higher indicating that the RTD curves were somewhat skewed to the right. These differences were, however, relatively smaller as compared with the difference observed while using vertical SSHE. This implies that RTD deviations from normality were more pronounced in the SSHE compared to the holding tube (pipe) flow due to the existence of radial and axial mixing induced by the rotor and blades.

Particle accumulation rate (B): Particle accumulation rates were significantly influenced holding tube length ($p < 0.001$) and particle size ($p < 0.01$), while starch concentration, flow rate and interactions were insignificant ($p > 0.05$) as in Table 4.4. Particle accumulation rates of carrot cubes, however, were significantly affected ($p < 0.01$) all main factors, except starch concentration ($p > 0.05$). Holding tube the major factor accounting for 74% of the variability (Table 4.4). The three-way interaction of holding tube-particle size-flow rate was the main contributor to the variability of carrot cubes (~8%) among all other interactions.

Duncan's multiple range tests showed that B increased with holding tube length, particle size and flow rate for both meat and carrot particles (Table 4.5), while it decreased with carrier fluid concentration, though insignificant ($p > 0.05$). Almost the same levels of B were observed for both meat and carrot cubes under similar conditions.

Multiple Regression Equations for Model Parameters

Coefficients for the multiple regression analysis for the model parameters and their associated coefficients of determination for meat and carrot cubes are shown in Table 4.6 with a plot of observed *vs* predicted values in Figure 4.8. Only the two-way interactions were included in the multiple regression analyses for simplicity. The model fitting is reasonably good, however, extrapolation of the model to ranges beyond those investigated should be avoided.

CONCLUSIONS

All test factors influenced the RTD parameters of meat and carrot cubes in the holding tube of a commercial pilot scale aseptic processing system. The RTD parameters (FPRT, FPNRT, MPRT and SRTD) of meat and carrot cubes increased with the holding tube length and starch concentration; and decreased with particle size and flow rate. FPRT and FPNRT of large meat cubes (20 mm) showed an opposite trend. Meat cubes travelled at about 1.6 times faster than the average carrier fluid, while carrot cubes travelled at approximately 1.4 times faster than fluid. On average, larger particles travelled faster than the smaller ones, a trend that is opposite to SSHE flow.

The logistic model well described the cumulative RTD data of meat and carrot cubes. Model parameters were responsive to test factors. Good fit was also obtained when the model-calculated cumulative RTD data was used to generate time-specific particle concentration. Predictive equations were developed to relate the model parameters to the system/carrier fluid/particle factors.

Table 4. 6. Multiple regression coefficients associated with the logistic model parameters for carrot and meat cubes in a holding tube.

	Regression Coefficients					
	HRT		HNT		B	
Variables	Meat	Carrot	Carrot	Meat	Meat	Carrot
Constant	-0.158	0.012	0.184	1.000	-35.66	18.40
Holding tube (H)	0.115	0.126	0.326	-0.025	0.623	1.636
Starch conc. (C)	0.056	0.093	0.074	0.049	7.958	-3.626
Particle size (P)	0.002	0.044	0.015	0.025	0.912	0.397
Flow rate (F)	0.021	-0.010	0.043	0.000	2.375	-0.787
H x C	0.000	0.007	-0.003	0.004	0.127	-0.070
H x P	0.000	-0.001	0.000	0.001	0.012	0.001
H x F	-0.004	-0.005	-0.002	-0.001	-0.007	0.011
C x P	0.000	-0.012	-0.002	-0.011	-0.146	0.094
C x F	-0.003	-0.001	-0.000	0.003	-0.564	0.227
P x F	0.001	0.000	0.001	-0.001	0.045	-0.031
R^2	0.940	0.981	0.512	0.703	0.617	0.772
Standard Error	0.066	0.072	0.088	0.078	7.086	4.182

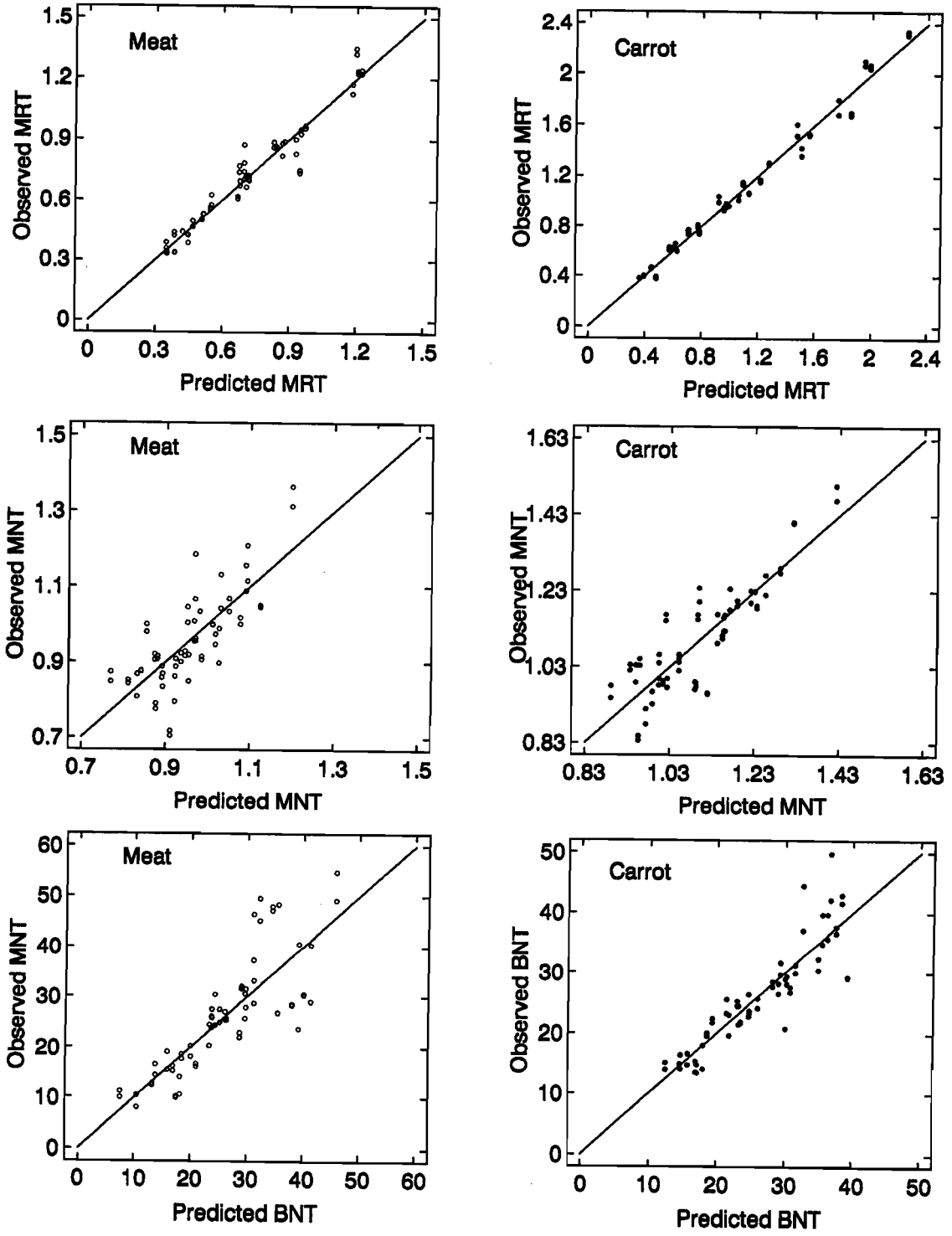

Figure 4.8. **Model-calculated *vs* observed values of half concentration internal age (HRT, HNT) and particle accumulation rate (B) of meat and carrot cubes in the holding tube of a pilot scale aseptic processing system.**

CHAPTER V

RESIDENCE TIME DISTRIBUTION (RTD) CHARACTERISTICS OF CARROT CUBES IN STARCH SOLUTIONS IN A PILOT ASEPTIC PROCESSING SYSTEM

ABSTRACT

Residence time distributions (RTD) of carrot cubes flowing in starch solutions were evaluated in a commercial pilot-scale aseptic processing system consisting of a scraped surface heat exchanger (SSHE) for heating, a set of holding tubes and another SSHE for cooling. A full factorial design of experiments was used employing flow rate (15 and 20 kg/min), temperature (80 and 100°C), holding tube length (1.50, 17.45 and 26.65 m), particle size (6 and 13 mm) and starch concentration (3 and 5% w/w) as factors. All factors significantly ($p < 0.05$) influenced the fastest particle residence time (FPRT) and its normalized version (FPNRT), mean particle residence time (MPRT), standard deviation of residence time (SRTD). While particle size, holding tube length and starch concentration increased the FPRT, fluid flow rate and temperature had the opposite effect. FPRT was lower than the average fluid retention time under all testing conditions. A logistic model was used to describe the RTD curves of carrot cubes which showed an excellent agreement with the experimental data ($r^2 > 0.95$). All test factors significantly ($p < 0.05$) influenced the logistic model parameters which could be predicted as a function of test factors.

INTRODUCTION

RTD behaviour of meat and carrot cubes were independently evaluated in the scraped surface heat exchanger (Chapter III) and the holding tube (Chapter IV). In a typical aseptic processing system, however, there will be, held in series, one SSHE for heating, a predetermined length of holding tube and one or two SSHEs for cooling prior to aseptic packaging. There has been no published information on the systemic influence on the RTD of particles. While RTD in holding tubes have been implicated in establishing processing criteria, it is often difficult to completely separate the RTD behaviour in these and other sections. In this chapter, an attempt has been made to evaluate the RTD of particles in the entire system. Keeping the SSHE combinations constant and varying the holding tube lengths, it has been attempted to elucidate the residence times of particles in the holding tubes by difference. A bio-validation test described in the next chapter (Chapter VI) is an attempt to provide justification for the systemic approach.

The objective of this study was to evaluate the RTD of food particles (carrot cubes) in a pilot scale aseptic processing system as influenced by selected factors related to particle (size), carrier fluid (flow rate, concentration) and system (temperature, holding tube length), develop a suitable model to describe the experimental RTD data of carrot cubes, and to relate the experimental factors (particle size,carrier fluid concentration and flow rate, operating temperature and holding tube length) to the model parameters.

MATERIALS AND METHODS

Materials

Carrots cubes (6 and 13 mm) and starch solutions (3 and 5% w/w) were prepared as described and detailed in Chapter III.

Aseptic Processing System

The system used consisted of two scraped surface heat exchangers (Contherm Division, Alfa Laval Inc., Newburyport, MA), one for heating and one for cooling, connected by a series of holding tubes, the length of which could be varied by selecting the number of turns (4.6 m/turn). The two heat exchangers were inter-connected directly with a short tube (1.5 m) in the no-holding tube situation, while half (2.5 turns, 17.45 m) and full lengths (4.5 turns, 26.65 m) were used as additional variables. A variable tri-lobe speed positive displacement pump (Albin Pump, Atlanta, GA) was used for feeding the system with the carrier fluid and food particles as regulated by a three-way valve. The layout of the experimental set-up is shown in Figure 5.1, while the dimensions of the system are detailed in Table 5.1.

At the beginning of a test run, the system was equilibrated with the carrier fluid without particles. Temperatures were measured by thermocouples installed at the entrance and exit of the heat exchangers and at different turns of the holding tube. Temperatures were monitored and regulated through the system control panel which ensured a constant exit temperature from the heating section SSHE in each experimental run. In any typical run, the maximum temperature drop across the holding tube was 2°C.

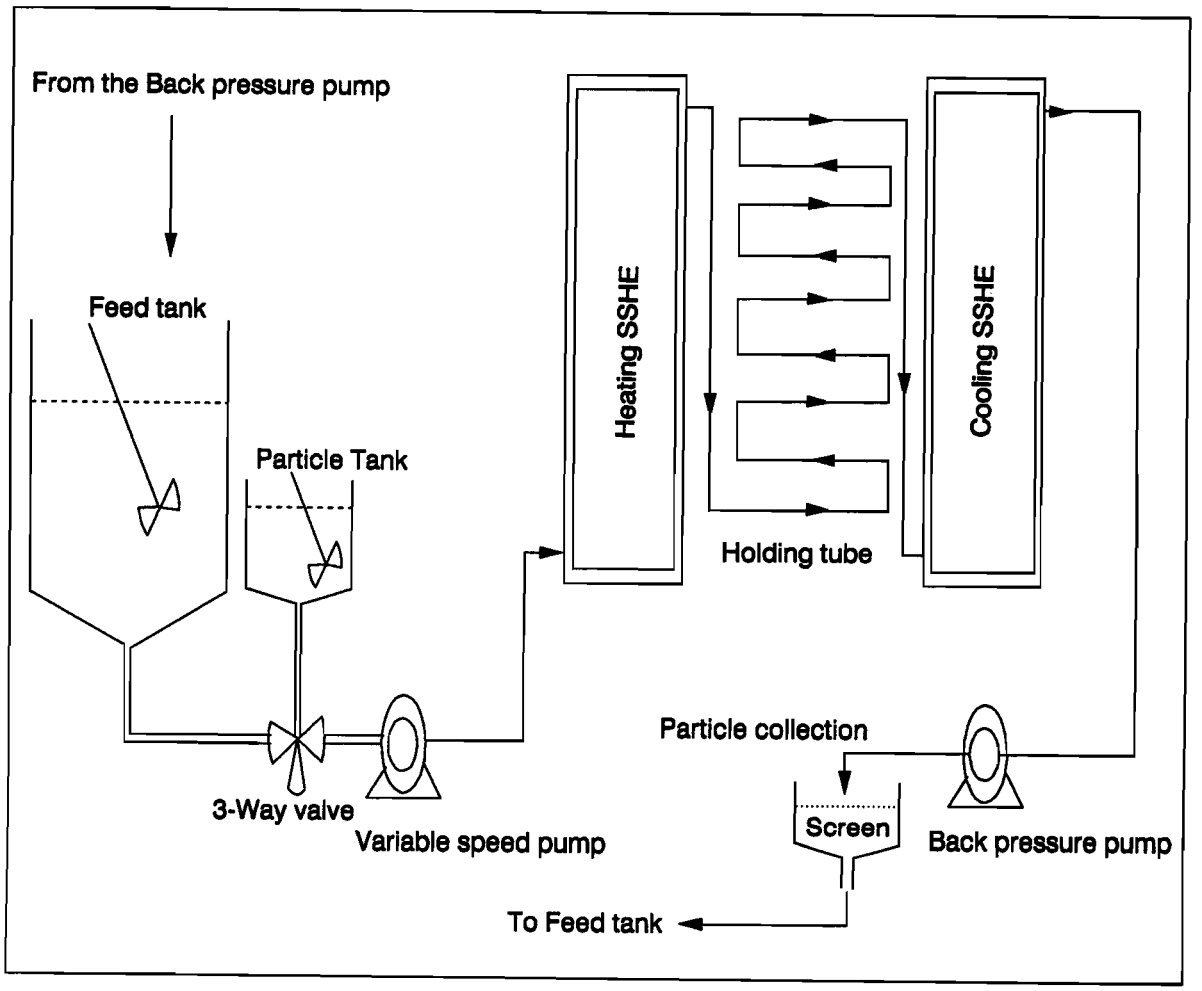

Figure 5.1. Schematic of the experimental set-up used for the RTD studies.

Table 5.1. Specifications of the aseptic processing system used to study the residence time distribution of carrot particles.

Parameter			Specification
Scraped surface heat exchanger			
Scraped surface heat exchanger			1.8 X 0.9 m
Internal diameter			0.152 m
Internal shaft diameter			0.076 m
Material of construction			Stainless steel
Dasher speed			300 rev/min
Heating medium			Steam at 150°C
Cooling medium			Water at 10°C
Holding Tube			
Total length	17.45	26.65	m
Internal diameter	0.0381		m
Carrier fluid	Therm-flo starch		
Concentration	3	5	% w/w
Density	1010	1014	kg/m^3
Flow behaviour index (80°C)		0.575	0.536
Flow behaviour index (100°C)	0.875	0.646	
Consistency coefficient (80°C)	0.353	0.657	Pas^n
Consistency coefficient(100°C)	0.052	0.554	Pas^n
Flow rate	15, 20	15, 20	kg/min
Particles	Carrots cubes		
Size	6	13	mm
Concentration	5		% w/w
Density	1040		kg/m^3

The fill mass of the system was determined by calculating the system volume and multiplying it by the measured density of the carrier fluid. The system volume included the void volume of heat exchangers (manufacturer's data) and volume of the tubing from the feed pump to the collection tank assuming no dead volume in the system. The volume in the tubing was calculated by measuring the total length of the tubing and multiplying it by the cross sectional area.

Operation

The procedure used for RTD evaluation was described in detail in Chapter III. In this study, 500 g of carrot cubes (6 mm or 13 mm) were thoroughly mixed with 9.5 kg of carrier fluid (3% or 5% w/w) and placed in the liquid/particle tank (5% w/w particle concentration). The time lapsed from when the particle/liquid mixture was introduced into the system to when the first particle emerged out of the system was measured and taken to represent residence time of the fastest moving particle. The fluid and particles emerging out were subsequently collected at 1 min intervals for up to 25 min and weighed separately. From this data, the concentration of particles in each-fraction was determined.

Experimental Design

A full factorial design of experiments was employed involving two levels of flow rate (15 and 20 kg/min), temperature (80 and 100°C), starch concentration (3 and 5% w/w), particle size (6 and 13 mm), and three levels of holding tube length (1.50, 17.45 and 26.65 m). All the experiments were performed in triplicate. Statistical analyses were

performed using the General Linear Model Procedure (SAS, SAS Institute Inc., Cary, NC; 1985) to test the effects of different factors on (1) the fastest particle real and normalized residence times (FPRT & FPNRT), (2) mean particle residence time (MPRT) and (3) standard deviation of residence time variance (SRTD). FPNRT was calculated by dividing FPRT by the average residence time (t_m) of the fluid (calculated as mentioned in Chapter III).

Estimation of the Model Parameters

Experimental data was fitted to the different distribution models using the Maximum Likelihood Program (MLP, Lawes Agricultural Trust, Rothamsted Experimental Station) as previously described in Chapter III.

RESULTS AND DISCUSSION

Carrot Cubes RTD

Influence of Particle Size: Typical normalized RTD curves (at two holding tube lengths, 1.50 and 26.65 m) for carrot cubes of two sizes are shown in Figure 5.2, with the top two curves representing the *F*-type distribution and the bottom two representing *E*-type distribution. The fastest moving particle of the smaller size generally had a shorter residence time than the mean residence time of the carrier fluid (average FPNRT = 0.81, Table 5.2), while particles of the larger size (13 mm) moved at about the average fluid flow rate (average FPNRT ~1, Figure 5.2 and Table 5.2). This also implied that the smaller

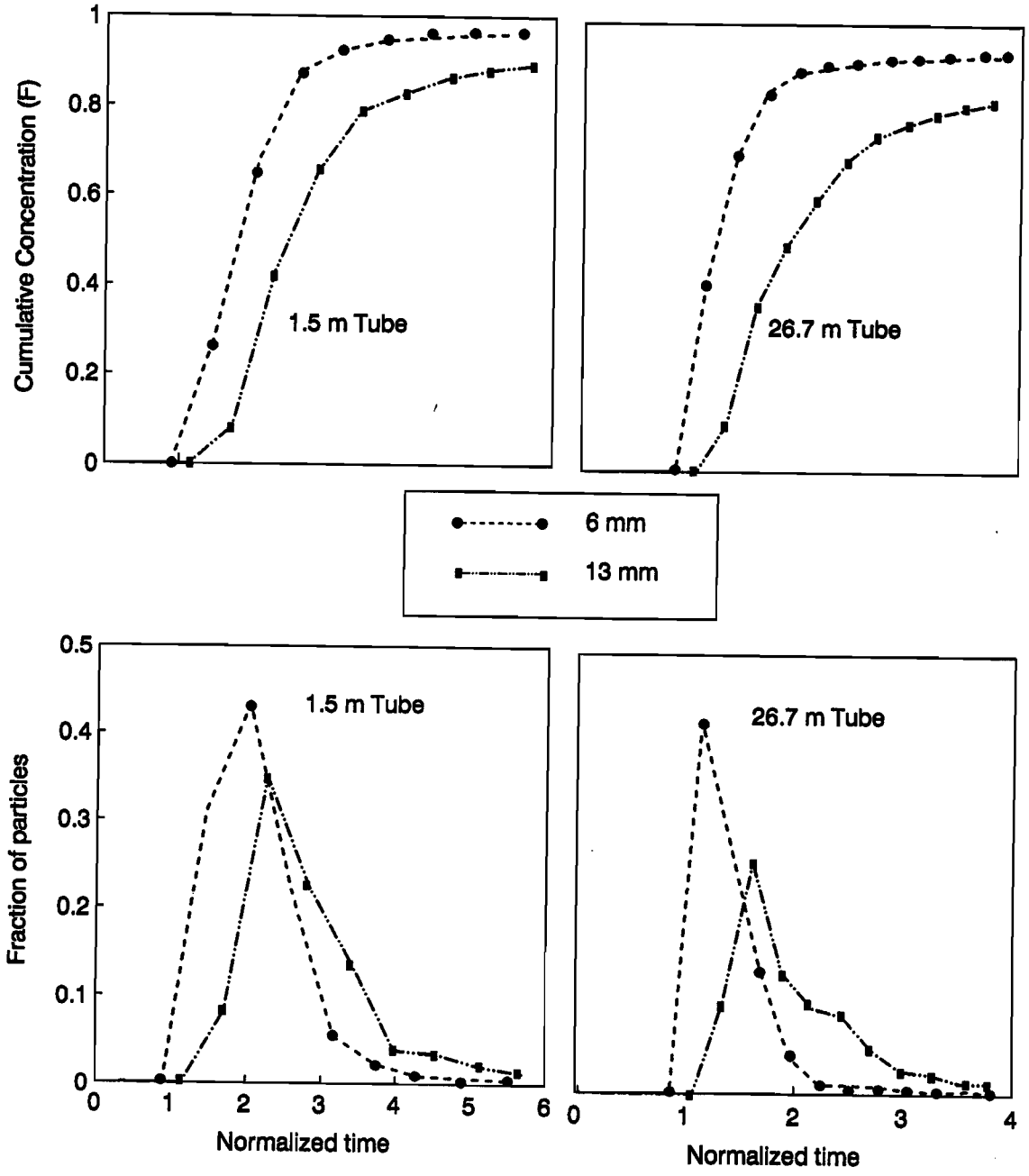

Figure 5.2. **RTD of carrot cubes as influenced by particle size and holding tube length (fluid flow rate 15 kg/min, 100°C and 5% w/w starch concentration).**

Table 5.2. Duncan's Multiple range test on the fastest particle residence time (FPRT, FPNRT), mean residence time (MPRT) and standard deviation of residence time (SRTD) for carrot cubes in a pilot scale aseptic processing system.

Levels	FPRT (min)	FPNRT	MPRT (min)	SRTD (min)
Particle Size (mm)				
6	1.99[a]	0.81[a]	4.24[a]	3.18[a]
13	2.44[b]	1.02[b]	4.73[a]	3.44[b]
Starch Concentration (% w/w)				
3	2.13[a]	0.88[a]	4.89[a]	3.78[a]
5	2.30[b]	0.95[b]	4.01[a]	2.77[b]
Flow Rate (kg/min)				
15	2.56[a]	0.93[a]	4.84[a]	3.36[a]
20	1.87[b]	0.90[a]	4.13[b]	3.27[b]
Temperature (°C)				
80	2.30[a]	0.95[a]	4.67[a]	3.58[a]
100	2.13[b]	0.88[a]	4.31[a]	3.03[a]
Holding Tube Length (m)				
1.50	1.50[a]	0.98[a]	3.96[a]	2.77[a]
17.45	2.34[b]	0.90[b]	4.61[b]	3.36[a]
26.65	2.80[c]	0.87[b]	4.90[b]	3.69[b]

[abc]Means with the same superscript within the levels of the same factor are not significantly different ($p > 0.05$).

particles had a maximum velocity about ~1.2 times the average fluid velocity. The FPRT of smaller cubes were lower than that of larger cubes. MPRT showed similar trends to FPRT. Smaller particles (6 mm) were also associated with a higher cumulative particle concentration as compared to particles of the larger size (Figure 5.2). A general conclusion from the above results is that particle residence time increases with particle size. As pointed out in Chapters III and IV results from some earlier studies (McCoy *et al.*, 1987; Ramaswamy *et al.*, 1992) showed an opposite trend. In order to support this observation, it may be argued that smaller particles were likely lifted up and travelled along the rapidly moving central stream while the larger particles dragged behind moving along the wall. The "frog-leap" (smaller particles) and "necklace" (larger particles) type of particle-particle interactions reported by Sastry and Zuritz (1987) and Dutta and Sastry (1990a,b) might also explain such flow behaviour. Another likely explanation based on Chapter III and IV is that RTD of larger particles in the SSHE were longer compared to the smaller particles. Thus, the heating SSHE determines when and which particle size entered the holding tube.

Carrier Fluid Concentration: Normalized F and E-type RTD curves for carrot cubes of two sizes as influenced by starch concentration at a given temperature, flow rate and holding tube length are shown in Figure 5.3. Considerable differences were observed for the flow behaviour of larger particles in the two fluids (3% and 5% starch solutions). It can be seen that the 13 mm particles flowing in 3% starch solution considerably lagged the fluid mean residence time, whereas the 6 mm particles showed only a small lag

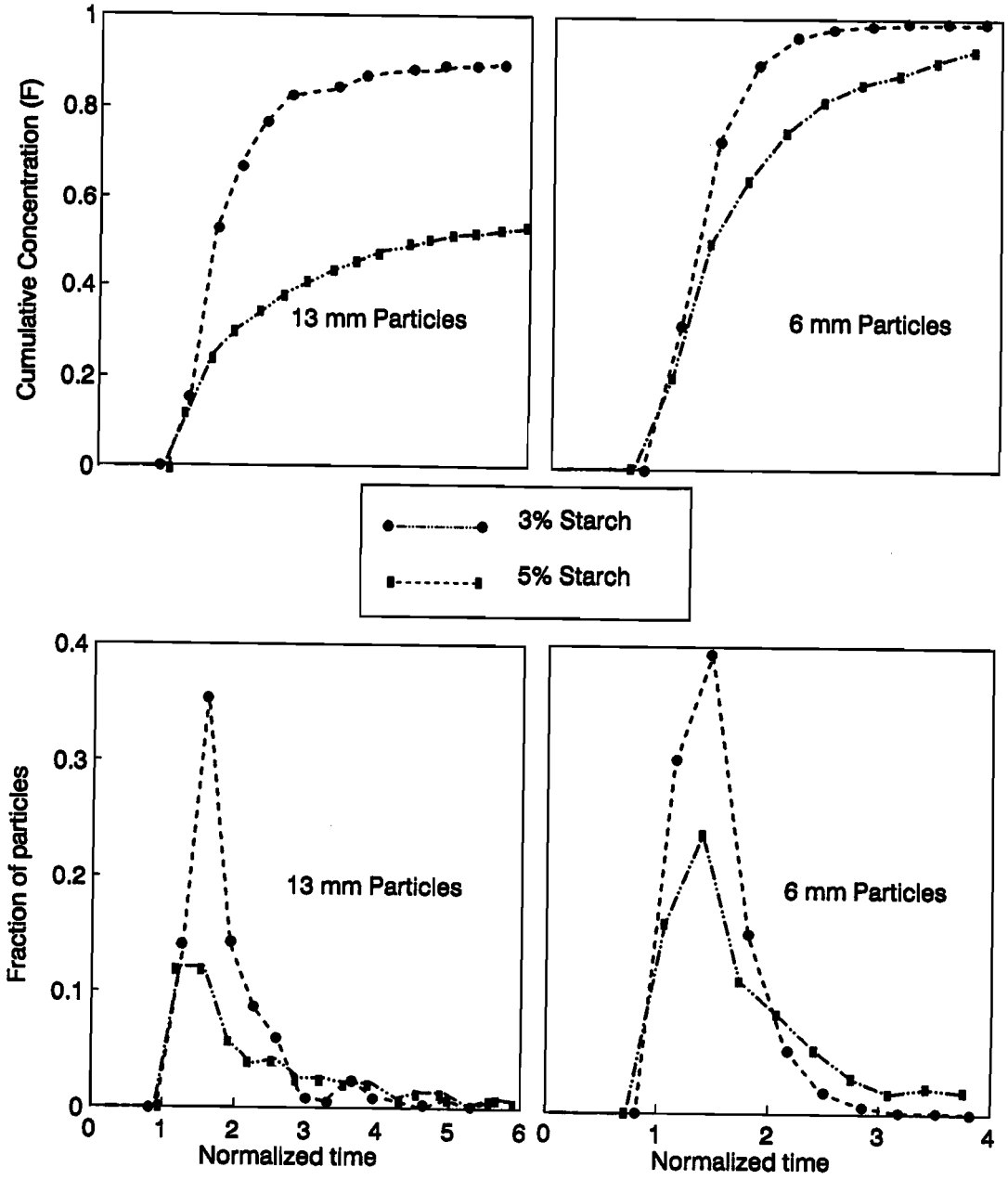

Figure 5.3. **RTD of carrot cubes as influenced by particle size and starch concentration (fluid flow rate 15 kg/min, 100°C and 17.45 m holding tube length).**

(Figure 5.3, *F* curves). Increasing the carrier fluid concentration to 5% w/w dramatically decreased the relative residence time of 13 mm particles to make them more comparable to that of smaller particles (Figure 5.3). The RT of small particles, on the other hand, was not affected only slightly by starch concentration.

Further, under all conditions tested, particles of smaller size were almost completely recovered after a normalized time of about 3.0 (in both 3% & 5% starch), whereas the recovery of larger particles was only partial and as low as ~60% in 3% starch at 15 kg/min flow rate even after a normalized time of 7.0. The cumulative particle concentration at the exit at any time was higher with the 5% starch solution than with 3% starch for particles of both sizes (Figure 5.3, *E* curves). On the average, the higher concentration of starch in the carrier fluid resulted in a small but significant ($p < 0.05$) increase in FPRT and decrease in MPRT while it dramatically decreased the SRTD. While the residence time distribution appeared to narrow down with an increase in starch concentration, it should be coupled with a higher flow rate in order that the associated fluid-to-particle heat transfer coefficient is not overly reduced as a result of viscosity increase.

Flow Rate: Normalized *F* and *E*-type RTD curves for carrot cubes of two sizes as influenced by fluid flow rate at a given temperature, starch concentration (3% & 5%) and holding tube length are shown in Figures 5.4 and 5.5. Again, considerable differences were observed for the flow behaviour of particles at the flow rates (15 and 20 kg/min) as shown with 3% starch in Figure 5.4. The higher flow rate decreased the residence time

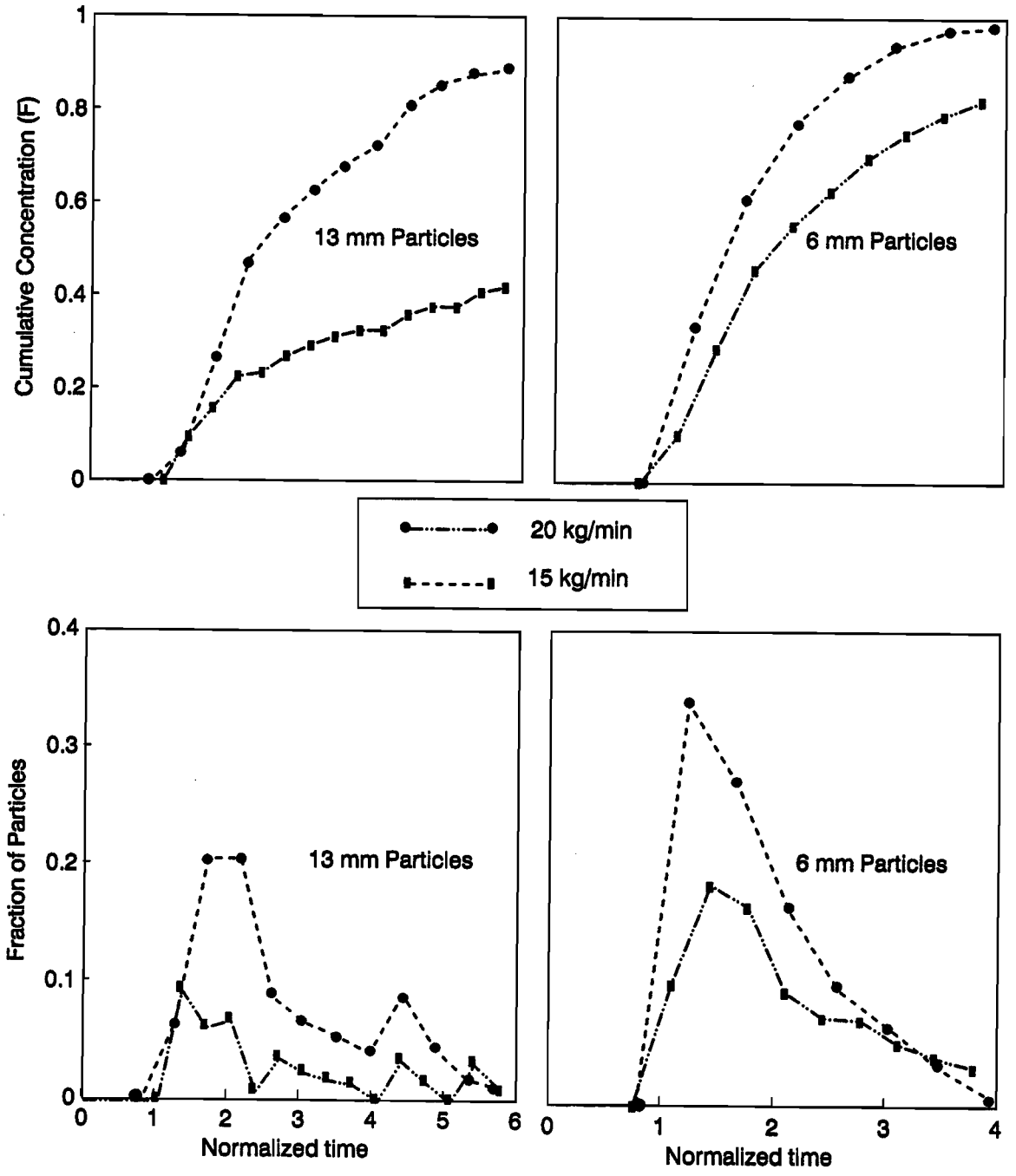

Figure 5.4. **RTD of carrot cubes in 3% starch solution as influenced by particle size and fluid flow rate (100°C and 17.45 m holding tube length).**

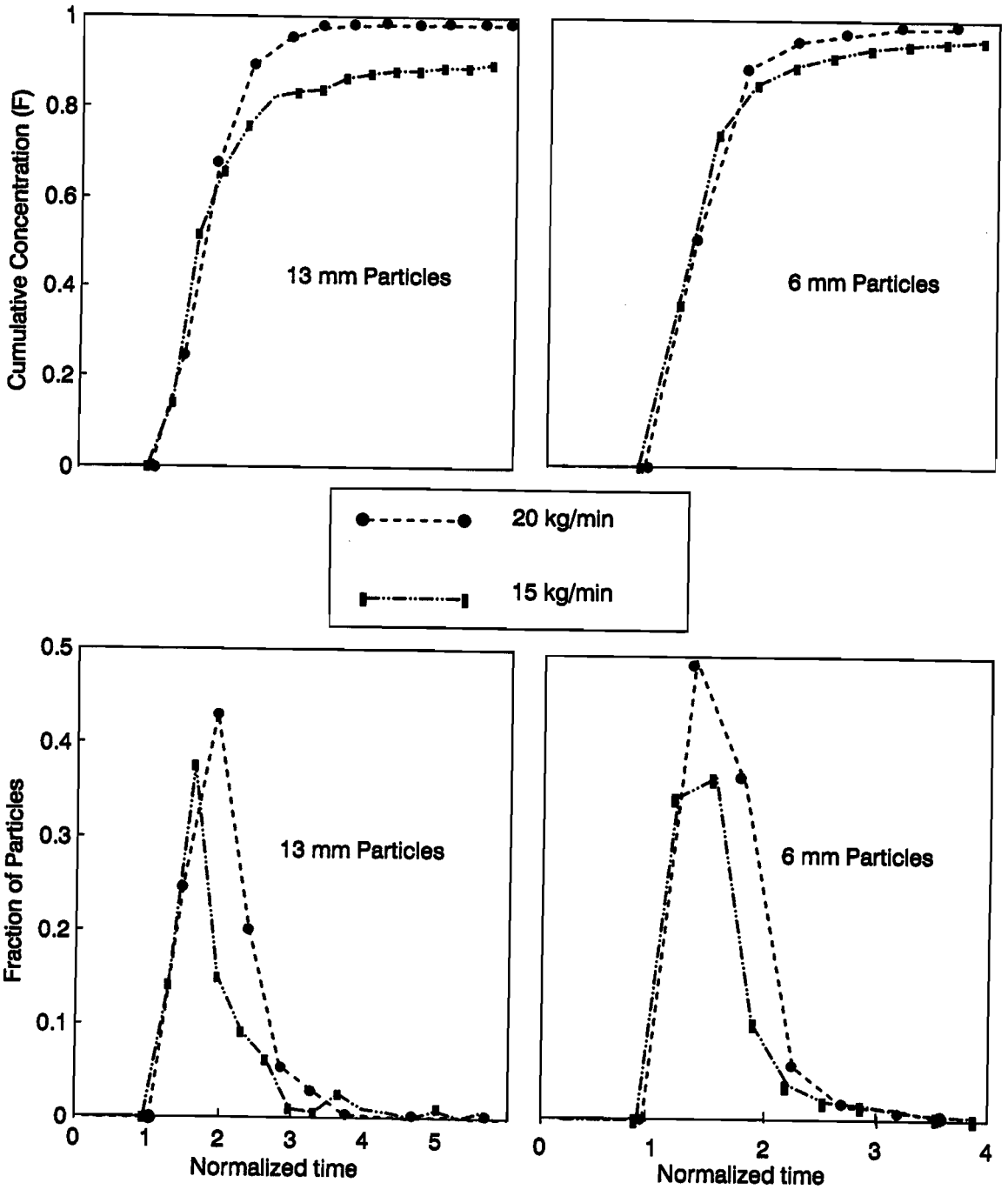

Figure 5.5. **RTD of carrot cubes in 5% starch solution as influenced by particle size and fluid flow rate (100°C and 17.45 m holding tube length).**

of particles with the larger ones showing a greater sensitivity than the smaller ones (Figure 5.4, *F* curve). At the higher starch concentration, RTD differences between the two flow rates virtually disappeared (Figure 5.5). The interaction between flow rate and starch concentration probably masked the effect of fluid flow rate on the particle RTD (Table 5.2). The general trend, however, was evident: higher the fluid flow rate, faster the particle flow.

Temperature: Normalized *F* and *E*-type RTD curves for carrot cubes of two sizes as influenced by fluid temperature at a given flow rate, starch concentration and holding tube length are shown in Figure 5.6. The higher temperature (100°C) resulted in a significantly shortened RT for the fastest particle (Figure 5.6). The higher temperature also shortened the MPRT and decreased the SRTD (Table 5.2). This may be attributed to increased turbulence at higher temperatures as a result of reduced fluid viscosity. In this study, it was found necessary to maintain fluid flow rates above 15 kg/min in order to maintain particles moving through the system. At such flow rates, an operating temperature higher than 100°C was not possible due to the lack of sufficient heat transfer surface on the SSHE. Even to reach this temperature, it was found necessary to maintain the inlet temperature of the fluid at ~60°C. Although temperatures used in this study are still below the commercial operating levels, it is a step in the right direction. The majority of particulate RTD studies reported in literature have been performed at room temperature, the main reason for this being the apparent difficulty in carrying out such studies in pressurized processing systems. As a compromise, temperature influence has generally

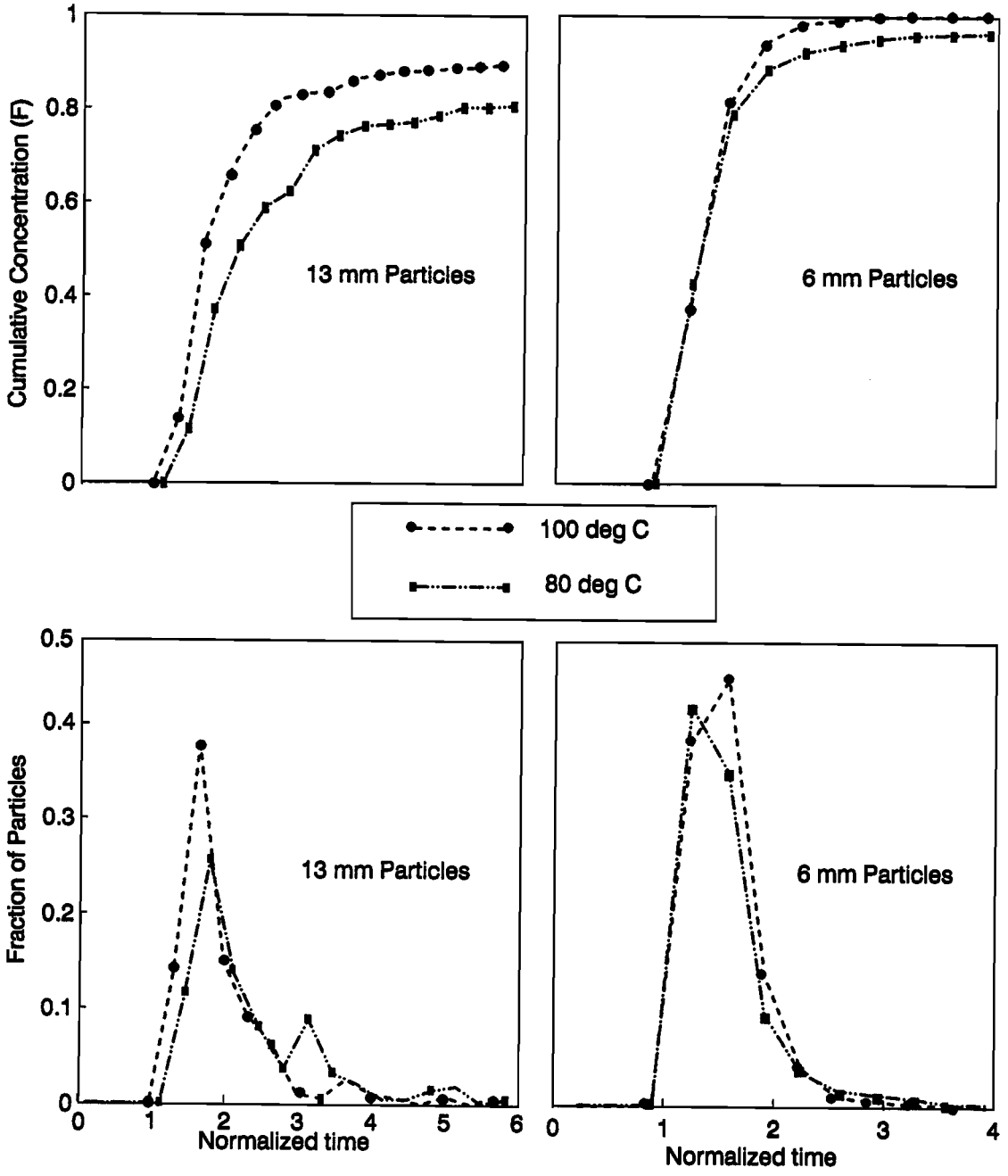

Figure 5.6. **RTD of carrot cubes as influenced by particle size and temperature (flow rate 15 kg/min, 5% starch concentration and 17.45 m holding tube length).**

been simulated by employing carrier fluids at lower viscosity levels characteristic to higher temperatures. In order to get a true effect, however, experiments need to be carried out under real processing conditions because the higher temperatures not only influence the fluid viscosity, but also the thermo-physical properties of both fluid and particle due to cooking.

Holding tube length: Normalized *F* and *E*-type RTD curves for carrot cubes of two sizes as influenced by the holding tube length at a given temperature, starch concentration and flow rate are shown in Figure 5.7. An increase in the holding tube length resulted in a small shift in both *F*- and *E*-curves indicating influence on the particle residence times. On the average, the fastest particle residence time increased with the holding tube length (Table 5.2). Relative to a no-holding tube situation, the FPRT with a 2.5-turn (17.45 m) holding tube was 55% higher and with a 4.5-turn (26.65 m) holding tube it was 85% longer, indicating a perfect linear relationship with the holding tube length. FPNRT of the particles also showed a decreasing trend as the holding tube length increased (Table 5.2). This tend to indicate the existence of some particle-to-particle interaction increasing with the length of the holding tube.

Statistical analysis of RTD

To test the significance of the results, statistical analysis was performed with Statistical Analysis System (SAS) using a General Linear models (GLM) procedure. The basic hypotheses which are the homogeneity of the variance and the normality of the

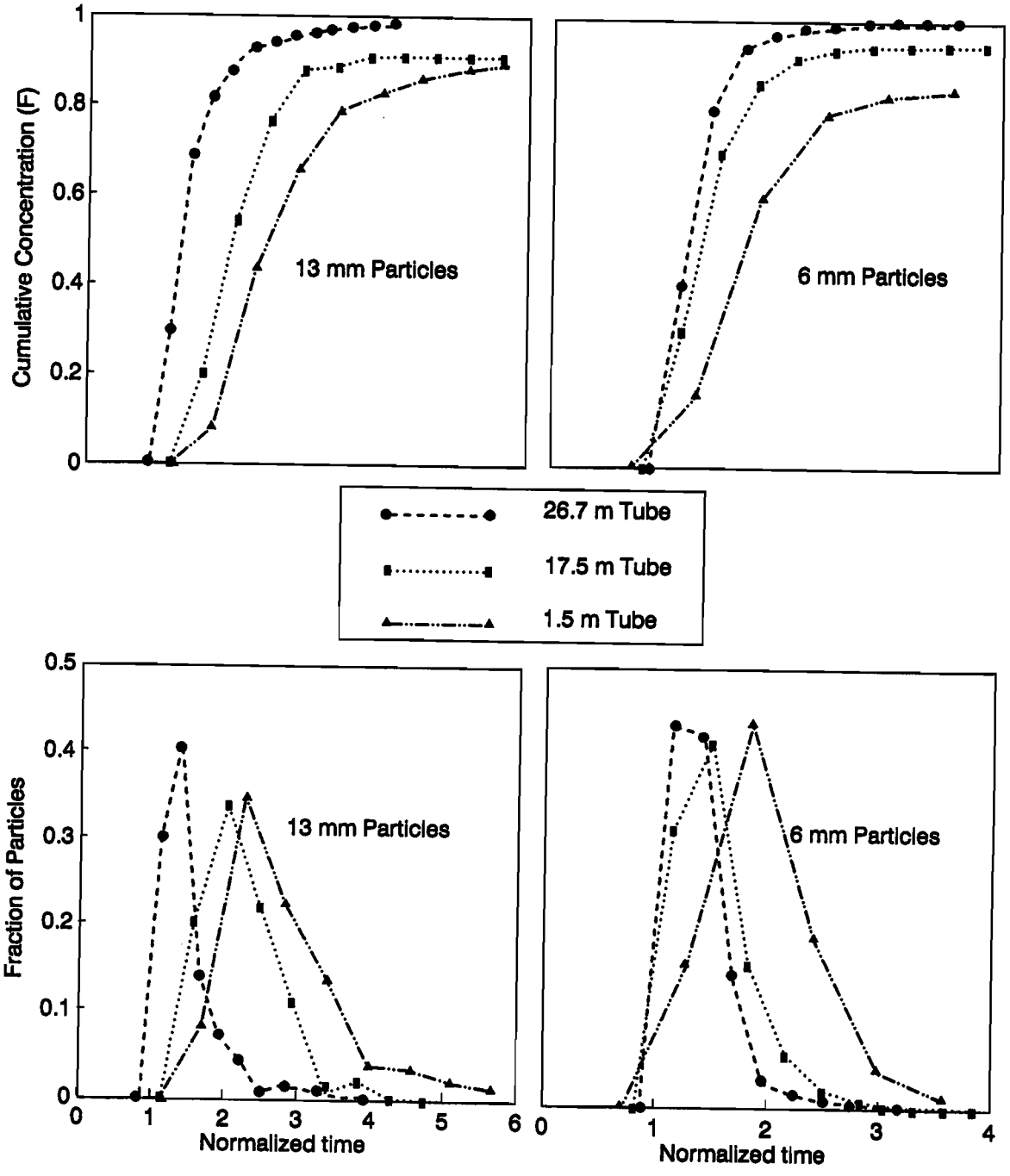

Figure 5.7. **RTD of carrot cubes as influenced by particle size and holding tube length (fluid flow rate 15 kg/min, 5% w/w starch concentration and 100°C).**

Table 5.3. **Analysis of variance for the fastest particle residence time (FPRT, FPNRT), mean residence time (MPRT) and standard deviation of residence time (SRTD) for carrot cubes in a pilot scale aseptic processing system.**

Source	FPRT[1]	FPNRT	MPRT	SRTD
Main effects	86.7	50.6	37.1	4.5
Starch Concentration (C)	11.0[a]	3.5[a]	11.0[a]	17.1[a]
Particle Size (P)	9.3[a]	35.7[a]	4.2[a]	2.3[c]
Hold tube (H)	53.3[a]	6.8[a]	10.7[a]	11.4[a]
Flow rate (F)	21.7[a]	0.4[n]	8.8[a]	1.1[n]
Temperature (T)	1.2[a]	4.2[c]	2.3[a]	2.3[c]
Interactions	3.4	10.4	17.6	18.6
P x H	----	6.0[a]	----	3.6[c]
C x H	0.5[c]	----	----	2.6[c]
C x P	----	----	1.8[c]	4.1[b]
P x F	0.6[b]	----	----	5.4[a]
C x F	----	----	9.1[a]	----
H x F	1.5[a]	----	3.4[a]	2.9[c]
P x T	----	----	----	----
F x T	0.4[c]	----	1.5[c]	----
C x T	----	----	----	----
Residual	9.9	39.0	45.3	46.9
Total	100.0	100.0	100.0	100.0

[1]Percentage contribution to the total sum of squares (%SS).
[abc]Significant at $p < 0.001$, $p < 0.01$, $p < 0.05$; respectively.
[n]Not significant $p > 0.05$.

distribution were verified. Analysis of variance results are summarized in Table 5.3. With few exceptions, all factors studied influenced ($p < 0.05$) the particle flow behaviour. The following are some specific details:

Fastest Particle Residence Time (FPRT & FPNRT): Analysis of variance results showed that all main effects except the flow rate on FPNRT were significant ($p < 0.05$) in influencing the fastest particle residence times. The major interaction was between the particle size and the holding tube length. Particle size was the dominant factor (accounting for ~36% of the total variability) influencing the normalized residence time. However, as expected, holding tube length and flow rate were the major factors with respect to residence time (FPRT), accounting for 53% and 22% of total variability, respectively, with particle size coming next at 9%. Some interactions were significant ($p < 0.05$, Table 5.3), however, individually they contributed no more than 2% of the total variability.

Mean Particle Residence Time (MPRT): MPRT was influenced ($p < 0.05$) by all variables with the exception of starch concentration. Starch concentration (11%), holding tube length (~11%), and flow rate (~9%) were major contributors of variability with respect to MPRT. The interactions terms were not significant ($p > 0.05$) with the exception of four two-way interactions ~18% to the total variability (Table 5.3).

Standard Deviation of Residence Time (SRTD): Standard deviation is an important parameter characterizing the spread of particle RTD. Analysis of variance on SRTD

showed that all main factors were significant ($p < 0.05$, Table 5.3) with the exception of flow rate. Double interaction terms contributed to almost 9% of total variability. Among main effects, holding tube length and carrier fluid concentration were major contributors (~17 and 11% of the total variability, respectively). It can be seen that the spread of particle RTD increased with particle size and decreased with carrier fluid concentration, flow rate, temperature and holding tube length.

Modelling RTD Curves of Carrot

Representative *F* curves, showing the RTD of carrot cubes under various conditions are shown in Figure 5.8. All experimental factors were set at base level (size, 6 mm; starch concentration, 5%; flow rate, 15 kg/min; temperature 100°C; holding tube length, 17.45 m) except for the variable in question. Similar curves for the three holding tube lengths are shown in Figure 5.9. In both these figures (5.8 & 5.9), data points represent experimental observations while lines indicate model fitting. Results indicated good agreement between the developed logistic model and experimental data. The associated r^2 values were generally higher than 0.95.

The time-specific particle concentration distribution (*E*-type) could easily be derived from the developed logistic model (which gives *F* distribution) by subtracting from the cumulative particle concentration at any given time the cumulative particle concentration at the preceding time interval. Again, good agreement was observed between experimental and calculated *E*-type distribution which are also shown under selected conditions in Figures 5.8 and 5.9. Some differences, however, were inevitable.

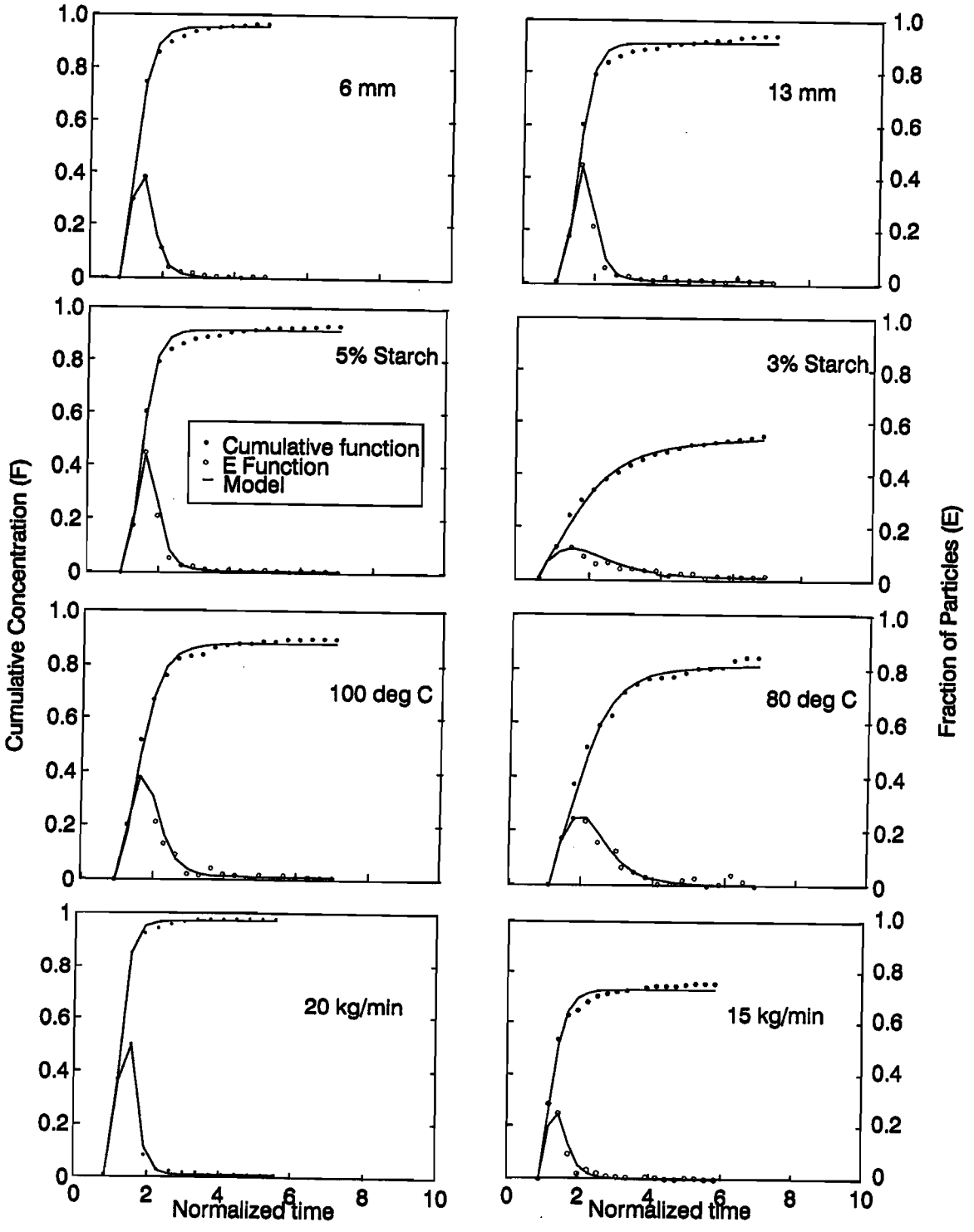

Figure 5.8. **Comparison between experimental and logistic model under aseptic processing various conditions (all factors except the one under study kept set at base level: particle size, 13 mm; starch concentration, 5%; fluid flow rate, 15 kg/min; temperature, 100°C; and holding tube length, 17.45 m).**

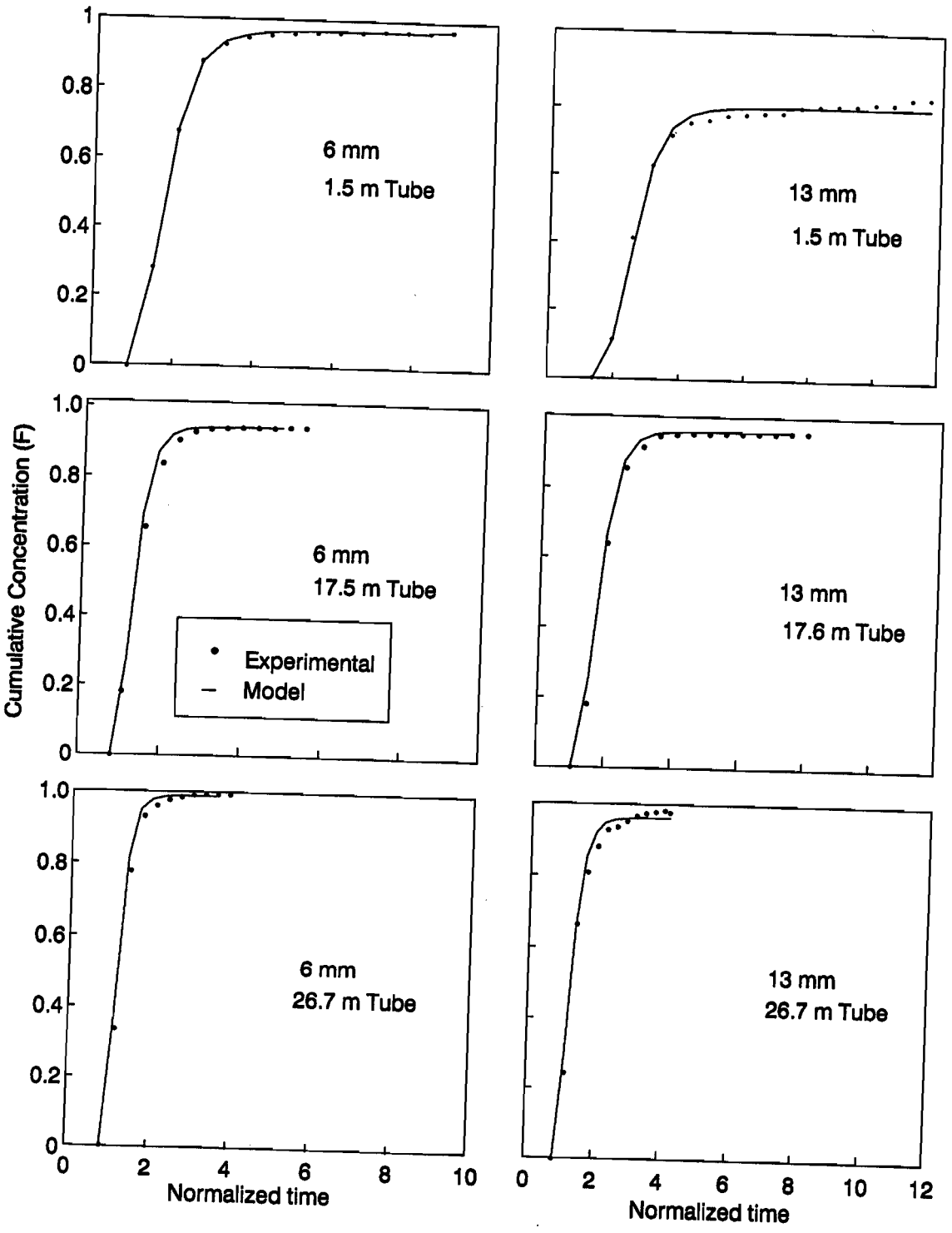

Figure 5.9. **Comparison between experimental and logistic model under aseptic processing with varying holding tube lengths (all other factors set at base level: particle size, 13 mm; starch concentration, 5% w/w; fluid flow rate, 15 kg/min; temperature, 100°C; and holding tube length, 17.45 m).**

The Effect of the Different Factors on Model Parameters

The general influence of the different test factors with the logistic model parameters are summarized in Table 5.4. Data in this table are based on Duncan's multiple range test which usually ignores the interactions between the test factors and therefore provide influence of only the main effects and not their interaction (some of which are significant) on model parameters. It was noted that the half concentration internal age associated with the real time (HRT) is different from that of the normalized time (HNT) on an average by the normalization factors of the carrier fluid average RT's. Normalization permits the use of dimensionless concepts with particle residence times expressed as a fraction (or ratio) of the fluid residence time. While, in general, normalization does not influence the general trends with respect to most factors, they could have a major influence on fluid flow rate and holding tube length as factors. With respect to particle accumulation rate (B) and the upper limit particle concentration (U), it was noted that normalization had no influence.

HRT and HNT values significantly increased with particle size, whereas B and U demonstrated a declining trend. The starch concentration, flow rate, temperature effects were entirely opposite: they resulted in decreased HRT and HNT, while increasing B and U parameters with the exception of U as influenced by temperature which was not significant (Table 5.4). Holding tube length increase also increased B, while U remained unaffected. The overall profiles of the RTD curves demonstrating the effect of the above factors are presented in Figure 5.10. The curves shift toward left with particles of smaller size, at the higher flow rate, at the higher temperature and with the holding tube of longer

Table 5.4. **Duncan's Multiple range test on the half concentration internal age (HRT, HNT), particles accumulation rate (B) and the upper limit particle concentration (U) associated with the logistic distribution model.**

Levels	HRT (min)	HNT	B	U
Particle Size (mm)				
6	3.55^{a}	1.48^{a}	13.6^{a}	0.98^{a}
13	5.13^{b}	2.19^{b}	11.0^{b}	0.80^{b}
Starch Concentration (% w/w)				
3	4.90^{a}	2.09^{a}	7.8^{a}	0.84^{a}
5	3.72^{b}	1.55^{b}	16.8^{b}	0.94^{b}
Flow Rate (kg/min)				
15	5.13^{a}	1.91^{a}	11.4^{a}	0.83^{a}
20	3.55^{b}	1.70^{b}	13.2^{b}	0.95^{b}
Temperature (°C)				
80	4.57^{a}	1.91^{a}	11.7^{a}	0.89^{a}
100	3.98^{b}	1.70^{b}	12.8^{b}	0.89^{a}
Holding Tube Length (m)				
1.50	3.72^{a}	2.24^{a}	10.7^{a}	0.88^{a}
17.45	$4.47^{a,b}$	1.70^{b}	$12.5^{a,b}$	0.88^{a}
26.65	$4.79^{b,c}$	1.51^{b}	13.7^{b}	0.90^{a}

^{abc}Means with the same superscript within the levels of the same factor are not significantly different ($p > 0.05$).

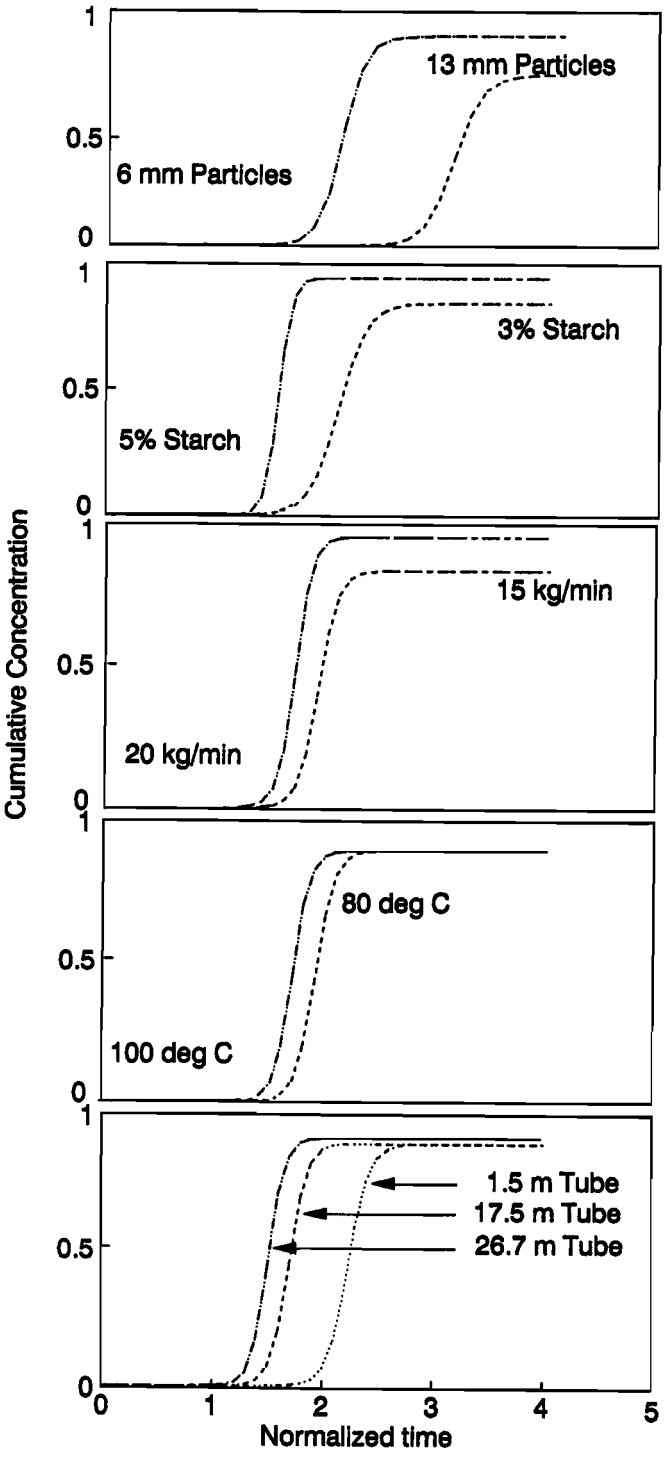

Figure 5.10. **Model predicted residence time distributions of carrot cubes as influenced by particle size, starch concentration, flow rate, temperature, and holding tube length.**

length; all indicative of lowered normalized or relative particle residence times. Two points need deliberation. Lowering of relative residence times indicate higher particle velocity at a given fluid flow rate. Normally, larger particles are expected to move faster than the smaller ones in holding tube since they can intercept a larger cross section of the parabolic velocity profile of the moving fluid. However, in these studies, smaller particles appear to move faster than larger particles possibly because of the effect of scraped surface heat exchanger (perhaps a better situation for aseptic processing because it will reduce particle overcooking). Longer holding tube also decreased the normalized residence times which is perhaps reasonable because in the majority of test runs, the particle residence was smaller than average fluid residence time.

Statistical Analysis of Model Parameters

The analysis of variance showing the influence of test factors on model parameters HRT, HNT, B and U are summarized in Table 5.5. Results showed that all main effects significantly ($p < 0.001$) influenced HRT and HNT. Particle size was the major factor influencing both HRT and HNT contributing over 24% to the total sum of squares. Flow rate (with HRT) or holding tube length (With HNT) are equally influential. Starch concentration comes next with about 14-18% contribution the sum of squares. Some interactions were significant with a total contribution of about 6%. The two way interaction between particle size and temperature was common to both HRT and HNT, both increasing as particle size increased and decreasing as temperature increased. The triple interaction involving particle size, flow rate and temperature was also common to

Table 5.5. **Analysis of variance of the half concentration internal age (HRT, HNT), particles accumulation rate (B) and the particles upper limit concentration (U) associated with the logistic distribution model.**

Source	HRT	HNT	B	U
Main effects	76.5	76.6	85.1	55.7
Starch concentration (C)	13.6[a]	17.7[a]	70.1[a]	8.21[a]
Particle size (P)	24.2[a]	30.0[a]	5.98[a]	32.4[a]
Holding tube (H)	8.85[a]	23.2[a]	5.35[a]	0.40[n]
Flow rate (F)	26.6[a]	2.40[a]	2.74[a]	14.5[a]
Temperature (T)	3.23[a]	3.28[a]	1.00[a]	0.14[n]
Interactions	9.20	10.6	7.17	29.8
C x P	1.42[a]	0.39[n]	0.28[n]	5.61[a]
C x H	0.08[n]	1.05[b]	0.18[c]	1.23[n]
P x F	0.01[n]	0.24[n]	0.01[n]	7.90[a]
C x F	1.43[b]	0.01[n]	0.39[c]	7.21[a]
H x F	0.09[n]	0.86[c]	0.59[c]	0.45[n]
P x T	1.46[b]	2.09[b]	1.33[b]	1.32[n]
C x P x H	0.07[n]	0.96[c]	0.03[n]	0.69[n]
C x P x F	0.73[c]	0.01[n]	0.65[b]	4.92[a]
P x F x T	1.05[b]	1.04[c]	0.12[n]	0.54[n]
C x P x H x F	0.28[n]	0.11[n]	1.06[b]	1.04[n]
P x H x F x T	0.10[n]	0.05[n]	0.53[b]	0.73[n]
Residual	14.3	12.8	7.69	14.5
Total	100.0	100.0	100.0	100.0

[a] [b] [c]Significant at $p < 0.001$, $p < 0.01$, $p < 0.05$; respectively.
[n]Not significant $p > 0.05$.

both HRT and HNT and indicated them to increase with particle size, decrease with temperature and flow rate.

Particles accumulation rate (B) was also significantly ($p < 0.001$) influenced by all main factors. The dominant factor was clearly the carrier fluid concentration which contributed more than 70% of the total sum of the squares (Table 5.5). Other factors individually contributed no more than 6% of the total variability. Interactions between the different factors jointly contributed to less than 5% of the sum of the squares. Only two quadriple interactions were significant: one between particle size, flow rate, holding tube length together with starch concentration in one and temperature in the second. One such interaction is illustrated in Figure 5.11 which indicates that the general trend as indicated by mean values in table 5.4 has some exceptions: B does not always increase as flow rate, starch concentration, holding tube length and temperature increases, and decreased as particle size increased.

With the cumulative particle concentration upper limit (U), only particle size, flow rate and starch concentration effects were significant ($p < 0.001$), with 32, 14 and 8%, respectively, contribution toward the total sum of the squares (Table 5.6). On the other hand, the interactions contribute to more than 25% of the total variability (three two way and one three-way interactions were significant). Normally, under proper operating conditions, U should be a constant indicating the upper limit which as a fraction of the initial concentration is always unity. However, as seen in this study, there are several conditions which combine low flow rate, low concentration of starch and larger particle

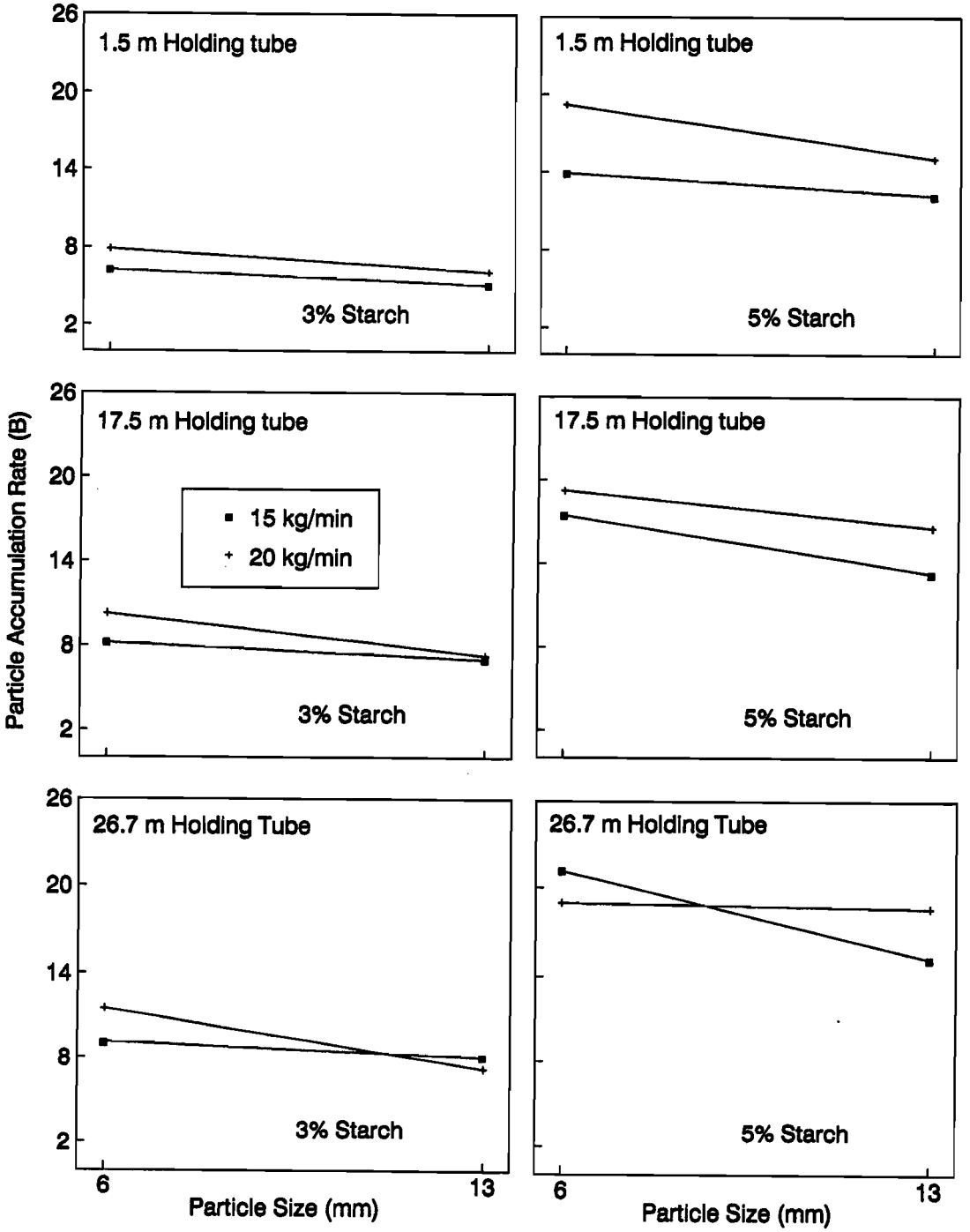

Figure 5.11. **Quadruple interaction of particle size, flow rate, holding tube length and starch concentration on particle accumulation rate (B).**

size which will not allow a smooth flow of the particles through the system. Within the short experimental time frame, these will show up as nonrecoverable particles with equilibrating to a value lower than unity (Figures 5.8-5.10).

Multiple Regression Equations for Model Parameters

Coefficients for the multiple regression relationships for the model parameters and their associated R^2 are presented in Table 5.6 with a plot of observed vs predicted values in Figure 5.12. Only two-way interactions, significant at $p < 0.01$, were included in the multiple regression analyses to keep equations simple. The model fitting appears to be reasonably good, however, extrapolation of the model to ranges beyond those covered in this study should be avoided. The determination coefficients of 0.78, 0.74, 0.87 and 0.80 for HRT, HNT, B and U, respectively.

Significance of the Systemic Approach

Under real aseptic processing situations it is very difficult to isolate the effects of the system components (heat/hold/cool) on RTD of food particles. The approach undertaken in this study was therefore based on the whole system together. Regulatory agencies approved only the RTD information collected through the holding tube, which can be obtained by difference taking the RTD within the SSHEs as the control. This procedure tends to cancel the effect of the SSHEs and account only for the holding tube. A perfect linear relationship was obtained when the holding tube length was varied under this systemic approach as shown in Figure 5.13 for all the test factors. This seems to

Table 5.6. Multiple regression coefficients of the half concentration internal age (HRT, HNT), particle accumulation rate (B) and the upper limit particle concentration (U) associated with the logistic model.

	Regression Coefficients			
Source	HRT	HNT	B	U
Regression Constant	11.82	0.8978	6.0721	0.5834
Starch concentration (C)	-2.0148	-0.1804	2.1418	0.2448
Particle size (P)	1.3835	0.6001	-1.9568	-0.1639
Holding tube length (H)	0.0106	-0.0200	0.0984	-0.0002
Flow rate (F)	-0.8589	-0.0532	0.0297	0.0438
Temperature (T)	0.0459	0.0232	-0.1139	0.0005
C x H	----	0.0028	----	----
C x P	-0.0925	-0.0299	----	0.0111
P x F	----	----	----	0.0053
C x F	0.1258	----	0.1330	-0.0173
H x F	----	----	-0.0039	----
P x T	-0.0085	-0.0041	0.0176	----
Determination coefficient (R^2)	0.78	0.74	0.87	0.80

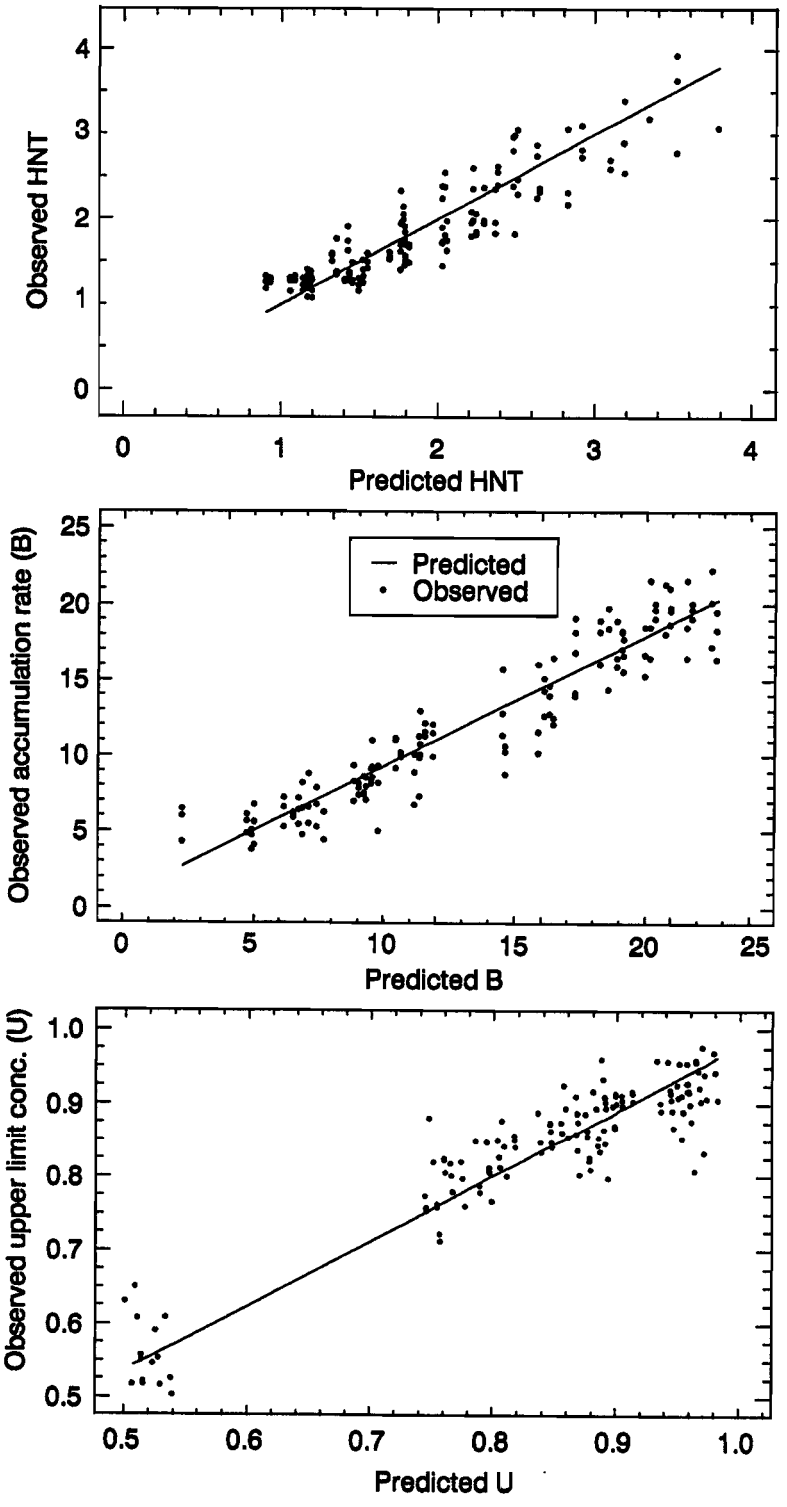

Figure 5.12. **Observed and model calculated values of half concentration internal age (HNT), particle accumulation rate (B) and particle concentration upper limit (U).**

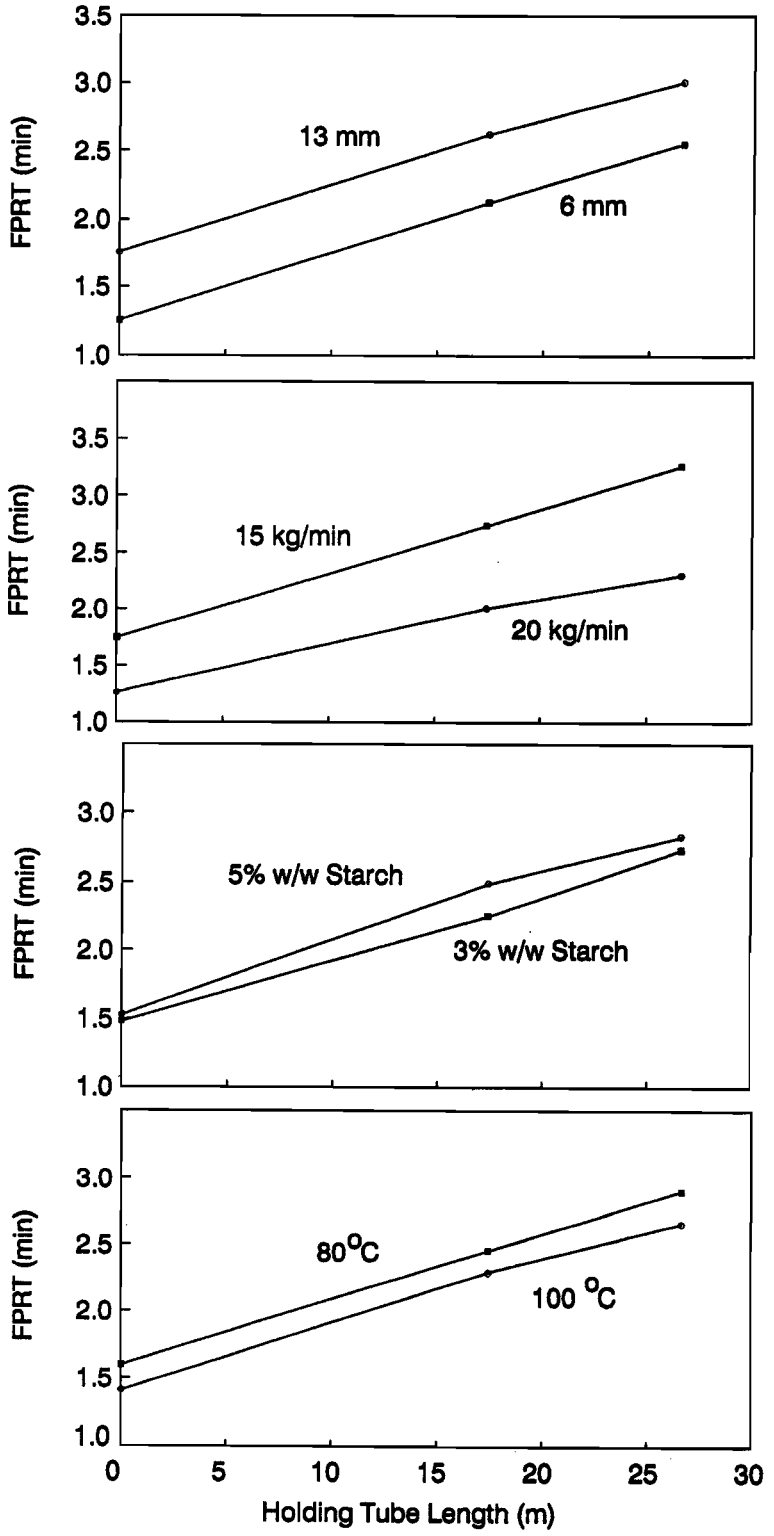

Figure 5.13. **Effect of the holding tube length on FPRT of carrot cube under the different test factors (particle size, fluid flow rate, starch concentration and temperature).**

agree with the results of RTD studies in the holding tube only (Chapter IV) which showed that RTD parameters linearly increased with holding tube length. Further investigation showed that the calculated particle velocity was about 0.34 m/s with the whole system and 0.26 m/s for the holding tube length only experiments. These magnitudinal differences were expected due to the different experimental conditions used, especially the operating temperatures. Ramaswamy *et al.* (1992) and Grabowski and Ramaswamy (1994) showed that increased temperature resulted in higher particle velocities, probably due to the temperature effect on viscosity. Even within the present experimental set-up lowering the temperature from 100°C to 80°C increased the residence time, and hence decreased the particle velocity by about 10% (Table 5.2). The higher starch viscosity at room temperature as compared with 80-100°C (Chapter VII) could easily account for the ~30% lower velocities associated with holding tube only experiments conducted at room temperature. Thus, using the systemic approach technique gave RTD information matching the RTD data obtained using the SSHE (Chapter III) and the holding tube (Chapter IV) individually. This implies that RTD within the holding tube can be estimated by difference between RTD within the SSHEs and the holding tube. The advantage of using the whole system for RTD studies is the possibility of employing pressurized situations similar to actual processing conditions taking into account all process variables.

CONCLUSIONS

Results from this study, using a modified pulse input procedure, give some insight into RTD of food particles as influenced by the particle (size), carrier fluid (concentration and flow rate) and system (temperature and holding tube length). Using the whole system approach gave RTD results similar to those obtained using only the SSHE and the holding tube. Tests were carried out employing the maximum attainable temperature under the flow conditions. All factors investigated have been found to influence the particle RTD. On an average, under the conditions of testing used in the present study, the fastest travelling food particle moved at a velocity of 1.25 times higher than or equal to the average velocity of the carrier fluid.

The logistic model can be used to characterize the RTD curves of carrot cubes passing through a pilot plant aseptic processing system based on scraped surface heat exchangers. The parameters correlated well with the factors influencing RTD.

CHAPTER VI

BIOLOGICAL EVALUATION OF PARTICULATE FOODS IN A PILOT SCALE ASEPTIC SYSTEM USING *BACILLUS SUBTILIS* SPORES IMMOBILIZED IN CARROT/ALGINATE CUBES

ABSTRACT

Biological evaluation of particulate foods was carried out in a commercial pilot scale aseptic processing system using *Bacillus subtilis* spores immobilized in 12 mm carrot/alginate cubes. Experiments were performed with 5% w/w starch as the carrier fluid at 15 kg/min flow rate at 120, 115, 110, 105, 100 and 95°C under two system configurations. In the first, the two SSHEs were connected with a short holding tube (1.5 m), while in the second a holding tube of 26.7 m was attached between the two SSHEs. The aseptic system data was compared to that obtained for *B. subtilis* embedded in carrot/alginate cubes, heated in a silicone oil for selected time-temperature combinations. All test factors were found to influence the destruction/survival of the *B. subtilis* spores.

INTRODUCTION

The production of high quality foods is the major driving force behind the adoption of aseptic processing for homogeneous liquid foods. The extension of this technology to liquid foods containing particulates is difficult due to lack of data on the fluid-to-particle interfacial heat transfer coefficient (h_{fp}) as well as the residence time

distribution for liquid/particle mixtures (Tucker and Withers, 1992). The time a particle spends in the holding tube of an aseptic processing system and the processing temperature dictate the lethality of the process. As the particles flow through the holding tube, they spend different length of time inside the system; thus creating a residence time distribution (RTD). The accumulated lethality can be calculated according to the time spent in the system from the expression:

$$F_0 = \int_0^t 10^{\frac{(T-T_{ref})}{z}} dt \qquad (46)$$

where F_o is the integrated centre-point lethality (min), T is the measured centre-point temperature (°C), T_{ref} is the reference temperature usually taken as 121.1°C, z = the number of degrees required to cause a decimal increase or decrease in the D value or the decimal reduction time (C°), t is the time (min). Lethality calculations must be based on centre-point temperature of the fastest particle travelling in the holding tube of the aseptic processing system in order to address the issue of public health concerns (Lee *et al.*, 1990a,b; Rao, 1992). With biological indicators, the integrated F-value (sterilization value of the process, F_s) which is a measure of the degree of lethality at isothermal conditions can be calculated according to Stumbo's (1973) formula:

$$F_s = D_{ref}(\log N_o - \log N) \qquad (47)$$

where F_s= sterilization value (min), D_{ref} = decimal reduction time at 121.1°C (min), N_o = initial number of microorganisms (CFU/mL), and N = final number of microorganisms at any time.

Assurance of microbiological safety requires an appropriate thermal process based on either accurate time/temperature data collected at the slowest heating point within the fastest moving particle, or a biological evaluation of the F_o-value achieved at the coldest point (Dignan *et al.*, 1989). Currently, the physical determination of the temperature at the centre of a particle moving in an aseptic system is not practical (Sastry, 1986; Sastry *et al.*, 1988; Gaze *et al.*, 1989). Several researchers (Berry *et al.*, 1989 & 1990; Weng *et al.* 1991 & 1992; and Kim and Taub, 1993) have employed chemical and enzymatic markers to validate aseptic processing of liquid/particle foods. But the regulations of the Food and Drugs Administration (FDA) require only inoculated-pack validation for aseptic process of viscous low-acid liquid foods containing particulates (Dignan *et al.*, 1989). Consequently, microbiological validation is considered as the only definitive test to address the uncertainties of the critical factors in aseptic processing of low-acid liquid/particulate foods (Segner *et al.*, 1989; Pflug *et al.*, 1990; Rao, 1992). Different techniques for immobilizing bacterial spores such as *Bacillus anthracis* in polymethylmethacrylate (perspex) beads (Hunter, 1972) and *Bacillus stearothermophilus* spores in alginate beads (Dallyn *et al.*, 1977) have been employed. In these studies only small particles (1.6-3.2 mm) were used. The alginate/spore beads were also utilized to investigate the lethality achieved in a pilot scale scraped surface heat exchanger in the temperature range of 138-142°C (Bean *et al.*, 1979) as well as that achieved in stationary large particles (0.8-2.4 cm cubes of alginate/potato, meat or pea) in a holding tube at the temperature range of 120-150°C (Brown *et al.*, 1984). Thus, available literature on biological validation is limited especially under commercial aseptic processing conditions.

The objectives of this study was to investigate the effects of particle residence time, holding tube length and process temperature on the inactivation of *Bacillus subtilis* spores immobilized in carrot/alginate cubes in a pilot scale aseptic processing system.

MATERIALS AND METHODS

Preparation of Carrot/Alginate Cubes

Carrot obtained from a local farm (St. Remi, Quebec) was manually washed, peeled and steam cooked at 121.1°C for 20 min in order to reduce the microbial load and soften the carrots to facilitate further treatments. A puree was prepared using 300 g of carrots and 100 mL distilled water in a laboratory blender. 100 g of the resulting puree was mixed with 0.075 g of tri-sodium citrate (BDH Chemical Inc., Toronto) and 6 g of sodium alginate powder (BDH Chemical Inc., Toronto) in a sterile laboratory blender for 5 min to ensure complete dissolution. The spore suspension (25 mL, 4.57 x 10^7/mL) of *Bacillus subtilis* (ATCC 6633) followed by 0.3 g of calcium sulphate (BDH Chemical Inc., Toronto) in 10 mL distilled water were aseptically added to the carrot/alginate puree, thoroughly mixed and immediately transferred into a 12 mm cube shaped mold. The mold was subsequently immersed in 2% w/v calcium chloride solution (Sigma Chemical Co., St. Louis, MO) for approximately 24 h at room temperature. The cubes were then removed from the mold and stored at 4°C in sterile distilled water for up to 3 days. Bacterial spore stocks were prepared by standard methods as described by the National Canners Association Research Laboratories (1968) using Campden sporulation agar (CSA). The components of this agar are listed in Table 6.1 (Gaze *et al.*, 1989).

Table 6.1. Compositional formulation of the sporulation (CSA) and recovery (YDTAS) media of *Bacillus subtilis* spores (ATCC 6633).

Components		Quantity (g/litre)
Campden Sporulation Agar (CSA)		
Tryptone	Oxoid L42	5.0
Bacterial peptone	Oxoid L37	5.0
Lab Lemco	Oxoid L29	1.0
Yeast extract	Oxoid L21	2.0
Calcium chloride	AnalaR	0.056
$MnSO_4.H_2O$	AnalaR	0.062
Glucose	AnalaR	1.0
Bacto agar	Difco	15.0
Yeast Dextrose Tryptone Agar Enriched with Starch (YDTAS)		
Bacteriological peptone	Oxoid L37	5.0
Tryptone	Oxoid L42	2.5
Beef extract	Oxoid L20	1.0
Glucose	AnalaR	1.0
Soluble starch	AnalaR	1.0
Bacto agar	Difco	15.0
Chemical Products Utilized in Carrot/alginate Cubes Preparation		
Sodium alginate of *Laminaria hyperborea* (BDH)		
Hydrated calcium chloride ($CaCl_2.2H_2O$)	AnalaR	
Sodium citrate, tribasic ($Na_3C_6H_5O_7.2H_2O$)	AnalaR	
Hydrated calcium sulfate ($CaSO_4.½H_2O$)	AnalaR	

pH was adjusted to 7.0.
Media was Autoclaved for 15 minutes at 121.1°C.

Aseptic Processing System

The system used for biological evaluation is described in Chapter V (Figure 5.1). The two heat exchangers were inter-connected directly with a short tube (1.5 m) in the no holding tube situation, while and full length (26.65 m) of the holding tube was used to represent the whole system. The dimensions of the system, carrier fluid and particle properties are detailed in Table 6.2. At the beginning of the experiments, the system was equilibrated with the carrier fluid (5% w/w starch) prepared as detailed in Chapter III.

Heating of the Carrot/Alginate Cubes

100 g of carrot/alginate cubes containing the *B. subtilis* spores (12 mm) were mixed in the liquid/particles tank with 1.9 kg of the carrier fluid (5% w/w starch) to obtain 2 kg of particle/liquid mixture (5% w/w particle concentration). In a typical run, the system was first equilibrated with the carrier fluid until the process temperature was attained (95, 100, 105, 110, 115 and 120°C) at a flow rate of 15 kg/min. The operational procedure is as described in Chapter III. The time interval from the moment the particle/liquid mixture was introduced into the system to when the first particle emerged out of the system was timed and taken to represent the fastest particle residence time. The fluid and the particles emerging out were subsequently collected at 20, 30 and 60 sec intervals for 120-105, 100 and 95°C, respectively. Only one carrot/alginate cube was drawn at the beginning of each interval to assess the survival/destruction of the *B. subtilis* spores. To have a comparison with conventional thermal processing, carrot/alginate inoculated cubes were placed in the same starch solution as above in a closed chamber

Table 6.2. Specifications of the pilot scale aseptic processing system used to study the biological evaluation of carrot/alginate inoculated cubes.

Parameter	Specification
Scraped surface heat exchanger	
Scraped surface heat exchanger	1.8 X 0.9 m
Internal diameter	0.152 m
Internal shaft diameter	0.076 m
Length of the heating surface	0.762 m
Material of construction	Stainless steel
Dasher speed	300 rpm
Heating medium	Steam at 150°C
Cooling medium	Water at 10°C
Holding Tube	
Total length	26.65 m
Internal diameter	0.0381 m
Carrier fluid	Therm-flo® starch
Gelatinization temperature	140 °C
Concentration	5% w/w
Density	1019 kg/m^3
Flow rate	15 kg/min
Particles	carrot/alginate cubes
Size	12 mm
Concentration	5% w/w
Density	1040 kg/m^3
Biological indicator	*Bacillus subtilis* (ATCC 6633)

heated by a silicone oil bath as shown in Figure 6.1. The heating temperatures were 95, 100 and 105°C. Heating times were the same as those experienced in the scraped surface heat exchangers (SSHE) with no holding tube. The minimum heating time was chosen to be equal to that of the fastest particle residence time. The proceeding time intervals were also chosen to be similar to those of the aseptic system.

Recovery Technique

Following heat treatment, complete dissolution of the carrot/alginate cubes to release the *Bacillus subtilis* spores was achieved by adding 4 mL of 1% w/v sodium citrate ($Na_3C_6H_5O_7.2H_2O$) per 1 g of the cubes in a specialized sterilized bags (Model "400" bags, Seward Medical, London, England) and shaking in a laboratory blender (Stomacher Lab-Blender 400, Model No. BA 6021; UL Laboratory Equipment, London, England). The pH of the citrate solution was adjusted to approximately 7.1 in order to obtain good recovery (Dallyn *et al.* 1977). 1 mL samples from each bag was aseptically transferred to sterile test tubes of peptone water and appropriate decimal dilutions were made. The dilutions made were then water-bath heated at 80°C for 10 min to activate the *Bacillus subtilis* survivors and eliminate vegetative cells (Russell, 1982). The survivor spores were enumerated by plating 1 mL aliquot from the serial dilutions on a petri dish containing approximately 15 mL of Bacto agar, YDTAS (Difco) supplemented with bacteriological peptone (Oxoid L37), tryptone (Oxoid L42), beef extract (Oxoid L20), glucose (AnalaR, BDH), and soluble starch (AnalaR, BDH) as listed in Table 6.1. The pH of the mix was adjusted 7.0, solubilized and autoclaved at 121.1°C for min.

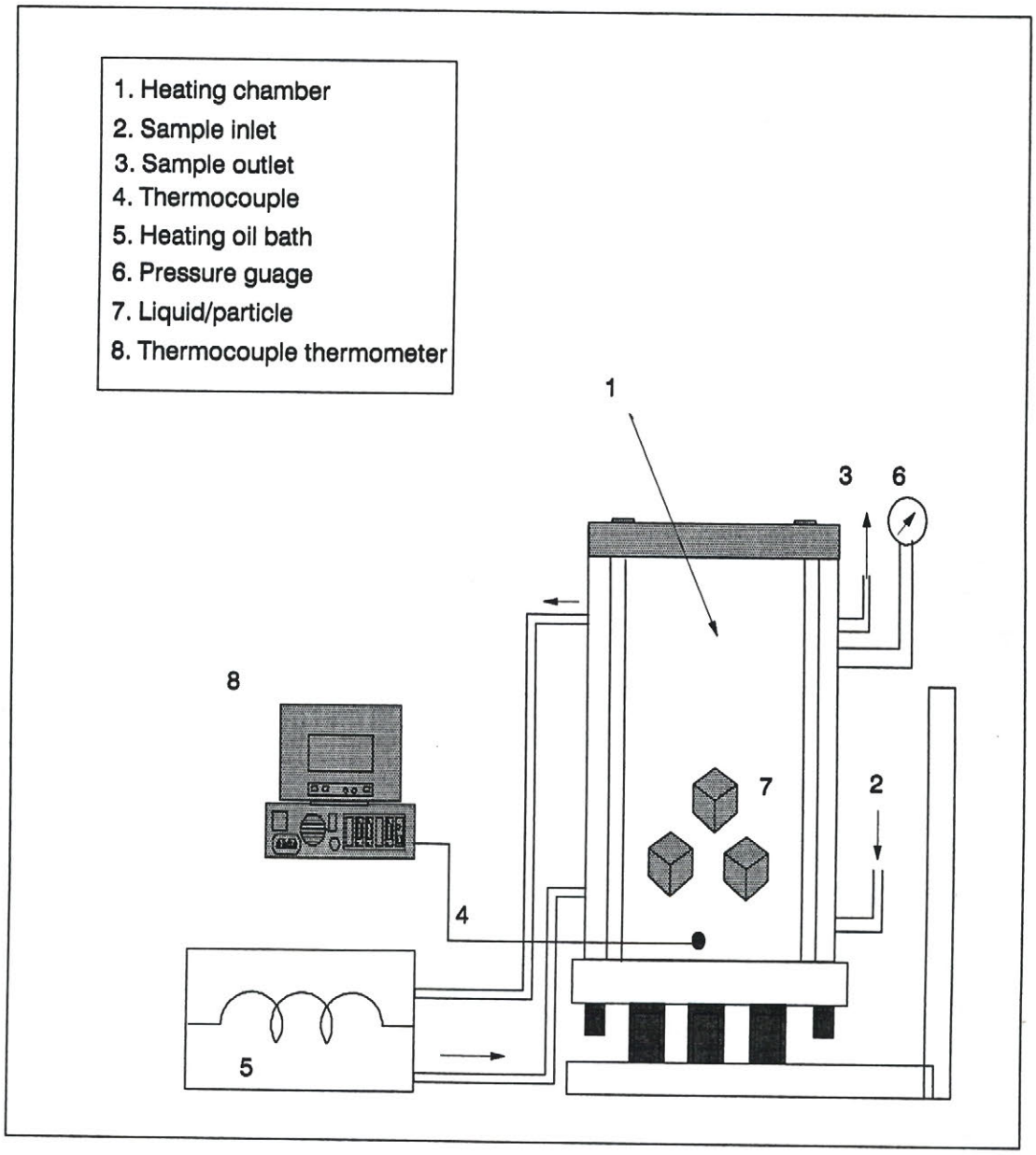

Figure 6.1. **Experimental set-up for the pressurized oil-heated chamber used for the evaluation of the heat resistance of *Bacillus subtilis* spores immobilized in carrot/alginate cubes.**

The plates were shaken and left to solidify for 30-45 min at room temperature and incubated at 35°C for 48 hr before counting colonies.

RESULTS AND DISCUSSION

Survivor curves for *Bacillus subtilis* spores immobilized in carrot/alginate cubes using heating and cooling scraped surface heat exchangers (SSHEs), i.e. no holding tube, at 95 and 100°C are presented in Figure 6.2. The higher temperature resulted in higher destruction levels of the *B. subtilis* spores especially particles exiting after longer residence times. Complete destruction of *Bacillus subtilis* spores was observed with the whole system (heat-hold-cool) at the temperatures of 110, 115 and 120°C. At 100 and 105°C, however, bacterial survival was noticed only with the fastest particle. A similar trend prevailed with the heat-cool system in the temperature range 110-120°C. The calculated sterilization values of the processes are summarized in Table 6.3 for the temperature range 95-105°C. Heat-hold-cool processes were obviously more effective than the heat-cool processes. Survivor curves shown for SSHE only *vs* SSHE-hold systems are compared in Figure 6.3 for processing at 95°C.

The bacterial load of the fastest particle was assumed as the final concentration of *B. subtilis* spores for sterilization value calculations using a decimal reduction time ($D_{121.1^oC}$) of 0.7 min reported by Fellows (1988). Moreover, the use of this technique for biological verification will only give integrated sterilization values because the calculations are not based on the slowest heating centre spore survival (Pflug *et al.*, 1990), besides the difficulty of obtaining the temperature profiles in these particles.

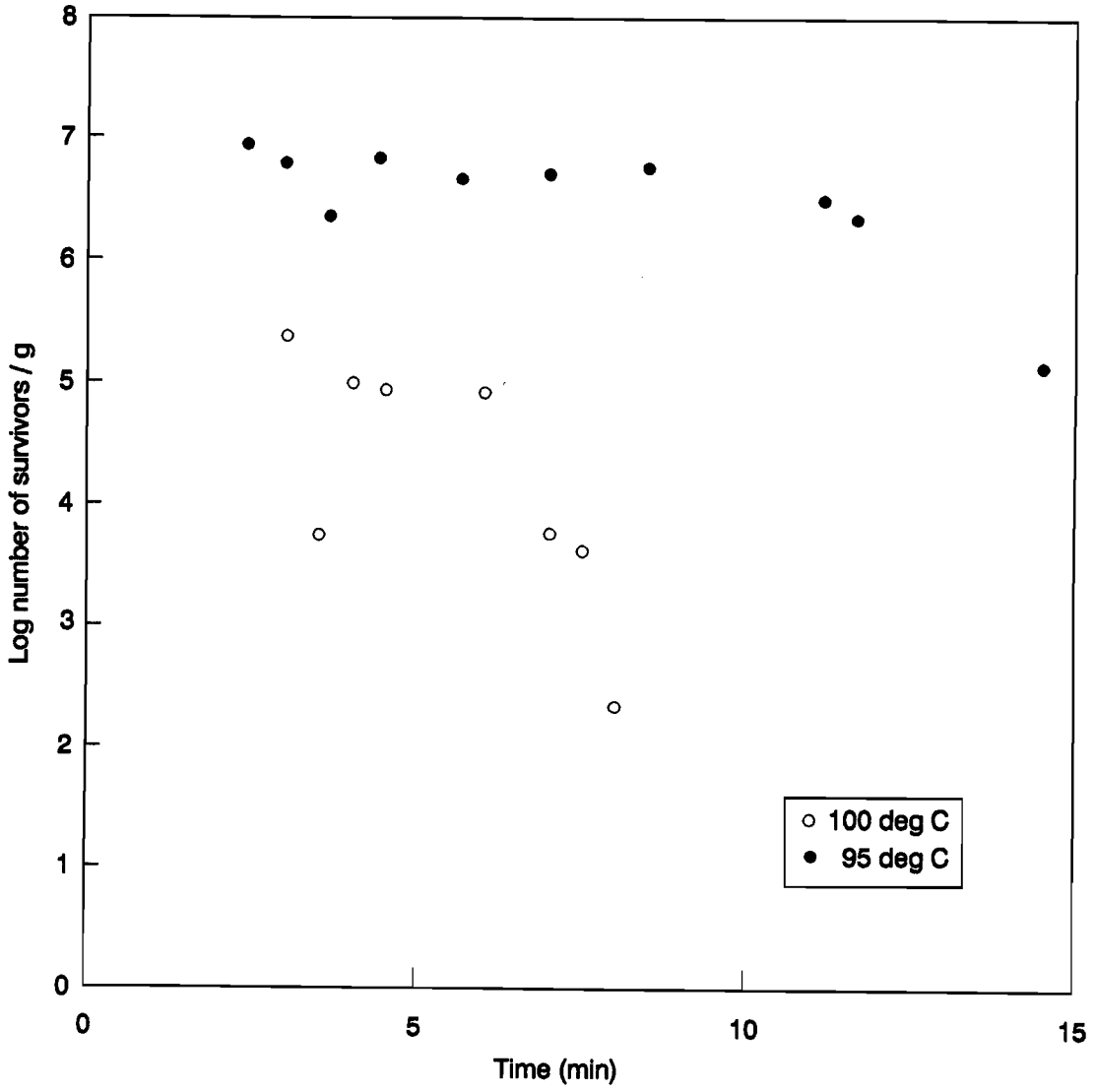

Figure 6.2. **Thermal destruction time curves for *Bacillus subtilis* spores exposed to a heat-cool cycle in a pilot scale aseptic processing system at 95 and 100°C under 15 kg/min flow rate and 5% w/w starch concentration.**

Table 6.3. Minimum sterilization values[a] of *Bacillus subtilis* (ATCC 6633) as influenced by the processing system under different processing temperatures ($D_{121.10C}$ = 0.7 min)

Heating Temperature (°C)	Minimum Sterilization Values (min)		
	heated chamber	Heat-cool	Heat-hold-cool
95	0.75 ± 0.11	1.15 ± 0.20	1.63 ± 0.15
100	1.05 ± 0.21	1.21 ± 0.12	3.95 ± 0.33
105	2.75 ± 0.19	3.12 ± 0.71	4.33 ± 0.26

[a]Calculated using Eqn 47.

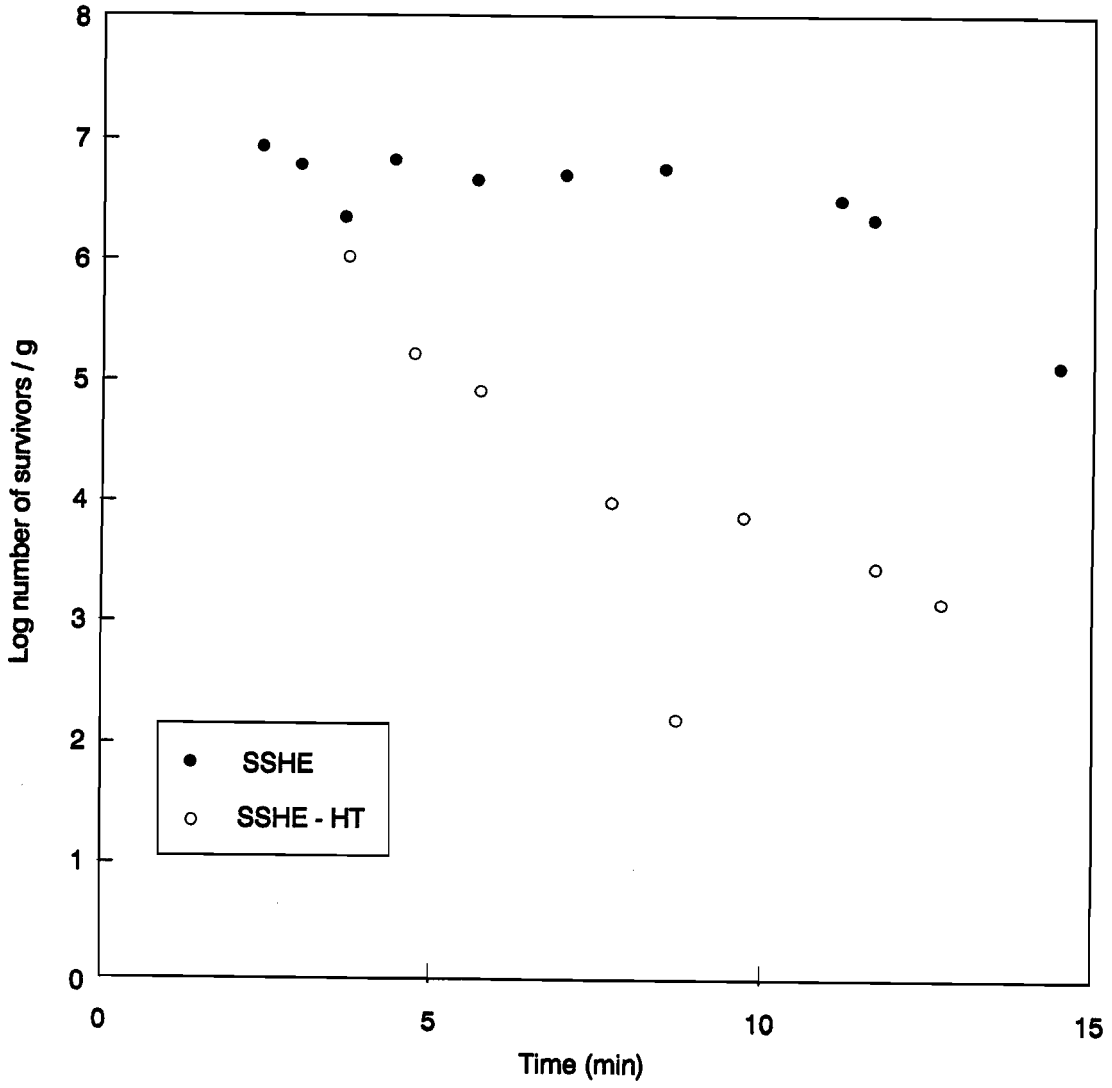

Figure 6.3. **Thermal destruction data for *Bacillus subtilis* spores exposed to a heat-cool and heat-hold-cool cycles in a pilot scale aseptic processing system at 95°C under 15 kg/min flow rate and 5% w/w starch concentration.**

Higher minimum sterilization values were achieved with the aseptic processing than when carrot/alginate cubes containing *Bacillus subtilis* spores were subjected to the same heating temperatures (95, 100 and 105°C) for the same length of time in pressurized silicone oil heated chamber (Figure 6.4). Radial mixing and agitation of liquid/particles mix by the scraper blades as well as the axial movement due to flow in the heating SSHE and the holding tube were considered the likely reasons for the enhanced heat transfer and increased lethal effects.

D-values were obtained for *B. subtilis* by two procedures. In the first one, the residence time in the holding tube was obtained as a difference between the RT in the heat-hold-cool system (RT_2) and the heat-cool system (RT_1). Similarly, the number of decimal reductions in bacterial spores in the holding tube was obtained by difference. The residence time was divided by the number of decimal reductions achieved. D-values calculated from such a procedure were 2.39, 0.43 and 0.32 min at 95, 100 and 105°C; respectively. D-values were also calculated for the heating chamber by tests, for fastest particle as time divided by logarithmic decimal reductions. The calculated values were 4.0, 2.88 and 1.09 min at 95, 100 and 105°C; respectively. These D-values are comparable to those of Harnulv and Snugg (1972) and Smith and Brown (1980) who reported D-values of 3 min for *B. subtilis* var. *globigii* (NCIB 8058) at 96°C and 4.8 min for *B. subtilis* (NCIB 8054) at 95°C; respectively, although experimental conditions and spore strains were different. Ronner (1990) employed a commercial 8 mm plastic spherical model bioindicator containing *Bacillus subtilis* (NCTC 10400) and reported thermal death time (TDT) values of 45, 5.7 and 1.7 min at 100, 110 and 116°C, respectively at the

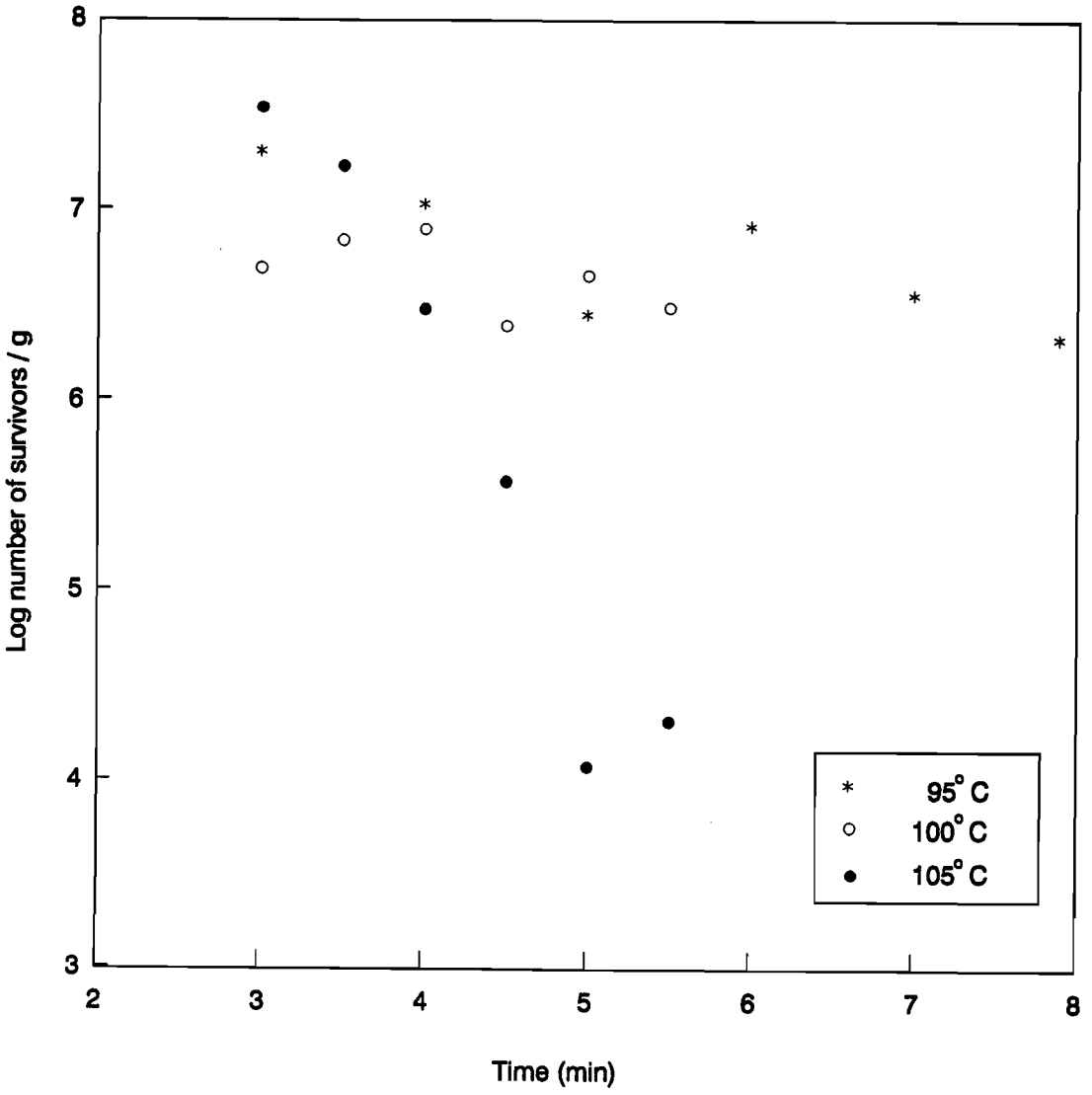

Figure 6.4. **Thermal destruction curves for *Bacillus subtilis* spores exposed to a heat-cool cycle in a pressurized silicone oil-heated chamber at 95, 100 and 105°C in 5% w/w starch solution.**

inoculation level of 10^6 CFU/g. No information was given on the exact chemical composition of this bioindicator. The TDT values can vary depending on product characteristics like viscosity, chemical composition, pH, processing equipment and the bacterial strain used. The high thermal sensitivity of *Bacillus subtilis* (ATCC 6633) may attributed to some of the chemical components used in the fabrication of the bioindicator especially at elevated temperatures. At room temperature chemical composition of carrot/alginate had no effect on the spores. According to Russell (1982) some divalent metallic cations can affect the heat resistance of bacterial spores.

Further kinetic studies and characterization on the *Bacillus subtilis* (ATCC 6633) bioindicator were not possible because of the relative heat sensitivity of this spore-former strain although the reported z-value was 4.1-7.2 C° (Fellows 1988).

CONCLUSIONS

Mesophilic *Bacillus subtilis* spores immobilized in carrot/alginate cubes were used for biological evaluation in a pilot scale aseptic processing system, but at relatively low temperatures (95-105°C) as no survivors were observed at high temperatures ≥ 110°C. Carrot/alginate cubes showed excellent mechanical strength withstanding pumping as well as the scraper blades beating without disintegration. Higher sterilization were achieved with aseptic system compared to the static heated chamber, although difficut to justify. Destruction of the spores increased with temperature and holding tube length.

CHAPTER VII

RHEOLOGICAL PROPERTIES OF STARCH AT ASEPTIC PROCESSING TEMPERATURES

ABSTRACT

Rheological properties of cross-linked waxy maize starch in the concentration range of 3 to 6% w/w were evaluated using a computer controlled rotational viscometer equipped with a high temperature/high pressure attachment and a magnetic coupling. The influence of temperature on the rheological parameters were evaluated in the temperature range 60-140°C. The flow curves essentially followed the power law model and both the consistency coefficient and flow behaviour index were sensitive to changes in temperature and concentration. Temperature dependency followed the Turian model.

INTRODUCTION

Lack of rheological data at elevated temperatures is one of the major problems facing modelling of aseptic processing of low acid liquid foods containing particulates. Most liquid foods are viscous and non-Newtonian in nature. Thermal process design for liquid foods with or without particulates require accurate information on the flow behaviour of the fluid as well as particle/fluid relative velocities in the holding tube to arrive at processing conditions which ensure safety and improve quality factor retention (Subramaniam and Zuritz, 1990; Alcairo and Zuritz, 1990). The relative velocity between

the liquid medium and the food particle may also influence the fluid to particle convective heat transfer coefficients in the holding tube (McCoy *et al.*, 1987; Hallstrom *et al.*, 1988; Dail and Steffe, 1990a,b).

Starches are widely used in foods as thickening agents which result from the swelling of starch granules occurring at the gelatinization temperatures (Self *et al.*, 1990). Recently, a modified cross-linked starch was used to simulate carrier fluids in liquid foods containing particulates (Chandarana *et al.*, 1989; Chandarana *et al.*, 1990). It has been recognized that the rheological properties of fluids depend on concentration of the active compound, temperature and shear rate (Harrod, 1989a). Consequently the rheological properties has to be based on: 1) a representative model product, 2) an appropriate measuring technique, 3) measurement of flow properties over wide ranges of shear rate, temperature and concentration, and 4) investigation of the time-dependency of the flow properties. In a useful contribution, Harrod (1989a,b,c) reported some effects of concentration, preparation method and time-dependency on the rheological properties of modified (cross-linked and esterified) potato starch.

Available data on cross-linked waxy starches at elevated temperatures is limited. Dail and Steffe (1990a,b) studied starch rheology under aseptic processing conditions using relatively low concentrations (1.82, 2.72 and 3.15% g dry starch/100g water) over a small range of shear rate (35-175 s^{-1}). Harrod (1989a) and Self *et al.* (1990), on the other hand, employed high concentrations, but at relatively low temperatures (10-90°C).

Most experimental data on residence time Distribution (RTD) and heat transfer studies pertaining to liquid foods containing particulates have been reported at near room

temperatures (Chandarana *et al.*, 1989; Chandarana *et al.*, 1990), probably due to the lack of rheological data obtained on starch at high temperatures. The objectives of this work were (1) to study the flow behaviour of modified cross-linked starch solutions (3-6% w/w) by evaluating the relationship the shear stress/shear rate data, and (2) to study the effects of temperature, concentration, preparation and gelatinization on starch rheology.

MATERIALS AND METHODS

Test samples of starch were freshly prepared using commercially available Therm-flo® starch Lot # HK 4254 (National Starch and Chemical Corp.; Bridgewater, NJ). This starch is a cross-linked waxy maize recommended for low acid foods that are thermally processed. Four concentrations (3, 4, 5 and 6% w/w) were used. Prior to measurements, starch samples were gelatinized at 140°C employing the aseptic processing system previously described in Chapter III.

Rheological measurements were carried out using a Haake rotational viscometer Model RV20 (Haake Mess-Technik GmbH u. Co., Karlsruhe, Germany), equipped with an M5 OSC measuring head used in the rotational mode and programmed via a computer controlled Rheocontroller RC20 module. A D100/300 rotor assembly sensor system capable of operation under high temperature/high pressure conditions of aseptic processing was used for measurements. The rotor inside the sample compartment is operated by a magnetic clutch assembled to the measuring head. The system was calibrated to cancel the effect of this magnetic coupling before performing the measurements by using silicone oil standards and the conventional MV1 sensor. The lay-out of the sensor system as well

as the experimental set-up is shown in Figure 7.1. The sample is kept inside a jacketed chamber heated by circulating silicone oil using an immersion circulator (PolyScience Model 800; Cole Parmer, Chicago, IL). Temperature of the sample was monitored by a platinum resistance thermometer installed at the bottom of the chamber and connected to the rheocontroller.

Test samples were loaded into the test chamber and allowed to equilibrate to the required temperature (~10 min). Samples were sheared at a programmed rate increasing from 0 to 500 s^{-1} in 10 min, held steady at 500 s^{-1} for 20 min, followed by a linearly decreasing shear from 500 to 0 s^{-1} in 10 min. A full factorial design of experiment with five levels of starch (3, 4, 5 and 6% w/w) and five levels of temperature (60, 80, 100, 120 and 140°C) was employed with three replicates. Experimental data of starch were corrected for magnetic coupling effect using the calibration factor obtained with silicone oil standards and MV1 sensor.

Flow Curve Evaluation

The flow curves (rheograms) were evaluated by using the power law, the Herschel-Bulkley, Casson, and the Bingham linear rheological models as previously described in Chapter II. The combined effect of temperature and concentration on the consistency coefficient (m) and the flow behaviour index was evaluated by a modified Turian approach (Turian, 1964) through a multiple regression analysis. Time dependency was evaluated using the modified Weltmann (1943) logarithmic model.

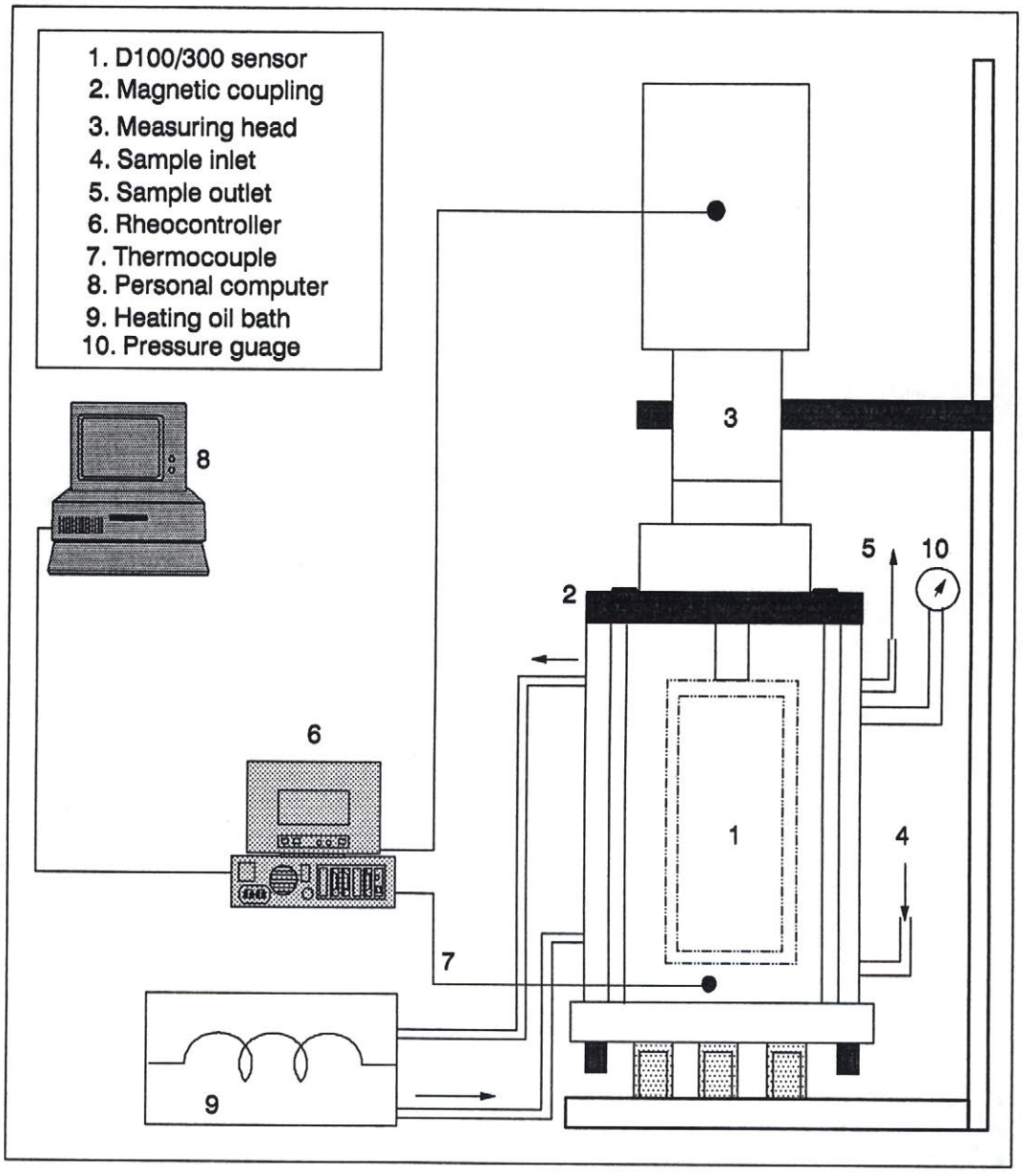

Figure 7.1. **Schematic diagram showing the experimental set-up for the high temperature/high pressure sensor system (D100/300).**

RESULTS AND DISCUSSION

Sensor Calibration

The D100/300 sensor system and the measuring head (M-5 OSC) of the viscometer are connected together by a magnetic coupling as explained earlier. Therefore the sensor has to be calibrated to cancel the effect of the magnetic clutch on the rheological data. Shear stress-shear data for D100/300 and MV1 sensors with silicone oil standards at 60°C are shown in Figure 7.2. Higher shear stress values were observed with the D100/300 sensor. This was corrected by correlating the shear stress data of the two senors and a correction factor of 10.33 was obtained for the three viscosity standards used. The corrected D100/300 data showed good agreement with the MV1 data (Figure 7.3) and the correction factor was used for correcting shear stress data of starch and CMC solutions (Chapter VIII).

Characterization of the Flow Curves

Typical flow curves for different concentrations (3, 4, 5 and 6% w/w) of Therm-flo® starch at 120 and 140°C under dynamic and steady shearing are presented in Figure 7.4. Higher concentrations of starch were associated with higher viscosities as indicated by the higher shear stress values under a given shear rate, while increased temperature resulted in decreased viscosities. Starch solutions showed only a negligible structural breakdown under a steady shear rate of 500 s^{-1} for 20 min (i.e. solutions were not strongly time-dependent). With 3% starch, there were some fluctuations with the shear stress-shear rate data, perhaps due to prevailing low viscosities.

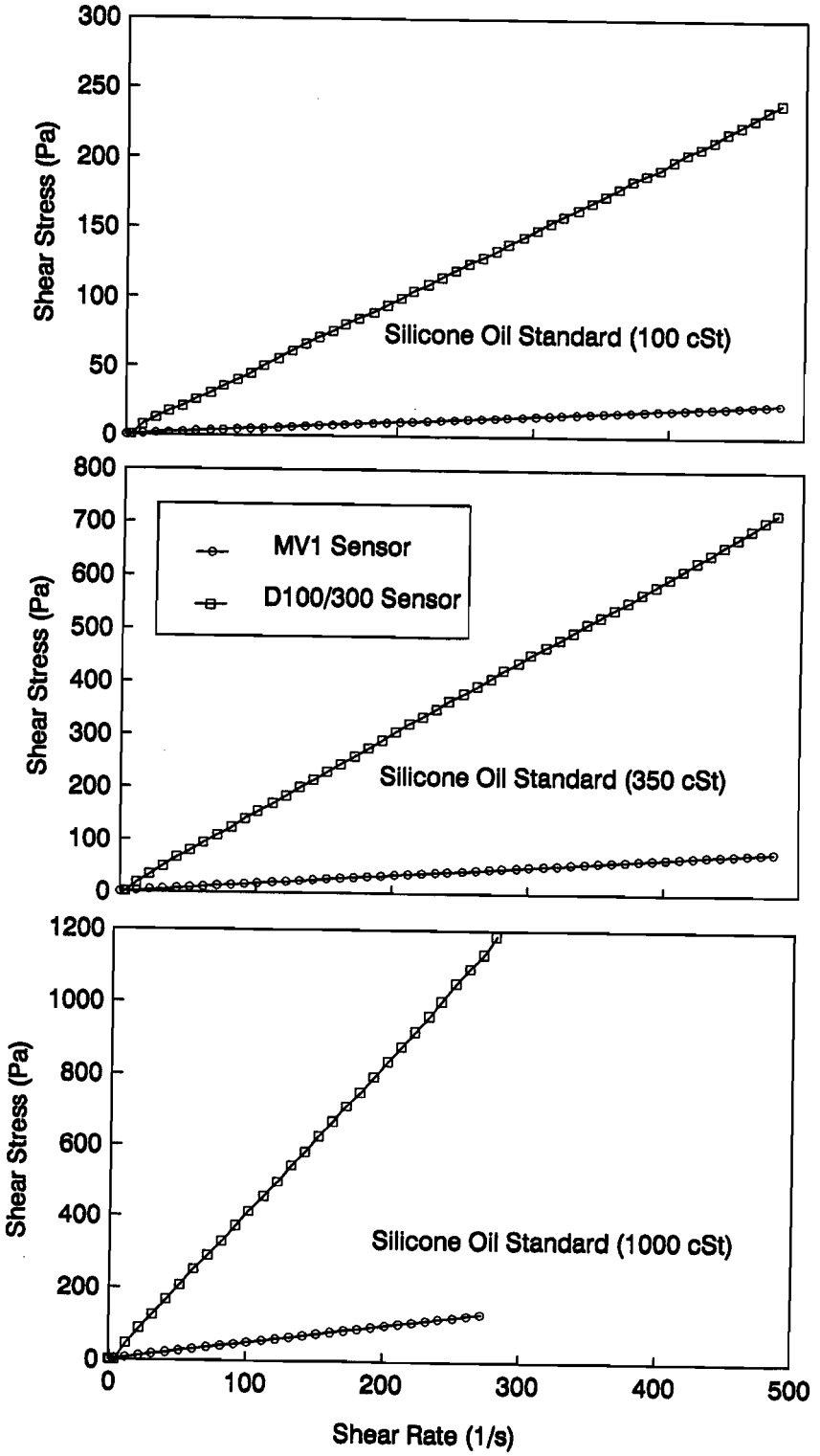

Figure 7.2. **Shear stress-shear rate data for silicone oil standards using D100/300 and MV1 sensor systems at 60°C under a dynamic shearing cycle.**

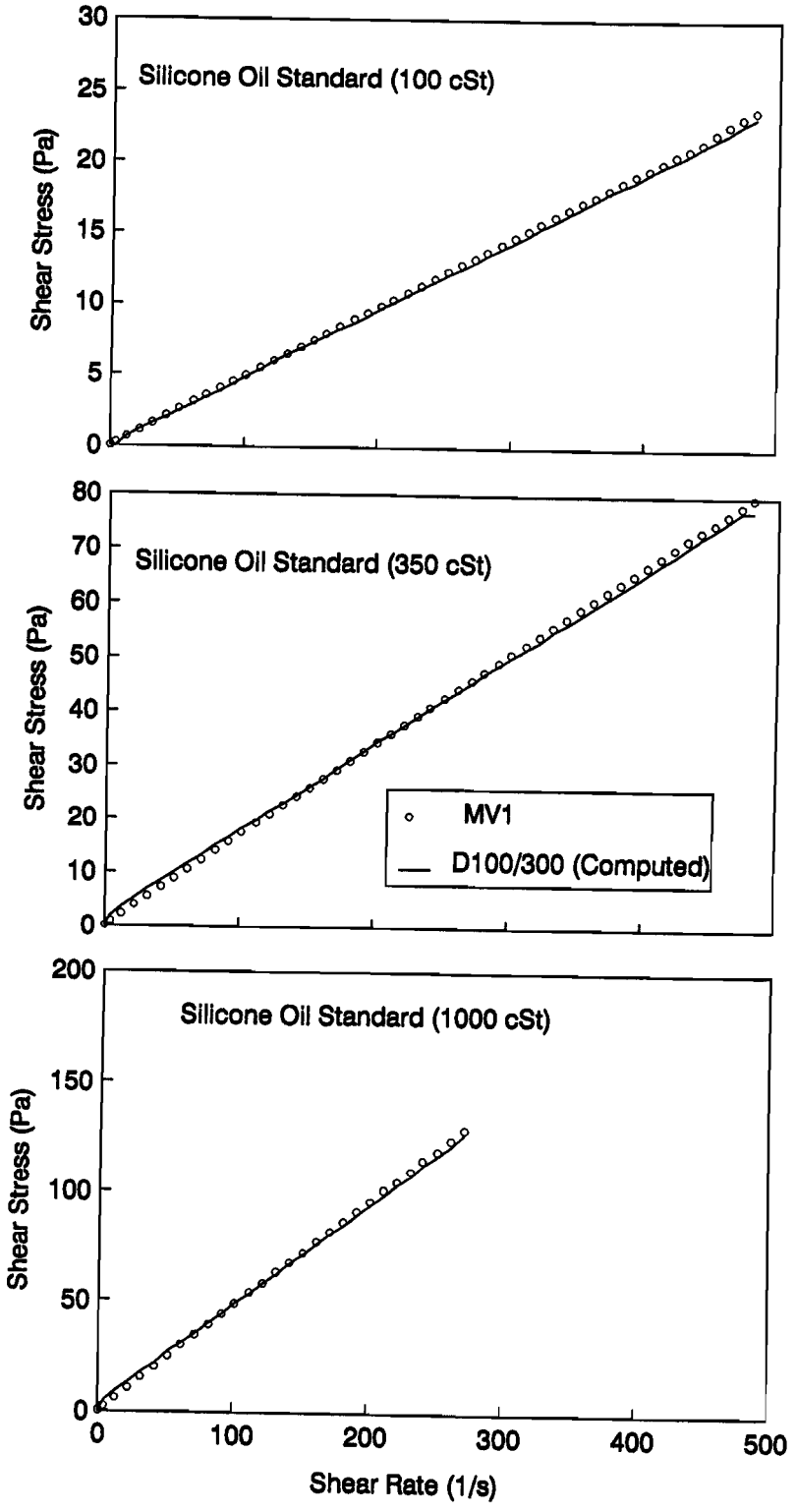

Figure 7.3. **Corrected Shear stress-shear rate data for the D100/300 system with silicone oil standards at 60°C under a dynamic shearing cycle.**

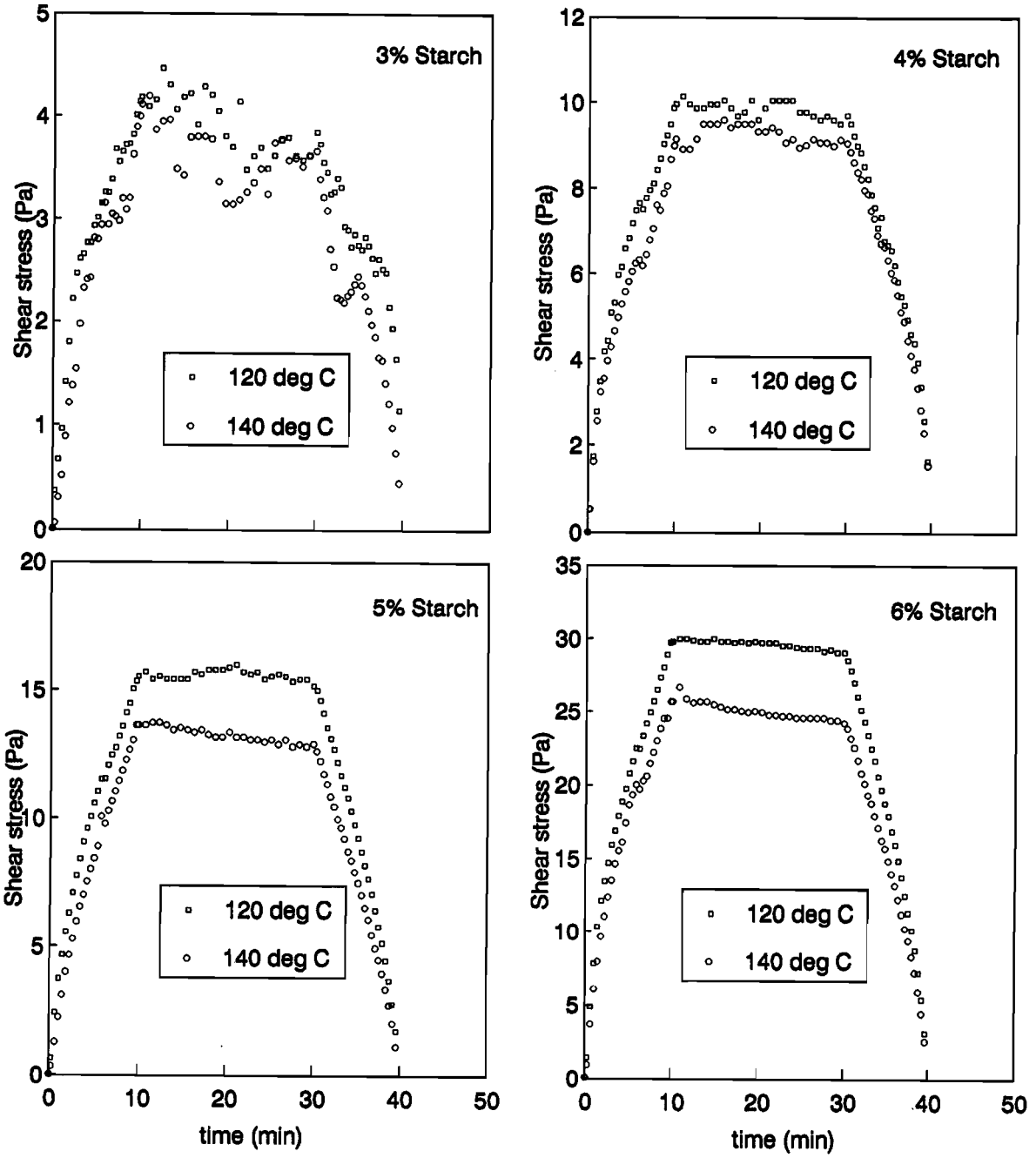

Figure 7.4. **Influence of concentration of shear stress-shear rate data of Therm-flo® starch at 120 and 140°C using the D100/300 sensor system.**

Rheological Models of Starch

The suitability of different rheological models was tested for fitting the shear stress/shear rate data of starch as detailed in Table 7.1. The Bingham model was suitable for the lower concentration of starch (3%) with determination coefficients (r^2) of 0.91 and 0.95 for the upward and downward flow curves, respectively. At high concentrations (4-6%), the power law model was dominant (r^2 = 0.99). Models such as Herschel-Bulkley and Casson also showed good fit over the data (r^2 = 1.00 for Herschel-Bulkley model) at the concentration of 6% (Table 7.1). However, throughout this study the power law model was used for the analysis because of its overall good fit of the data and its inherent compatibility as far as engineering calculations are concerned (Lalande *et al.*, 1991).

Effects of Temperature and Concentration

The mean and standard deviations of the power law parameters (m and n) of starch are presented in Table 7.2. Analysis of variance showed that m and n were significantly ($p < 0.001$) influenced by concentration, temperature and their interactions. Starch concentration effects were dominant. The same was true, to a lesser extent, with the flow behaviour index. The general trend was that: consistency coefficient (m) increased with concentration and decreased with temperature, while the effects were reversed with the flow behaviour index as indicated by the means plots (Figure 7.5).

Time-Dependent Flow Behaviour

Time-dependent or thixotropic behaviour of Therm-flo® starch was investigated

Table 7.1. Different rheological models parameters of the different Therm-flo® starch concentrations (3-6% w/w) at 120°C.

Conc. (%)	Model	Upward Curve			Downward Curve		
		Intercept	Slope	r^2	Intercept	Slope	r^2
3	Linear	0.1018	0.066	0.91	0.1576	0.041	0.95
	Power Law	0.2037	0.530	0.76	0.1812	0.527	0.75
	H. Bulkley	0.0349	1.185	0.77	0.0243	0.759	0.87
	Casson	0.0241	0.053	0.86	0.1041	0.015	0.89
4	Linear	3.6510	0.126	0.98	3.3793	0.120	0.99
	Power Law	0.2110	0.686	0.98	0.1467	0.664	0.97
	H. Bulkley	0.1176	0.722	0.96	0.1290	0.682	0.96
	Casson	2.1620	0.055	0.96	2.0500	0.051	0.97
5	Linear	4.9260	0.257	0.98	4.2690	0.257	0.98
	Power Law	0.2165	0.689	0.99	0.1429	0.728	0.99
	H. Bulkley	0.1970	0.826	0.88	0.1455	0.822	1.00
	Casson	2.6063	0.130	0.99	2.0810	0.140	1.00
6	Linear	9.0000	0.506	0.97	9.6700	0.443	0.99
	Power Law	0.7315	0.565	0.99	0.5390	0.650	0.99
	H. Bulkley	0.2398	0.784	0.95	0.4982	0.731	1.00
	Casson	4.4180	0.274	0.99	5.4340	0.209	1.00

Table 7.2. The rheological parameters of Therm-flo® starch as affected by concentration and temperature using the power law model[1].

Conc. (%)	Temperature (°C)	Upward Curve		Downward Curve	
		m^2	n^2	m	n
3	60	0.511 ± 0.033	0.411 ± 0.003	0.475 ± 0.010	0.384 ± 0.020
3	80	0.421 ± 0.011	0.420 ± 0.010	0.311 ± 0.043	0.451 ± 0.024
3	100	0.293 ± 0.037	0.467 ± 0.007	0.224 ± 0.017	0.497 ± 0.045
3	120	0.204 ± 0.011	0.530 ± 0.023	0.181 ± 0.005	0.527 ± 0.021
3	140	0.151 ± 0.033	0.550 ± 0.029	0.150 ± 0.022	0.601 ± 0.010
4	60	0.464 ± 0.030	0.480 ± 0.015	0.411 ± 0.030	0.498 ± 0.010
4	80	0.398 ± 0.032	0.495 ± 0.015	0.287 ± 0.016	0.573 ± 0.028
4	100	0.279 ± 0.052	0.609 ± 0.063	0.201 ± 0.022	0.593 ± 0.011
4	120	0.211 ± 0.027	0.686 ± 0.029	0.147 ± 0.025	0.664 ± 0.108
4	140	0.182 ± 0.005	0.692 ± 0.068	0.093 ± 0.014	0.836 ± 0.018
5	60	0.694 ± 0.028	0.647 ± 0.018	0.498 ± 0.048	0.695 ± 0.017
5	80	0.489 ± 0.025	0.648 ± 0.006	0.327 ± 0.028	0.707 ± 0.011
5	100	0.356 ± 0.028	0.672 ± 0.021	0.221 ± 0.017	0.719 ± 0.014
5	120	0.217 ± 0.032	0.689 ± 0.011	0.143 ± 0.021	0.728 ± 0.043
5	140	0.193 ± 0.029	0.697 ± 0.027	0.116 ± 0.020	0.750 ± 0.026
6	60	1.154 ± 0.081	0.527 ± 0.004	0.898 ± 0.054	0.639 ± 0.018
6	80	0.897 ± 0.025	0.608 ± 0.004	0.721 ± 0.033	0.647 ± 0.010
6	100	0.790 ± 0.013	0.594 ± 0.008	0.646 ± 0.020	0.648 ± 0.010
6	120	0.732 ± 0.010	0.565 ± 0.011	0.539 ± 0.038	0.650 ± 0.015
6	140	0.678 ± 0.019	0.614 ±0.006	0.432 ± 0.023	0.662 ± 0.007

[1]$\sigma = m\,\gamma^n$

[2]Results are means of triplicates.

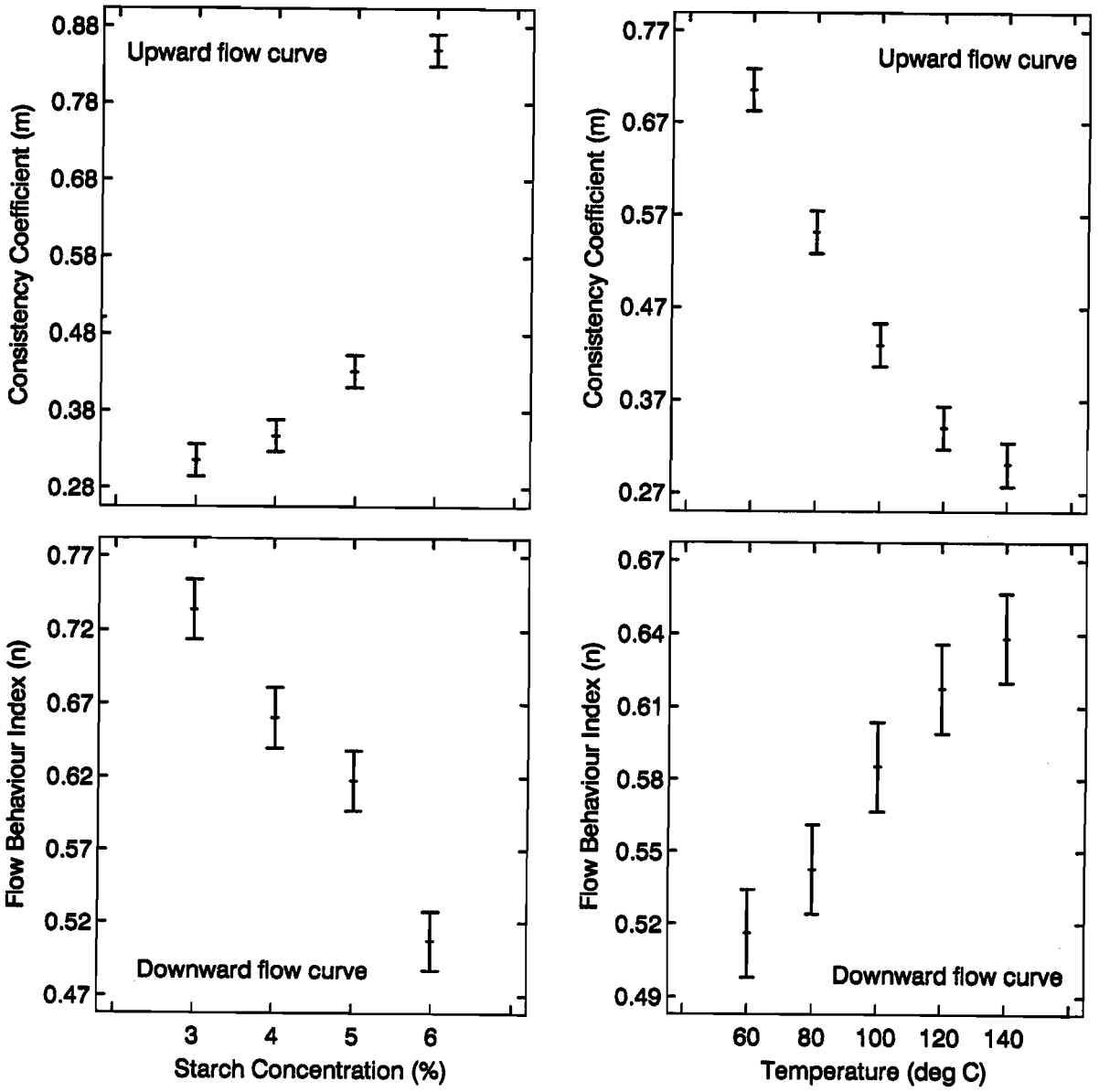

Figure 7.5. **Means plots for consistency coefficient (m) and flow behaviour index (n) of the starch solutions for upward and downward flow curves as influenced by concentration and temperature.**

using Weltmann (1943) model. Structural breakdown was minor (Figure 7.2). Melting and swelling of starch was probably complete at 140°C, and obvious structural build-up or loss was not observed. Time-dependent flow behaviour is usually associated with partially gelatinized starches, due to the on-going melting and swelling of the starch granules as a result of the exposure to higher temperatures (Donovan *et al.*, 1983; Biliaderis, 1991).

A Combined Model For Starch Rheology

From a practical stand-point, it will be very useful to describe the effect of temperature and concentration by only one equation. In literature, the Arrhenius model was frequently used to model effects of temperature, while the effect of concentration was described by either an exponential or a power relationship (Fichtali *et al.*, 1993). However, Arrhenius was not suitable for Therm-flo® starch especially at lower starch concentrations. The alternative was a modified Turian approach taking into account the effects of both temperature and concentration on consistency coefficient and flow behaviour index. Both m and n were related to temperature and concentration by multiple regression analysis. The following predictive equations involving various temperature and concentration functions were introduced for the upward flow curve (R^2 =0.91 and 0.76 for $\log_{10}$ (m) and n, respectively):

$$\log(m) = -3.50 + C\left[0.39 + 0.0013T + \frac{2.01}{T}\right] + \frac{7.16}{C} - 0.01T \quad \textbf{(48)}$$

$$n = 1.83 - C\left[0.091 + 0.001T + \frac{0.86}{T}\right] - \frac{3.86}{C} + 0.004T \quad \textbf{(49)}$$

Similar predictive equations were developed for the downward flow curve (R^2 were 0.91 and 0.79 for $\log_{10}$ (m) and n, respectively):

$$\log(m) = -5.06 + C\left[0.57 + 0.001T + \frac{1.91}{T}\right] + \frac{10.1}{C} - 0.01T \quad \textbf{(50)}$$

$$n = 1.67 - C\left[0.063 + 0.001T\right] - \frac{4.00}{C} + 0.006T \quad \textbf{(51)}$$

Plots for the observed *vs* predicted m and n are presented in Figure 7.6. The model will be useful as rheological data at aseptic processing conditions is very limited.

CONCLUSIONS

The use of magnetic coupling with D100/300 sensor system allowed adequate evaluation of starch rheology at high temperatures. The power law model showed an overall good fit over starch flow curves. The rheological properties were temperature and concentration sensitive. These effects need to be taken into consideration using Therm-flo starch as a carrier fluid for particulate foods while designing continuous aseptic processing.

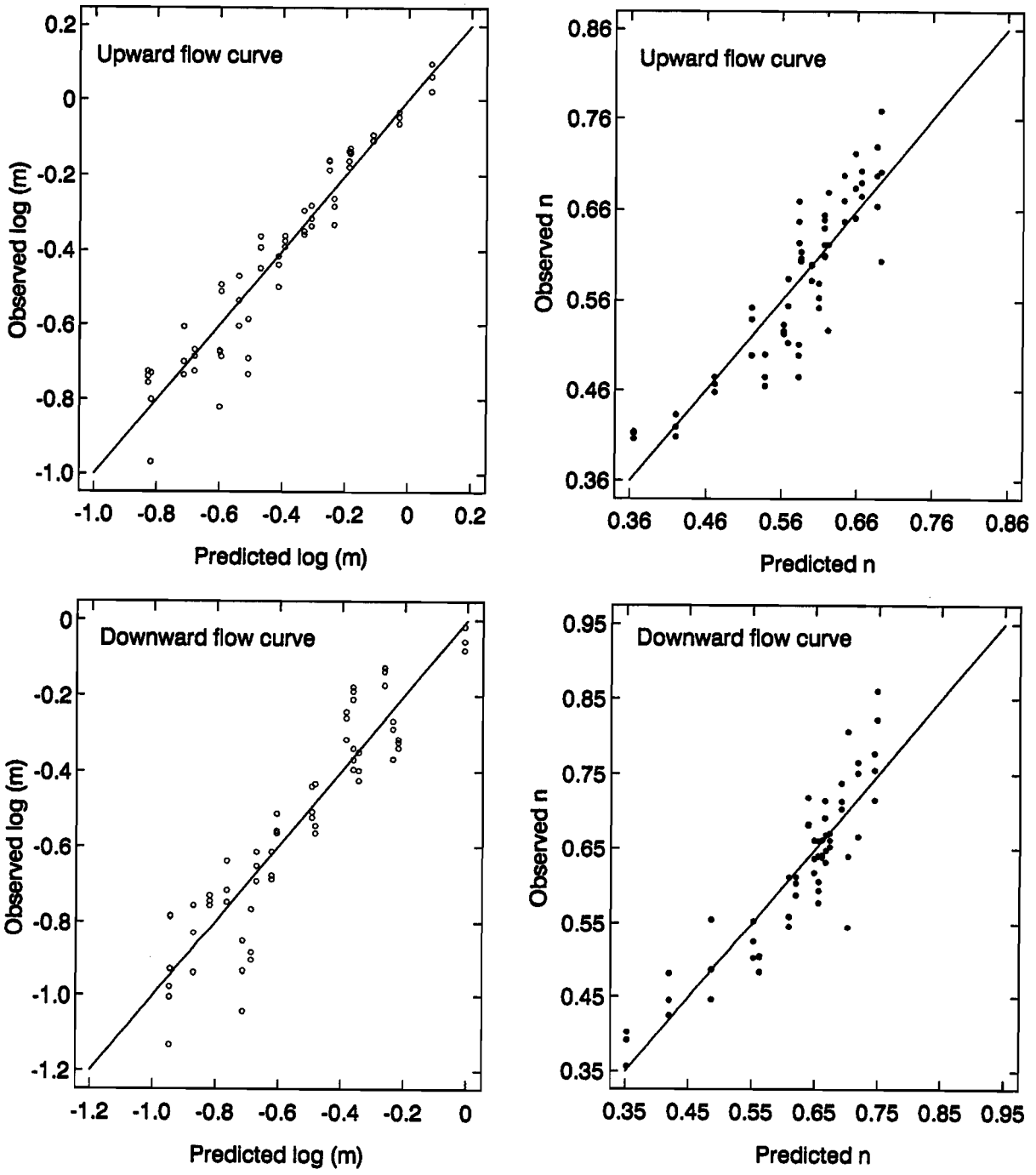

Figure 7.6. **Plots for experimental *vs* calculated m and n of the starch solutions for the upward and downward flow curves.**

CHAPTER VIII

RHEOLOGY OF CARBOXYMETHYL CELLULOSE (CMC)

ABSTRACT

Rheological properties of 0.5 to 2.0% carboxymethyl cellulose (CMC) solutions were evaluated using a computer controlled rotational viscometer under a dynamic upward and downward linear-ramp shearing sequence. Experiments were performed in the temperature range 60-140°C with a high temperature/high pressure sensor system. Rheological properties were significantly ($p < 0.01$) influenced by both temperature and concentration. Rheological parameters are evaluated using different models (power law, Herschel-Bulkley, Casson and linear), while the temperature and time dependency of these parameters were evaluated by fitting the Turian and the Weltmann models, respectively.

INTRODUCTION

Designing of food processing operations (mixing, pumping, heating, cooling) requires accurate data on rheological properties. Flow characteristics of pumpable food products are dependent on the fluid viscosity and density. Calculation of thermal treatment times for aseptic processing of liquid foods containing particulates is complex because the residence time distribution (RTD) of particulates and carrier fluid are influenced by concentration and type of the carrier fluid, size, shape and density of particles and interactions between particles and fluid as well as other process parameters (time, temperature, pressure, and the system configuration). Data on rheological characteristics,

relative velocity between fluid and particles, and fluid to particle heat transfer coefficients are needed for optimizing the heat exchanger and holding tube designs in aseptic processing of liquid foods containing particulates (Dail and Steffe, 1990a,b).

Carboxymethyl cellulose (CMC) has been widely used as a pseudoplastic carrier fluid for low acid particulate foods in aseptic processing simulations (McCoy *et al.*, 1987; Alhamdan and Sastry, 1990; Dutta and Sastry, 1990a, b; Lee and Singh, 1991; Awuah *et al.*, 1993), but there is little data on its rheological properties and most experimental data on RTD using CMC as carrier fluid have been reported at temperatures below 100°C (Castell-Perez and Steffe, 1990; Abdelrahim *et al.*, 1994). By carrying out experiments at 30°C before and after processing, Rao *et al.* (1981b) were able to show that thermal processing has detrimental effects on the structure of CMC.

In aseptic processing operations, the carrier fluid will be subjected to different shear rates in different sections, e.g. low shear rates in straight holding tube and high shear rates while passing through pumps and scraped surface heat exchangers. The apparent viscosity in continuous flow situations for most engineering applications is generally computed using Eqn 52 as described by McCabe *et al.* (1985):

$$\eta_{ap} = \frac{2^{(n-3)}\, m \left[\frac{3n-1}{4n}\right]^{n}}{u_f^{(1-n)}\, D^{(n-1)}} \qquad (52)$$

The objective of this work was to study the rheology of aqueous CMC as influenced by concentration (0.5-2.0%) in the temperature range 60-140°C; as well as its shear thinning time dependent behaviour (thixotropy) at the aseptic processing conditions.

MATERIALS AND METHODS

Four completely dissolved concentrations (0.5, 1.0, 1.5, and 2.0% w/w) of commercial high viscosity CMC sodium salt (0.65-0.85 degree of substitution and 2×10^5 molecular weight; Sigma Chemical Co., St. Louis, Mo) were prepared by hydrating in distilled water overnight followed by vigorous hand mixing and standing for 24 h to release air bubbles. Test samples were then gently and carefully mixed before measurements to avoid the air entrapment.

Rheological measurements (shear rate-shear stress data) were made using a rotational viscometer (Haake Model RV20; Haake Mess-Technik, Karlsruhe, Germany), equipped with an M-5 OSC measuring head and a D100/300 sensor system assembly interfaced to a microcomputer for control and data acquisition. The D100/300 rotor assembly sensor system is capable of operation under high temperature/high pressure conditions of aseptic processing (as described in Chapter VII).

Experimental Procedure

Test samples of CMC were filled into the sample cup and loaded on to the jacketed chamber through a cylindrical spindle. The sample was allowed to rest in the test chamber for 20 min to equilibrate to the desired temperature preset on the circulating bath. Test samples were treated similar to starch solutions (Chapter VII). Temperature effects at different concentrations were evaluated in the range 60-140°C (20°C intervals) with three replicates. The resulting flow curves (rheograms) were evaluated using the power law, Herschel-Bulkley, Casson and the linear models described in Chapter II.

Effect of Temperature

Turian (1964) approach was also employed to evaluate the temperature sensitivity of the rheological parameters, m and n as described in Chapter II. A combined model for CMC solution was developed for $\log_{10}$ (m) and n with temperature and concentration using multiple regression analyses based on Turian approach (Chapter II).

Thixotropic behaviour of CMC was also evaluated using a modified Weltmann (1943) logarithmic model (Eqn 36, Chapter II).

RESULTS AND DISCUSSION

Characterization of the Flow Curves

Typical flow curves for the different concentrations of CMC (1.0, 1.5 and 2.0% w/w) at high temperatures (120 and 140°C) are presented in Figure 8.1 (A, B, C). High concentrations of CMC were associated with high viscosities as indicated by the higher shear stress values at a given shear rate, while increasing the temperature from 120 to 140°C decreased the shear stresses (lower viscosities). Unlike with starch solutions, structural breakdown was observed with CMC solutions under all temperatures, when they were constantly sheared at 500 s^{-1} for 20 min (Figure 8.1)

Rheological Models of CMC

The suitability of different rheological models previously defined was tested for fitting the shear stress/shear data of CMC. The power law model gave a better fit especially at lower temperatures (60 and 80°C) for all CMC concentrations. The Bingham

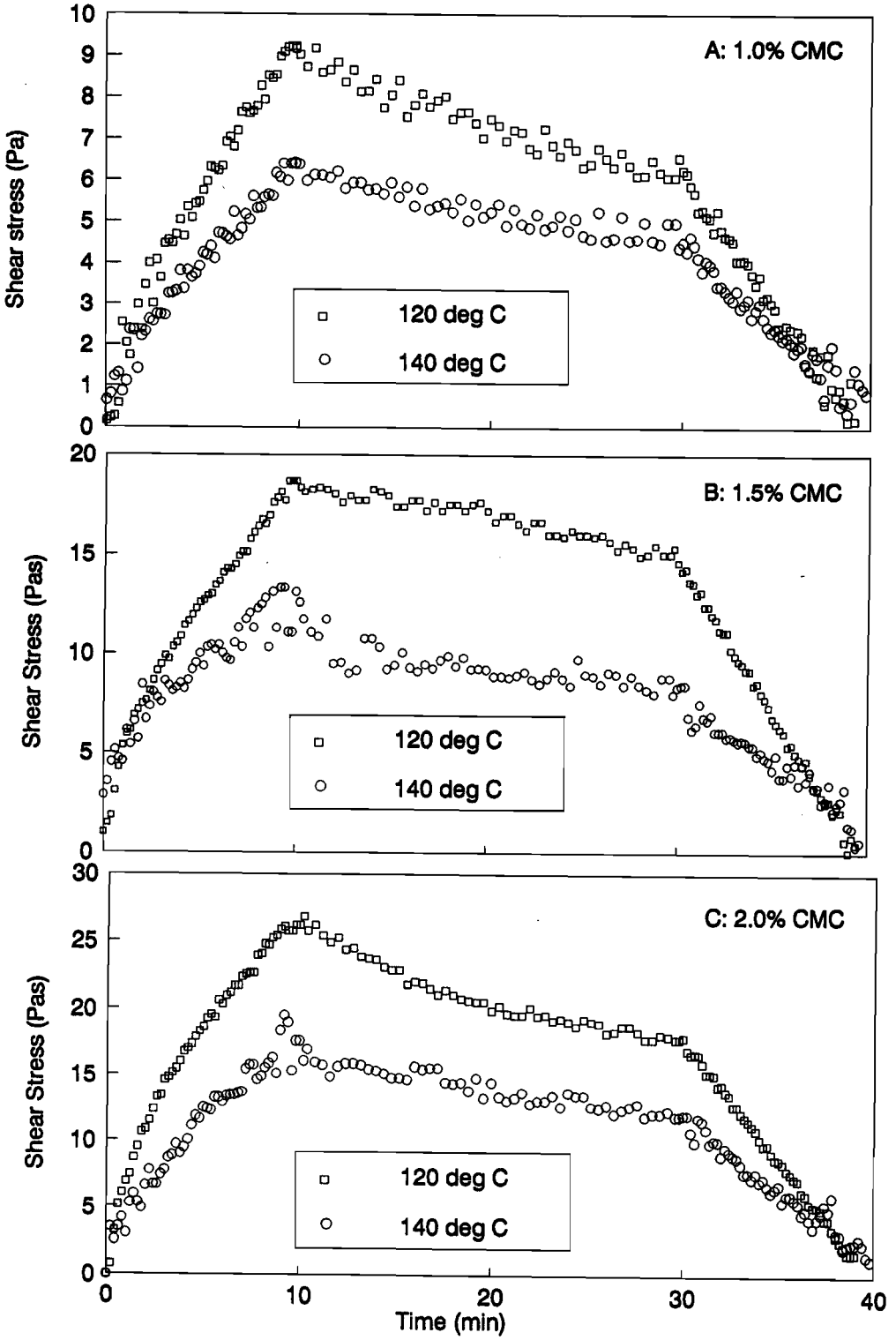

Figure 8.1. **Shear stress-shear rate curves for CMC solutions during a programmed 40 min run under dynamic and constant shearing (A: 1.0%, B: 1.5%, C: 2.0% w/w) using the D100/300 sensor system.**

linear model showed the best fit at temperatures exceeding 100°C and at the lower concentrations (0.5 and 1.0%) particularly for the downward flow curve. However, the power law model was chosen for the subsequent analysis and discussion of the data in this study because of its overall good fit and its inherent compatibility for engineering calculations (Lalande *et al.*, 1991).

Effect of Temperature and Concentration

The mean values and standard deviations of m and n for various concentrations of CMC at different temperatures are presented in Table 8.1. Analysis of variance showed that m and n of CMC were significantly influenced ($p < 0.001$) by temperature, concentration and their interaction. In this case temperature was the major contributor to the variability of m for both the upward and downward flow curves. The differences between the two flow curves may be attributed to the differences in the shear history for the downward flow curve of CMC samples following the steady shear at 500 s^{-1} for 20 min. The general trend, however, was that the consistency coefficient (m) increased with concentration and decreased with temperature, while the opposite trend was observed with the flow behaviour index (n).

Time Dependency (thixotropy)

Thixotropic behaviour of CMC solutions was examined using the modified Weltmann logarithmic model as presented in Figure 8.2 for 1.0, 1.5 and 2.0% w/w at 100, 120 and 140°C. The Weltmann A value (A_w), which is the measure of initial resistance

Table 8.1. **The power law model parameters of CMC solutions as influenced by concentration and temperature determined using the D100/300 sensor system.**

		Upward Flow Curve		Downward Flow Curve	
Conc. (%)	Temp. (°C)	m[1, 2]	n[1, 2]	m	n
0.5	60	0.252 ± 0.033	0.681 ± 0.026	0.073 ± 0.002	0.904 ± 0.028
0.5	80	0.170 ± 0.010	0.802 ± 0.043	0.062 ± 0.001	0.982 ± 0.026
0.5	100	0.084 ± 0.012	0.874 ± 0.029	0.029 ± 0.005	1.041 ± 0.059
0.5	120	0.054 ± 0.007	1.036 ± 0.029	0.012 ± 0.001	1.043 ± 0.043
0.5	140	0.024 ± 0.007	1.215 ± 0.076	0.006 ± 0.003	1.168 ± 0.044
1.0	60	1.191 ± 0.101	0.560 ± 0.029	1.032 ± 0.077	0.560 ± 0.018
1.0	80	0.364 ± 0.049	0.673 ± 0.030	0.320 ± 0.041	0.681 ± 0.021
1.0	100	0.129 ± 0.032	0.924 ± 0.067	0.078 ± 0.060	0.820 ± 0.009
1.0	120	0.105 ± 0.022	0.991 ± 0.005	0.046 ± 0.002	1.159 ± 0.043
1.0	140	0.044 ± 0.007	1.010 ± 0.002	0.045 ± 0.004	1.189 ± 0.056
1.5	60	3.559 ± 0.123	0.481 ± 0.028	2.581 ± 0.111	0.564 ± 0.012
1.5	80	1.443 ± 0.053	0.565 ± 0.002	1.336 ± 0.144	0.653 ± 0.015
1.5	100	1.045 ± 0.045	0.750 ± 0.014	0.635 ± 0.049	0.779 ± 0.010
1.5	120	0.619 ± 0.025	0.787 ± 0.025	0.234 ± 0.018	1.020 ± 0.023
1.5	140	0.255 ± 0.019	0.867 ± 0.054	0.130 ± 0.007	1.062 ± 0.075
2.0	60	15.55 ± 0.554	0.412 ± 0.032	13.65 ± 0.781	0.445 ± 0.016
2.0	80	6.767 ± 0.354	0.498 ± 0.016	6.784 ± 1.212	0.553 ± 0.018
2.0	100	1.990 ± 0.236	0.607 ± 0.013	1.948 ± 0.141	0.686 ± 0.018
2.0	120	1.134 ± 0.081	0.695 ± 0.018	0.878 ± 0.101	0.861 ± 0.045
2.0	140	0.642 ± 0.068	0.744 ± 0.018	0.490 ± 0.025	0.882 ± 0.015

[1] $\sigma = m \gamma^n$

[2] Results are means ± standard deviations of triplicates.

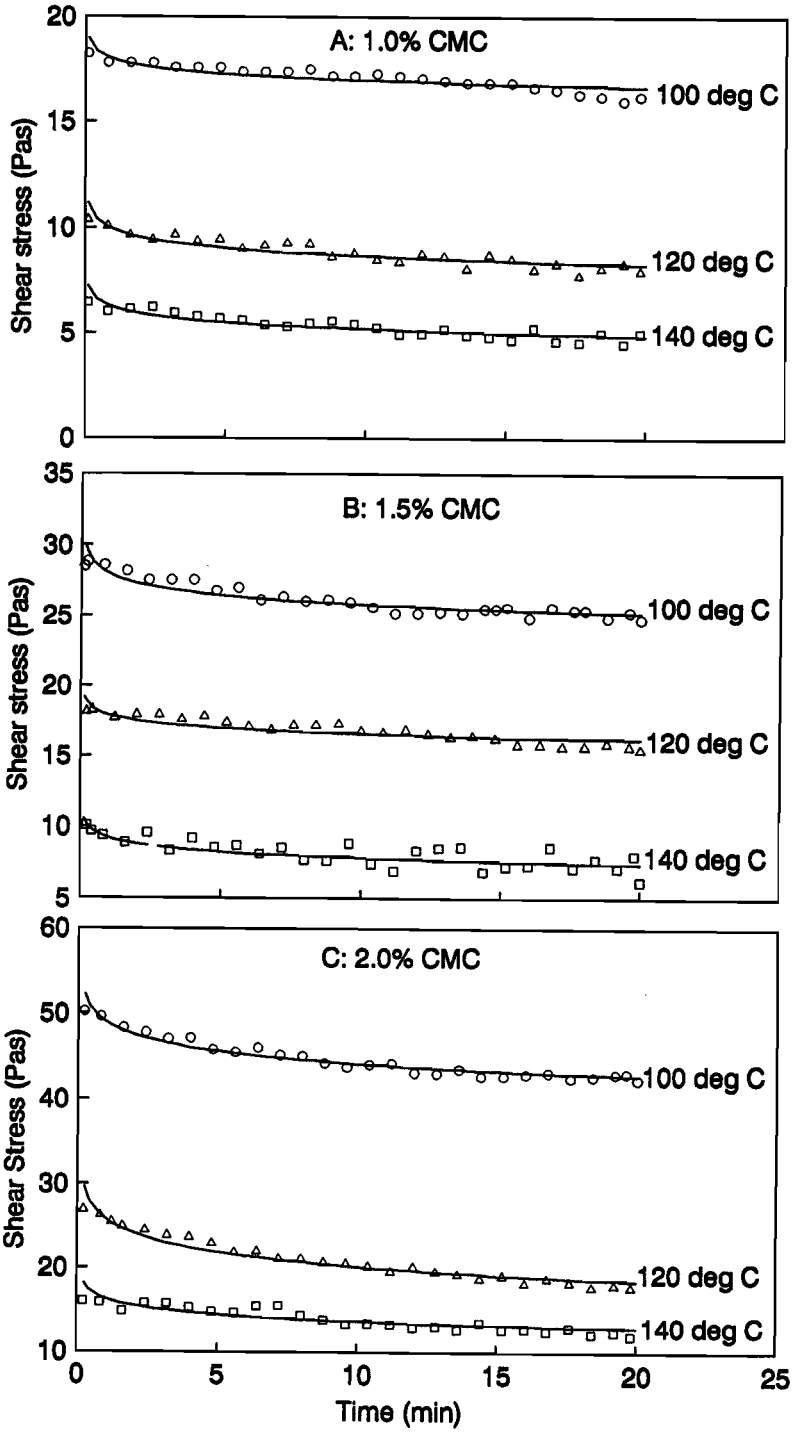

Figure 8.2. **Time dependency plots for 1.0-2.0% CMC solutions during a programmed 20 min constant shearing (500 s^{-1}) at 100, 120 and 140°C (A: 1.0%, B: 1.5%, C: 2.0%).**

to shearing force was significantly affected by concentration, temperature and their interaction ($p < 0.001$). A_w value increased with concentration and decreased with temperature which has a more dominant effect than concentration. The susceptibility of CMC solutions to structural breakdown with shearing time was measured by the Weltmann B value (B_w coefficient of thixotropic structural loss). B_w value was significantly ($p < 0.001$) influenced by concentration and temperature and their interactions. B_w value also increased with concentration and decreased with temperature, but the effect of concentration was more dominant. Multiple regression equations for both A_w and B_w values are given below:

$$A_w = -29.77 + 101.42C + T[0.216 - 0.703C] \quad (R^2 = 0.88) \qquad \textbf{(53)}$$

$$B_w = 1.191 + 1.139C + T[0.005 - 0.014C] \quad (R^2 = 0.96) \qquad \textbf{(54)}$$

Both A_w and B_w increased with concentration and decreased with temperature, but the effects of concentration and temperature were more pronounced with A_w as presented in response surface plots (Figure 8.3).

Regression Coefficients for CMC Rheological Parameters

A modified Turian model was used to accommodate the effects of concentration and temperature on CMC rheological parameters using multiple regression analyses. The predictive equations for $\log_{10}$ (m) and n employing various functions of temperature and concentration are given below for the upward flow curve (R^2 was 0.97 and 0.94 for $\log_{10}$ (m) and n; respectively):

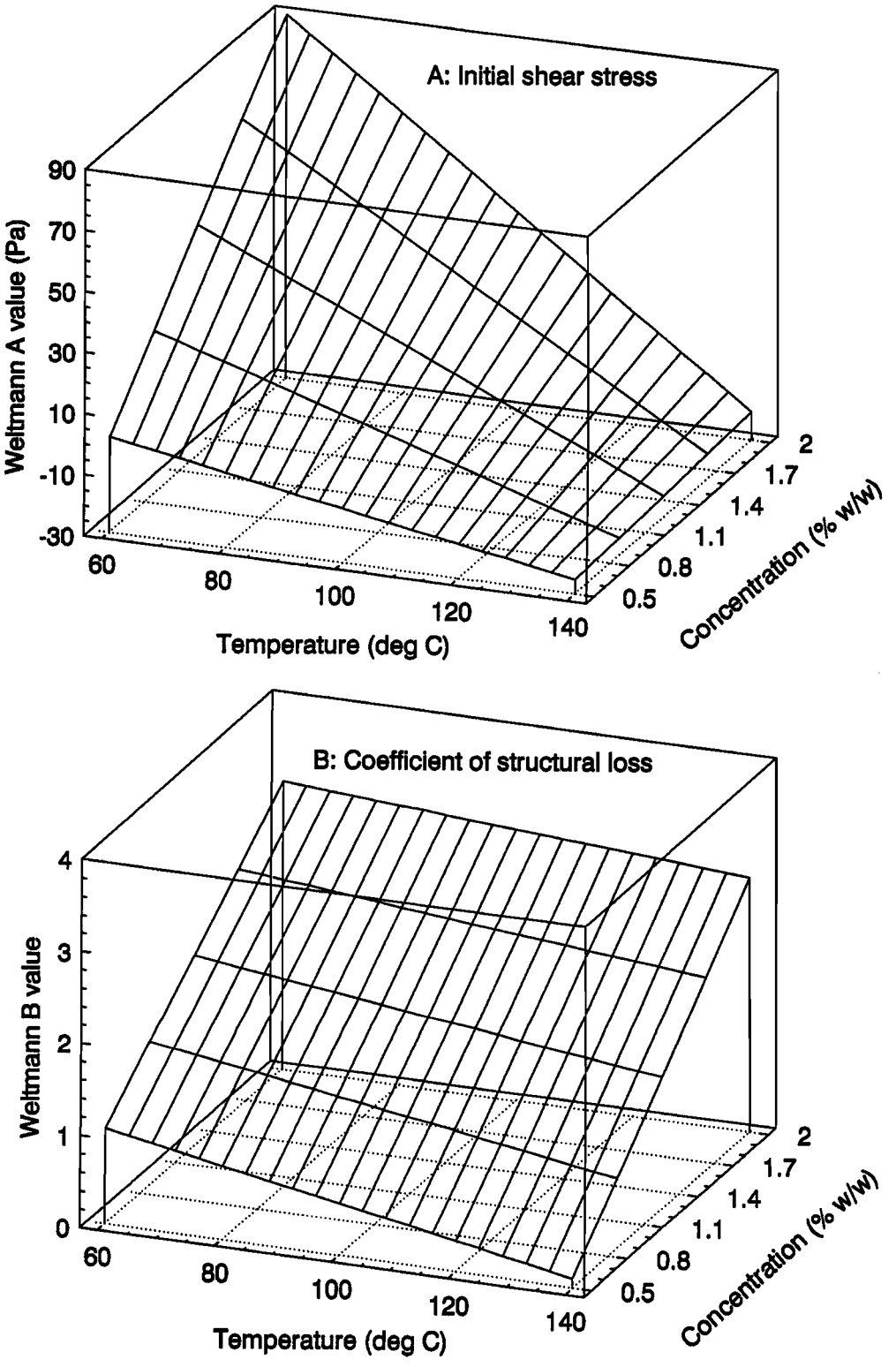

Figure 8.3. **Multiple regression generated response surface plots for the A and B values of the Weltmann model for CMC solutions sheared at 500 s^{-1}.**

$$\log_{10}(m) = -0.66 - 0.013T + \frac{0.159}{C} + 0.005TC + \frac{59.5C}{T} \quad (55)$$

$$n = 0.329 + 0.007T - 0.002TC - \frac{4.088C}{T} \quad (56)$$

and similarly for the downward flow curves (R^2 was 0.97 and 0.87 for $\log_{10}$ (m) and n; respectively):

$$\log_{10}(m) = -0.195 - 0.018T - \frac{0.133}{C} + 0.006TC + \frac{54.5C}{T} \quad (57)$$

$$n = 0.647 + 0.005T - 0.001TC - \frac{14.32C}{T} \quad (58)$$

The pooled data for experimental m and n *vs* those predicted by the above regression relationships are presented in Figure 8.4 show an overall good distribution of data points along the perfect diagonal line.

CONCLUSIONS

The flow behaviour of CMC can be adequately described by the power law model using high temperature viscometry. The physical isolation of rotor and sensor (D100/300) assembly with the coupling achieved through a magnetic clutch permits evaluating the rheological properties at the temperature conditions of aseptic processing. Both m and n of CMC were sensitive to changes in concentration and temperature. A modified Turian approach was used to describe the combined influence of temperature and concentration. The Weltmann model adequately described the CMC time dependency.

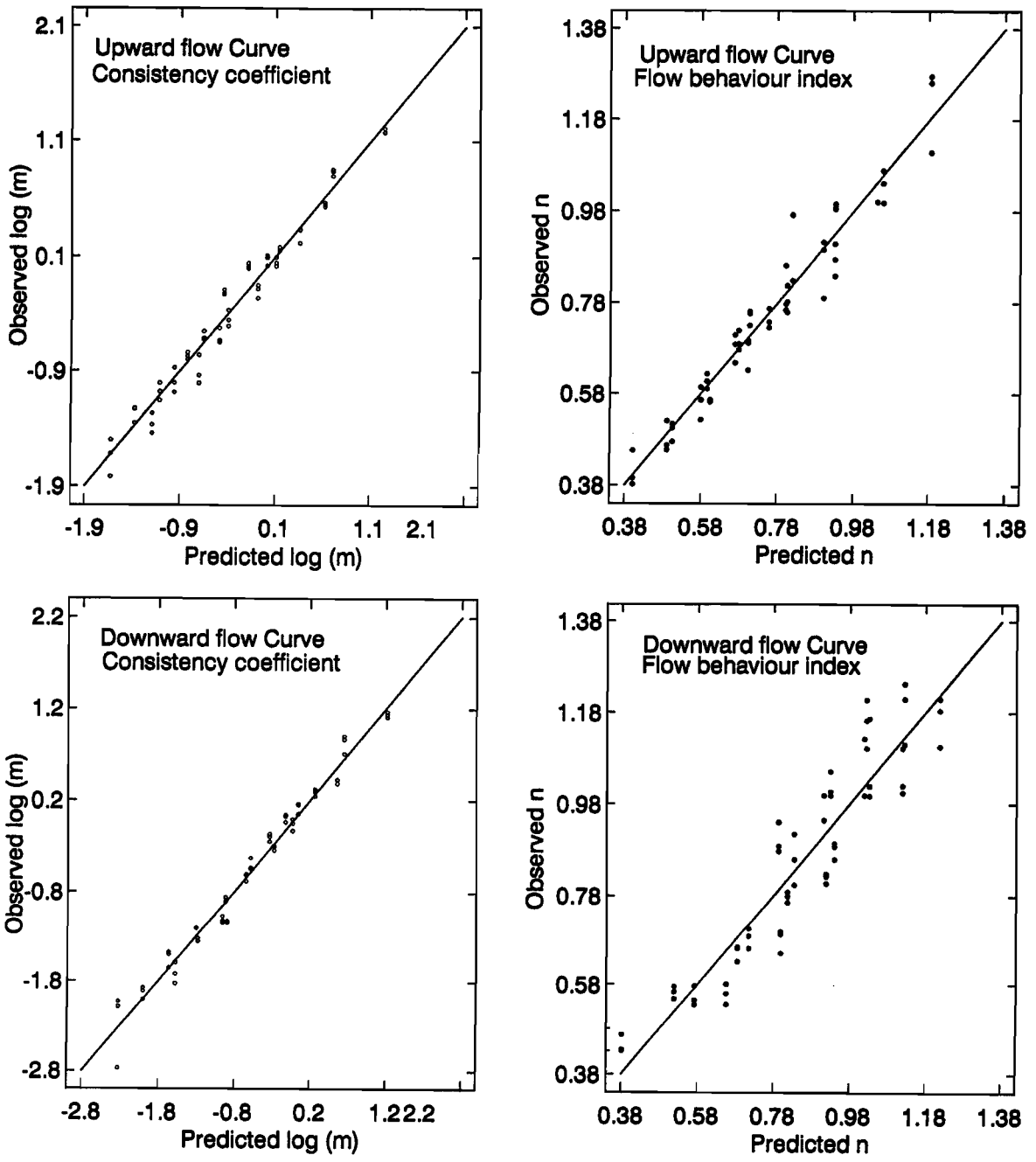

Figure 8.4. **Experimental *vs* multiple regression generated data for consistency coefficient (m) and flow behaviour index (n) of CMC solutions under the influence of concentration and temperature for the upward and downward flow curves.**

aseptic processing of low acid liquid foods containing particulates (Subramaniam and Zuritz, 1990; Subramaniam *et al.*, 1991; McKay *et al.*, 1992; Sandeep and Zuritz, 1993). From safety stand-point, the fastest particle residence in aseptic systems is required for process calculations. Dutta and Sastry (1990a, b) showed that the relative velocity of particles was influenced by particle Froude number and dimensionless viscosity. Particle density was also an important parameter in two-phase flow (Yang and Swartzel, 1992). Recently, there has been a growing research interests in residence time distribution analysis of food particles in SSHEs (Alcairo and Zuritz, 1990; Lee and Singh, 1991, 1993) and holding tube (Zhang *et al.*, 1992; Ramaswamy *et al.*, 1992; Grabowski and Ramaswamy, 1994). Richardson and Holdsworth (1989) indicated the existence of a critical velocity (u_c) which ensures a uniform suspension and flow in two-phase systems. They also showed that there was a linear relationship between the mean velocity of the particles and that of the solid/liquid mixture. Very little information is available on using dimensionless correlation required to understand the generalized flow behaviour of food particles in different aseptic systems under various experimental conditions.

The objectives of this study was to develop dimensionless correlations to describe the flow behaviour of food particles (meat and carrot cubes) in the SSHE, holding tube and the whole assembly of a pilot scale aseptic processing system.

THEORETICAL BACKGROUND

When a particle (sphere) is dropped into a viscous fluid, it experiences a change of momentum equal to the sum of imposed forces: gravitational, buoyancy, drag and fluid

inertia forces (Govier and Aziz, 1972; Denn, 1980; Smoldyrev, 1982). Thus the relative velocity of the particle is dependent on the following (Eqn 59):

$$Fr_p,\ Re_p,\ \frac{u_p}{u_f} = \phi\ (\ GRe_f,\ Fr_f,\ a,\ Ar_{f,p},\ \frac{d_e}{D}\ ) \qquad (59)$$

where ϕ represents a function of the various dimensionless numbers defined below:

$$GRe_p = \frac{u_p\, d_e\, \rho_f}{\eta_{ap}}\ , \qquad GRe_f = \frac{u_f\, D\, \rho_f}{\eta_{ap}} \qquad (60)$$

$$Fr_p = \frac{u_p^2}{g\, d_e}\ , \qquad Fr_f = \frac{u_f^2}{g\, D} \qquad (61)$$

$$a = \left| \frac{\rho_p}{\rho_f} - 1 \right| \qquad (62)$$

$$Ar_p = \frac{a\, g\, d_e^3\, \rho_f^2}{\eta_{ap}^2}\ , \qquad Ar_f = \frac{a\, g\, D^3\, \rho_f^2}{\eta_{ap}^2} \qquad (63)$$

In the above defined dimensionless numbers, due to the non-Newtonian character of the starch solutions (power law fluids), the viscosity term is replaced by apparent viscosity defined by MaCabe *et al.* (1985) is used (Eqn 52; Chapter VIII).

MATERIALS AND METHODS

Materials and Experimental System

Food particles (meat and carrot cubes) and starch solutions were prepared as described and detailed in Chapter III. Experimental set-up for obtaining RTD data for

meat and carrot cubes in the SSHE, holding tube and the whole aseptic system were as described in Chapters III, IV and V, respectively.

Regression Analysis

Experimental data for the fastest particle residence time (FPRT) was used to calculate the dimensionless numbers (GRe, Fr, Ar, u_p/u_f, a, d_e/D) for carrier fluid, as well as meat and carrot cubes in both the scraped surface heat exchanger (SSHE) and the holding tube where applicable. The residence time data was converted to velocity of the fastest moving particle by dividing the path length by FPRT for calculation of dimensionless numbers. The above dimensionless numbers were chosen to represent the major parameters influencing residence time distribution of food particles in aseptic processing systems: particles size (included in d_e/D), density of the particle and carrier fluid (included in Ar), viscosity of the carrier fluid (in GRe, Ar), flow rate and velocity profiles of the particle and carrier fluid (in Fr, GRe).

RTD data for the fastest particle in the holding tube were also obtained from test runs with the whole aseptic processing system was obtained by subtracting the residence times spent in the SSHEs as discussed in Chapter V. The ranges of the parameters related to the carrier fluid/particles and experimental system used are summarized in Table 9.1 and 9.2, respectively. All necessary rheological data were obtained using Haake rotational viscometer as described in Chapter VII. Step-wise multiple regression analysis was performed using the different dimensionless correlations with particle relative velocity (u_p/u_f), or Reynolds (GRe_p) and Froude numbers (Fr_p) as the dependent variables.

Table 9.1. Properties of the carrier fluid and food particles.

Parameter	Specification					
Carrier fluid	Therm-flo® starch					
Gelatinization temperature	140					°C
Concentration	3	4	5	6		% w/w
Density	1010	1014	1019	1026		kg/m^3
Adjusted density	1077					kg/m^3
Flow behaviour index (25°C)	----	0.621	0.484	0.466		
Flow behaviour index (80°C)	0.575	----	0.536	----		
Flow behaviour index (100°C)	0.875	----	0.646	----		
Consistency coefficient (25°C)	----	0.571	3.795	10.52		Pas^n
Consistency coefficient (80°C)	0.353	----	0.657	----		Pas^n
Consistency coefficient(100°C)	0.052	----	0.554	----		Pas^n
Flow rate	10, 15, 20, 25					kg/min
Particles	Carrots		Meat			cubes
Particle size (d_e)	0.07	0.16	0.12	0.19	0.25	m
Concentration	5		5			% w/w
Density	1040		1110			kg/m^3

Table 9.2. Specifications of the different experimental set-ups used.

Parameter	Specification
Scraped surface heat exchanger	
Scraped surface heat exchanger	1.8 X 0.9
Length of the heating surface	0.962 m
Internal diameter	0.152 m
Internal shaft diameter	0.076 m
Annulus	0.076 m
Material of construction	Stainless steel
Dasher speed	150-300 rpm
Heating medium	Steam at 150°C
Cooling medium	Water at 10°C
Holding Tube	
Whole System (80 & 100°C)	
Total length	26.65 m
Lengths used	17.45 26.65 m
Internal diameter	0.0381 m
Holding Tube Only (25°C)	
Total number of turns	4
Length of the turn	4.6 m
Lengths used	4.6 9.2 13.8 18.4 m

RESULTS AND DISCUSSION

Scraped Surface Heat Exchanger (SSHE)

Based on step-wise multiple regression of various dimensionless numbers and related functions, the following two equations gave the best fit for the experimental data on particle Froude and Reynolds numbers (R^2 were 0.97 and 0.99; respectively):

$$Fr_p = 0.23\ (Fr_f)^{0.60}\ (\frac{d_e}{D})^{-0.48}\ (GRe_f)^{0.50}\ (Ar_f)^{-0.24} \qquad (64)$$

$$GRe_p = 2.69\ (Fr_f)^{0.31}\ (\frac{d_e}{D})^{-0.27}\ (GRe_f)^{0.23}\ (Ar_p)^{0.39} \qquad (65)$$

Figure 9.1 shows the plots for the experimental *vs* calculated particle Froude (Fr_p) and Reynolds (Re_p) for carrot and meat cubes in the SSHE. Both Fr_p and Re_p were influenced by the particle-to-tube diameter ratio (d_e/D, $Ar_{f,p}$) and the carrier fluid velocity (Fr_f, Gre_f), density and viscosity (GRe_f, $Ar_{f,p}$). Further analysis on the different dimensionless numbers by equating the powers of the various components (Eqns 64 &65) showed that maximum particle velocity (u_p) increased with fluid velocity and decreased with particle size and viscosity. But the effect of viscosity was relatively small, probably due to the absence of temperature effects ($u_p = \Phi\ (\eta^{-0.01})$) as this study was carried out at room temperature.

Fr_p decreased and Re_p increased as the Archimedes number increased. Although density simplex (a) was a significant factor, it was difficult to draw concrete conclusions with respect to its effect as the absolute density difference between both meat/carrot cubes and adjusted carrier fluid density was about the same. Particle Froude and Reynolds

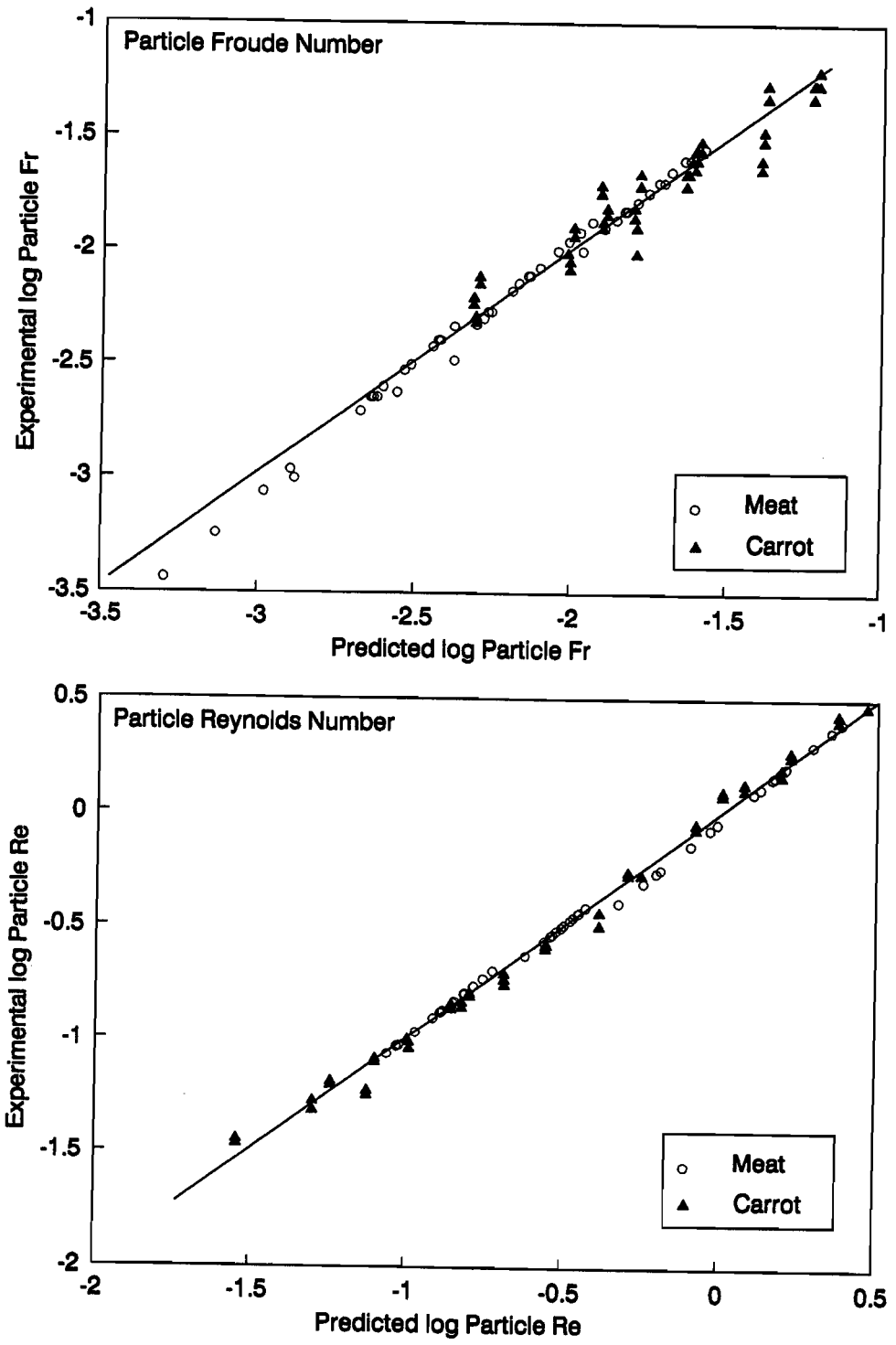

Figure 9.1. **Experimental *vs* regression predicted Froude (Fr_p) and Reynolds (Re_p) numbers of both carrot and meat cubes in the vertical SSHE of a pilot scale aseptic processing system at 25°C.**

numbers were uniformly distributed, with meat cubes associated with lower Froude numbers. This could probably be attributed to larger sizes of meat cubes which would have lower inertial forces in comparison to the gravitational forces. This is clearly illustrated in Figure 9.2 which shows that increased particle size resulted in lower particle Froude numbers. Particle Reynolds number (Re_p) generally increased with GRe_f, and particle size showed similar trends (higher inertial forces compared to the viscous forces (Figure 9.2). Poor correlations were obtained for relative velocities of both meat and carrot cubes due to the cancellation effect of the different factors by normalization.

Holding Tube

A step-wise regression was performed on RTD data of meat and carrot cubes in the holding tube obtained at room temperature. Plots of the experimental *vs* regression predicted values for Fr_p and Re_p of meat and carrot cubes are presented in Figure 9.3. The fit was excellent and the experimental data showed a reasonable uniform distribution. The significant dimensionless numbers for Fr_p and Re_p were the particle-to-tube diameter ratio, Fr_f, GRe_f and Ar_p as shown in the following correlations (R^2 were 0.84 and 0.99; respectively):

$$Fr_p = 1.48\,(Fr_f)^{0.70}\left(\frac{d_e}{D}\right)^{-0.65}(GRe_f)^{0.15}(Ar_p)^{-0.08} \qquad \textbf{(66)}$$

$$GRe_p = 6.65\,(Fr_f)^{0.34}\left(\frac{d_e}{D}\right)^{-0.29}(GRe_f)^{0.10}(Ar_p)^{0.45} \qquad \textbf{(67)}$$

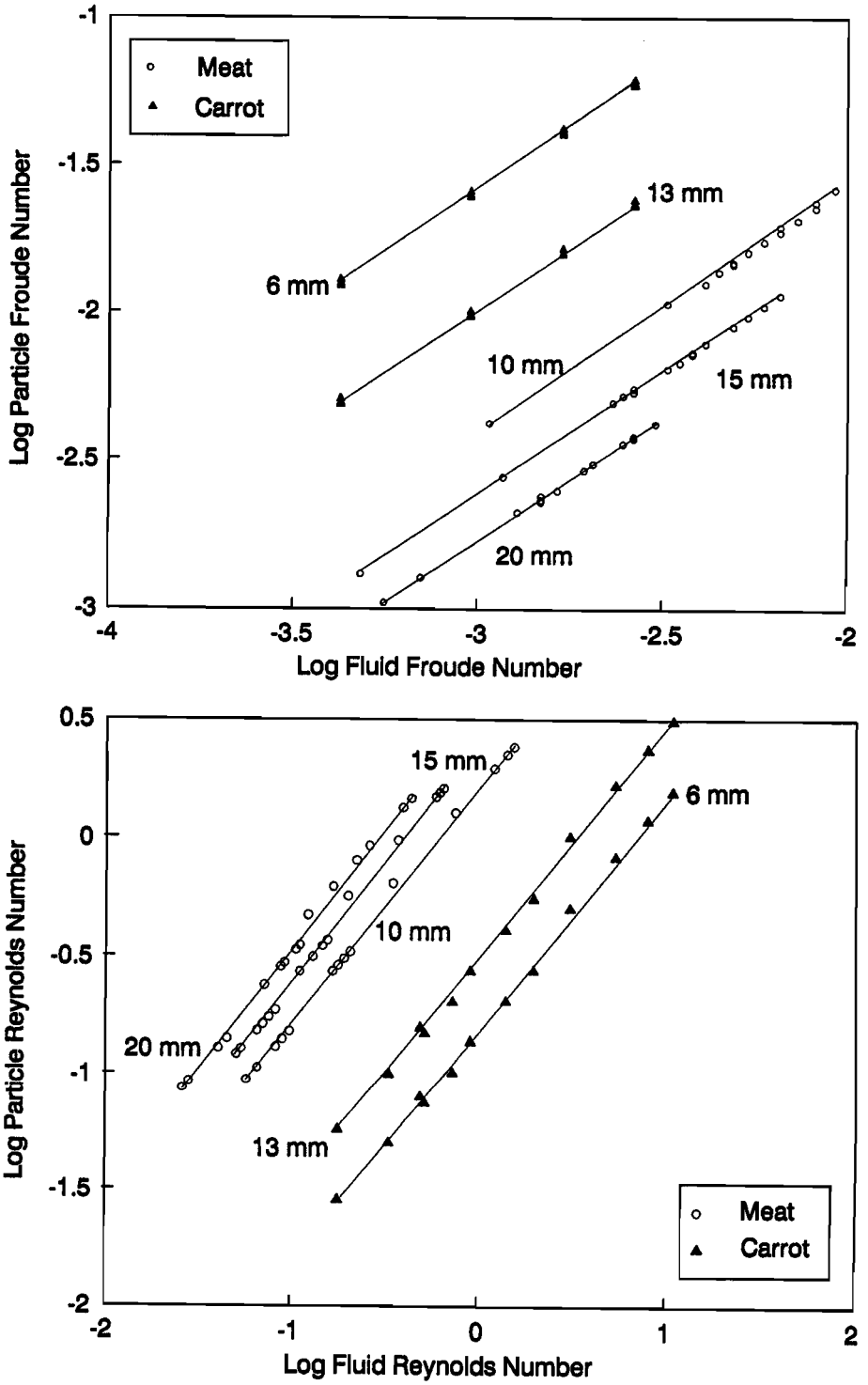

Figure 9.2. **Effect of carrier fluid Froude and Reynolds numbers on carrot and meat cubes Froude number and Reynolds numbers in the vertical SSHE of a pilot scale aseptic processing system at 25°C.**

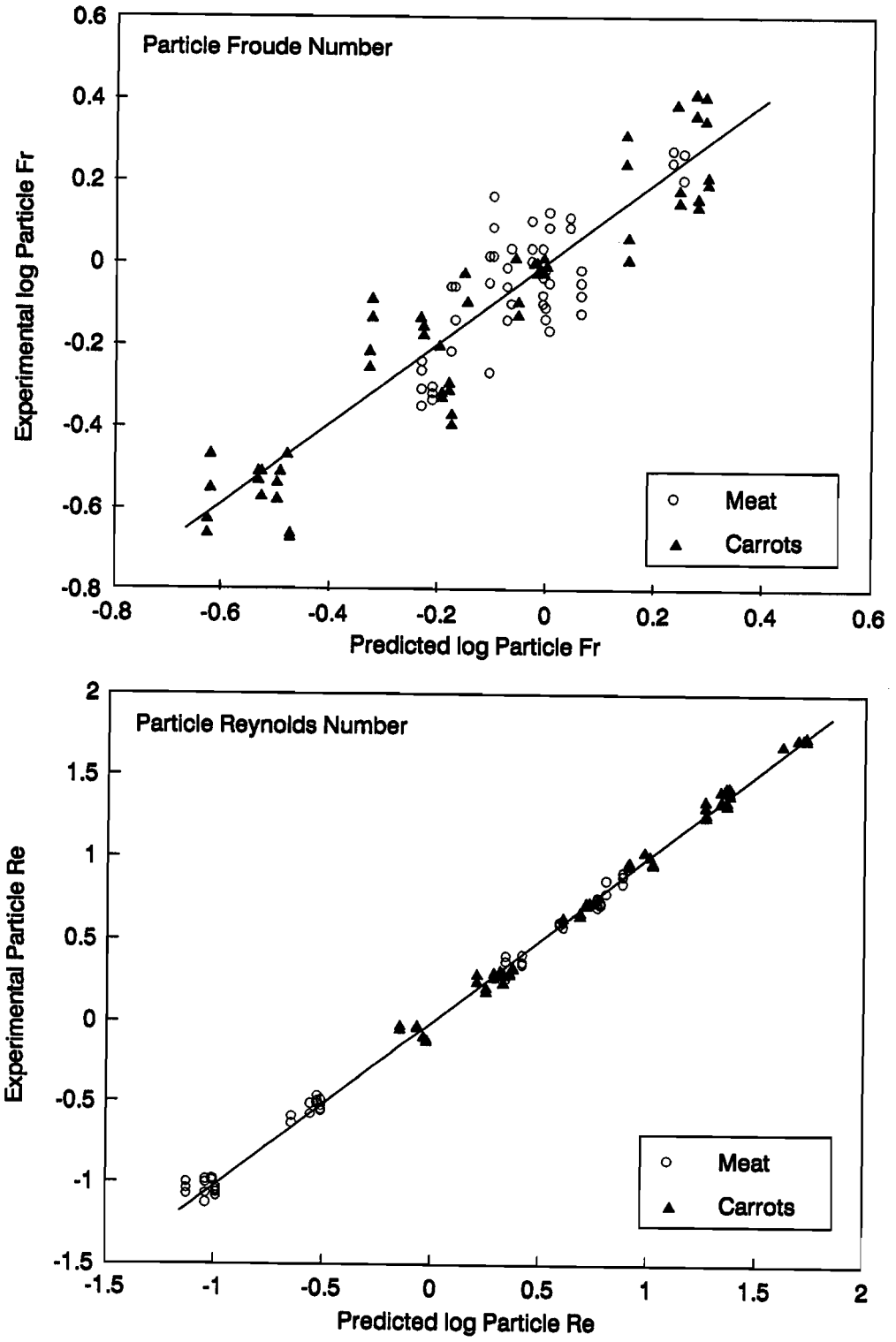

Figure 9.3. **Experimental *vs* regression predicted Froude (Fr_p) and Reynolds (Re_p) numbers of both carrot and meat cubes in the holding tube of a pilot scale aseptic processing system at 25°C.**

Particle relative velocities again showed poor correlations with these dimensionless numbers as with the SSHE. Fr_p increased with Fr_f and GRe_f and decreased with d_e/D and Ar_p. GRe_p had similar trends except that it increased with Ar_p. Particle Reynolds number showed a better fit over the data compared to the Froude number. Further close analysis of Eqns 66 & 67 indicated that the maximum particle velocities increased with particle size and fluid velocity. Carrier fluid apparent viscosity showed no effect on u_p, again because of the non-existence of temperature influence. This is in agreement with the findings of Grabowski and Ramaswamy (1994) who showed that carrier fluid apparent viscosity was influential only when the density difference between particles and fluid was large. Thus the flow behaviour of food particles in the holding tube was different from that of the SSHE, as larger particles were associated with higher Froude and Reynolds numbers (Figure 9.4) because the inertial forces were dominant over the gravitational and viscous forces.

Whole Aseptic System

Experiment with the whole aseptic processing system were conducted at 80 and 100°C only with carrot cubes. Fastest particle RT data was obtained for the holding tube by difference. Scraped surface heat exchangers (SSHEs) connected directly was taken as the control from which the FPRT for 17.5 and 26.7 m holding tubes connected to the SSHEs was obtained by difference. Both particle relative velocity (u_p/u_f) and Froude number gave poor correlations with the dimensionless numbers, but Re_p showed a better correlation (Figure 9.5).

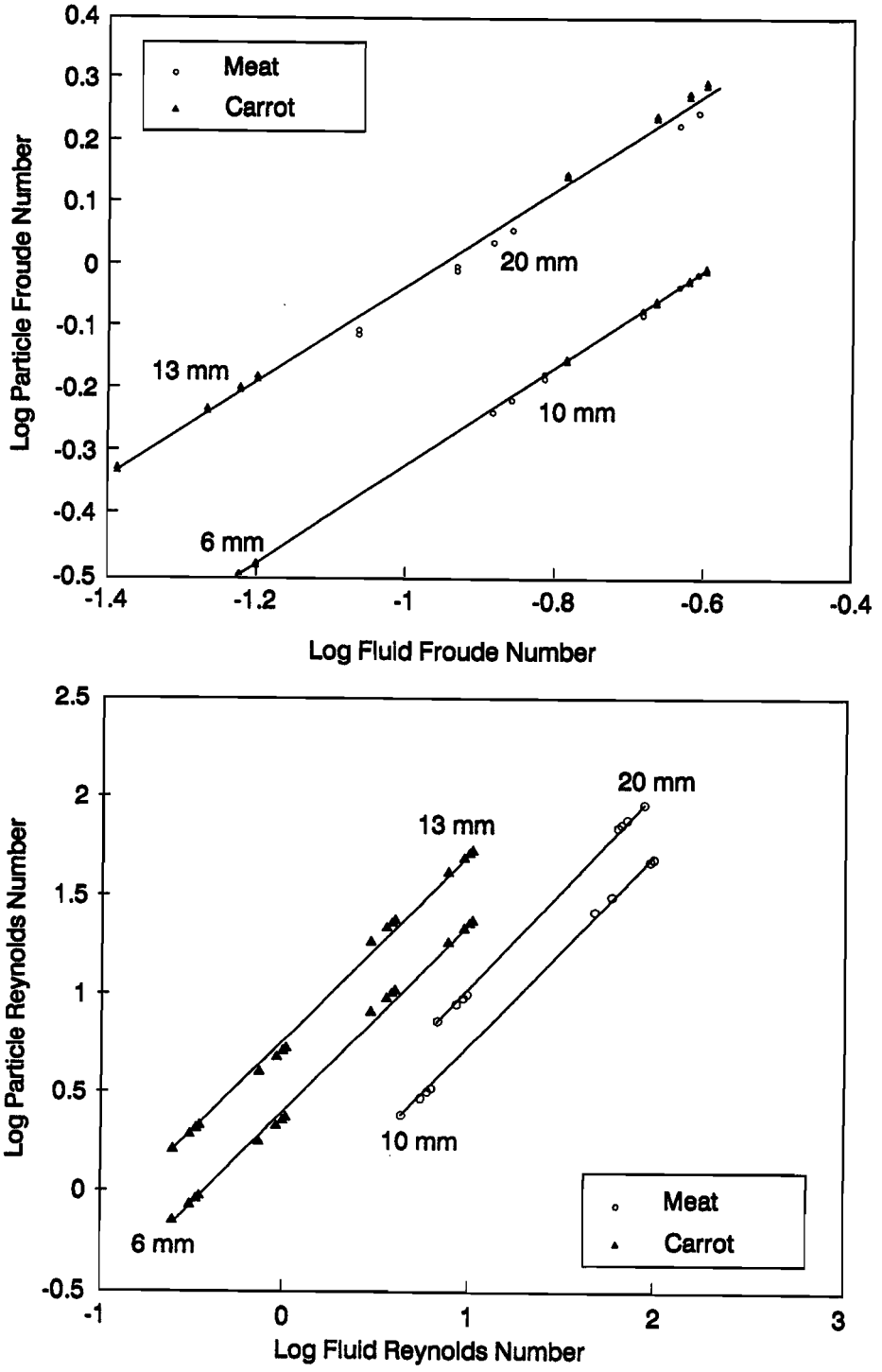

Figure 9.4. **Effect of carrier fluid Froude and Reynolds numbers on carrot and meat cubes Froude number and Reynolds numbers in the holding tube of a pilot scale aseptic processing system at 25°C.**

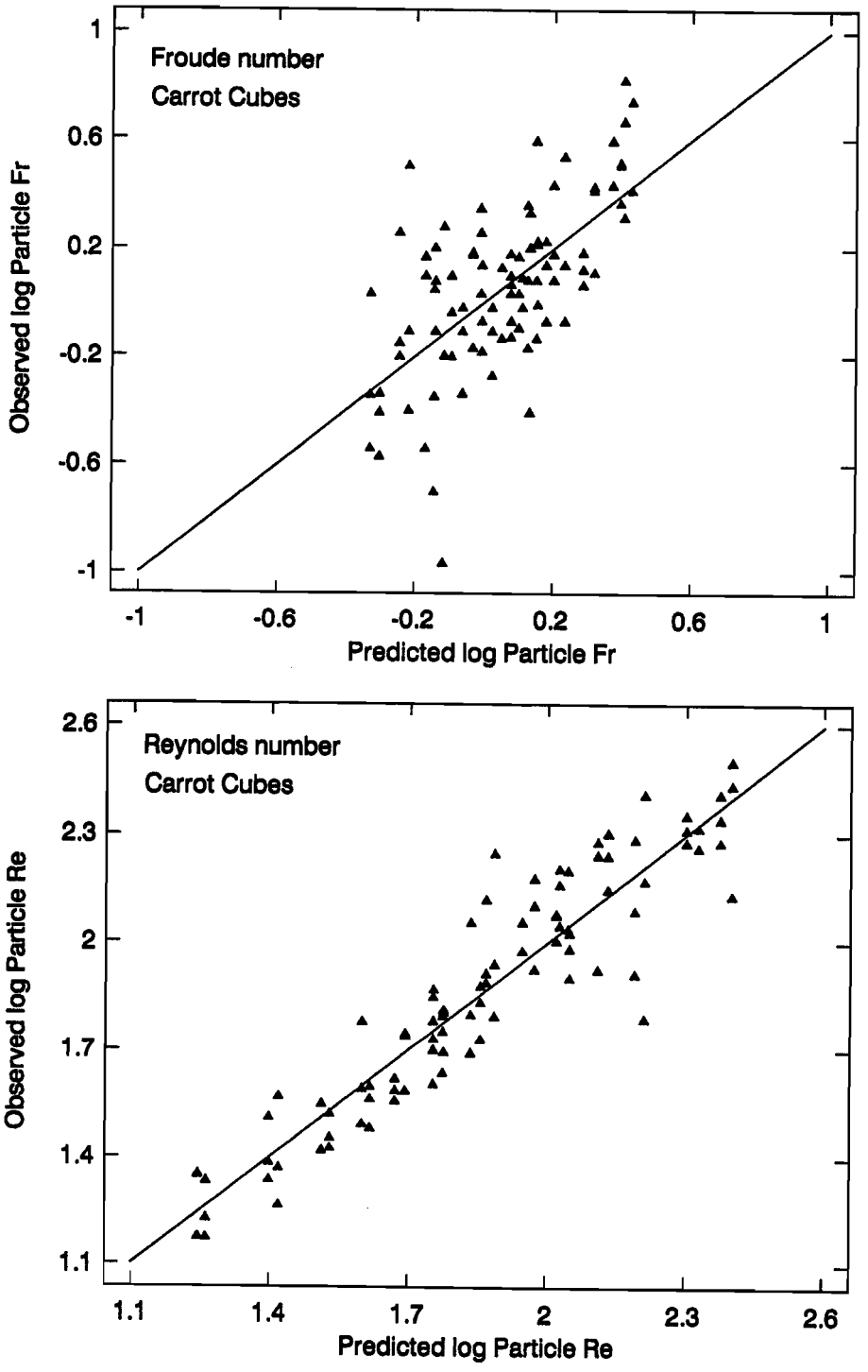

Figure 9.5. **Experimental *vs* regression predicted Froude (Fr_p) and Reynolds (Re_p) numbers of carrot cubes in the holding tube of a pilot scale aseptic processing system at elevated temperatures (80 and 100°C).**

The resulting equations for particle Froude and Reynolds numbers of carrot cubes were:

$$Fr_p = 0.17\ (Fr_f)^{0.69}\ (\frac{d_e}{D})^{-0.88}\ (GRe_f)^{0.37},\ [R^2 = 0.41] \quad \textbf{(68)}$$

$$Re_p = 0.41\ (Fr_f)^{-0.15}\ (\frac{d_e}{D})^{1.06}\ (GRe_f)^{1.19},\ [R^2 = 0.87] \quad \textbf{(69)}$$

The following correlations were obtained for Froude and Reynolds numbers for the holding tube at room temperature (25°C) by re-evaluating Eqns 66 & 67 using data for only carrots:

$$Fr_p = 1.17\ (Fr_f)^{0.74}\ (\frac{d_e}{D})^{-0.89}\ (GRe_f)^{0.07},\ [r^2 = 0.90] \quad \textbf{(70)}$$

$$Re_p = 10.8\ (Fr_f)^{-0.13}\ (\frac{d_e}{D})^{1.05}\ (GRe_f)^{1.04},\ [r^2 = 0.99] \quad \textbf{(71)}$$

The above correlations appear to be consistent with that of carrot cubes in the holding tube *only* situation, except for the expected numerical differences due to the different experimental conditions especially the temperature and starch concentration. More importantly to mention is that the RTD of carrot cubes within the heating SSHE appeared to determine how and when these particles entered the holding tube. From the components analysis of Fr_p and GRe_p for systemic approach (Eqns 68 & 69) and holding

tube *only* situation (Eqns 70 & 71), it appeared that u_p was influenced by u_f somewhat similarly. The major differences were observed with the d_e/D and η_{ap}. The indices of the diameters ratio were higher with the holding tube only ($u_p = \Phi \{d_e^{0.6} D^{0.11}\}$) compared to the systemic approach ($u_p = \Phi \{d_e^{0.06} D^{0.28}\}$). The reason for these differences is that the entrance of carrot cubes to the holding tube was governed by the SSHE in which case smaller particles emerged faster than the larger ones, thus causing a somewhat diffusion effect of particle size within the holding tube. Particle maximum velocity was strongly related to apparent viscosity with the systematic approach ($u_p = \Phi \{\eta_{ap}^{-0.19}\}$) compared to holding tube only situation ($u_p = \Phi \{\eta_{ap}^{-0.04}\}$). Temperatures employed with the systematic approach were significantly higher (80 and 100°C), and caused apparent viscosity to decrease. As a result, higher particle velocities were achieved with the systemic approach, perhaps due to the effect of temperature on viscosity and consequently carrier fluid velocity ($u_p = \Phi \{u_f^{0.78}\}$ and $\Phi \{u_f^{0.88}\}$ for holding tube and systemic approach; respectively). This seems to confirm and justify the importance of performing RTD studies at elevated temperatures conditions of aseptic processing rather than using holding tube or SSHE only at room temperature. It is worthwhile noting, that the emphasis of this chapter was the particle maximum velocities because of its importance from the public health safety stand-point, but similar dimensionless analysis can be performed on particle average velocities since they are important for food quality assessment.

CONCLUSIONS

Particle maximum velocities in aseptic processing were mainly influenced by particle size, fluid flow rate (GRe_f, Fr_f), as well as particle and fluid densities (Ar). Generalized correlations for both meat and carrot cubes were introduced, with equations showing excellent agreement with the experimental data. The trends were somewhat different between the SSHE and the holding tube. Increasing particle density and size decreased particle velocity in the SSHE, whereas the opposite was true for the holding tube. Influence of apparent viscosity was very small in the case of the holding tube and SSHE when experiments performed at room temperature, but the effect was more pronounced with the systemic approach experiments carried out at higher temperatures.

CHAPTER X

GENERAL CONCLUSIONS

1. RTD parameters associated with meat and carrot cubes in the vertical SSHE were influenced by particle size, carrier fluid concentration and flow rate. Residence time of meat particles (FPRT, FPNRT, MPRT, SRTD) decreased with starch concentration and flow rate, while increase with particle size. Similar trends were observed with carrot cubes except starch concentration which resulted in an increase in RTD. Density differences between meat and carrot cubes were considered to be the primary reason for this behaviour. Relatively, carrot cubes had a much shorter RT than meat cubes in the SSHE.

2. RTD parameters (FPRT, FPNRT, MPRT and SRTD) of meat and carrot cubes increased with the holding tube length and starch concentration; and decreased with particle size and flow rate. FPRT and FPNRT of large meat cubes (20 mm) showed an opposite trend. Meat cubes travelled, on average, at about 1.6 times faster than the carrier fluid, while carrot cubes travelled at approximately 1.4 times faster than fluid. On average, larger particles travelled faster than the smaller ones, a trend that is opposite to SSHE flow.

3. In the systemic approach, RTD of food particles were influenced by the factors related to the particle (size), carrier fluid (concentration and flow rate) and system (temperature and holding tube length). Using the whole system approach gave RTD results similar to those obtained using only the SSHE and the holding tube. Tests were carried out employing the maximum attainable temperature under the flow conditions. On an average, under the conditions of testing, the fastest travelling food particle moved at a velocity higher than or equal to the average velocity of the carrier fluid. However, more studies are needed to evaluate the effects increased particle-to-fluid concentration, mutator speed and the effect of particle and carrier fluid densities on RTD. Employing higher aseptic processing temperatures for RTD determination cannot be over-emphasized in order to compensate for the temperature effects on viscosity.

4. The logistic growth model well characterized the RTD curves of meat and carrot cubes passing through a vertical scraped surface heat exchanger, holding tube and a whole assembly of a commercial pilot scale aseptic processing system. Good fit was obtained when the model-calculated cumulative RTD data was used to generate time-specific particle concentration. Predictive equations were developed to relate the model parameters to the system/carrier fluid/particle factors based on test factors found to have significance influence.

5. Mesophilic *Bacillus subtilis* spores were used for biological evaluation of RTD in aseptic processing of liquid foods containing particulates, but at relatively lower

temperatures (95-105°C) as no survival was observed at high temperatures (110-120°C). Carrot/alginate cubes showed an excellent mechanical strength as they withstand pumping as well as the scraper blades without disintegration. Higher degrees of sterilization were achieved with aseptic system compared to the static heated chamber. Destruction of the spores increased with temperature and holding tube length. More controlled experiments are needed to adequately address the issues of food safety with absolute certainty as well as quality factor retention using heat resistant enzymes and vitamins. Transparent windows are needed before and after each of the *heat-hold-cool* sections to monitor the particles as the enter and leave each section to estimate the RTD of food particles precisely and hence the kinetics.

6. The physical isolation of rotor and sensor (D100/300) assembly with the coupling achieved through a magnetic clutch permits evaluating the rheological properties of starch and CMC at the temperature conditions of aseptic processing. The power law model showed an overall good fit over starch and CMC flow curves. The rheological properties were temperature and concentration sensitive. A modified Turian approach was used to describe the combined influence of temperature and concentration. The Weltmann model adequately described the CMC time dependency, while starch thixotropy was negligible.

7. Particle maximum velocities in aseptic processing were mainly influenced by particle size, fluid flow rate (GRe_f, Fr_f), as well as particle and fluid densities (Ar). Generalized correlations for both meat and carrot cubes were introduced, with the equation showing an excellent agreement with the experimental data. The trends were somewhat different between the SSHE and the holding tube. Increased particle density and size decreased particle velocity in the SSHE, whereas the opposite was true for the holding tube. Influence of apparent viscosity was very small in the case of the holding tube and SSHE only experiments performed at room temperature, but the effect was more pronounced with the systematic approach experiments carried out at higher temperatures. More studies are required to clearly address the influence the density difference between the food particles and the carrier fluid.

8. The ramifications of this work if carried to its potential, would mean that RTD of food particles in aseptic processing can be determined by using a systemic approach involving the whole system. The use of the systemic approach will give the advantages of operating at aseptic processing temperatures, use of real commercial systems with real food system, and proper opportunities for biological evaluation and validation of aseptic processes for low acid particulate foods. Rheological properties determined at the aseptic processing conditions will serve as a useful tool in mathematical modelling and further process verification.

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