# STUDIES ON RECIPROCATING AGITATION THERMAL PROCESSING OF CANNED PARTICULATES IN NON-NEWTONIAN FLUID

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Suggested short title:

## STUDIES ON RECIPROCATING AGITATION THERMAL PROCESSING

Dedicated to My Family

#### ABSTRACT

This research focuses on evaluating heat transfer coefficients (using model particles) and quality of real food particulates in non-Newtonian fluids undergoing thermal processing in a labscale reciprocating retort. Evaluation of the heat transfer coefficient is the first step in any heat transfer study. During reciprocating agitation thermal processing (RA-TP) very rapid heating conditions exist which make evaluation of overall (U) and fluid-to-particle ( $h_{\rm fp}$ ) heat transfer coefficients difficult and unreliable. Hence, a new methodology was proposed for evaluating U and  $h_{\rm fp}$  by using an average retort temperature and heat penetration parameter. The proposed method was compared with the conventional methodology and was validated for various reciprocating frequencies and amplitudes. The new method was found to be robust and produced reliable and consistent values of U and  $h_{\rm fp}$ , under reciprocating agitation conditions.

Subsequent to this, the effect of various process and product variables on U and  $h_{fp}$  during reciprocating agitation thermal processing were evaluated using a response surface methodology. The variables chosen for this analysis were: operating temperature (110-130 °C), reciprocation frequency (1-4 Hz), reciprocation amplitude (5-25 cm), can headspace (6-12 mm), liquid viscosity (0-1% CMC solution), particle size (15-25 mm), particle density (Polypropylene, Nylon, Teflon), particle concentration (20, 30, 40% v/v), and particle shape (cube, sphere, and cylinder). Results showed that with an increase in temperature, frequency, amplitude, and headspace, associated U and h<sub>fp</sub> values increased, but with an increasing liquid viscosity and particle size, U and h<sub>fp</sub> decreased. For particle density, U & h<sub>fp</sub> followed the order: Nylon (1128 kg/m<sup>3</sup>)> Teflon (2210 kg/m<sup>3</sup>)> polypropylene (830 kg/m<sup>3</sup>). Amongst the three particle shapes studied, both heat transfer coefficients followed the trend: cube > sphere > cylinder. With an increase in particle concentration, U and  $h_{fp}$ increased up to a maximum value and then decreased. In order to minimize quality loss due to particle motion (agitation intensity) and thermal damage (severity of thermal processing), optimization of processing conditions was also carried out. A certain reciprocation intensity was found to be critical for efficient RA-TP. It was concluded that a reciprocation intensity less than 18 ms<sup>-2</sup> is best suited for products susceptible to agitation induced quality losses, and vice-versa.

Following the heat transfer analysis, quality of canned green beans processed in RA-TP was studied. Green bean processed at higher temperatures and higher reciprocation frequency resulted in greener green beans with a better chlorophyll and better antioxidant activity retention. However, results also showed that very high reciprocation frequency can cause texture damage to food particulates (breakage of beans, increased turbidity of can fluid and leaching of chlorophyll etc.). In order to reduce the impact of agitation and to minimize the quality damage due to rapid back and forth motion of cans, a new concept of stopping agitations mid-way (after sufficient development of cold-spot temperature) during process was proposed. Three agitation time (viz. a) full cycle agitation - continuous agitation throughout the process; b) equilibration time agitation - agitation stopped at zero-log temperature difference between cold-spot and operating temperature, and c) partial time agitation - agitation stopped at one-log logarithmic temperature difference between cold-spot and operating temperature) were considered and corresponding operator's process time to impart 10 min lethality and agitation time were calculated using heat penetration results. Results showed that stopping agitation after sufficient development of cold-spot temperatures is an effective way of reducing product damage caused by agitation (ex. breakage of beans and its leaching into liquid). Agitation continued until achieving a product temperature 10°C lower than the retort temperature gave the best color, texture and visual product quality for low viscosity liquid-particulate mixture and extended agitation till equilibration time was found to be best for high viscosity products.

Finally, artificial neural network (ANN) models were developed for predictive modeling of heat transfer coefficients and process time in reciprocating agitation thermal processing. The optimal configuration of ANN model consisted of two hidden layers with eight neurons in each hidden layer and was trained using the delta learning rule, tangent sigmoid transfer function, Levenberg-Marquardt back-propagation training function and 60,000 learning runs. The developed ANN model was found to accurately predict the heat transfer coefficients for canned green beans. An ANN model coupled with finite difference (ANN-FD model) was also developed for prediction of process time to provide desired lethality to a thermally processed product. This ANN-FD model gave process times comparable to Ball's formula method matched for most processing scenarios.

## RÉSUMÉ

Cette recherche se concentre sur l'évaluation des coefficients de transfert de chaleur et de la qualité des particules en conserve dans des fluides non-Newtoniens subissant un traitement thermique dans un autoclave alternatif. La première étape de l'étude du transfert de chaleur consiste en l'évaluation du coefficient de transfert de chaleur. Cependant, au cours du traitement thermique sous agitation, la vitesse de chauffage augmente largement, rendant l'évaluation du coefficient de transfert de chaleur entre le fluide et les particules (h<sub>fp</sub>) difficile et peu fiable. Par conséquent, une nouvelle méthodologie, utilisant la température moyenne de l'autoclave et les paramètres de pénétration de la chaleur, a été proposée afin d'évaluer U et h<sub>fp</sub>. La méthode proposée a été comparée à la méthode conventionnelle et a été validée pour diverses valeurs des fréquences et amplitudes du mouvement alternatif. La nouvelle méthode a été prouvée vigoureuse et fournit des valeurs fiables et cohérentes de U et h<sub>fp</sub>, selon les conditions d'agitation réciproque.

Par la suite, l'effet de divers procédés et produits sur U et h<sub>fb</sub>, pendant le traitement thermique par l'agitation réciproque, a été évalué en utilisant la méthodologie de surface de réponse. Les variables choisies pour cette analyse étaient: la température de fonctionnement (110-130 °C), la fréquence de l'agitation réciproque (1-4 Hz), l'amplitude de l'agitation réciproque (5-25 cm), l'espace de tête (6-12 mm), la viscosité du liquide (0-1 % solution CMC), la taille des particules (15-25 mm), la densité des particules un (polypropylène, nylon, téflon), la concentration des particules (20-40% v/v), et la forme des particules (cube, une sphère, et le cylindre). Les résultats ont montré qu'avec une augmentation de la température, la fréquence, l'amplitude et de l'espace de tête, les valeurs de U et h<sub>fp</sub> ont augmenté. Cependant, avec l'augmentation de la viscosité et de la taille des particules, les valeurs de U et hfp ont diminué. En ce qui concerne la densité des particules, U & hfp suivent l'ordre suivant: Nylon (1128 kg/m<sup>3</sup>)> Téflon (2210 kg/m<sup>3</sup>)> polypropylène (830 kg/m<sup>3</sup>). Parmi les trois formes de particules étudiées, les deux coefficients de transfert de chaleur ont suivi la tendance suivante: Cube>Sphère>Cylindre. Avec l'augmentation de la concentration de particules, U et h<sub>fp</sub> ont augmenté jusqu'à avoir atteint un maximum puis ont diminué. Afin de minimiser les pertes de qualité dues aux mouvements de particules (intensité de l'agitation) et des dommages thermiques (gravité du traitement thermique), l'optimisation des conditions de traitement a également été réalisée. Une intensité de mouvement alternatif de 18 ms<sup>-2</sup> a été jugée essentielle afin de résoudre les pertes de qualité d'origine thermique ou causées par l'agitation. Ainsi, il a été conclu qu'une intensité de mouvement alternatif de moins de 18 ms<sup>-2</sup> est la mieux adaptée pour les produits sensibles à l'agitation.

Suite à l'analyse du transfert de chaleur, la qualité des légumes en conserve (haricots verts) soumis au traitement d'agitation thermique réciproque a été étudiée. Les haricots verts soumis à des températures élevées et à une fréquence de déplacement alternatif, présentaient une teneur plus élevée en chlorophylle ainsi qu'une meilleure rétention de l'activité des antioxydants. Cependant, les résultats ont également montré que la fréquence très élevée de la réciprocité pourrait endommager la texture des particules (bris de haricots, augmentation de la turbidité du liquide de conserve et la perte de la chlorophylle, etc.). Afin de réduire l'impact de l'agitation et de minimiser les pertes de la qualité qui pourraient résulter du mouvement rapide de va-et-vient des boîtes de conserve, un nouveau concept a été proposé. Celui-ci consiste à arrêter les agitations à mi-chemin pendant le processus, après un développement suffisant de la température au point froid. Trois durées d'agitation (incluant une agitation de temps plein- agitation continue tout au long du processus, agitation au temps d'équilibration – une agitation arrêtée à une différence de température logarithmique nulle entre le point froid et la température de fonctionnement, et, une agitation de temps partiel - agitation arrêtée à une différence de température logarithmique de 1 entre point froid et la température de fonctionnement) ont été prises en compte. Le temps de traitement de l'opérateur permettant de conférer 10 min de létalité, ainsi que le temps d'agitation ont été calculés en utilisant les résultats concernant les taux de pénétration de la chaleur. Les résultats ont montré que l'arrêt des agitations, après un développement suffisant de la température au point froid, est un moyen efficace afin de réduire les dommages causés au produit en raison de l'agitation (ex. rupture des haricots et leur déversement dans le liquide). Les agitations dont la différence de température logarithmique est inférieure ou égale à 1, ont résulté en une meilleure couleur du produit ainsi qu'en une amélioration de la texture et de la qualité du produit. Ce dernier est un liquide contenant des particules ayant une faible viscosité. Cependant, les agitations prolongées, allant jusqu'au temps d'équilibrage ont été trouvées plus efficaces pour les produits ayant une grande viscosité.

Enfin, le modèle artificiel de réseau de neurones (ANN) a été développé afin de réaliser une modélisation prédictive des coefficients de transfert de chaleur ainsi que du temps de traitement

recommandé pour un traitement thermique d'agitation. La configuration optimale du modèle ANN est composée de deux couches cachées contenant huit neurones chaque. Celle ci a été formée en utilisant la règle du delta, la fonction tangente sigmoïde de transfert, la fonction de formation de rétro propagation Levenberg-Marquardt, ainsi que 60000 pistes d'apprentissage. Les modèles ANN développés ont été prouvés utiles pour prédire avec précision les coefficients de transfert de chaleur des boîtes de conserve de haricots verts. Un modèle ANN couplé avec le modèle ANN-FD a également été développé afin de prédire la durée recommandée du processus qui permettrait d'obtenir la létalité désirée du produit soumis au traitement thermique.

#### **CONTRIBUTION OF AUTHORS**

Several presentations have been made based on the thesis research and some manuscripts have been prepared for publication. Some authors were involved in the manuscripts in addition to Anika Singh, the PhD candidate, and their contributions are detailed as follows:

Anika Singh is the PhD candidate who planned and conducted all the experiments, in consultation with her supervisor, gathered and analyzed the results, and drafted all the manuscripts for scientific publications.

Dr. Hosahalli S. Ramaswamy is the thesis supervisor, under whose guidance the research was carried out, and who assisted the candidate in planning and conducting the research as well as in correcting, editing, reviewing and processing the manuscripts for publications.

Anubhav Pratap Singh is another PhD candidate who was involved with the global project on reciprocating agitation thermal processing under the supervision of Dr. Ramaswamy. He worked together with the candidate to fabricate the reciprocating assembly in the retort and also helped her to carry out the experiments using reciprocating retort. His thesis research was focussed on evaluating heat transfer coefficients with Newtonian liquids.

#### LIST OF PUBLICATIONS AND PRESENTATIONS

# Part of this thesis has been published and/or prepared as manuscripts for publications in refereed scientific journals:

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- Singh, A., Singh A. P., & Ramaswamy, H. S. (2015). A refined methodology for evaluation of heat transfer coefficients in canned particulate fluids under rapid heating conditions. *Food and Bioproducts Processing*, 94, 169-179.
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- Singh, A. & Ramaswamy, H. S. (2015). Predictive modelling of heat transfer coefficients and process time for reciprocating agitation thermal processing using artificial neural networks coupled with finite difference model (ANN-FD). *Computers and Electronics in Agriculture*, Draft submitted.
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- Singh A., & Ramaswamy H. S. (2014). A refined method for evaluating heat transfer coefficients in canned particulate fluids under rapid heating conditions. Institute for Thermal Processing Specialists (IFTPS) Annual Meeting, March 11-14, Orlando, FL, USA. [Won second place in graduate paper competition].

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- Singh A., & Ramaswamy H. S. (2014). Overall and fluid-to-particle heat transfer coefficients associated with canned particulate non-Newtonian fluids during reciprocating agitation thermal processing. The Northeast Agricultural and Biological Engineering Conference (NABEC), July 27-30, Kemptville, ON, Canada.
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## NOMENCLATURE

Aorig	absorbance of original CMC solution before processing
A <sub>sample</sub>	absorbance of CMC solution in processed cans
AT <sub>ETA</sub>	agitation time during equilibration time agitation, min
AT <sub>FTA</sub>	agitation time during full time agitation, min
AT <sub>PTA</sub>	agitation time during partial time agitation, min
α	thermal diffusivity, m <sup>2</sup> /s
a	radius of sphere, m
А	total external surface area, m <sup>2</sup>
c <sub>p</sub>	specific heat capacity, J/kg °C
$C_0$	cook value, min
CUT	come-up time of retort, min
EQT	equilibration time, min
$\mathbf{f}_{\mathbf{h}}$	heating rate index, min
Fo	process lethality, min
h <sub>fp</sub>	fluid to particle heat transfer coefficient, W/m <sup>2o</sup> C
Jch	heating lag factor
jcc	cooling lag factor
k	thermal conductivity, W/mºC
m	mass, kg
Nafter	number of whole green beans in processed cans
N <sub>before</sub>	number of whole green beans before processing
n	number of samples
Pt	operator's process time, min
rpm	rotations per min
Т	temperature, °C
Torig	transmittance of original CMC solution before processing
$T_{\text{sample}}$	transmittance of CMC solution in processed cans
$TPT_{ETA}$	total process time/cook time during equilibration time agitation, min
TPT <sub>FTA</sub>	total process time/cook time during full time agitation, min

TPT <sub>PTA</sub>	total process time/cook time during partial time agitation, min
t	time, s
τ	Fourier number,
U	overall heat transfer coefficient, W/m <sup>2o</sup> C
X	value of a variable,
$\bar{x}$	mean of the values,
Z	z-value, °C
$\Delta a^*$	redness
$\Delta b^*$	yellowness
ΔΕ	total color change
$\Delta L^*$	lightness

# Subscripts

c	can
crt	constant/average retort temperature
i	initial condition
1	liquid
р	particle
ps	particle surface
pred	predicted
r	retort
S	surface
ps	particle surface

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#### Chapter 1. INTRODUCTION

Thermal processing, or canning, has been defined as the application of heat for the purpose of shelf-life extension and promotion of food safety (Stumbo et al., 1975). Thermal processing is one of the most important industrial technologies for the manufacture of shelf stable canned foods (Teixeira et al., 1969). Due to their numerous applications and economic importance, this topic has been investigated by many researchers (Ramaswamy and Marcotte, 2005; Holdsworth and Simpson, 2007; Erdogdu, 2008). Despite the emergence of various novel non-thermal approaches, thermal processing continues to be the most widely used method for preserving foods and ensuring product safety. Unfortunately, thermal treatment is not limited to destruction of microbes alone, but it can also destroy many nutrients and degrade food quality. Minimizing quality losses during thermal processing while ensuring the minimal desired sterility is one of the challenges of the canning industry (Abdul Ghani et al., 2002). For products heated by convection, such as liquid food, high temperature-short time processing (HTST) has proven to be useful in acquiring a balance between a safe and high quality product. HTST approach is successful because, compared with microorganisms, the nutrients in foods have a higher resistance to thermal destruction and a lower sensitivity to temperature changes (Dwivedi, 2008). In semi-solid products (i.e. liquid-particulate foods), agitating the container to enhance mixing and the use of thin profile packages (retortable pouches) are some other approaches used to promote better quality. For agitating the containers during sterilization/canning, rotary retorts have been widely utilized industrially. Rotation of containers enables mixing of contents inside the can, and thus results in rapid and uniform heating of the product. Hence, process times are shorter for rotary processing (as compared to conventional heating in static cans), resulting in better product quality (Meng, 2006; Dwivedi, 2008).

The objective of thermal processing is to impart a predetermined minimal process lethality (Fo) to inactivate microorganisms of spoilage and public health concerns at the slowest point of heating (cold-spot). The process time to impart this minimal lethality is often estimated through mathematical modeling of the sterilization process, which requires data on heat transfer coefficients. In liquid-particulate systems, overall heat transfer coefficient (U – at can wall) and fluid-to-particle heat transfer coefficient ( $h_{fp}$  - at liquid-particle interface) are needed to predict the heat transfer rates to the particle and liquid (Singh *et al.*, 2015a). Numerous studies (Lenz and Lund, 1978; Lekwauwa

and Hayakawa, 1986; Deniston *et al.*, 1987; Fernandez *et al.*, 1988; Stoforos and Merson, 1990, 1991, 1992 and Sablani and Ramaswamy, 1995, 1996, 1997, 1999) have been undertaken for the evaluation of heat transfer coefficients U and  $h_{fp}$  during rotary agitation thermal processing. It has

been generally accepted that rotational speed, retort temperature, headspace volume, system geometry, liquid viscosity, rotation radius, particle size, and particle density are key factors during agitation thermal processing (Lenz and Lund, 1978; Naveh and Kopelman, 1980; Hassan, 1984; Anantheswaran and Rao, 1985; Lekwauwa and Hayakawa, 1986; Deniston *et al.*, 1987; Rao and Anantheswaran, 1988; Sablani and Ramaswamy, 1995, 1996, 1999; Meng and Ramaswamy, 2007a,b; Dwivedi and Ramaswamy, 2010a, 2010b).

Most of available heat transfer studies on canned liquid particulate food were conducted with low viscous Newtonian fluids. However, in reality, most of canned fluid foods containing particulates are non-Newtonian in nature. For many widely used thickening agents in real foods, such as starch and sodium Carboxymethyl cellulose, their aqueous solutions exhibit non-Newtonian flow behavior (Rao *et al.*, 1981). Heat transfer studies of canned particulates in non-Newtonian liquids are limited. Anantheswaran and Rao (1985a) studied heat transfer coefficients of aqueous guar gum solutions without particles during end-over-end rotation. In axial rotating cans, Meng and Ramaswamy (2005, 2007a, 2007b) conducted heat transfer studies with nylon particles in non-Newtonian fluids (CMC). However, they used the concept of apparent overall heat transfer coefficient U<sub>a</sub> and, apparent fluid to particle heat transfer coefficient h<sub>ap</sub> to account for the effect of large temperature gradients found within the system for non-Newtonian fluids.

Although thermal processing is a well-established process, it also has a detrimental effect on the nutrients and other important quality attributes (Stumbo *et al.*, 1975; Rattan and Ramaswamy, 2014a). Newer concepts such as agitation processing have been found very effective in achieving the desired objective of reducing quality degradation in various studies (Lee and Ammerman, 1974; Hayakawa and Timbers, 1977; Abbatemarco and Ramaswamy, 1993, 1994; Lebovka *et al.*, 2004; Moyano *et al.*, 2007; Garrote *et al.*, 2008; Rattan and Ramaswamy, 2014a, 2014b). Plenty of research has been carried out analyzing the texture and color changes during thermal processing of canned vegetables (Abbatemarco and Ramaswamy, 1994; Garrote *et al.*, 2008; Rattan and Ramaswamy, 2014a, 2014b). Most of the practical research on canned vegetables has involved

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optimizing the various processing conditions so as to achieve a greener product with a better texture (Greve *et al.*, 1994). Additionally, some researchers have monitored changes in chlorophyll content of canned vegetables to investigate the quality degradation in terms of loss in green color of vegetables (Abbatemarco and Ramaswamy, 1994; López-Ayerra *et al.*, 1998; Almela *et al.*, 2000). Moreover, minimizing texture loss is another objective in order to get a good quality product (Steele, 1987; Abbatemarco and Ramaswamy, 1994; Ávila *et al.*, 2006; Taherian and Ramaswamy, 2009; Delchier *et al.*, 2012; Iborra-Bernad *et al.*, 2013). Apart from color and texture, antioxidants (vitamins and phenolic compounds) have also been found to be significantly influenced by thermal processing. Effect of thermal processing on antioxidant activity has been extensively investigated by various researchers and it is generally believed that a severe heat treatment (such as sterilization) can cause depletion of naturally occurring antioxidants in processed vegetables (Nicoli *et al.*, 1999; Hunter and Fletcher, 2002; Turkmen *et al.*, 2005; Delchier *et al.*, 2012).

Although rotary agitation has significant effect in increasing the heat transfer, it has been observed that increase of rotation speed beyond a certain limit (when centrifugal forces become dominant) is futile (Hassan, 1984). At higher rotation speeds, centrifugal forces clump the particulates against the can wall and thereby reduce product mixing (Stoforos, 1988; Sablani and Ramaswamy, 1996; Dwivedi, 2008). In order to further increase the heat transfer, more recently, a new mode of agitating the cans called reciprocating agitation (Shaka) was introduced by Walden and Emanuel, (2010). The system promises to effectively increase the heat transfer rates beyond the scope of rotary agitation and reduce the thermal exposure time. Walden and Emanuel (2010) found that reciprocating agitation could reduce process times more than 10-fold compared to a rotary process. However, there exists a paucity of detailed heat transfer analysis and scientific process validation studies on reciprocating agitation thermal processing. In a very recent study, Singh *et al.*, (2015b) investigated reciprocating agitation thermal processing, yet, they used glycerin as the covering fluid which is Newtonian in nature. To the best of our knowledge, no attempt has been made to investigate and understand the phenomenon of heat transfer and to evaluate the quality of canned non-Newtonian fluid particulates subjected to reciprocating agitation thermal processing.

Based on the available knowledge and recognizing the need for heat transfer and quality studies on reciprocating agitation thermal processing involving canned particulates suspended in non-Newtonian fluids, the following objectives were formulated for this study:

### 1.1 Objectives

## 1.1.1 Overall objective

The general objective of this work is to this study the heat transfer and quality of canned particulates in non-Newtonian fluid subjected to Reciprocating Container Agitation Thermal Processing.

## 1.1.2 Specific objectives

- i. To develop a methodology for the evaluation of U and h<sub>fp</sub> during reciprocating container agitation thermal processing.
- To study the effect of process variables on U and h<sub>fp</sub> in canned particles in non-Newtonian fluid during reciprocating agitation thermal processing.
- To study the effect of particulate characteristics on U and h<sub>fp</sub> in canned particulates in non-Newtonian fluid during reciprocating agitation thermal processing.
- iv. To study the effect of reciprocating agitation thermal processing on quality of canned vegetables (green bean).
- v. To study the effect of controlled agitation for improving the quality of canned green beans subjected to reciprocating agitation thermal processing.
- vi. Predictive modeling of heat transfer coefficients and process time for reciprocating agitation thermal processing using artificial neural networks (ANN) coupled with finite difference model.

#### **PREFACE TO CHAPTER 2**

Hear transfer characteristics and product quality are two factors that are of concern when investigating a new system like reciprocating agitation thermal process. The rate of heat transfer plays an important role in calculating the required minimal thermal treatment and hence, affects the final quality of the product. The quality attributes of foods can be evaluated by sensory analysis or by using various analytical instruments. The required thermal treatment with maximum possible quality retention is the main focus of food processing industries. To estimate the minimum thermal exposure required, knowledge of several factors such as, pH of food, D and z values of target microorganism, thermal properties of food, storage conditions, container specifications etc. are required. Using data on these coupled with time temperature profiles, one can estimate heat transfer coefficients by employing previously established formulas. Also, various methodologies have been adopted to analyze the time-temperature data collected for estimation of heat transfer coefficients and development of predictive models to characterize the thermal process.

This chapter reviews the various methods and models available in literature for the evaluation of heat transfer coefficients for liquid particulate canned food mixtures subjected to thermal processing. This chapter also discusses research studies dealing with the effect of various operational parameters on heat transfer coefficients subjected to agitation thermal processing. Further, the effect of various modes of thermal processing and their effect of final product quality have also been studied.

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This review was carried out by the candidate under the supervision of Dr. H. S. Ramaswamy.

#### Chapter 2. LITERATURE REVIEW

Thermal processing, or canning, has been defined as the application of heat for the purpose of shelf-life extension and promotion of food safety (Stumbo *et al.*, 1975). It is one of the most promising technology for the manufacture of shelf stable canned foods and continues to be an effective method for preserving foods and ensuring product safety (Sun, 2012). In-container sterilisation involves heating foods in hermetically sealed containers at specific temperature and for a specific time to eliminate the microbial pathogens that endanger public health and microorganisms and enzymes that deteriorate food during storage (Ramaswamy and Marcotte 2005).

#### 2.1 Target microorganisms and process Lethality

In order to design a thermal process for a particular product the first step would be to identify the target microorganism upon which the process should be based. In hermetically sealed canned foods, the achieved environment inside is anaerobic. The aerobic microorganisms, therefore, have no supporting environment to exist. However, in such conditions, any microorganisms which can grow in low oxygen conditions are the target microorganisms for food processors. It is well known that growth rate of anaerobic microorganisms is pH dependent, hence the target microorganism has been related to food pH as well. For the purpose of thermal processing, foods are divided into three pH groups: high-acid, medium-acid, and low-acid foods (Ramaswamy and Marcotte, 2005).

In canned food, the target microorganism is C. *botulinum*, which is highly heat resistant, rod shaped, spore forming, and anaerobic pathogen. It could produce a deadly botulism toxin under anaerobic conditions for a large range of storage temperatures (10-40 °C). It has been generally recognized that C. *botulinum* does not grow and produce toxin in high acid foods (pH < 4.5). Hence, the dividing pH between the low acid and acid groups is set at 4.5 such that in the medium-acid and high acid foods (pH < 4.5) it is not necessary to worry about C. *botulinum*. Other anaerobic microorganisms that are more heat resistant, such as Bacillus *stearothermophilus*, B. *thermoacidurans* and C. *thermosaccolyaticum*, although low-acid and spore-forming, are of little concern specially if the processed cans are stored at temperatures below room temperature (30°C), as they are generally thermophilic in nature and hence, can only grow at higher temperature (50-55°C).

The equivalent time of heating at reference sterilization temperature (121 °C) which shall produce the same amount of microbial kill is defined as process lethality (Fo). The desired degree of lethality in terms of an equivalent time at a reference temperature is generally pre-established for a given product, and processes are designed to deliver a minimum of this pre-set value at slowest heating point in the food held in the container. Mathematically;

$$F_o = F * 10^{\frac{T - T_0}{Z}} \tag{2.1}$$

where, F is the lethality at temperature T and  $F_o$  is the equivalent lethality at reference temperature  $T_{o}$ .

In general, for low-acid foods, a minimum Fo = 3 min (for 12-D reduction of Clostridium botulinum spores) is necessary (National Canners Association Research Laboratories, 1968; NFPA, 1971, 1982, 1985; Stumbo *et al.*, 1975; Institut-Appert, 1979; Nelson and Tressler, 1980; Montville and Sapers, 1981; Barrett *et al.*, 2004). However, often many low-acid foods are heated beyond this point to ensure destruction of microorganisms which are more heat resistant, even though they may not be of public health concern. Different kinds of products like fish, meat, milk and dairy products etc. require different levels of minimal treatments due to specific regulatory laws in place for them.

#### 2.2 Heat penetration parameters

For establishing a thermal processing schedule for any fluid particulate food system, heat penetration data is needed. Heat penetration profile is generated by measuring the temperature of retort, liquid and particle inside the can using thermocouples. In thermal processing applications either needle type or flexible wire copper-constantan thermocouples are used by various processors. Mostly, in agitating cans, a needle type thermocouple is used for liquid temperature measurements while flexible wire thermocouples are used for measuring temperature at a particle center (Ramaswamy and Abbatemarco, 1996; Sablani, 1996; Meng, 2006; Dwivedi, 2008). In fluid particulate canned food, the heat transferred from the heating medium to the particle center (cold-spot) is a complex process. It is well expected that during a heating cycle, the retort heats much faster than the liquid and particulate in the can. This difference is attributed to the fact that the particle heats by conduction, and the liquid heats up by convection. The temperature profile of a

food product undergoing thermal processing depends on various factors like, heating process (still vs agitated; in container vs aseptic), heating medium (steam, hot water, steam-air), heating conditions (retort temperature, initial temperature of food), product type (solid, semi-solid, liquid, particulate liquid, thermo-physical properties of product) and container's specifications (Holdsworth, 1997).

Heat penetration parameters *viz*. heating and cooling rate indices, and heating and cooling lag factors from the heat penetration are calculated from heat penetration curve shown in Figure 2.1. For both the heating and cooling portion of the time temperature curve a separate curve is plotted as shown in Figures 2.2a and 2.2b, which is used for the estimation of the heat penetration parameters. Heating rate index ( $f_h$ ) and heating lag factor ( $j_{ch}$ ) is calculated from the heating curve and the cooling rate index ( $f_h$ ) and the cooling lag factor ( $j_{ch}$ ) are calculated from cooling curve. The heating and cooling rate index ( $f_h$ ) is equal to the negative reciprocal of the slope of the respective curves (Figure 2.2).



Figure 2.1: A Typical Heat Penetration Profile (Ewoldt, 2012)

The heating lag factor  $(j_{ch})$  measures the delay in the beginning of the uniform heating profile of the product. It is expressed by the ratio between the difference of retort temperature and pseudoinitial product temperature (intercept of the straight line portion of heating curve (Figure 2.2a) on yaxis) and the difference of retort temperature and actual initial product temperature. Mathematically it is expressed as:

$$j_{ch} = \frac{T_r - T_{pih}}{T_r - T_{ih}}$$
(2.2)

where,  $T_r$  is retort temperature;  $T_{ih}$  is actual initial product temperature;  $T_{pih}$  is y-intercept of heat penetration curve (pseudo-initial temperature). Similarly, the cooling lag factor (j<sub>cc</sub>) is calculated from the cooling curve (Figure 2.2b) as:

$$j_{cc} = \frac{T_w - T_{pic}}{T_w - T_{ic}}$$
(2.3)

where,  $T_w$  is the cooling water temperature;  $T_{ih}$  is actual temperature when cooling starts;  $T_{pih}$  is the y-intercept of the cooling curve.

#### a. Heating curve

#### b. Cooling Curve



#### Figure 2.2: A typical heating and cooling curve

The lag in heating is due to the slow come up time (CUT) of the retort. This is accounted by determining the new zero time for the process. It was found experimentally that the 42% of the CUT is actually effective and it should be added to the time at the operating temperature (Ball and Olson, 1957).

#### 2.3 Thermal process calculation

Thermal process calculation methods are broadly divided into two classes: (1) General methods and (2) Formula methods. The General methods is based on graphical/numerical integration

of the lethality calculated from heat penetration data. On the other hand, Formula methods, make use of heat penetration parameters ( $f_h$ ,  $j_{ch}$ ,  $j_{cc}$ ) to integrate the lethal effects by employing mathematical procedures. These process calculation approaches have made possible the required control over different modes of thermal processing and the development of quality assurance procedures to establish government regulations for ensuring the safety & reliability of thermal processes (Ramaswamy and Marcotte, 2005).

#### 2.3.1 General method

The General method (Bigelow *et al.*, 1920) is the basic foundation for all the thermal process calculation methods. It is a graphical procedure which integrates the total lethally calculated from the heat penetration data. The main requirements for application of the general method are (a) a reference TDT value and z value related to the target microorganism, (b) representative product heat penetration data, and (c) either the required sterilization value of the process (for calculating the process time) or process time (for calculating the sterilization value). The TDT is defined as the lethal rate at the corresponding operating temperature. The sterilization value is used to express the severity of a given thermal treatment (Ramaswamy and Marcotte, 2005). The product of lethality and the corresponding heating time gives the accomplished sterilization value which indicates that the heating time given is equivalent to the TDT at that temperature. The sterilization values below 1.0 indicated under processing and value above 1.0 indicates over-processing of samples. Furthermore, the Improved General method employing a graphical approach was suggested by Ball (1923). In this approach, a hypothetical thermal destruction curve is drawn which is parallel to the thermal resistance curve of the target microorganism (i.e., with same z value) and having an F value of 1 min at 121.1°C. This approach facilitated the comparison of processes with different lethalites.

#### 2.3.2 Formula methods

Formula methods are more efficient, easier and faster than the General method to estimate the required processing time. These methods are proven very efficient in analysing the effects of various processing conditions on the process time. These methods can be used to determine the process time if the target process lethality (Fo) value is known; and to calculate the delivered lethality of a given
process. Since, the formula methods uses heat penetration data in the parametric form ( $f_h$  and  $j_{ch}$  values), it can easily calculate new processes such as for different size containers and changes in the heating medium temperature (Tr) or the initial product temperature (Ti) by directly using available parameter conversion procedures. Some modifications to the formula methods were also done by some researchers. Later Pham (1989) developed a formula for calculating process times in conduction heated foods.

#### 2.3.2.1 Ball formula method

The Ball formula is one of the simplest and most widely used techniques for process calculations. The Ball Formula method calculates the Ball process time, B (min), based on the following equation:

$$B = f_h \log(\frac{j_{ch} I_h}{g_c})$$
(2.4)

where, B is the process time;  $f_h$  is the heating rate index;  $j_{ch}$  is the lag factor;  $I_h$  is the initial temperature difference ( $T_r$ - $T_i$ ); and  $g_c$  is the temperature difference at the end of cook period ( $T_r$ - $T_i$  at t=B).

Ball (1923) provided the relationship between  $f_h/U$  and  $g_c$  which can be easily interpreted from the given tables and figures. Further, U value can be obtained as:-

$$\mathbf{U} = \boldsymbol{F}_{\boldsymbol{o}} \boldsymbol{F}_{\boldsymbol{i}} \tag{2.5}$$

$$F_i = 10^{\frac{250 - T_i}{z}}$$
(2.6)

where,  $F_i$  is the number of minutes at the retort temperature equivalent to 1 minute at 121°C. One of the limitation of this method is the use of a constant cooling factor (j<sub>cc</sub>) at 1.41 at z=18°F and f<sub>h</sub> = f<sub>c</sub>.

#### 2.3.2.2 Stumbo formula method

Stumbo formula is similar to Ball process, except that it uses several tables for thermal process evaluation to take into account process with different  $j_{cc}$  values. This approach allows process calculations to become more versatile in accounting for the thermal effects of cooling (Stumbo and Longley, 1966). Revised tables were later improved through the use of computer

integration of thermal histories generated from finite difference simulations of heat transfer equations (Stumbo, 1973; Stumbo et al., 1975). It was observed that these tables were able to calculate process times that are often in better agreement with those obtained from the General method of calculation than the similar calculations using Ball's method.

#### 2.3.2.3 Pham's formula method

Pham (1987) proposed a set of simple algebraic equations and simplified tables for thermal process calculations for conditions U/f<sub>h</sub> >1 and U/f<sub>h</sub> <1. The main advantage of Pham formula method is that, instead of using 57 tables of Stumbo formula, it uses only one table and hence, facilitates thermal processing calculations at least as accurate as Stumbo formula. This method could also be used for mass-average lethality, similar to Stumbo's method (1973). Later, Pham amended his equations to cover situations in which the heating and cooling rates were not equal (Pham, 1989) and the accuracy of the modified formulae were reported to be as good as the ones reported earlier for  $f_h = f_c$  situations.

#### 2.4 Computational techniques used in heat transfer modeling of canned foods

Although, there are many methods which researchers have used to calculate the heat transfer coefficients associated with thermal food processes, yet overall they involve the same theoretical background. The first basic step is calculating the overall thermal balance on a liquid-particulate food system to calculate the associated convective heat transfer coefficients. The liquid-particulate system is governed by two stages of heat transfer resistances, one across the can wall and the other across the liquid-particle boundary. Thus, the entire phenomenon can be modeled by knowing the values of the overall heat transfer coefficient (U), at the can wall, and the fluid-to-particle heat transfer coefficient (h<sub>fp</sub>), at the liquid-particle interface. Energy balance equations across the two surfaces can be used to get the values of these parameters (Sablani and Ramaswamy, 1999):

Across the can wall: 
$$UA_c(T_R - T_f) = m_f c_{pf} \frac{dT_f}{dt} + m_p c_{pp} \frac{dT_p}{dt}$$
(2.7)

Across the liquid-particle boundary:  $m_p.c_{pp}.\frac{dT_p}{dt} = h_{fp}A_p(T_f - T_{ps})$  (2.8)

Equation (2.7) is the energy balance equation for a liquid particulate canned system according to which the total heat which is coming inside the can is used for increasing the temperature of liquid and particle. Equation (2.8) is the energy balance at the liquid particle interface. These transport equations can be solved analytically or numerically for a given boundary and initial conditions. The following assumptions have been used to solve these Equations (Sablani and Ramaswamy, 1999): 1) Uniform initial temperatures for liquid and particle; 2) Constant heat transfer coefficients; 3) Constant physical and thermal properties for both fluid and particles; and 4) No energy accumulation in the can wall.

Equations (2.7-2.8) form the basic framework to calculate U and  $h_{fp}$ . These equations can be solved by using the collected time-temperature data of the retort, liquid and particle surface to estimate U and  $h_{fb}$ . Equation (2.7) and (2.8) can be individually solved only when the particlesurface temperature profile is available. However, it is difficult to collect particle-surface temperatures using traditional thermocouples in liquid particulates as the thermocouple tip is exposed to liquid temperatures also. Rather, it is better to collect particle-center temperatures by the use of thermocouples fixed to the particle center which provide a radial symmetry. Particle-center temperature measurements are advantageous because they represent cold-spot lethality for conduction heating particles and hence, are used for establishing a thermal process. However, the disadvantage of the particle-center temperature measurement is that thermocouple attachment may prevent particle movement, leading to possible changes in heat transfer coefficient and the temperature measurements, accordingly. During particle movement in agitation processing, thin flexible wire thermocouples fixed to the particle-center have been used to collect particle-center temperatures (Dwivedi and Ramaswamy, 2010a). These flexible wire thermocouples are light and flexible so move along with the particle and thus allow particle motion. However, there is certainly some change in particle motion but it is generally considered trivial as the thermocouples are very thin. Although, other non-invasive techniques available like wireless thermocouples, biological indicators etc. have also been used to measure particle temperatures, yet, they pose a problem as these have appreciable weight and do influence the particle thermo-physical properties. So there is always some compromise. Hence, thermocouple based measurements are still the most common mode of collecting temperature profile. If particle-center temperatures are used, it is important to

estimate the particle-surface temperatures to be used in Equation (2.11). For this, conduction heat transfer equation is coupled with Equations (2.7-2.8) to get an estimate of U and  $h_{fp}$ . For a spherical particle, assuming a 1-D heat transfer along the radial direction, the heat flow is described by the partial differential Equation (2.11) with initial and final boundary conditions prescribed by Equations (2.9-2.12) (Alhamdan *et al.*, 1990):

$$\frac{\partial T}{\partial t} = \alpha_p \left( \frac{\partial^2 T}{\partial r^2} + \frac{2}{r} \frac{\partial T}{\partial r} \right)$$
(2.9)

$$\Gamma(\mathbf{r}, 0) = T_i at t = 0$$
 (2.10)

$$\frac{\partial T(r,t)}{\partial r} = 0 \text{ at } r = 0 \tag{2.11}$$

$$k_p \frac{\partial T(r,t)}{\partial r} = h_{fp} (T_f - T_{ps}) at r = a$$
(2.12)

The  $h_{fp}$  value can then be calculated by solving Equation (2.8) iteratively using the experimental particle-center and liquid temperature profiles and U value can then be obtained by solving Equations (2.7) and (2.8) using the particle-surface temperature profile generated by using Equation (2.9). Solving the partial differential Equation (2.9) with these boundary conditions is not very straightforward, as there is no simple analytical solution available. Most of the solutions to Equation (2.9) assume a constant liquid temperature, but these temperatures are transient. For liquid-particulate canned systems, researchers have used several approaches to solve Equation (2.9). Some of these approaches are discussed in the following sections, while the basics of these approaches are described in Table 2.1.

#### 2.4.1 Lumped parameter model

This approach is only valid for a thermally thin substance (with Biot number less than 0.1). For them, the evaluation of the heat transfer convective coefficients is based on the assumption of a negligible internal resistance. For the lumped parameter model to be valid either  $h_{fp}$  must be very low or the size of particle must be very low or the thermal conductivity of the particle must be very high.

# Table 2.1: Solution Methodology for Solving Heat Transfer Equation (2.9) using Analytical and Numerical Solution Techniques for

**Thermal Processing of Liquid Particulate Foods** 

Analytical solution techniques						
Method of Separation of Variables <sup>a</sup>	Laplace Transformation method <sup>a</sup>	Duhamel's Theorem <sup>b</sup>				
• Equation (2.8) is solved as a product of space X(x) and a time T (t) (viz.T(x,t) = X(x), T(t) = [D cos bx + E sin bx]e <sup>-b<sup>2</sup>t</sup> ). Separating terms of X and T and equating each side to a constant, one can obtain solutions for X and T. Particular solution is found using Fourier series. • Using a step function for surface temperature of the particle: $T^* = (2a/\pi r) \sum_{n=1}^{\infty} (-1)^{n+1} \sin(n\pi r/a) e^{-n^2 \pi^2 F_0}$ • For the case of external heat transfer: a) $\sum_{n=1}^{\infty} \frac{T^* = (2r/2n^2) (2r/2n^2)}{2n^2 n^2 n^2 n^2} e^{-Mn^2 F_0}$	<ul> <li>Equation (2.8) is multiplied by e-<sup>bt</sup> and integrated from t=0 to ∞. The solution of the transformed equations can then be reverse transformed to the original system to get solution.</li> <li>However, finding reverse transform is not always easy (general transforms available in Ozisik, 1985).</li> <li>Fourier sine transform is prescribed for BCs, while Henkel transform and Bessel functions are used for cylindrical geometry. Some special transcendental functions also exist.</li> </ul>	<ul> <li>Equation (2.8) is valid for constant liquid temperatures, but they are transient. Duhamel's theorem is used to handle time-dependent BC.</li> <li>Also used to predict data for different experimental conditions at same BC by decoupling temperatures.</li> <li>In this method, a step-response solution is converted to required solution using Duhamel's theorem.</li> </ul>				
n=_	Numerical Solution Techniques					
<ul> <li>Finite Difference Method <sup>c</sup></li> <li>Equation (2.8)'s derivative functions are approximated at discrete points (nodes) using forward, backward or central difference techniques.</li> <li>Developed equations are algebraic and easily solvable using linear algebra.</li> </ul>	<ul> <li>Finite Element Method <sup>d</sup></li> <li>Equation (2.8)'s complex PDE is approximated at discrete subdomains (elements) using calculation of variations. All element equations are combined using coordinate transformation and solved together.</li> <li>Developed equations are algebraic (solved by linear algebra) for steady-state and ODEs (solved by numerical integration) for transient problem.</li> </ul>	<ul> <li>Finite Volume Method <sup>e</sup></li> <li>Equation (2.8) is approximated for small volumes using volume-integral. All divergent terms are converted to surface integrals using the divergence theorem.</li> <li>Developed equations are easily solved as fluxes at the surface of each finite volume.</li> </ul>				
<sup>a</sup> Holdsworth and Simpson (2007), Ramaswamy and Zareifard (2002), Welty <sup>b</sup> Hayakawa et al. (1997), Stoforos et al. (1997), Welty et al. (2007).	et al. (2007).					

<sup>e</sup> Erdogdu (2008), Holdsworth and Simpson (2007), Smith, (1999).

<sup>d</sup> Strang and Fix (2008).

<sup>e</sup> LeVeque (2002); Toro (1999).

Various researchers (Sastry *et al.*, 1985; Fernandez *et al.*, 1988; Alhamdan *et al.*, 1990; Alhamdan and Sastry, 1990; Stoforos and Merson, 1991; Rahman and Al-Saidi, 2009; Cariño-Sarabia and Vélez-Ruiz, 2013) have used this approach while studying systems with biot number less than 0.1. Most of these systems involve the use of metal particles. However, Cariño-Sarabia and Vélez-Ruiz (2013) had reported that the validity of lumped system approach was rather low, as it was not possible to apply this formulation with low thermal conductivity materials like real foods.

Under the aforementioned assumption, the rate of increase of thermal energy within the food system is considered equal to the rate of heat addition by convection, and is formulated using the following equations:

$$\frac{m_p c_{pp}}{A_p} \ln(T^*) = -h_{fp} t \tag{2.13}$$

From the temperature history of the particle, plots of ln T\* vs. time can be obtained and the slope of this plot can be calculated. Thus, the convective heat transfer coefficient may be evaluated from the following equation:

$$h_{fp} = -\text{slope} * \left(\frac{m_p c_{pp}}{A_p}\right)$$
 (2.14)

The advantage of this formulation is that it leads to a simple first-order differential equation which describes heat transfer in these systems. Having a Biot number smaller than 0.1 labels a substance as thermally thin, and the temperature can be assumed to be constant throughout the material volume, and thus the use of a lumped parameter approach can be justified. However, this formulation is also valid only for constant liquid temperature. For liquid-particulate mixtures, the liquid temperature is transient and hence, Duhamel's theorem (Table 2.1) is used in conjunction with the lumped parameter analysis. But still, this approach provides a quick and simple analytical solution for thermally thin substances.

# 2.4.2 Analytical solution techniques

When the Biot number is greater than 0.1, a lumped parameter approach cannot be used. For this more complicated heat transfer equations for "transient heat conduction" will be required to describe the time-varying & non-spatially-uniform temperature within the material body. In this case, the evaluation of the convective coefficient is based on finite surface and internal resistances. The solution to Equation (2.9) for a sphere, under convective boundary conditions defined by Equations (2.10-2.12) has been given as Equation (2.15) (Ramaswamy and Zareifard, 2002).

$$T^* = (2Bi) \sum_{n=1}^{\infty} \frac{[M_n^2 + (Bi-1)^2] \sin M_n}{M_n^2 [M_n^2 + Bi(Bi-1)]} \cdot \frac{\sin(M_n r/a)}{(r/a)} e^{-M_n^2 F_0}$$
(2.15)

Here  $M_n$  is the n<sup>th</sup> positive root of convective boundary condition (Equation. 2.16).

$$M\tan M = Bi \tag{2.16}$$

After sufficiently long heating time ( $F_0 > 0.2$ ), the series solution of Equation (2.16) converges rapidly and can be approximated as the first term. Thus, using M as the first root of the above equation and abbreviating all parameters behind the exponential term as A, Equation (2.17) can be rewritten as Equation (2.17).

$$\log_{10} T^* = \log_{10} A - \left(\frac{M^2 \alpha t}{2.303 L_c^2}\right)$$
(2.17)

Again, Equation (2.15) assumes a constant liquid temperature like lumped parameter approach solution. Hence, Duhamel's theorem needs to be used to handle transient liquid temperatures. Solution to Equation (2.15) has been further simplified for different geometries by various researchers (Ramaswamy *et al.*, 1982; Ramaswamy and Sreekanth, 1999).

There are many techniques (Table 2.1) to arrive at such analytical solutions for the heat transfer equation. For different shapes like sphere, infinite and semi-infinite plates and cylinders, general analytical solutions based on these techniques are available in literature (Holdsworth and Simpson, 2007; Welty *et al.*, 2007). Generally, method of separation of variables is the most

preferred method, yet transformation methods are easier for simple problems. However, for complex boundary conditions and geometries such as those encountered during canning of liquid particulates, finding reverse transform is not always easy and may be numerically very intensive. Thus, a combination of Duhamel's theorem with the analytical solution is preferred in heat transfer studies on liquid particulate mixtures (Stoforos and Merson, 1991; Cariño-Sarabia and Vélez-Ruiz, 2013).

Once, we have the temperature profile of the retort, liquid and the particle, the values of U and  $h_{fp}$  can be evaluated by fitting the analytical profile with the experimental one using least sum of squared temperature difference (LSTD) or least accumulated lethality different (LALD) approach. Although, analytical solutions are easy to work with, yet their applicability is restricted to a limited number of situations, and cannot be obtained for all conditions and geometries of the particles. Under these situations, numerical solution techniques are applied.

# 2.4.3 Numerical solution techniques

In a real life situation like in food canning, it is not always possible to provide a simple analytical solution to a problem. Hence, numerical solutions provide an alternate to solving these problems. Numerical methods don't require solving complex transcendental equations, so they are most suited to modern computing fashion (Singh *et al.*, 2015c). These methods are based on iterative estimations of temperatures using approximate methods. There are three types of numerical techniques for these kinds of problems: the finite differences (FD); the finite element (FE); and, the finite volume (FV) methods. The principles for using these techniques to solve Equation (2.16) for liquid particulate mixtures have been visited in Table 2.1 and some of the studies based on using these techniques have been presented in Table 2.2. Generally, FE schemes are computationally most demanding, while FD schemes are easiest to implement. Yet, FV schemes are the ones which guarantee convergence and are believed to be the fastest.

FD scheme is the most common numerical method used for liquid particulate mixtures (Wang and Sun, 2003). They have been widely applied in thermal processing to predict sterilizing value, process time, process lethality and nutrient retention of foods (Tucker, 2004). FD schemes are easiest to implement and preferred for simple geometries. With regards to the stability of these

schemes, there are two methods which are generally used. The fastest method is the explicit method in which nodal values are replaced by values taken from previous time interval. However,  $F_0 < 0.25$ is the stability criterion for considering a node for explicit methods. A more stable, but computationally more demanding, method is the implicit method in which the nodal points are replaced by the values taken at the same time-interval, rather than previous one. Apart from these methods, there are several other methods which lie in between the implicit and explicit methods and are known as semi-implicit methods like Crank-Nicolson method, Jacobi method, Gauss-Seidel method and over-relaxation methods. However the major disadvantage of FD schemes is that computer programs are specific for each system, and need to be rewritten if geometries or boundary conditions change. Teixeira et al. (1969) were the first to use the numerical methods for canned foods. They used FD method to predict transient temperature profiles during natural convection heating. Recently, researchers have developed simulation programs based on FD methods for particle-center temperature and lethality prediction during thermal process design for canned liquid particulates (Chen and Ramaswamy, 2002a, 2002b, 2002c; Welty et al., 2007). Numerical solutions based on finite differences have also been used to evaluate U and hfp for thermal processing of liquid particulate mixtures (Weng et al., 1992; Sablani and Ramaswamy, 1999; Mohamed, 2003, 2007; Meng, 2006; Palazoglu, 2006; Dwivedi and Ramaswamy, 2010b).

FE methods have been used for modeling the thermal processing of baby foods (Smith, 1999), irregular foods like mushrooms (Sastry *et al.*, 1985; Mohamed, 2003), thick and viscous foods like lasagna (Nicolai *et al.*, 1995) etc. These models are frequently generic and can be adjusted easily for different geometries. However, FE methods are computationally most demanding and are preferred only where meshing (the discretization procedure) is easy. Meshing can be done using Hrennikoff's approach (using lattice analogy for creating elements) or Courant's approach (using finite triangular sub-regions for creating elements) to solve second order elliptic partial differential equations. FE methods have also been used to solve tough problems involving stochastic initial and boundary conditions, for perturbation analysis or sensitivity analysis of the variation of parameters in thermal processing, and for estimation of cooling temperatures (Naveh *et al.*, 1983; Nicolai and De Baerdemaeker, 1997). Basically (and with many caveats), FE provides more degrees of freedom and therefore accuracy; but is more memory-intensive. Yet, with the advances of computing power these

days, this isn't much of a problem and FE is being used more and more, even in CFD packages like Adina, Ansys, COMSOL Multiphysics, FEMLAB etc. Still, FE is considered best for solving diffusion equation, while FV is preferred for solving fluid mechanics problems.

FV methods are most commonly used technique in heat transfer module of various CFD packages. Due to low computation time involved, these are best for problems for which FE methods require large memory and time (high Reynolds number flow and source term dominated flows). These conditions are usually encountered during heating of liquid-particulate mixtures. Many researchers have studied simulation during sterilization of viscous liquid foods in can using FV methods (Abdul Ghani *et al.*, 1999). Recently, researchers have used this approach for numerical simulation of natural convection heating of canned foods (Abdul Ghani *et al.*, 2002). Because the flux entering a given volume is identical to that leaving the adjacent volume, these methods are conservative. Another advantage of the FV method is that it is easily formulated to allow for unstructured meshes. Apart from these 3 methods, there are other methods like spectral element method, boundary element method, high-resolution discretization schemes etc. available for numerical solution methodology.

Thus, researchers have used various analytical and numerical models for studying heat transfer in liquid particulate mixtures. Some of the major works have been highlighted with the methodology used in Table 2.2. In most of these studies, either radial symmetry for the particle or axisymmetric approach along the y-axis of the vertical can was assumed to reduce the computation time. However, with development of advanced computational technologies, researchers are now focusing on developing 3-dimensional models for studying heat transfer in cans (Abdul Ghani *et al.*, 2001). More recently, researchers have attempted to combine mass and heat transfer equation simultaneously to study the temperature and velocity profiles during canning of solid-liquid mixtures (Kiziltas *et al.*, 2010; Erdogdu and Tutar, 2011; Tutar and Erdogdu, 2012). Thus, with the progress in computation powers, researchers are relying on more accurate but computationally more demanding methodologies. With the advent of numerous easy-to-use and user-friendly CFD packages, the numerical and analytical methods are slowly giving way to advanced simulation studies (Boz et al., 2014). Yet, there are many groups of researchers still relying on conventional analytical and FD based numerical methods due to their lower time-complexity and simplicity.

Table 2.2: Mathematical models for modeling the heat transfer during thermal processing of liquid particulate foods (Singh et al.,2015c).

Food/Food Models	Mode Agitation	of	Model solved	Reference
Carrot/Orange soups in cans			3-d Numerical Solution using FV scheme (CFD simulation on	Abdul Ghani (2006)
Carrot/Orange soups in pouches	-		PHOENICS)	Abdul Ghani et al. (2001)
Permeable and impermeable pineapple	Still		Simultaneous Numerical Solution of Mass, Energy &	Abdul Ghani and Farid
slices in water			Momentum balance using FV scheme (CFD simulation on	(2006)
Carrot/Orange soups in horizontal cans			PHOENICS)	Abdul Ghani et al. (2002)
Mushroom shaped particles in CMC	Still		Lumped capacity model	Alhamdan and Sastry (1990)
Mushroom shaped particles in water				Alhamdan et al. (1990)
Canned mussels in brine solution	Still		Transfer function based analytical MATLAB model coupled with optimization routine to search for retort temperature profile for optimum quality index	Ansorena and Salvadori (2011)
Canned pork puree	Still		Reduced-order FD model on MATLAB coupled with optimization routine (ICRS/DS) for thermal process design	Balsa-Canto et al. (2002)
Canned pork puree	Still		Optimization routine ICRS/DS for optimal control problems	Banga et al. (1991)
Horizontal cans containing water	Still		2-d and 3-d CFD simulations of continuity, energy and momentum equations using Ansys CFX 12.1	Boz and Erdogdu (2013)
Sterilization of canned foods	Still		Numerical solution using FD scheme coupled with receding horizon optimal control strategy for optimum retort profile	Chalabi et al. (1999)
Liquid-particulates in water	Still		Numerical solution using FD scheme on Microsoft Visual Basic 6.0 coupled with ANN-GA based optimization routine for CRT/VRT processing	Chen and Ramaswamy (2002b, 2002c)
			ANN-GA based multiple ramp-variable retort temperature control	(2004)

Mushrooms, tomatoes, potatoes in Newtonian and non-Newtonian fluids	Still	Analytical solution using Lumped parameter approach	Cariño-Sarabia and Vélez- Ruiz (2013)	
Asparagus in 4% NaCl solution	Still	3-d CFD simulation using FLUENT using COUPLED, SIMPLE and PISO algorithms for pressure-velocity coupling	Dimou and Yanniotis (2011)	
Peach halves in sugar solution			Dimou et al. (2014)	
Nylon particles suspended in CMC solution	Biaxial rotation	Numerical solutions based on finite difference model	Dwivedi and Ramaswamy (2010a)	
Sphere and cylinder in water	Still	Multi-objective optimization using complex method and lexicographic ordering	Erdogdu and Balaban (2003)	
Sterilization of water and air in cans	Still	2-d CFD simulation on Fluent	Erdogdu and Tutar (2011)	
Canned Green peas in 2% NaCl and 1.5%	End over and	Numerical solution based on FD scheme using Crank-	Garrote et al. (2006a,	
sucrose solution	Ella-over-ella	Nicolson algorithm	2006b)	
Aluminum in sucrose syrup	A vial rotation	Lumped capacity and Use of Duhamel's theorem	Formandaz et al. (1099)	
Beans in water and sucrose solutin	Axiai iotatioli	Numerical solution using FD scheme	remanuez et al. (1900)	
Potatoes in water	End-over-End	Numerical solution and	Hayakawa et al., (1997)	
	rotation	Duhamel's theorem		
Sterilization of 0.85% CMC simulating	lg Still	Numerical solution based on FV scheme using CFD (CFX	Kannan and Gourisankar	
tomato puree	Still	v5.7)	Sandaka (2008)	
Canned peas in water		2-d axisymmetrical CFD simulation using Ansys V11	Kiziltas et al. (2010)	
Cylindrical particulates in CMC	Still	Numerical solution based on FV scheme using CFD (CFX v5.7)	Kurian et al. (2009)	
Nylon Particles in CMC	End-over-End rotation	Numerical simulation based on FD scheme	Meng and Ramaswamy (2007)	
Canned pork puree	Still	Global optimization algorithm for convex problem using Tuy's algorithm	Miri et al. (2008)	
Tomato Sauce in cans & 8% bentonite suspension	Still	Numerical solution based on alternating direction implicit FD method in FORTRAn	Mohamed (2003)	
8% bentonite suspension	Still	Numerical solution based on Crank-Nicolson implicit FD method in FORTRAN	Mohamed (2007)	

Potato and polymethylpentene cubes in water	Still	Numerical simulation based on FD model	Palazgolu (2006)	
Eggs in water	Still & axial rotation	3-d CFD simulations using Ansys Inc and SIMPLE algorithm for pressure-velocity coupling	Ramachandran <i>et al.</i> (2011)	
Model liquid-particulate mixtures in cans	Still	Simplified analytical solution	Ramaswamy <i>et al.</i> (1982, 1997)	
Canned potatoes in 1% CMC	Free axial & end-over-end	Optimization using numerical solution based on FD scheme	Rattan and Ramaswamy (2014b)	
<i>Nylon &amp; polypropylene spheres in water &amp; oil</i>	End-over-end	Numerical solutions based on FD model	Sablani and Ramaswamy (1999)	
Mushroom-shaped aluminum in water	Still	Lumped capacity approach	- Sastry <i>et al.</i> (1985)	
Mushrooms in water	Still	3-d FEM based Heat & Mass Transfer Model		
Canned Tuna	Still	Multi-objective optimization (NBI-based weighted Tchebycheff approach) for simultaneous maximization of nutrient & quality and minimization of process time	Sendin <i>et al</i> . (2010)	
Nylon spheres in glycerin	Reciprocating agitation	Numerical solutions based on FD model	Singh <i>et al.</i> (2015b)	
Potato and PTFE spheres in deionized water and aluminum spheres in silicone fluid.	Axial rotation	Analytical solution	Stoforos and Merson (1991)	
Canned 3.5% corn starch dispersion	Axial rotation	Numerical solution based on FEM scheme using FIDAP	Tattiyakul et al. (2001)	
Sterilization of water and air in cans	Axial rotation	Numerical solution using volume of fluid (VEF) element method coupled with FV scheme using FLUENT 2007	Tutar and Erdogdu (2012)	
Tomato/carrot/green bean/apple/banana purees in CMC	Still	CFD simulation using CFX v5.6	Varma and Kannan (2005)	
Polyacetal spheres and cubes in water	Agitated water bath	Numerical solution	Weng <i>et al.</i> (1992)	

# 2.4.3.1 Use of computational fluid dynamics in thermal processing

Use of advanced modeling techniques, such as computational fluid dynamics (CFD), reduces the complexity in solving the non-linear partial differential equations of heat transfer. CFD is based on numerical methods that can simplify the geometrical representation of models, hence, also cut down pre-processing and solving time. Coupled with other technological advancements, CFD has shown great potential to achieve improvement in both quality and safety of the food, with reducing energy consumption and the amount of empiricism associated with a design process (Norton and Sun, 2007). CFD can easily be applied to conventional and novel thermal food processes ranging from experimental models to the scaled-up systems (Ramachandran *et al.*, 2011). CFD models are being used in thermal processing studies on liquid-particulate systems such as pineapple slices in juice (Abdul Ghani and Farid, 2006), peas in water (Kiziltas *et al.*, 2010), asparagus in brine (Dimou and Yanniotis, 2011) etc.

Furthermore, CFD is an excellent tool to understand transient heat transfer phenomena occurring inside the can. Using CFD analysis, it is easy to find the slowest heating zone inside the can, which can be further used for calculating the process time required for that zone (Abdul Ghani, 2006). CFD simulations have been used to generate the data required for modeling and optimization to predict sterilization time. Abdul Ghani and Farid (2006) studied simulation in the sterilization process of a solid-fluid mixture in a can, and concluded that the position of the food in the can has a large influence on the sterilization times. Varma and Kannan (2005) studied the effect of container shape on the efficiency of the sterilization process and observed that conical shaped vessels pointing upwards was fast to reach appropriate sterilization temperature. Subsequently, they investigated the effect of the geometry and orientation of the conventional cylindrical cans on the rate of thermal sterilization of pseudo plastic food using CFD simulations. In another study, Kannan and Gourisankar Sandaka (2008) provided insight into the thermal sterilization process in relatively larger cans. Dimou *et al.* (2014) studied the effect of can-orientation on peach halves and concluded that horizontal cans exhibited fastest heating rates.

CFD was also found very effective for optimization of heating rates during agitation thermal processing. In recent study, Erdogdu and Tutar (2011) studied the effect of rotation speed and

physical properties computationally, by applying a finite volume method for axial rotation of can. Differences in the heat transfer rates of air–water and air–food material systems are attributed to the development of rotational forces compared to the gravitational buoyancy forces. They observed negative effect of increasing rotation speed on the temperature increase. Tutar and Erdogdu (2012) studied numerical simulation for heat transfer and velocity field characteristics in axially rotating horizontal canned system. They observed two-phase flow patterns significantly influenced by increasing rotational speeds that lead to distinguishable flow patterns in air–liquid interface characteristics. Using this, headspace air bubble movement through the liquid phase and its effect on mixing of liquid particulates has also been modeled (Erdogdu and Tutar, 2011; Tutar and Erdogdu, 2012) and it was found that the size of headspace bubble significantly affected the heat and mass transfer happening in the system.

In most of the studies on CFD, axisymmetric approach was used to reduce the computational time. Abdul Ghani *et al.* (2002) suggested using 3-D approach since the axis-symmetric approach is not possible to apply in all systems. However, 2-D approach is also important and more useful for simpler systems as it reduces the computation time for rapid decision and optimization purposes. Depending upon the geometry and orientation of the can, cans can be classified into bottom heated (where heat transfer from bottom is significant) and side heated (where heat transfer from sides is significant). Considering this, Boz and Erdogdu (2013) studied heat transfer simulation in horizontal cans of liquid products in 2-D and 3-D scenarios to reduce the computation time significantly. Thus, it is seen that numerous CFD studies (Table 2.2) have been undertaken to determine and understand the actual physical phenomena happening inside the can during the heating and cooling processes, which led to the development of more realistic heat transfer correlations between process variables.

# 2.5 Predictive modelling of heat transfer using artificial neural networks (ANN).

Artificial Neural Networks (ANNs) are basically computer models, which are biologically inspired by the function of the human brain. Similar to the brain, ANN models modify themselves constantly during the learning phase from the provided set of input/output variables until they are able to generate satisfactory predictive results. ANNs are very much applicable to complex systems like food because they do not need any prior knowledge about the mechanism of the process and rely solely on the relationship between the input and output data. ANN models can replace missing data

as well as can overcome inconsistencies in data, rendering them more flexible compared to any other empirical models. ANN models can also accommodate multiple-input and multiple-output systems at the same time. Certainly, this makes ANN an excellent choice for use in thermal processing calculation when on line process control and fault corrections are desired (Goncalves *et al.*, 2005). Also, they do not require information about the product's thermo physical properties at all, which can vary in real foods. However, the major disadvantage of ANN models is that it is difficult to extract the process going underneath with regards to the decisions the ANN is making. Also, ANN models are not portable and are specific to the system for which they are made.

The working of ANN is mainly divided into three parts: selection and optimization of the various parameters, training or learning from a set of experimental data and finally, validation from another set or subset of the experimental data. ANN models require data from the experimental setup for which predictions are to be made, and usually these models cannot be extended to predict data for other operating conditions and product properties like shape, density, concentration etc. More details about the working of ANN and procedures involved in the development of a model can be found in literature (Baughman and Liu, 1995). Many researchers have developed predictive models in thermal processing using ANNs. Some of these are discussed to explore the benefits of using artificial intelligence.

Sreekanth *et al.* (1999) developed ANN models for evaluation of surface heat transfer coefficient for tackling inverse heat conduction problems with sphere and a finite cylinder. The error associated with the developed model was almost negligible (< 3 %) with those calculated using conventional numerical/analytical techniques. ANN models were developed to predict thermal process evaluation parameters, g (difference between retort and product temperature at steam-off time) and  $f_h/U$  (ratio of time taken for one log reduction in temperature difference to the required lethality) (Mittal and Zhang, 2002). The parameters g and  $f_h/U$  are used to predict thermal process time and thermal process lethality respectively while using formula methods. The temperature change required for the thermal destruction curve to reduce one log cycle (z), heating lag factor (j<sub>ch</sub>) and  $f_h/U$  were input variables which were used for predicting g, while z, j<sub>ch</sub> and g were inputs for predicting  $f_h/U$ . Data available from previous studies were used as input to ANN model for learning

as well for testing the prediction accuracy. They predicted value of g and  $f_h/U$  with mean absolute error < 2%.

Afaghi *et al.* (2001) developed ANN model to use Stumbo method for thermal process calculations and compared the Stumbo method with the Ball method used earlier by Sreekanth *et al.* (1999). Apart from g and  $f_h/U$ , Stumbo model also requires the value of cooling lag factor (j<sub>cc</sub>). Thus j<sub>cc</sub> was another parameter considered in this model. ANN based Ball models had an average error of 1% for the validation data set while the ANN based Stumbo models had a slightly higher 3% average error for process time and process lethality calculations. The smaller size of data set and a wider range of parameters associated with Stumbo tables were considered to be the reason for the associated higher errors with the ANN based Stumbo models. Afaghi *et al.* (2001) used ANN model to compare the effectiveness of Pham model in addition to Stumbo's method of process calculation. The error associated with the developed ANN models was found to be lower than that predicted using Pham or Stumbo's model. Thus, it is seen that ANN models demonstrate a good potential for application in process calculations with advantages of accuracy, simplicity and even on-line compatibility.

In another work (Sreekanth *et al.*, 2000), the effectiveness of ANN based models in predicting full factorial data from the fractional data corresponding to some of the commonly used experimental designs such as L8, L9, L15, L18, Box and Behnken schemes etc. were evaluated. In general, it was found that ANN assisted L8, Box and Behnken, L15, and L18 designs predicted the full factorial data reasonably well with errors less than 5%.

Chen and Ramaswamy (2002a) studied dynamic modeling of retort processing using two neural network approaches (moving-window and hybrid neural network). Model was developed for modeling cumulated average lethality and average quality retention under constant retort temperature processing. Both neural network models were found significant for describing average lethality and quality retention. However, it has been observed that required number of neurons in the hidden layer and learning runs for training was more in case of moving-window ANN models than the hybrid ANN models.

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Sablani *et al.* (1997b) applied neural network models, trained using data from a finitedifference program, to predict optimum thermal processing conditions i.e. to predict optimal sterilization temperatures, which resulted in least quality degradation. Sablani *et al.* (1997c) compared the ANN models with conventional dimensionless models for modeling fluid to particle heat transfer in agitation processing and found the former to be more accurate and versatile. They developed multi-layer feed forward back propagation neural networks with seven input and two output variables (neurons) for a single particle in a can, and six input and two outputs variables (neuron) for multiple particles in a can.

Dwivedi and Ramaswamy (2010b) developed ANN models for overall heat transfer coefficient and the fluid to particle heat transfer coefficient in canned Newtonian fluids with and without particles, and compared model performances with the dimensionless correlations for free and fixed axial modes of agitation. They calculated average errors in calculation of predicted overall heat transfer coefficient and the fluid to particle heat transfer coefficient as a function of the ANN variables i.e. number of hidden layers, number of neurons in each hidden layer, learning rule, transfer function and number of learning runs. Error values were not significantly different with number of hidden layers between one and three, and minimum with one hidden layer and 8 neurons in each layer. Mean relative errors with ANN models were very low as compared with dimensionless correlations i.e. 75-78% reduction observed for fluid to particle heat transfer coefficient transfer coefficient in figure and 66% for overall heat transfer coefficient U in fixed and free axial mode with particulate in liquid. However without particulates, in comparison with dimensionless correlations, the error for ANN models were 37% lower in end-over-end mode and 76% lower for free axial mode. Overall, ANN models yielded much higher R<sup>2</sup> values than dimensionless correlations.

Chen and Ramaswamy (2002a, 2002b, and 2002c) have developed models for constant retort temperature and variable retort thermal processing using neural networks. Models were developed for process time, average quality retention, surface cook value, equivalent energy consumption, final temperature difference at can center, and lethality ratio. Data for training and testing ANN models were obtained from a FD computer simulation program. ANN-based prediction models were found to be very efficient for describing the relation between input/output variables ( $R^2$ > 0.98). For variable retort processing, they used with ramp time and step temperatures as input in four

consecutive stages of retort processing and concluded that compared to constant retort, variable retort process could improve process time significantly. Similarly, during end-over-end processing of particulates in viscous fluids, Meng and Ramaswamy (2010) conducted studies on neural network modeling in a pilot-scale rotary retort. A portion of experimental data obtained for the associated heat transfer coefficients were used for training while the rest were used for testing. The optimum configuration parameters for the Newtonian fluids were two hidden layers, five neurons in each hidden layer, the delta learning rule, a sine transfer function and 40,000 learning runs, while for the non-Newtonian fluids, one hidden layer, six neurons in each hidden layer, the delta learning rule, a hyperbolic tangent transfer function and 50,000 learning runs ware obtained. The developed ANN models were significantly better than those obtained from the dimensionless correlations. They also included algebraic solutions in their results, which could be used to predict the heat transfer coefficients without requiring an ANN.

# 2.6 Types of agitation retorts

Agitation in the cans can be achieved by either rotation (end over end, free axial, fixed axial) or by lateral motion (Shaka system). Agitating retorts have many advantages over still retorts most important of all is that, they offer better and fast heat transfer to the food products which ensures better quality retention during the processing as the time required in processing assisted with agitation is lower than the time required for processing in still mode.

# 2.6.1 Rotary agitation

In industries there are 3 different types of rotary agitations are used viz. end-over-end, axial and bi-axial mode of can rotation. In end-over-end rotation, the cans are held in a vertical position in the cage and rotated in such a way that the head space bubble moves in longitudinal direction and causes mixing of can contents as shown in Figure (2.3a). This type of agitation was first used by Clifcorn *et al.* (1950) in order to find methods to improve heat transfer to canned food products. In fixed axial mode, the cans are held horizontally in the cage and rotated as it can be seen in Figure (2.3b). This mode of rotation is not common in the industries because of its minimal advantages

compared to other modes of agitation. The bi-axial (Figure 2.3c) mode of rotation consists of two phases of rotation of the cans which are mutually in opposite directions.



Figure 2.3: Different modes of agitation in a retort (Rattan, 2012)

## 2.6.2 Shaka system

The Shaka retorts are a relatively new technology and still in the development phase. This system actually uses reciprocating agitation in addition to gravity and hence, accelerates the mixing inside the cans (Walden, 2010). This system facilitates the rapid back and forth motion of can held containers to shorten total heat treatment times and increase the quality of the final products with less energy consumption. It has been claimed by Walden and Emanuel (2010) that the Shaka process can lower the thermal exposure time almost 20-fold compared to a still process and 10-fold compared to a rotary process. This happens because Shaka process can enhance the heat transfer higher than what is possible in rotary agitation. However, Shaka process can only provide processing in batches and lacks continuity of process which can be easily achieved in rotary retorts (Dwivedi, 2008) such as end over end & axial agitation retorts. Also, there is lack of scientific studies available which can describe the effects of the Shaka process on the quality of real food.

#### 2.7 Factors affecting heat transfer in agitating retorts

There is a great potential in thermal processing via agitation. There is an enhanced heat transfer rate to both liquid and particles, leading to improvement in quality retention and reduction of processing time. From previous studies, it was discovered that the most relevant factors effecting heat transfer rates to liquid particulate canned foods processed under agitation are the mode of

agitation, the agitation speed, headspace, fluid viscosity, particle size, particle properties, and particle concentration (Lenz and Lund, 1978; Hassan, 1984; Lekwauwa and Hayakawa, 1986; Deniston *et al.*, 1987; Fernandez *et al.*, 1988; Stoforos and Merson, 1991, Sablani and Ramaswamy, 1995, 1996, 1997, 1999). The following parameters are very important in studies on agitation.

#### 2.7.1 System related parameters

The system related parameters such as operating temperature, rotation speed, fluid and particle characteristics plays an important for estimating the heat transfer rates of agitating retorts. The heat transfer and the lethality of canned liquid foods containing particles processed in Steritort (reel type retort-axial rotation) have been studied by Lenz and Lund (1978), and it was found that changing the reel speed from 3.5 to 8 rpm resulted in an average increase in  $h_{fp}$  by 150 W/(m<sup>2</sup>°C). Hassan (1984) measured the convective heat transfer coefficients of all three types of particles, and found that varying the can speed from 9.3 to 101 rpm had more of an effect on U than on  $h_{fp}$ . Stoforos (1988) explained the effect of rotational speed on  $h_{fp}$  when he reported that at high speeds (100 rpm) his canned contents behaved as a solid mass and therefore provided little agitation inside the can, and further explained that revolutions per minute would affect h<sub>fp</sub> so long as the increase affected the relative particle to fluid velocity. Stoforos and Merson (1992) showed that by increasing the rotational speed, the U and h<sub>fp</sub> values could be increased as well. Sablani (1996) observed that when the rotational speed increased from 10 to 20 rpm, on average, hfp values increased by 56% for oil and 53% for water, and the U value increased by 24% for oil and 13% for water. Meng (2006) reported that while using canned particles/Newtonian fluids, the effect of rotational speed was significant, and the associated hap (Apparent fluid-to-particle heat transfer coefficient) values increased as well. Dwivedi (2008) reported in his study of canned Newtonian (glycerine: 80%-100%) particulate food that U and h<sub>fp</sub> in free axial mode increased from 448 to 907 and 477 to 1075 W/ ( $m^{2\circ}C$ ), respectively, with the increase in revolutions per minute from 4 to 24.

Further, Lenz and Lund (1978) found that water had higher values for both U and  $h_{fp}$  than a 60% aqueous sucrose solution, indicating a role of fluid viscosity in the values of U and  $h_{fp}$ . Later, Hassan (1984) found that increasing the fluid viscosity decreased the overall heat transfer coefficient, and that the same was true for the particle–liquid film heat transfer coefficients, except in

the case of large aluminum spheres (3.17 cm diameter), processed with particle volume fraction ( $\epsilon$ = 0.21). Sablani (1996) studied U and h<sub>fp</sub> values with nylon particles in water and oil, and found that larger h<sub>fp</sub> and U values were obtained from water. He also found that the U and h<sub>fp</sub> values improved with the decrease in liquid viscosity. Meng (2006) computed the heat transfer coefficients and found that the associated h<sub>fp</sub> values ranged from 215 to 376 W/(m<sup>2</sup>K) and U values ranged from 112 to 293 W/(m<sup>2</sup>K). The U values decreased with an increase of the liquid viscosity, which could be explained by the thickness of the associated boundary layer around the particle and inside the surface of the can in higher viscosity fluids. Dwivedi (2008) reported that with an increase in glycerine concentration, it was observed that U and h<sub>fp</sub> decreased by 26% and 40%, respectively. They attributed the decrease to the larger thickness of the associated boundary layer in the higher viscosity liquids.

Furthermore, the mode of agitation is also a major factor that affects the heat transfer. The effect is generally the most predominant at the early stages of the heating where temperature gradients are higher. Tsurkerman *et al.* (1971) reported that maximum heat transfer rates were obtained when there was a 45° angle for the axis of rotation. Under the intermittent agitation, when the direction of rotation was reversed every 15–45 s, Hotani and Mihori (1983) reported that heating rates and uniformity were increased and that there was no significant difference between EOE & axial rotation. Using canned Newtonian liquid, Dwivedi (2008) reported that the overall heat transfer coefficient (U) was significantly higher in the case of free axial mode (340 W/m<sup>2</sup>K) than in the EOE mode (253 W/m<sup>2</sup>K), which was contrary to what Hotani and Mihori (1983) found out.

### 2.7.2 Product related parameters

Particle to fluid concentration, particle density, shape and size are four main factors which are studied by various researchers. The fraction of total effective can volume occupied by the particles is defined as the ratio of the particle volume to the sum of the particle and liquid volume, and can be expressed as  $\varepsilon$ . Hassan (1984) and Deniston *et al.* (1987) separately studied the effects of  $\varepsilon$  on the heat transfer rates and found that for  $\varepsilon$  at 0.107, the average U value for triplicate experiments was 1300 W/(m<sup>2</sup>K). U<sub>o</sub> exhibited a maximum value of 1640 W/m<sup>2</sup>K for  $\varepsilon = 0.400$  and subsequently U declined, having value of 1360 W/m<sup>2</sup>K at  $\varepsilon = 0.506$ . Deniston *et al.* (1987) summarized that since the spheres were tightly packed in the can at high  $\varepsilon$  values, the particles were not free to move and the functional dependency of U and h<sub>fp</sub> probably differed compared to loosely packed particles. Sablani and Ramaswamy (1997) found that U increased 20% for oil and 5% for water at 29% concentration when compared to a single particle, but a further increase in concentration to 40% resulted in a decrease of h<sub>fp</sub> by 12% for oil and 7% for water. Meng (2006) found that with a rotational speed of 15 rpm, headspace of 9% (v/v), retort temperature of 120°C, and a rotation radius of 120 mm in 0.9% CMC concentration, particle concentration had significant effect on U values (p < 0.05). Dwivedi (2008) also reported that the particle concentration has a significant influence (p < 0.05) on the heat transfer coefficients U and h<sub>fp</sub>. For a particle size of 25 mm, U increased with particle concentration ranging from 20% to 30%, while further increasing the concentration to 40% would decrease U and h<sub>fp</sub>. This phenomenon is explained by the fact that the presence of particulates inside the canned liquid affects the heat transfer coefficient and causes secondary agitation of the liquid. This results in better mixing and distribution of heat inside the can, leading to increase in U and h<sub>fp</sub> values. However, if the concentration increases too much, the mixing in liquid is tremendously decreased, leading to subsequent decline.

Hassan (1984) found that the increase in the diameter of potato spheres processed in water from 2.22 to 3.49 cm, resulted in greater process times for fluid and particles. He was also able to determine that for the larger particles, the effect of rotational speed on the particle heating times was higher than for smaller particles, and found similar results when he used Teflon and aluminum particles processed in water or silicon liquids. The overall heat transfer coefficient, however, was reported to have the highest values at all rotational speeds when particles of an intermediate size (2.86 cm) were used. The data collected by Deniston (1984) showed that  $h_{fp}$  was not greatly influenced by increasing particle size. Lenz and Lund (1978) reported that increasing particle size resulted in higher overall heat transfer coefficients, but Sablani and Ramaswamy (1997) found that U values decreased by about 9% in oil and 6% in water as the size of nylon particle increased from 19.05 to 25 mm diameter, and concluded that particle size influenced more  $h_{fp}$  than it did U. Meng (2006) reported no significant effect of particle size on Ua and  $h_{ap}$ . Dwivedi (2008) reported that particle size had a significant effect on U and  $h_{fp}$ . The U values in free axial mode for the three concentrations observed (20%, 30%, and 40%) were found to decrease by 33%, 25%, and 29% as the size of the particles increased from 19.05 to 25 mm. Under the same increasing size conditions,  $h_{fp}$  also decreased, probably due to the thicker boundary layers associated with larger particle diameters. It can be seen that the increasing diameter had a greater influence on  $h_{fp}$  than on U ( $h_{fp}$  decreased by 32%, 34%, and 41% as the size went from 19.05 to 25 mm in the three concentrations).

Ramaswamy and Sablani (1997b) used nylon particles of various shapes such as cylinders, cubes, and spheres, and found that when there were multiple particles inside the can, the U values for the cube-shaped particles were about 6% lower than those of the cylinder, and 6% lower than that of the spheres. When studied in oil, the average U value of the cubes became 3% lower than the cylinders and 6% lower than the sphere-shaped particles. This was because different shapes created different void areas between the particles, allowing for different levels of mixing taking place inside the can.  $h_{fp}$  was determined to be affected by shape as well: in experiments conducted by Ramaswamy and Sablani (1997a) using multiple particles submerged in oil,  $h_{fp}$  increased by 6% with cubes compared to that of a cylinder which in turn increased by 20% compared to that of sphere, and the influence of shape was even more noticeable in water. They explained this in terms of more disturbances in the flow field near the surface of the cylinder and the cube shaped particles.

Particle density can affect the particle fluid motion pattern inside the can, thereby affecting the heat transfer coefficient. When using a single particle inside a can, Sablani (1996) found a significant effect of density on the  $h_{fp}$  value. He noted that particles of greater density settled in the can faster, resulting in more motion inside the can due to the higher particle/liquid relative velocity and creating a higher  $h_{fp}$ . When the particles with densities close to each other (nylon and acrylic) had similar  $h_{fp}$  values in both oil and water, he concluded that particle thermal properties did not influence  $h_{fp}$ . Meng (2006) reported that the  $h_{fp}$  values increased with the increase in the particle density. Dwivedi (2008) studied the influence of particle density on U and  $h_{fp}$  in free axial mode and found that at 20% particle concentration, polypropylene particles are shown to have the lowest  $h_{fp}$  (495 W/m<sup>2</sup> °C) and Teflon particles the highest (922 W/m<sup>2</sup> °C), while nylon particles fell in between with  $h_{fp}$  of 764 W/(m<sup>2</sup> °C).

#### 2.8 Optimization in thermal processing

Thermal processing is one of the most complex systems to study optimization. One has to consider the efficiency of thermal treatment in terms of microbial kill and product quality deterioration simultaneously, due to the application of heat. Predictive models, computational techniques and CFD simulations are developed to model food systems for satisfaction of variety of thermal processing objectives like selection of optimum processing condition, maximizing product quality etc. Traditionally, thermal processing optimization problems are solved in two steps. First, one is to develop the objective function of the problem using mathematical approaches, regression methods, theoretical analysis and differential equations; and second is, using an appropriate search method for finding optimum values of different variables. It is a complex task to develop a comprehensive prediction model which can cover all variables simultaneously over the available ranges of variables. The diversity of thermal processing objectives impose different optimal requirements to sterilization processing, which can be determined by analytical or numerical procedures and/or based on sophisticated software (Abakarov and Nuñez, 2013). Also, a number of methods has been developed for the prediction of the quality and microorganism degradation for constant temperature thermal processing. Due to development of artificial intelligence techniques, modeling and optimization becomes easy, especially in situations involving nonlinear and complicated mathematical approaches as compared with conventional methods.

During the last four decades various optimization techniques have been carried out to optimize the quality and safety of thermal processes (Erdogdu, 2008a). Holdsworth (1985) has presented a general review of mathematical models for optimizing the effects of heat processing food products. These studies were focused on microbial destruction, nutrient destruction, cooking value and loss of quality. Durance (1997) has also investigated about optimization procedures adopted for improving food quality during variable retort temperature canning. Banga *et al.* (2003) presented the computer-aided optimization techniques used to improve food processing, in context of recent developments using modern optimization techniques. Their potential for industrial applications was discussed with use of several important examples. Finally, future trends and research needs are outlined in the study as well. Erdogdu (2008b) has evaluated the potential uses and the limitations of optimization techniques for food processing, including conventional methods, artificial intelligence - genetic algorithms based methods, multi-objective optimization procedures, and computational fluid dynamics based techniques.

Teixeira *et al.* (1969) were the first who developed FD model for thermal processing optimization in the conduction heating foods. Since then, several other researchers have carried similar studies in this area (Sastry *et al.*, 1985). The search methods for optimization were described by Saguy (1983), who evaluated optimum temperature-time profiles using the maximum principle. Edgar and Himmelblau (2001) have suggested that nonlinear programming is far better in terms of efficiency as compared to the maximum principle for many variables problems. Banga *et al.* (1991) developed an optimization algorithm, Integrated Control Random Search (ICRS/DS) for variable retort thermal processing and tested these algorithms for maximizing the overall nutrient retention and quality factor at the surface of the food, and for minimizing process time. This model has later been used by various researchers (Balsa-Canto *et al.*, 2002) for solving various optimization problems. Apart from this, various MATLAB based sub-routines (Balsa-Canto *et al.*, 2002; Ansorena and Salvadori, 2011; Alonso *et al.*, 2013) are also very popular.

On line predictive control in processing of canned foods is gaining popularity these days. Optimization routines have been efficiently applied to achieve this objective for obtaining better quality products. Chalabi et al. (1999) proposed the on-line implementation of an optimal control solution for thermal sterilization. However, the controller only made use of the heat transfer model for the product without consideration of the dynamics of the retort. Similar problems were considered by Balsa-Canto et al. (2002) to explore the potentialities in dynamic optimization of reduced order models based on POD (Proper Orthogonal Decomposition) expansions of the product heat transfer equation. More recently, optimal control problems for thermal sterilization oriented to quality maximization have been undertaken in the context of multi-objective optimization (Erdogdu and Balaban, 2003; Abakarov et al., 2009; Sendin et al., 2010), local and global optimization algorithms (Chen and Ramaswamy, 2002b, 2002c; Miri et al., 2008; Ansorena and Salvadori, 2011; Enitan and Adeyemo, 2011), and adaptive search techniques (Miri et al., 2008; Simpson and Abakarov, 2011). From a control point of view, advanced model-based predictive control methods (MPC) are the appropriate framework capable of producing optimal temperature and pressure profiles on an uncertain environment (Banga et al., 2008). Alonso et al. (2013) have presented a real time optimization method for quality control of batch thermal sterilization of prepackaged foods.

Apart from numerical optimization techniques, evolutionary algorithms coupled with neural networks have also been utilized for optimization of thermal processes in the form of genetic algorithms based approaches (Enitan and Adeyemo, 2011). However, very limited work has been done using GA as an optimization technique in the field of thermal processing. This is mainly because there is no absolute assurance that a genetic algorithm will find a global optimum. It happens very often when the population is very large, which is a reality in thermal processing of liquid particulates. However, there are techniques available (ex. combination of genetic algorithm with another search method) which assure global minima for a large number of scenarios. This unfortunate genetic algorithm property limits the genetic algorithms' use in real time applications. Further, like other artificial intelligence techniques, the GA cannot assure constant optimization response times. Therefore, it is unreasonable to use genetic algorithms for on-line control in real systems without testing them first on a simulation model. Yet, one of the very useful study for CRT (constant retort temperature) thermal processing optimization was carried out by Chen and Ramaswamy (2002b), who evaluated the application of genetic algorithms for optimization of constant retort temperature (CRT) thermal processing. In their study they coupled the ANN models and GA to search for optimal quality retention and corresponding retort temperature, and investigated the effects of main processing condition parameters on both optimal quality retention and retort temperature. In a continuation of their research (Chen and Ramaswamy, 2002c) they selected their objective to optimize variable retort thermal process using GA approach. The objective of their study was to develop ANN-GA based procedures for the selection of the optimal retort temperature profile under multistage ramp-variable (MRV) retort temperature control for optimizing thermal processing. They used ANN for developing dynamic prediction models for process time, average quality retention and surface cook value. GA-ANN based optimization procedure has also been used for searching the best combination of retort ramp sequence to give a continuous variable temperature profile that achieves the optimization objective under imposed constraints conditions (Chen and Ramaswamy, 2004). These works demonstrate that despite the limited number of studies, ANN and ANN-GA based models have the potential to be developed and used extensively as a possible algorithm for process optimization during thermal processing.

# 2.9 Quality changes induced during thermal processing

The nutritional components all together with sensory attributes, contributes to the final quality of processed foods. The main objective of thermal processing is to minimize the quality losses, while providing the sufficient thermal treatment required. The term quality is basically referred to the characteristics of foods that play an important role in acceptability of that product such as color, texture and flavour. Quality in general is a relative term and has no dimensions and constraints (Rattan and Ramaswamy, 2014a, 2014b). However, it can be evaluated by sensory analysis or by using various analytical instruments. Effects of thermal processing applications, our primary focus was only to destroy the spoilage causing microorganisms and quality concerns were not given much importance until recently when consumer awareness and desire for good quality food increased.

Thermal degradation of food quality due to heat exposure is widely studied by various researchers (Stumbo, 1973; Leonard *et al.*, 1986; Holdsworth, 1997). The food industry is seeking alternatives to minimize quality deterioration of thermally processed foods in wake of increasing consumer demands for high quality products (Awuah *et al.*, 2007). It has been also shown that the susceptibility of microorganisms to thermal destruction is much more than that of enzymes and quality attributes. This difference in susceptibility is a key to develop and optimize the thermal process which can assure high quality retention without compromising commercial sterility of the food product (Leonard *et al.*, 1986). Quality factor degradation follows first order degradation kinetics just similar to microbial destruction. The quality factor determination is needed so as to model the quality factor changes that are obtained from analytical methods.

#### 2.9.1 Color

Color is a primary factor that influences consumers' buying behavior (Rao *et al.*, 1985). Heat treatment destroys pigments present in vegetables, such as chlorophyll, xanthophyll, carotenoids, and lycopene and produces changes in color (Abbatemarco and Ramaswamy, 1994; Shin and Bhowmik, 1995). Green vegetables are most susceptible to color loss and they turn bright green to olive brown

(chlorophyll is converted to pheophytin) depending on the severity of heat treatment due the conversion of chlorophyll through a series of reactions (Woolfe, 1979). Furthermore, any change in food color as a result of heat treatment may be induced by some reactions such as degradation, oxidation and/or Maillard reaction (Krokida *et al.*, 1998; Baini and Langrish, 2009). In food stuffs in which the color is due to pigments such as carotenoids and anthocyanins, it is observed that color degradation by thermal treatments is less as compared to green colored foods (Clydesdale, 1991). In case of anthocyanins it is found that they break down by pH and oxygen changes and in anaerobic conditions the pH effect also becomes very small hence, the color changes are less (Clydesdale, 1991).

To measure color, instrumental methods are used based on the fact that color is a mathematical combination of three primary colors viz. red, green and blue (Clydesdale, 1991). Tristimulus colorimeters, Hunter L\*a\*b and CIE L\*a\*b are some of the instruments used to quantify the color in mathematical form. A mathematical representation of color makes it possible to compare the color of different foods on the same scale. In food industries a Hunter L\*a\*b model is used for analyzing color changes. Where L\* depicts the lightness, a\*depicts greenness and redness and b\* depicts blueness and yellowness of a sample. The total color change can then be estimated by using parameter  $\Delta E$  which is mathematically evaluated from the equation:

$$\Delta E = \left[ (\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2 \right]^{0.5}$$
(2.18)

#### 2.9.2 Texture

Apart from color change, texture softening occurs during processing of some vegetables, such as potatoes, radish, peas, beans etc. (Huang and Bourne, 1983). Although this is advantageous in some case as they can be consumed easily and can have lower cooking times, texture changes in most fresh vegetables are undesirable and affect consumer acceptance (Alvarez *et al.*, 2001). Thermal processing of vegetables degrades the pectin substances present in cell wall and interlamellar region and results in tissue softening. Lund (1982) described that thermal softening can be attributed to two main factors. First, is due to changes in cell wall matrix polysaccharides, which depend on many factors like pH, types and amounts of various salts present in the plant cell wall. As

an example, presence of calcium chloride can result in harder texture of the food and water uptake by polysaccharides results in reduction of cohesiveness of cell wall matrix thereby resulting in lower adhesion. Second reason for softening is the loss of turgor pressure which is nothing but pressure of cell components against the cell wall (Rao and Lund, 1986). Previous studies show that the extent of softening varies for different foods if they are subjected to similar treatments due to difference in their compositions (Rao and Lund, 1986). The textural changes can be analyzed by using either sensory evaluation or by instrumental methods. Szczesniak and Hall (1975) first developed the instrumental texture profile analysis using computer assisted texture analyzer. With advent of computer technology these methods have become very common. The texture analyzer generates texture profile by compressing/cutting a bite sized food particle, which basically simulates the mastication process of humans to give force *vs* time curves as a function of deformation. Using texture profile analysis, texture parameters such as hardness, firmness, toughness, springiness, gumminess, adhesiveness, chewiness etc. can be evaluated from texture profile generated using the computer assisted texture meters.

# 2.9.3 Antioxidants

Antioxidants present in vegetables (glutathione, ascorbic acid,  $\beta$ -carotene, a-tocopherol, and other flavonoids) are also sone of the most important quality parameters to be considered when subjected to thermal processing (Larson, 1988). Thermal processing at high temperature is generally believed to be responsible for depletion of naturally occurring antioxidants in processed vegetable (Nicoli *et al.*, 1999). Effect of thermal processing on antioxidant activity has been extensively investigated by various researchers (Hunter and Fletcher, 2002; Turkmen *et al.*, 2005; Delchier *et al.*, 2012). Antioxidant compounds are susceptible to high temperature, although few studies have showed that a less severe heat treatment has some beneficial effect on increasing the antioxidant activity. Patras *et al.* (2009) showed that tomato puree subjected to thermal treatments (70 °C for 2 min) had significantly higher antioxidant activity (p < 0.05) than in untreated samples. Similarly Dewanto *et al.* (2002) showed that the antioxidant activity of raw tomatoes was 4.13 µmol vitamin C equiv/g of tomato which was further increased to 5.29 µmol of vitamin C equiv/g of tomato after thermal treatment at 88 °C for 2-30 min.

# 2.10 Conclusions

Analysis of the heat transfer associated with thermal processing of liquid particulate mixtures is a complex phenomenon. The governing heat transfer coefficients (U and h<sub>fp</sub>) depend on several factors and when investigating a new process those factors need to be critically examined. Various computational techniques have been extensively employed for evaluation of heat transfer coefficients and for better understanding of the process. Computational techniques used in thermal processing can be categorized into three categories, viz. a.) Mathematical models for heat transfer modeling of the process, b.) Predictive models for development of correlations to predict the process and c.) Optimization procedures to predict optimal processing conditions for various objective functions (using mathematical and evolutionary search algorithms). While, analytical and numerical modeling techniques have been extensively employed for evaluation of heat transfer coefficients and for better understanding of the process, for predictive modeling of data, neural network models can be effectively used by correlating the experimental results to develop predictive models. Although, many techniques have been used in each of these categories, yet there is still a huge scope of development of these domains. Moreover, consumer demands for high-quality products have inspired researchers and the food processing industries to explore alternative methods for innovative and promising techniques for providing high quality foods. Hence, it is necessary to make intelligent use of all available mathematical techniques to predict and develop a safe thermal process yielding high quality shelf stable food products. Each processing technique is unique in itself and development of new systems, like reciprocating agitation, requires additional heat transfer and quality studies. Although, new processing concepts such as vigorous agitation using reciprocating agitation have potential to provide both the economy and quality of thermally processed foods yet, extensive validation and verification, optimization, accuracy, cost effectiveness and overall quality, are some of the key elements that will justify the adoption of the developed system.

#### **PREFACE TO CHAPTER 3**

For proper investigation of the heat transfer phenomenon prevalent during reciprocating agitation thermal processing, knowledge of heat transfer coefficients (U and  $h_{fp}$ ) is needed. Although, several studies (Dwivedi, 2008; Meng, 2006; Garrote et al., 2006b) have investigated heat transfer in other modes of processing (static, end-over-end, axial, biaxial etc.), no such work has been done for the reciprocating mode of agitation. High intensity agitation induced during reciprocation necessitates improvement in the application of conventional methodologies for U and  $h_{fp}$  evaluation. Hence, the work was undertaken to develop a suitable methodology for calculating U and  $h_{fp}$  under these atypical conditions.

The conventional methodology was refined for calculating U and  $h_{fp}$  from heat penetration parameters of the collected time-temperature profile. This enabled calculation of U and  $h_{fp}$  using the heating rate indices of the liquid and cold-spot temperature profiles. The refined methodology was compared with the conventional methodology for Newtonian and non-Newtonian fluids. This work was useful in the calculation of U and  $h_{fp}$  to study heat transfer aspects in further chapters in this study.

Parts of this chapter won the second prize in **Charles Stumbo Graduate Student Paper Competition** and a poster presentation was made in the Annual Meeting of the Institute for Thermal Processing Specialists (IFTPS) held on Mar 11-14, 2014 in Orlando, Florida.

Parts of this chapter have been presented in form of poster presentation at Institute of Food Technologists (IFT) Annual Conference held from 21-24 June'2014 at New Orleans, Louisiana.

Parts of this chapter have been published as an original research article entitled "A refined methodology for evaluation of heat transfer coefficients in canned particulate fluids under rapid heating conditions" in Food and Bioproducts Processing, 94, 169-179.

The experimental work and data analysis was carried out by the candidate under the supervision of Dr. H. S. Ramaswamy.

# Chapter 3. DEVELOPMENT OF METHODOLOGY FOR EVALUATION OF HEAT TRANSFER COEFFICIENTS DURING RECIPROCATING AGITATION THERMAL PROCESSING

# 3.1 Abstract

The existing methodology was refined for calculating the heat transfer coefficients (U and  $h_{fp}$ ) under rapid heating conditions during reciprocating agitation thermal processing. The conventional methodology of  $h_{fp}$  evaluation became unreliable and/or non-reproducible under rapid heating conditions such as those encountered during reciprocating agitation thermal processing. This was due to fluctuations in gathered transient temperatures during rapid reciprocating motion and due to very short equilibration times (EQT) (heat-up time to 1°C below operating temperature). In this method, U and  $h_{fp}$  were calculated using a temperature profile predicted by using an average retort temperature (after the come-up period) and heat penetration parameters, instead of numerous data points from the experimental temperature profile used in the conventional methodology. The method was validated for various reciprocation frequencies (0-3 Hz) and amplitudes (0-20 cm). Results revealed that the refined methodology was robust and produced reliable and consistent values of U and  $h_{fp}$ , under both slow and rapid heating conditions. Also, the heat transfer coefficient values, using the proposed method, consistently matched the results and trends observed with conventional methods. A parameter sensitivity analysis was also conducted and the effects of perturbations of different parameters employed in the refined methodology were reported.

#### 3.2 Introduction

During thermal processing of liquid-particulate canned foods, it is essential for the cold-spot (e.g. particle-center in conduction-heated foods) to achieve the minimum process lethality ( $F_o$ ) required for destroying or inactivating the microorganisms of health or spoilage concern. Thermal processing is usually applied on the basis of a predetermined minimum heat treatment necessary to assure a specified minimum  $F_o$  value. In order to properly design and successfully optimize a thermal process, it is essential to devise an appropriate methodology to accurately predict heat transfer rates so that reliable estimates of particle-center temperatures and resulting lethality could be made. This is needed to establish the optimum processing conditions (process time and operating

temperature). For canned particulate liquid foods, these heat transfer rates are expressed by means of an overall heat transfer coefficient U (the heat transfer coefficient between retort and liquid across the can wall), and a fluid-to-particle heat transfer coefficient  $h_{fp}$  (the heat transfer coefficient between liquid and particle surface).

Majority of methods used for prediction of heat transfer coefficients involve only canned liquid foods and hence, evaluate only U. Rao and Anantheswaran (1988), in their review, detailed various methodologies for evaluation of U in liquid foods. Initial studies on heat transfer in particulate foods involved the use of a low-Biot number scenario (Bi < 0.01) and thereby the use of a lumped capacity approach for calculating h<sub>fp</sub> (Lenz and Lund, 1978; Fernandez et al., 1988). Ramaswamy et al. (1982) developed an analytical methodology to measure surface heat transfer coefficients for heat transfer from a fluid to a solid. Using this method, Awuah et al. (1995) proposed a methodology to estimate h<sub>fp</sub> by fitting the values of the heating rate index (fh) of the experimental particle and those obtained from the analytical solution. They compared it with another approach involving comparison of temperature gradient ratios and found that the heating rate index approach gave more consistent and conservative results. Stoforos and Merson (1990) estimated heat transfer coefficients in a liquid-particulate mixture by only considering the liquid temperatures and ignoring the particle-temperatures. Sablani and Ramaswamy (1995, 1996) evaluated U and hfp during agitation thermal processing by collecting particle-center temperatures using flexible-thin wire thermocouples during end-over-end mode of rotation. Sablani and Ramaswamy (1995, 1996) calculated h<sub>fp</sub> by minimizing the difference between the experimental temperature profile and predictions from a numerical solution using a finite difference computer simulation program. The values of U were then calculated using a heat balance approach across the can-liquid and liquidparticle interface. Using similar concepts, Balasubramaniam and Sastry (1995) calculated hfp in a non-Newtonian fluid during continuous flow. Meng and Ramaswamy (2005) evaluated the heat transfer in canned particulate non-Newtonian fluid for end-over-end processes and found that for high viscous non-Newtonian fluids, due to the larger temperature gradients in the liquid, the methodology used by Sablani and Ramaswamy (1996) failed. Hence, Meng and Ramaswamy (2005) introduced an apparent overall heat transfer coefficient Ua and apparent fluid-to-particle heat transfer coefficient, hap. While U and h<sub>fp</sub> were based on the transient temperature difference between the canned fluid and the particle, Ua and hap were based on the temperature difference between the heating medium and the particle. This methodology showed better agreement between calculated and estimated lethality. Garrote *et al.* (2006b) also used a slight modification of the conventional methodology used by Sablani and Ramaswamy (1996) by using Laplace transform to solve the differential equation and compared this with lumped parameter methodology. Garrote *et al.* (2006b) found that U values were higher for the conventional method which considered variation of retort temperature during come up time. Cariño-Sarabia and Vélez-Ruiz (2013) compared the solutions of the heat transfer equations using a lumped parameter and an analytical approach for Newtonian and non-Newtonian fluids. Cariño-Sarabia and Vélez-Ruiz (2013) found that h<sub>fp</sub> was higher for heating in Newtonian fluids and concluded that the lumped parameter approach is easier and faster, but when an accurate evaluation of h<sub>fp</sub> is needed, the analytical approach should be utilized.

Thus, several studies are available in literature analyzing heat transfer mechanisms in liquid/particulate food for different rotary processing scenarios. All these methodologies rely on experimental data from the particle-surface or particle center. Under rapid heating conditions, when the Biot number increases beyond 10, small inaccuracies in the experimental temperature profiles result in large deviations in the value of U and h<sub>fp</sub> (Ramaswamy and Sablani, 1997a). These rapid heating conditions are desirable for reduction of process time and improvement in product quality as they help in increasing heat transfer rates and reduce the time required for cold-spot to reach operating temperature (Dwivedi and Ramaswamy, 2010a). For rotary processing, biaxial mode of agitation has been found to provide the most rapid heating condition (Dwivedi and Ramaswamy, 2010b). For this mode of agitation, Dwivedi and Ramaswamy (2010a) developed a new empirical methodology for measuring the U and h<sub>fp</sub> in canned liquid/particle mixtures. They hypothesized that under similar processing conditions, h<sub>fb</sub>/U quotient would remain the same between free-axial and fixed axial mode of heat transfer. To solve this problem, the h<sub>fb</sub>/U quotient value was obtained from the fixed axial mode using conventional methodology (Sablani and Ramaswamy, 1996). Fluid to particle heat transfer coefficient ( $h_{fp}$ ) in biaxial mode was then calculated by multiplying  $h_{fp}/U$ quotient with the U value obtained from lumped capacity approach for the free axial mode. However, with further developments in the field of retort technology and with the introduction of reciprocating agitations, the heat transfer rates have tremendously improved. By enhancing agitation,

reciprocation of containers can effectively provide very rapid heat transfer conditions (Walden and Emanuel, 2010). With increase in heat transfer, even small deviations in the retort temperature profiles, especially during the come up time (CUT), can considerably affect the values of U and  $h_{fp}$  calculated by previous methodologies. Therefore, a suitable method capable to predict U and  $h_{fp}$  under these rapid heating conditions in a reliable and reproducible manner is required.

The objective of this chapter was to develop a robust methodology for evaluation of U and  $h_{fp}$  during reciprocating container agitation thermal processing. For this, the conventional methodology (Sablani and Ramaswamy, 1996) was refined by fitting 'simulated' profiles (from numerical simulation of heat transfer) with 'predicted' profiles (using average operating temperature and heating rate indices), instead of actual 'experimental' profiles. The refined methodology was compared with the conventional methodology and a sensitivity analysis was conducted to study the effect of perturbations in different parameters on the values of U and  $h_{fp}$ .

### 3.3 Materials and methods

#### 3.3.1 Lab-scale reciprocating steam retort

A lab-scale steam retort (Figure 3.1) was used in this study. The retort was earlier modified to include a reciprocating cage (Singh *et al.*, 2015b), which could move back and forth on horizontal rails. The cage was designed to hold 4 vertical cans (307x409) along the diameter of the retort. It was connected to a slider crank assembly through a reciprocating rod inserted into the retort. This rod was connected to the rotating shaft on the slider crank assembly through a crank. The slider crank assembly was powered by a  $\frac{1}{2}$  hp direct current magnetic motor. The speed of rotation of the motor was controlled through an external voltage controller. In order to pivot the crank on the rotating shaft, fifteen different positions were provided, through which amplitude of reciprocation could be varied. Thus, both speed and amplitude of reciprocations could be controlled. The schematic of the modified retort is shown in Figure 3.2. Further details about modification mechanisms and voltage calibration are described in Singh *et al.* (2015b).


Figure 3.1a: Static Steam Retort



Figure 3.1b: Reciprocating Cage



Figure 3.1c: Reciprocating Cage

Figure 3.1: Modified Reciprocating retort (Singh *et al.*, 2015b)



Figure 3.2: A schematic representation of the vertical steam reciprocating retort (Singh et al., 2015b)

# 3.3.2 Test particulates and fluid

A food model system consisting of liquid and particles was used. Using real food introduces uncertainties in the thermo physical property data as they can vary widely across a temperature range. Thus, nylon spheres were used as food simulating particles (rather than real food), as nylon has thermal properties most relevant to the food, and its thermal properties do not change with temperature (Sablani and Ramaswamy, 1996). Spherical Nylon particles (Small Parts Inc., Miami, FL), 19 mm in diameter, were used as test particulates. Water was used to simulate a low-viscous Newtonian fluid, while pure glycerin (Fischer Scientific, Montreal, PQ) was employed to simulate a high viscous Newtonian fluid. Moreover, a 1% (w/w) solution of Carboxymethyl cellulose (CMC) in water was used to simulate a Non-Newtonian fluid. The CMC solution was prepared by dissolving the required weight of commercial grade CMC (Sigma, St. Louis, MO) in distilled water with simultaneous stirring and leaving it for 24 hours for complete dispersion of the lumps and elimination of dissolved air (Meng and Ramaswamy, 2007). The densities of glycerin and CMC solution were determined from the mass of a known volume of the solution. Thermal properties of water, glycerin and CMC solutions at different concentrations were taken from the literature available (Meng, 2006), and summarized in Table 3.1. Cans of nominal size 307 x 409 (87.3 mm diameter x 115.9 mm height) (Home Canning Co., Montreal, QC) were used in all the experiments.

Particle-center temperature was measured using flexible CNS copper constantan wire thermocouples (d = 0.127 mm, TT-T-36-100, Omega Engineering Corp., Stamford, CT). A fine hole was drilled to introduce the thermocouple tips to the center of the particle. Because of the thin wire, the thermocouple sensor lag and errors were minimal. The hole was then sealed by applying a small amount of epoxy glue at the surface. The prepared particle was left to dry overnight. The small amount of glue would not affect the temperature profile, but a 5% variation was considered in the sensitivity analysis. To measure the temperature of the can fluid, test cans were fitted with needle-type thermocouples (CNS copper-constantan, Ecklund-Harrison Technologies Inc., Miami, FL) with their tips positioned at the can center. Output was recorded using a data acquisition system (HP34970A, Hewlett Packard, Loveland, CO). Thermocouple signals were recorded at one second intervals. Additional details about can preparation and temperature measurements can be found from

Sablani and Ramaswamy (1996). To overcome the variations in initial and retort temperature, data were normalized to an initial temperature of 25°C and respective retort temperatures according to the procedures given by Stumbo (1973).

Material	Density (kg/m³)	Heat capacity (J/Kg ºC)	Thermal conductivity (W/mºC)	Thermal diffusivity (m²/s)
Nylon	1128	2073	0.369	$1.52 \times 10^{-7}$
Water	1000	4180		
Pure Glycerin	1260	2430		
CMC (1%)	1000	4180		

Table 3.1: Thermo-physical properties of test materials (Meng, 2006).

### 3.3.3 Experimental design

To evaluate the applicability of the conventional method during reciprocating container agitation, experiments were conducted with cans filled with 30%v/v Nylon particles in pure glycerin, water or CMC (1% w/w) at various frequencies and amplitudes of reciprocation. The experiment for each can fluid (glycerin, water and CMC) was carried out according to a full factorial design consisting of two reciprocating factors: frequency (1.5-3 Hz), and amplitude (10-20 cm), varied over two levels. These experiments were conducted at 121.1°C. Apart from this, separate experiments were performed under non-agitated conditions, for each can fluid. All experiments were conducted in triplicate to analyze the variation in the values of U and  $h_{fp}$ . For each experimental run, 2 duplicate cans were placed in the reciprocating cage, and the remaining two spaces were occupied by dummy cans filled with water. The cans were placed at the same spot equidistant from the center in each run. The temperatures at the two positions inside the retort may be considered to have same temperature by assuming radial symmetry across the retort.

# 3.3.4 Conventional methodology for evaluation of U and h<sub>fp</sub>

According to the methodology detailed in the literature (Sablani and Ramaswamy, 1996; Dwivedi and Ramaswamy, 2010a; and Hassan *et al.*, 2012), the experimental particle and liquid transient temperatures were directly used for evaluating h<sub>fp</sub>. For this, the experimental temperature profile was fitted with a 'simulated' particle-center temperature profile generated from experimental liquid temperatures by solving the system of partial differential equations (Equations 3.1-3.4) describing unsteady heat conduction in a spherical particle immersed in liquid.

$$\frac{\partial T}{\partial t} = \alpha_{p} \left( \frac{\partial^{2} T}{\partial r^{2}} + \frac{2}{r} \frac{\partial T}{\partial r} \right)$$
(3.1)

$$T(r,0) = T_i \tag{3.2}$$

$$\frac{\partial T(r,t)}{\partial r} = 0$$
(3.3)

$$k_{p}\frac{\partial T(r,t)}{\partial r} = h_{fp}(T_{l} - T_{ps}) \text{ at } r = a$$
(3.4)

$$F_0 = \int_0^t 10^{\frac{T-121.1}{z}} dt$$
(3.5)

where, T is the temperature of the particle at distance r and time t; a is the radius of particle (m); t is the time instant (s); r is the distance from the center (m);  $\alpha_p$  is the thermal diffusivity of the particle (m<sup>2</sup>/s);  $k_P$  is the thermal conductivity of the particle (W/m<sup>2</sup> °C);  $T_{Ps}$  is the temperature (°C) at particle-surface at any time instant;  $F_o$  is the lethality (min); z is the z-value of microorganisms for lethality calculation (~ 10 °C for most microorganisms – Stoforos and Merson, 1992); and,  $h_{fp}$  is the fluid to particle heat transfer coefficient (W/m<sup>2</sup> °C).

In order to solve the heat transfer problem presented above, a Finite Difference scheme was implemented on Matlab. An initial guess for the value of the  $h_{fp}$  was made based on previous studies and the resulting particle-center temperature profile was simulated. The objective function (difference between 'simulated' and 'experimental'  $F_0$  calculated using Equation 3.5) was minimized iteratively by implementing the algorithm described in Figure 3.3, to obtain the value of  $h_{fp}$ . The

objective function was chosen according to the LALD (Least Accumulated Lethality Difference) approach, as lower temperatures have little effect on the actual lethality of the process (Sablani and Ramaswamy, 1996).



Figure 3.3: Process flow chart showing the conventional methodology for U and h<sub>fp</sub>.

The final  $h_{fp}$  for which the 'simulated' and 'experimental'  $F_0$  matched to a pre-set value of 10 min was used to represent the processing conditions. Subsequently, U was calculated by solving the energy balance equation (Equation 3.6) derived by assuming that the heat transferred across the can wall was equal to the heat absorbed by the particle and can liquid (Sablani and Ramaswamy, 1996):

$$UA_{c} \int_{0}^{pt} (T_{r} - T_{l}) dt = m_{l} c_{pl} \int_{0}^{pt} dT_{l} + h_{fp} A_{p} \int_{t=0}^{t=pt} (T_{l} - T_{ps}) dt$$
(3.6)

where, U is overall heat transfer coefficient (W/m<sup>2</sup> °C);  $A_c$  and  $A_p$  are the surface area of can and particle respectively (m<sup>2</sup>);  $T_r$ ,  $T_l$ , and  $T_{ps}$  are the retort, liquid and particle surface temperatures, respectively (°C);  $m_l$  and  $c_{pl}$  are the mass (kg) and specific heat capacity of the liquid (J/kg °C) in can;  $\alpha_p$  and  $k_P$  is the thermal diffusivity (m<sup>2</sup>/s), and thermal conductivity (W/m<sup>2</sup> °C) of particle; Pt is the process time to achieve lethality of 10 min.

### 3.3.5 Proposed methodology for evaluation of U and h<sub>fp</sub>

When the conventional method as detailed in the preceding section was used, evaluated U and  $h_{fp}$  showed large variance or could not be determined at higher frequency and amplitudes of reciprocation (Table 3.2) due to frequent fluctuations in temperature measurements under very rapid heating conditions, especially during the early part of the heating regime (Figure 3.4a). In order to solve this problem, an alternate methodology was proposed. This methodology was based on an accepted hypothesis, traditionally used in thermal processing applications, that at specific processing conditions, the heat penetration profiles are generally characterized by an average heating rate index ( $f_h$ ) and a lag factor ( $j_{ch}$ ) (Hayakawa, 1977; Sablani and Ramaswamy, 1996). The  $f_h$  is calculated as the negative reciprocal of slope of the logarithmic temperature vs time, by considering the heating regime after the initial lag (Hayakawa, 1977). Once the initial conditions are streamlined (usually shortly after the come-up period), steady heating rate profiles are established from which one can determine the  $f_h$  and  $j_{ch}$ . This can be visualized from the heat penetration curve in Figure 3.4b and  $f_h$  and  $j_{ch}$  can be evaluated by fitting the equation of the straight line (for liquid or particle-center) with Equation (3.7).

$$U \log_{10}(T_{crt} - T) = -\frac{t}{f_h} + \log_{10}(j_{ch} * (T_{crt} - T_i))$$
(3.7)

Where, U is overall heat transfer coefficient (w/m<sup>2</sup> °C);  $T_{crt}$  is constant retort temperature (°C); T is the temperature of particle (°C) at distance from center r and time t;  $f_h$ , is the heating rate index (s); and  $j_{ch}$  is the heating lag factor.

The concept of  $f_h$  enables one to avoid the temperature fluctuations during the early part of a process involving rapid heating conditions. There are two issues to consider. First is to get average retort temperature and average heating conditions (in form of average heat penetration parameters) to represent the process. The average retort temperature was taken as the average temperature during the cook period (excluding the come-up period), as commonly employed in thermal process calculations for generating heat penetration curves (Figure 3.4b), and values of  $f_h$  and  $j_{ch}$  were computed (using Equation 3.7). The second is to 'predict' the temperature profile for the steady heat transfer rate section of the process (excluding the CUT) from the average retort temperature and heat penetration parameters by using Equation 3.8. The 'predicted' temperature profiles will match the real time-temperature profiles in the region when the heating profiles are fully developed (Figure 3.4c).

$$T_{\text{pred}} = T_{\text{crt}} - (T_{\text{crt}} - T_i) * 10^{\left(-\frac{t-t_0}{f_h}\right)} \qquad \forall \tau > 0.2$$

$$= T_i \qquad \forall \tau < 0.2$$
(3.8)

where,  $T_{pred}$ ,  $T_{crt}$  and  $T_i$  are the predicted temperature profile, constant retort temperature and initial temperatures (°C) respectively;  $f_h$  is the heating rate index (s); t is the time instant (s);  $t_o$  is the time (s) at which Fourier number  $\tau$ >0.2.

The fully developed state of heating is based on the concept of using the first term approximation of the infinite summation series of temperature ratio vs time charts (Hayakawa, 1977; Ramaswamy *et al.*, 1982) in conventional heat transfer applications. In order to approximate this, the heating period has to be longer than a minimum heating time. In heat transfer models, the heating time is represented in dimensionless form as Fourier number ( $\tau = \alpha t/a^2$ ). Equation (3.8) is the simplified form of the one-term approximation solution of a transient heat conduction problem and is valid for Fourier number  $\tau > 0.2$ . Hence, in our study, the 'predicted' profiles of the particle will be valid only for time longer than 2 min (this value may vary with geometry and materials used) to satisfy the  $\tau > 0.2$  criterion.



Figure 3.4: Steps involved in evaluation of U and h<sub>fp</sub> by refined methodology: a) Experimental time temperature profile; b) Heat penetration curve of liquid and particle together with the fitting for the corresponding heating rate indexes; c) 'Predicted' and 'Simulated' time temperature profile; d) 'Predicted' v/s 'Simulated' lethality



Figure 3.5: Process flow chart showing refined methodology for estimation of U and h<sub>fp</sub>

U and hfp are then calculated by matching the 'predicted' profiles obtained from heat penetration parameters (Equation 3.8) with the 'simulated' profiles obtained from the heat transfer simulation (Equations 3.1-3.6) by employing the same procedures as that used in conventional methodology (Section 3.3.4), but by replacing the 'experimental' profiles with 'predicted' profiles, as depicted in Figure 3.5. While the conventional methodology was based on matching 'simulated' and 'experimental' profiles, the refined methodology relied on fitting 'simulated' profiles and 'predicted' profiles. Since the 'predicted' profiles were obtained from heat penetration parameters, unlike 'experimental' profiles, they were free from the fluctuations in temperature signal during vigorous reciprocation or due to movement of the thermocouple embedded particle and were better suited for computing U and hfp.

### **3.3.6** Error analysis

In order to study the variance in the results obtained by the two methodologies, standard deviation from the mean values were calculated using the formula depicted in Equation (3.9). If the standard deviation was more than 50% of the mean value, the data was assumed to be inconsistent and non-reliable and was reported as "indeterminate".

$$sd = \sqrt{\frac{\sum(x-\bar{x})^2}{n-1}}$$
(3.9)

A parameter sensitivity analysis was also performed to study the effect of perturbations in various parameters used in the refined methodology. For this, a relative uncertainty of  $\pm 5\%$  was introduced with respect to thermocouple location, heating rate index of liquid and particle center, constant operating temperature, particle thermal diffusivity and liquid heat capacity and its effect on the values of U and h<sub>fp</sub> were recorded. The sensitivity analysis was carried out only with extreme conditions (lower range and higher range of values) of calculated U and h<sub>fp</sub> values.

### 3.4 Results and discussions

### **3.4.1** U and h<sub>fp</sub> calculation using conventional methodology

U and  $h_{fp}$  values evaluated by the conventional methodology during different reciprocating conditions for all the three liquids are summarized in Table 3.2. It was found that the conventional methodology failed to predict  $h_{fp}$ , during rapid heating conditions when the CUT/EQT quotient (ratio of equilibration time and retort come up time) increased beyond 0.25. Under still conditions and under low reciprocation intensity, when the heat transfer is slow, the conventional method produced satisfactory results. But at higher frequencies, when the heat transfer rate is increased, evaluation of  $h_{fp}$  became difficult and almost not possible. Values of  $h_{fp}$ , where standard deviations varied more than 50%, were considered indeterminate. Out of the five operating conditions for water and only one, the still mode, for CMC.

However, U was evaluated in all the cases for all the fluids. This is because under rapid heating conditions, even with indeterminate values of  $h_{fp}$ , it was possible to get a value of U in accordance with Equation (3.6) (as the second term on the right hand side becomes zero when liquid temperature becomes equal to the particle surface temperature at very large or infinite values of  $h_{fp}$ ). However, this value of U is unreliable as it is based on indeterminate  $h_{fp}$ .

The standard deviation in the values of U and  $h_{fp}$  shown in Table 3.2 can also be used to assess the reliability and reproducibility of the method. It was observed that the standard deviation in the value of U and  $h_{fp}$  became larger as the frequency and amplitude of reciprocation increased. While under still conditions, the standard deviations in value of U for water, glycerin and CMC were 3.33%, 8.02% and 7.44% respectively of the mean value, under maximum reciprocating conditions (20 cm amplitude and 3 Hz frequency), these values were 24.01%, 28.33% and 23.85% for water, glycerin and CMC respectively.

	Frequency	Amplitude		$h_{fp}^{a}$	CUT/EQT
	(HZ)	(cm)	(W/m <sup>2</sup> °C)	(W/m <sup>2</sup> °C)	
Water	Still	Still	300(±10)	201(±17)	0.166
	1.5	10	730 (±43)	456(±121)	0.258
	3	10	975 (±70)	Indeterminate	0.465
	1.5	20	970 (±90)	Indeterminate	0.451
	3	20	940 (±225)	Indeterminate	0.672
Pure Glycerin	Still	Still	185 (±15)	112(±18)	0.139
	1.5	10	415 (±21)	500(±101)	0.242
	3	10	500 (±43)	662(±133)	0.260
	1.5	20	590 (±85)	822(±321)	0.336
	3	20	670 (±189)	Indeterminate	0.445
CMC (1% w/w)	Still	Still	245 (±18)	58(±10)	0.154
	1.5	10	465 (±30)	Indeterminate	0.306
	3	10	625 (±56)	Indeterminate	0.362
	1.5	20	660 (±111)	Indeterminate	0.401
	3	20	700 (±167)	Indeterminate	0.584

Table 3.2: Overall heat transfer coefficient (U) and fluid to particle heat transfer coefficient (h<sub>fp</sub>) as predicted by the conventional methodology during various operating conditions.

<sup>a</sup> values in brackets denote the standard deviation from the mean value

Standard deviations in  $h_{fp}$  were even larger than that of U, and in numerous scenarios, the values were recorded as indeterminate (standard deviation > 50%). This rise in the standard deviation is indicative of the fact that conventional methodology started becoming non-reliable under rapid heating conditions. Ramaswamy and Sablani (1997a) also noted that the error in  $h_{fp}$  values in the model can vary 50-150% when Biot number was greater than 15. Under rapid agitation scenario, the Biot number was increased tremendously due to increase in  $h_{fp}$ , and thereby equilibration time decreased. As a result, due to lower data-points available for  $h_{fp}$  calculation, the robustness of the conventional methodology decreased. Thereby, it was seen that conventional methodology was not very suitable for rapid heat transfer conditions.

### 3.4.1.1 Problems associated in calculation of U & h<sub>fp</sub> using conventional methodology

Reciprocation of the container increased the heat transfer rates by rapid back and forth motion of the container and hence, reduced the total process time to achieve the required lethality. It is graphically shown in Figure 3.6 that in still mode the particle equilibration time (EQT) for glycerin was 26 min as compared to just 7 min at maximum reciprocation intensity (3 Hz-20 cm) and for CMC, 18 min as compared to 4 min at highest reciprocation intensity. The number of data points available for evaluation of U and h<sub>fp</sub> depends on the length of equilibration time in conventional methodology. In still process, where EQT and hence, the number of data points available for calculations is very large, small errors in data gathering didn't have much effect on robustness of calculations. However with reduction in EQT during reciprocation, even the small errors in temperature data gathering magnified to a large extent and resultantly the standard deviation in the calculated values of U and h<sub>fp</sub> were quite large, as discussed in Section 3.1 and in Table 3.2. Thus, the robustness of conventional methodology decreased appreciably for rapid heating conditions prevalent during reciprocating agitation thermal processing.



Figure 3.6: Time temperature profile of the particle-center showing differences in particle equilibration time between processing at 121.1°C under still conditions and reciprocating conditions (3 Hz frequency and 20 cm amplitude) for 1% CMC and glycerin.

The ratio of come up time to equilibration time (CUT/EQT) was used to assess the rapidity of heat transfer. CUT/EQT increased under rapid heat transfer scenario due to decrease in EQT for reciprocating cans. This ratio is presented in Table 3.2 for still and reciprocating conditions. At still mode, the ratio was 0.169, 0.153 and 0.139 respectively for water, CMC and glycerin. However, with increase in rate of heat transfer, EQT decreased and thereby this ratio increased. A high ratio of CUT/EQT indicates that the retort and particle profiles are very close. The liquid and particle-center profiles are even closer. Under these situations, a slight non-uniformity in liquid or retort temperature, could lead to very large changes in the values of U and  $h_{fp}$  computed by the conventional methodology. While analyzing the respective values, it was noticed that U and  $h_{fp}$  calculations became difficult and unreliable when the ratio of CUT/EQT increased beyond 0.25.

The major source of the temperature measurement errors during reciprocating agitation comes from disturbances in temperature signals recorded by vigorously moving the thermocouple equipped particle. This led to inaccurate experimental temperature measurements. Furthermore, there is also a possibility of manual errors during fixing the thermocouples at particle-center. These errors cannot be neglected, particularly in high heat transfer scenario (CUT/EQT > 0.25), as with a Biot number greater than 15, the associated errors with the conventional model is very high (Ramaswamy and Sablani, 1997a). Moreover, for our test conditions Biot number was found even higher (Bi > 40) which made h<sub>fp</sub> values indeterminate. Thus, without a very high degree of accuracy in temperature measurement, it was not possible to use the conventional methodology effectively for rapid heat transfer conditions. Therefore, it was desirable to develop a technique for evaluation of U and h<sub>fp</sub> under these rapid heating conditions.

# 3.4.2 U and h<sub>fp</sub> calculation using refined methodology

U and  $h_{fp}$  calculated using liquid and particle-center profiles predicted by the refined methodology using predicted profiles instead of experimental data are shown in Table 3.3. These 'predicted' profiles were obtained from the  $f_h$  values of the liquid and particle-center experimental profiles as discussed in Section 2.5.U and  $h_{fp}$  values varied between 330-995 and 225-1625 W/m<sup>2</sup> °C for water, 205-670 and 185-835 W/m<sup>2</sup> °C for glycerin, and, 245-730 and 195-1550 W/m<sup>2</sup> °C for

CMC solution respectively. The lower limits of the values are for still mode, while the upper limits are for the maximum reciprocation intensity (3 Hz-20 cm).

Table 3.3: Overall heat transfer coefficient (U) and fluid to particle heat transfer coefficient (h<sub>fp</sub>) as predicted by the refined methodology during various operating conditions.

	Frequency (Hz)	Amplitude (cm)	fh-liquidª (min)	fh-particle <sup>a</sup> (min)	U <sup>a</sup> (W/m <sup>2</sup> ºC)	h <sub>fp</sub> <sup>a</sup> (W/m <sup>2</sup> °C)
Water	Still	Still	5.9(±0.6)	7.1(±0.4)	330 (±20)	225 (±24)
	1.5	10	2.5(±0.3)	4.4(±0.3)	730 (±22)	525 (±15)
	3	10	1.9(±0.4)	3.1(±0.2)	955 (±34)	995 (±34)
	1.5	20	2.0(±0.2)	3.4(±0.2)	925 (±24)	920 (±60)
	3	20	1.8(±0.01)	2.4(±0.1)	995 (±22)	1625 (±52)
Glycerin	Still	Still	7.3(±1)	10.8(±1.3)	205 (±15)	185 (±6)
	1.5	10	3.4(±0.5)	5.2(±0.4)	435 (±30)	430 (±21)
	3	10	2.8(±0.3)	4.2(±0.3)	525 (±30)	585 (±15)
	1.5	20	2.4(±0.4)	3.9(±0.3)	600 (±40)	605 (±23)
	3	20	2.2(±0.1)	3.4(±0.2)	670 (±28)	835 (±12)
	Still	Still	6.8(±0.9)	8.9(±0.6)	245 (±12)	195 (±12)
	1.5	10	2.6(±0.5)	4.9(±0.4)	505 (±12)	535 (±18)
CMC (1% w/w)	3	10	2.2(±0.3)	3.8(±0.3)	670 (±16)	765 (±21)
	1.5	20	2.1(±0.1)	3.2(±0.2)	710 (±18)	920 (±12)
	3	20	1.9(±0.03)	2.6(±0.1)	730 (±10)	1550 (±54)

<sup>a</sup> values in brackets denote the standard deviation from the mean value

For still mode, the values of U and  $h_{fp}$  obtained by the refined methodology were similar to the values obtained by other researchers (Anantheswaran and Rao, 1985; Sablani and Ramaswamy, 1995; Dwivedi and Ramaswamy, 2010b). However, U and  $h_{fp}$  during reciprocating agitation were higher than those observed by various researchers during rotary agitation (Anantheswaran and Rao, 1985; Sablani and Ramaswamy, 1995, 1996; Garrote *et al.*, 2006b; Dwivedi and Ramaswamy, 2010b). This suggests that reciprocation was more advantageous as compared to other modes of agitation for enhancement of the heat transfer happening in the system. Further, the decrease in the equilibration time of the particle (EQT) and increase in the CUT/EQT ratio also suggests a rapid heating condition.

The proposed method was found to produce reliable and reproducible results with low standard deviations for both U and  $h_{fp}$ , for all processing conditions. As depicted in Table 3.3, the standard deviations in the values of U and  $h_{fp}$  were within 3-8% of the mean value. The low values of standard deviations can be attributed to the fact that this methodology was free from any fluctuations or non-uniformity in temperature profiles during reciprocation, unlike the conventional methodology. The refined methodology was efficient in eliminating errors during data gathering by transforming the real situation into a constant operating temperature condition. Also, as this methodology was based only on the average operating temperature and heating rate index, it can be easily used to develop computer models for evaluating U and  $h_{fp}$  for a wide variety of retorts and operating conditions.

### 3.4.2.1 Effect of reciprocation frequency

The effects of the frequency and amplitude of reciprocation on U and  $h_{fp}$  using the proposed methodology were analyzed. Figure 3.7 shows the values of U and  $h_{fp}$  for different set of processing conditions with water, CMC and glycerin. It has been found that doubling the frequency of reciprocation from 1.5 to 3 Hz results into an increase of 31%, 21% and 32% in the value of U for water, glycerin and CMC respectively. Similarly, it is clear from Figure 3.7b that by increasing the reciprocation frequency, the  $h_{fp}$  value increased for all the three liquid-particulate mixtures. At same reciprocation amplitude (20 cm), increasing the reciprocation frequency from 0 to 3 Hz increased  $h_{fp}$ by 7 times with water, 8 times with CMC and 4.5 times with glycerin. This increase can be explained by the improved mixing obtained at higher reciprocation speed due to higher degree of turbulence, resulting in increased relative particle to fluid velocity (Anantheswaran and Rao, 1985; Sablani and Ramaswamy, 1996). Other researchers have also documented the similar effect of rotational speed on U and  $h_{\rm fp}$  in rotary retorts (Lenz and Lund 1978; Meng and Ramaswamy, 2007a, 2007b; Dwivedi and Ramaswamy, 2010b). These observations in the literature conform to the findings of the present study with reciprocation mode of agitation.



Figure 3.7: Effect of reciprocating conditions on heat transfer coefficients in reciprocation agitation thermal processing: a) Overall heat transfer coefficient (U); b) fluid-to-particle heat transfer coefficient (h<sub>fp</sub>).

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### **3.4.2.2** Effect of reciprocation amplitude

For 1.5 Hz frequency, doubling the amplitude from 10-20 cm resulted in an increase in the value of U by 26% for water, 37% for glycerin and 40% for CMC (Figure 3.7a). Similar trends of higher  $h_{fp}$  were observed at higher amplitudes (Figure 3.7b). This can be justified by the fact that even if the frequency is constant, higher amplitude means a higher velocity of the cage as it has to move a longer distance in the same time. Thus, both increase in frequency and amplitude of reciprocation essentially result in higher cage velocity and thus a higher heat transfer scenario. Hence, we can conclude that increasing the reciprocation frequency and amplitude has great potential to enhance the fluid to particle heat transfer rates by increasing the relative motion between liquid and particles.

### 3.4.2.3 Effect of liquid viscosity

It was observed that at any combination of reciprocation frequency and amplitude, the values of U and h<sub>fp</sub> with glycerin (highly viscous Newtonian fluid) were lower as compared to water and CMC (Figure 3.7). This phenomenon has also been studied previously by other researchers (Ramaswamy and Sablani, 1997a) and it was found that due to the higher thickness of the boundary layer associated with higher viscous fluids, the heat transfer rate between can-liquid and particle-liquid interface is retarded. Ramaswamy and Sablani (1997a) studied U and h<sub>fp</sub> values with nylon particles in water and oil (100cst at 38°C), and found larger U and h<sub>fp</sub> values with water (lower viscosity) than with oil (higher viscosity), which is similar to the results in the present study. More recently, similar trends were observed (Meng and Ramaswamy, 2007b) with highly viscous fluids (CMC, glycerin).

### 3.4.3 Sensitivity analysis

Sensitivity analysis results are summarized in Table 3.4. Since the lowest values of U and  $h_{fp}$  were for glycerin hence, the sensitivity analysis was conducted with glycerin at the lower end. Due to the same reason, the analysis was conducted for water at the higher end. A thermocouple misplacement error of 0.095 mm from the center of the particle reduced the calculated U and  $h_{fp}$ 

values by only 1.7 and 3.4% at the lower end respectively and at the higher end the reduction was 1.9 and 5.4% respectively. Low perturbations due to thermocouple displacement indicate the reliability of the refined methodology. This is because,  $f_h$  was not affected much due to change in the position of the thermocouple. The errors in temperature measurement to thermocouple displacement at the particle center will be even lower due to symmetry of temperature distribution around the center (Sablani and Ramaswamy, 1996).

Average heating rate index and the average operating temperature are the two important parameters used in the refined methodology. Perturbations in  $f_h$  of liquid reduced U and  $h_{fp}$  values by 2-5.6%. However, a similar change in the value of  $f_h$  of particle-center had a lower impact on U and higher impact on values of  $h_{fp}$ . This indicated that the  $f_h$  of liquid affected U and  $f_h$  of the particle-center affected  $h_{fp}$  more predominantly. Changing the constant operating temperature had little effect (less than 1%) on the values of U. However, the values of  $h_{fp}$  were altered significantly (1-8%) for higher operating temperatures. This trend can be attributed to the fact that the target lethality of 10min was achieved faster at higher temperature and vice-versa. Since the  $f_h$  of liquid and particle were kept constant, the model gave a higher value of  $h_{fp}$  to reach the target lethality in the shorter time for a +5% change in operating temperature. Hence, it is important to choose the constant operating temperature and the heating rate indices employed in Equations (3.7-3.8), while evaluating U and  $h_{fp}$  using this methodology.

Increasing the liquid heat capacity increased the values of U and vice-versa by 2-2.5%, while it did not affect  $h_{fp}$  at all. U is dependent on liquid heat capacity because with higher liquid heat capacity, a higher heat transfer was required to get the same values of  $f_h$  of liquid and particle-center. Also, due to this, the first term on the right hand side of Equation (3.6) increased, which resulted in higher U values. However, changing liquid heat capacity did not affect  $h_{fp}$  as the values of liquid heat capacity were not required for evaluation of  $h_{fp}$ . Perturbations in the particle thermal diffusivity affected both U and  $h_{fp}$  inversely by 2-3%. Thus, it is important to carefully evaluate the values of respective thermo-physical properties carefully while employing the refined methodology.

Also, in Table 3.4, it is seen that the changes in the parameter values influenced the values at higher end more significantly than at lower end. This was because of the use of water as covering

fluid for higher end values. For a more viscous liquid like glycerin or CMC, these deviations can be expected to be lower than the reported values.

Table	3.4:	Results	of	sensitivity	analysis	for	the	refined	methodology	as	influenced	by
pertu	rbatio	ons in ch	ara	cteristic pa	rameters	•						

	Level	Lower end of U	Higher end of U	Lower end of h <sub>fp</sub>	Higher end of h <sub>fp</sub>
		(%)	(%)	(%)	(%)
Thermocouple Misplacement	0.095mm (5% of particle diameter)	1.7	1.9	3.4	5.4
<i>a</i> , 1, , , 1	+5%	4.4	2.0	2.6	5.0
th-liquid	-5%	5.1	2.2	2.2	5.5
fh norticle	+5%	0.1	1.2	5.2	6.2
m-particle	-5%	0.4	1.3	7.8	5.9
Constant operating Temperature	127.2 °C (+5%)	0.4	0.7	3.3	15.0
	115 °C (-5%)	0.8	0.9	1.1	7.8
Liquid Heat	+5%	2.4	2.2	0	0
Capacity	-5%	2.4	2.2	0	0
Particle Thermal	+5%	2.3	2.2	3.1	12.1
Diffusivity	-5%	2.6	5.0	2.1	10.0

# 3.4.4 Comparison of conventional methodology and refined methodology

The values of U obtained by employing both methodologies, compared in Figure 3.8, were generally in agreement. Yet, conventional method was generally found to under-predict the value of U by 5-10% for higher agitation frequencies (Figure 3.8). This may be because at higher agitation,

the conventional methodology failed to measure  $h_{fp}$  (indeterminate values) and hence, calculation of U using this  $h_{fp}$  was erroneous. The underestimation occurs because the conventional methodology neglects the second term on the right hand side of Equation (3.6) under very large values of  $h_{fp}$ . U and  $h_{fp}$  for glycerin agreed with each other for both methodologies due to slow heat transfer. However, for less viscous fluids,  $h_{fp}$  values could not be predicted properly by the conventional methodology at faster heating rates. On the other hand, the refined methodology produced reliable, reproducible and more robust results with low standard deviations. Values of U and  $h_{fp}$  calculated under the slow heating or still conditions by the two methodologies also agreed with each other (Table 3.2 and Table 3.3). Thus, it is observed that while the conventional methodology failed under high heat transfer scenarios, the refined methodology provided reliable and robust results at both slow and fast heating conditions and was not affected very significantly by perturbations in various parameters as depicted in previous sections.



U (w/m<sup>2</sup> °C) By conventional methodology

Figure 3.8: Comparison between the values of overall heat transfer coefficient (U) obtained from conventional and refined methodology.

### 3.5 Conclusions

A refined methodology based on the average heating rate index and operating temperature has been developed for evaluation of U and h<sub>fp</sub> for thermal processing of liquid-particulate mixtures with reciprocating container agitation. Since this method used temperature profiles 'predicted' from the heat penetration parameters, instead of actual 'experimental' temperature profiles, it was able to overcome the effects of fluctuations in temperature during vigorous back and forth motion of cans. The refined method was found to be more reliable than the conventional method and gave consistent results under both slow and rapid heating conditions. An analysis of the evaluated values of U and h<sub>fp</sub> revealed that both the reciprocation frequency and amplitude were significant factors in increasing the heat transfer rates in liquid particulate canned system. This methodology does not depend on the come-up portion of the retort and considers only the steady-state portion of the heating curve to compute the heat penetration parameters, from which U and h<sub>fp</sub> are evaluated. Thus, it has the potential to be used as a standard to compare the effectiveness of heat transfer at various processing conditions in different retorts with different heating mediums. Also, numerical simulations using this methodology would be simpler, as it utilizes only the average operating temperature and heating rate indices of liquid and particle-center, instead of many data points from the entire experimental profile. Thus, it would be possible to use the refined methodology in various optimization or neural network subroutines, which require rigorous regression.

### **PREFACE TO CHAPTER 4**

In Chapter 3, the conventional methodology to evaluate heat transfer coefficients in reciprocating agitation was refined and was found able to predict consistent values of U and  $h_{fp}$ . Results showed that the reciprocation agitation can increase the values of heat transfer coefficients to a very large extent as compared to still mode. It was also observable that the values of heat transfer coefficients are highly dependent on the processing conditions such as operating temperature, can headspace, intensity of reciprocation etc. Thus, a study was undertaken to evaluate this effect in canned non-Newtonian fluid particulate foods.

This chapter elucidates the effect of various processing variables on heat transfer coefficients in canned liquid particulates subjected to reciprocating agitation. Five most influencing process parameters were selected by performing preliminary experiments. Subsequently, the optimization of reciprocation intensity (function of process variables viz. reciprocation amplitude and frequency) was carried out in order to reduce the quality damage under high speed agitation (impact induced losses to canned food) without compromising the benefits of reciprocation agitation (reducing thermal damage). This study shall help in selection of process variables in industrial applications for process establishment purposes.

Parts of this chapter have been presented in the form of an oral presentation at The Northeast Agricultural and Biological Engineering Conference (NABEC) held on July 27-30 at Kemptville, Ontario.

Parts of this chapter have been published as an original research article entitled "*Heat* transfer coefficients during thermal processing of canned particulate non-Newtonian fluids undergoing reciprocating agitation" in LWT-Food Science and Technology, 65, 185–196.

The experimental work and data analysis was carried out by the candidate under the supervision of Dr. H. S. Ramaswamy.

The full manuscript as presented in Chapter 4 does contain some sections already presented in Chapter 3. Rather than deleting them, they have been left in place to provide continuity.

# Chapter 4. EFFECT OF PROCESS VARIABLES ON HEAT TRANSFER COEFFICIENTS IN CANNED NON-NEWTONIAN FLUID PARTICULATE FOODS DURING RECIPROCATING AGITATION THERMAL PROCESSING

### 4.1 Abstract

Overall (U) and fluid-to-particle heat transfer coefficients (h<sub>fp</sub>) in canned particulate non-Newtonian fluid (CMC solutions) mixtures undergoing reciprocation agitation thermal processing was evaluated in a pilot-scale vertical steam retort. Effect of five influencing process variables on U and h<sub>fb</sub> were evaluated using a central composite rotatable design (CCRD) and a 3x3x2 full-factorial design. Reciprocation frequency (1-4 Hz), being the most influential parameter, was selected as common factor in both designs. Apart from frequency, temperature (110-130°C) and liquid viscosity (contributed by 0-1% CMC solution) were varied in CCRD design, while amplitude (5, 15 and 25 cm) and can headspace (6 and 12 mm) were varied in full-factorial design. Cans (307x409 size) were filled with 30% (v/v) Nylon particles in CMC solution and processed under the preselected conditions. U values varied from 525 to 1125 W/m<sup>2</sup>  $^{\circ}$ C and h<sub>fp</sub> values varied from 550 to 1610 W/m<sup>2</sup> °C depending upon process conditions. Analysis of variance showed frequency, amplitude, liquid viscosity, headspace and temperature to be significant factors for h<sub>fp</sub>, and frequency, amplitude and liquid viscosity for U (p < 0.001). Increasing the reciprocation frequency from 1 to 4 Hz almost doubled the value of both the heat transfer coefficients. Similarly increasing the reciprocation amplitude from 5 to 25 cm, resulted in 30-35% increase in the values of heat transfer coefficients. Overall with increase in temperature, frequency, amplitude, and headspace, associated h<sub>fb</sub> and U values also increased, but with increasing liquid viscosity, both h<sub>fb</sub> and U showed a decrease. Finally, optimization of processing conditions was carried out to minimize quality losses due to particle motion (agitation intensity) and thermal damage (severity of thermal processing).

### 4.2 Introduction

Thermal processing industries have shifted their interest from conventional processing to new technologies using high-temperature short-time (HTST) processing, aseptic processing, processing under container agitation, or processing in thin profile formats (Ramaswamy *et al.*, 1993; Singh *et al.*, 2015a) to produce high quality canned products. HTST processes offer the potential to give the

same level of sterility but with reduced quality loss, as the activation energy of the reactions which results in microbial death are higher than those which result in quality loss. Agitation, in form of rotation (Dwivedi and Ramaswamy, 2010a) or oscillation (Sablani and Ramaswamy 1999), has been extensively used to achieve HTST processing. These agitations have also been used industrially where various spiral cookers and rotary retorts are available. Rotary modes of agitation have shown very significant role in increasing heat transfer rates based on various processing conditions; however, they are effective only up to certain rotation speed, after which centrifugal forces become dominant and no further increase in heat transfer is encountered (Ramaswamy and Dwivedi, 2011). More recently, a new form of container agitation, namely, reciprocation agitation, is being promoted to improve associated heat transfer rates (Walden and Emanuel, 2010). Reciprocating agitation has been found to be effective in reducing process times and quality loss indicator due to the rapid rate of heating (Singh *et al.*, 2015b). However, reciprocating agitation may result in quality loss in some situations due to excessive shear on food particles at high reciprocation intensity. Thus, it is necessary to optimize the reciprocating agitation process to minimize quality losses due to agitation in addition to that due to the time-temperature effect.

Data on overall heat transfer coefficient (U) and fluid-to-particle heat transfer coefficient (h<sub>fp</sub>) are needed for modeling of heat transfer in a liquid particulate system (Stoforos and Merson, 1992). Recent studies on thermal processing have focused on evaluation of U and h<sub>fp</sub> and effect of process variables on these coefficients. Most recently, Singh *et al.* (2015a) extensively reviewed the effect of these parameters on U and h<sub>fp</sub> in still and rotary mode of agitation. It has been generally recognized that rotational speed, retort temperature, head space volume, system geometry, rotation radius, liquid viscosity, particle shape, particle size, particle density etc. are key factors (process variables) in end-over-end (Anantheswaran and Rao, 1985; Sablani and Ramaswamy, 1996, 1999 and Meng and Ramaswamy, 2007a, 2007b) and axially (Lenz and Lund, 1978, Deniston *et al.*, 1987 and Dwivedi and Ramaswamy, 2010a) rotating cans. However, there is a serious lack of research work on U and h<sub>fp</sub> during reciprocation mode of agitation, although it is claimed that this process can reduce process times more than 20-fold compared to a still process and more than 10-fold compared to a rotary process time during reciprocating agitation compared to still mode and also quantified

the effect of some operating parameters on U and  $h_{fp}$  during reciprocating agitation. However, they used glycerin as the covering fluid which is Newtonian, but most of the real fluid foods are non-Newtonian in nature.

Hence, focus of this chapter was to investigate the effect of various process variables such as reciprocation frequency, reciprocation amplitude, retort temperature, fluid viscosity and can headspace on U and  $h_{fp}$  associated to reciprocation thermal processing of canned particulate foods. Further this chapter shall attempt to optimize the reciprocation intensity (function of reciprocation amplitude and frequency) in order to reach a balance between thermal and agitation induced losses to achieve better quality products.

### 4.3 Material and methods

### 4.3.1 Modified steam retort for facilitating reciprocation agitation

A pilot-scale vertical steam retort modified to include reciprocating agitation was used in this study. For this, a reciprocating cage (inside the retort) connected to a slider crank assembly (outside of retort) was installed in the existing retort (Figure 4.1a). The slider crank assembly converted rotation of motor into reciprocating motion (back and forth motion) of cage. The system was designed in a way that accommodated horizontal reciprocation of the cage (12 cm, length 35 cm and height 17 cm) placed inside a vertical retort (diameter 62 cm and depth 100 cm) up to a maximum frequency of 5 Hz. The reciprocating cage was designed to hold one level of 4 cans (No. 2) along the radial direction of the retort (cans could be placed at horizontal or vertical, parallel or perpendicular to the axis of agitation). In the present study. Two cans were placed on each side of the axis of the reciprocating crank on the rotating shaft of the slider crank assembly. The system was powered by a  $\frac{1}{2}$  hp direct current magnetic motor. The motor speed (and thus the frequency of reciprocation) was controlled through an external voltage controller.





Figure 1b.



Figure 4.1: a). Schematic of the experimental setup; b) schematic showing thermocouple equipped particle mounted inside the can using a brass connector

### 4.3.2 Materials used

Metal cans of size 307×409 (Home Canning Co., Montreal, QC) were used in the study. Manually operated seaming machine (Home Canning Co., Montreal, QC) was used for sealing the cans. Solutions of Carboxymethyl cellulose (CMC, Sigma, St. Louis, MO) were used as a model non-Newtonian fluid. Different concentrations of CMC solution (0, 0.2, 0.5, 0.8 and 1 %w/w), ware used to provide various range of starting viscosities. To overcome uncertainty in thermo-physical properties associated using real foods, Nylon spheres (Small Parts Inc., Miami, FL) of 19 mm diameter were used as food simulating particles (rather than real food), as nylon's thermal properties (heating behavior) are most relevant to real food, as seen in Table 4.1, and they do not change with temperature, unlike real foods.

Material	Density (kg/m³)	Heat capacity (J/kg °C)	Thermal conductivity (W/m ºC)	Thermal diffusivity (m²/s)
Nylon <sup>a</sup>	1128	2073	0.369	$1.52 \times 10^{-7}$
Real Foods <sup>b</sup>				
Potato	1189	3603	0.563	$1.44 \times 10^{-7}$
Carrot	1029	3849	0.569	$1.44 \times 10^{-7}$
Shrimp		3688	0.543	$1.52 \times 10^{-7}$
Tuna		3356	0.468	$1.52 \times 10^{-7}$

Table 4.1: Properties of Nylon and real particulate foods used in canning

<sup>a</sup>Murakami, (1997)

<sup>b</sup> Meng & Ramaswamy, 2005

### 4.3.3 Physical properties of CMC

Rheological properties of different CMC solutions were determined with AR 2000 rheometer (TA Instruments, New Castle, DE) at a temperature of 25°C. The power law model  $\tau = k\gamma^n$  was used to characterize the flow behavior, where,  $\tau$  (N/m<sup>2</sup>) is the shear stress, k (Pa s<sup>n</sup>) is the consistency coefficient, n is the flow behavior index and  $\gamma$  (s<sup>-1</sup>) is the shear rate. Because the

concentrations of CMC aqueous solutions were small, specific heat of all CMC concentrations were assumed to be same as water (Anantheswaran and Rao, 1985).

### 4.3.4 Temperature measurement

Cans were filled with the prepared liquid and 30% (v/v) Nylon particles (CMC solution and particles) to the required headspace. Liquid temperatures inside the can were monitored using CNS copper-constantan needle-type thermocouples (locking connector, C-10, Ecklund Harrison Technologies, Inc. Cape Coral, FL) with tips located at the geometric center of the can (Figure 4.1b). Particle-center temperatures were measured using flexible CNS copper-constantan wire thermocouples (d = 0.0762 mm, Omega Engineering Corp., Stamford, CT) introduced into the particle center through a fine hole and fixed by a small amount of epoxy glue (Figure 4.1b). Thermocouple signals were recorded at 1 second intervals. Additional details about can preparation and temperature measurements can be found from Sablani and Ramaswamy (1996). To overcome the variations in initial and retort temperature, data were normalized to an initial temperature of 25 °C and respective retort temperatures according to the procedures given by Stumbo (1973). A schematic of temperature measurement and experimental setup is shown in Figure 4.1a.

### 4.3.5 Thermal processing

For thermal processing, duplicate cans were placed vertically (Figure 4.1a) in the modified reciprocating retort equidistant from the axis of reciprocation. The empty space in the cage was filled with dummy cans to provide ballast. Retort was closed from the top and locked. The reciprocating crank was pivoted to the proper position on the rotating shaft to achieve the required amplitude. Motor was turned on at the required reciprocation frequency. Steam was turned on and retort was heated up to processing temperature. During the sterilization cycle, steam temperature and system pressure were maintained at the pre-set value through the retort control system. After completing the process, steam was turned off and water inlet to the retort was opened while the reciprocation was still on. Cans were then cooled by circulating cold water in the retort, until all temperatures reached below 30°C.

# 4.3.6 Experimental design and statistical analysis

Based on some preliminary experiments, the five most influencing process variables were selected: retort temperature, reciprocation frequency, reciprocation amplitude, CMC concentration, and can headspace. The influence of these variables were studied using two experimental designs (a central composite rotatable design – CCRD, and a full-factorial design). The CCRD design involved 20 experiments for five levels of retort temperature (110, 114, 120, 126, 130 °C), CMC concentration (0.0, 0.2, 0.5, 0.8 and 1.0%) and reciprocation frequency (1, 1.6, 2.5, 3.4, and 4 Hz). The fixed variables were: can headspace of 12 mm, and amplitude of reciprocation of 15 cm. The 3x3x2 full factorial design involved 18 experiments with three levels of reciprocation frequency (1, 2, 3 Hz), three level of reciprocation amplitude (5, 15, 25 cm), and two headspace levels (6 and 12 mm). The fixed variables were: operating temperature of 121.1°C, and CMC concentration of 1%. All experiments were conducted with duplicate cans and each test run was replicated twice. Statistical significance of the terms in the regression equations was examined. Analysis of variance (ANOVA) was used to determine significant terms in the model for each response. The adequacy of model was checked by looking at the R<sup>2</sup> and adjusted-R<sup>2</sup> values and by ensuring that the lack of fit was not significant and the coefficient of variation was below 10%.

### 4.3.7 Methodology for evaluation of U and h<sub>fp</sub>

Heating rate index of liquid ( $f_{h-l}$ ) and particle ( $f_{h-p}$ ) were evaluated from the gathered timetemperature data as negative reciprocal slopes of semi-logarithmic heat penetration curves and these were used to compute U and  $h_{fp}$  using a methodology developed earlier in chapter 3. In this methodology, values of  $h_{fp}$  were estimated by matching the predicted temperature profile of the coldspot (particle-center) with simulated temperature profile of the cold-spot under constant retort temperature conditions ( $T_{crt}$ ) for Fourier number greater than 0.2. The temperature-time data for liquid ( $T_{pred-L}$ ) and particle-center ( $T_{pred-P}$ ) under constant retort operating conditions were predicted from heating rate indices of liquid and particle (Equation 4.1); while the simulated temperature profile of the particle-center was generated from equilibrated liquid profile ( $T_{pred-L}$ ) using a finite difference simulation of the unsteady state heat conduction problem (Equation 4.2) of a spherical particle immersed in liquid. For fitting the predicted and simulated profiles, an initial guess of  $h_{fp}$  was assumed and then difference between the  $F_0$  values of the two profiles ( $T_{pred-L}$  and  $T_{pred-P}$ ) were minimized by sequentially changing the value of  $h_{fp}$  such that both matched a pre-set value of 10 min. U was then calculated using the energy balance equation derived by assuming that the heat transferred across the can wall is equal to the heat absorbed by the particle and can liquid (Equation 4.3).

$$T_{pred} = T_{crt} - (T_{crt} - T_i) * 10^{\left(\frac{t}{f_h}\right)}$$
 (4.1)

$$\frac{\partial T}{\partial t} = \alpha_{p} \left( \frac{\partial^{2} T}{\partial r^{2}} + \frac{2}{r} \frac{\partial T}{\partial r} \right)$$
(4.2)

$$UA_{c} \int_{0}^{Pt} (T_{crt} - T_{pred-L}) dt = m_{L} c_{pL} \int_{Ti}^{Tcrt} dT_{pred-L} + h_{fp} A_{p} \int_{t=0}^{Pt} (T_{pred-L} - T_{Ps}) dt$$
(4.3)

where,  $T_{pred}$ ,  $T_{crt}$ ,  $T_i T_{pred-L}$ ,  $T_{Ps}$  and T are the predicted temperature profile, constant retort temperature, initial temperature, liquid temperature, particle surface temperature, and temperature of particle (°C) at distance from center r and time t, respectively;  $m_l$  and  $c_{pl}$  are the mass (kg) and specific heat capacity of the liquid (J/kg °C) in can;  $\alpha_p$  and  $k_P$  is the thermal diffusivity ( $m^2/s$ ), and thermal conductivity ( $W/m^2$  °C) of particle;  $F_o$ ,  $f_h$ , and Pt is the lethality, heating rate index (s) and process time to achieve lethality of 10 min respectively; z is the z-value of microorganisms for lethality calculation (~ 10 °C for most microorganisms – Stoforos and Merson, 1992) and,  $A_c$  and  $A_p$  are the surface area of can and particle respectively ( $m^2$ ). More information about methodology to calculate heat transfer coefficients is detailed in chapter 3.

### 4.3.8 Optimization

Optimization was conducted for minimization of quality loss due to rapid agitation and due to thermal damage. Within the range of the heat transfer conditions studied, the normalized thermal quality loss was calculated using Equation 4.4.

Normalized thermal quality loss 
$$(TQL_{norm}) = \frac{\left(\frac{1}{U}\right) - \left(\frac{1}{U}\right)_{min}}{\left(\frac{1}{U}\right)_{max} - \left(\frac{1}{U}\right)_{min}}$$

$$(4.4)$$

where, (1/U) is the overall heat transfer resistance, and  $(1/U)_{max}$  and  $(1/U)_{min}$  are the maximum and minimum values of overall heat transfer resistance.

Normalized agitation quality loss (quality losses due to high speed agitation) was calculated using Equation 4.5 from the reciprocation intensity (Equation 4.6).

$$AQL_{norm} = \frac{\text{RI}}{RI_{max}} \tag{4.5}$$

$$RI = 2\pi^2 f^2 a \left( 1 + \frac{a}{2l} \right) \tag{4.6}$$

where, f, a and l are the values of reciprocation frequency, reciprocation amplitude and length of crank (=40cm) in slider crank unit.

The final quality loss indicator (QLI) was considered to be a function of both  $TQL_{norm}$  and  $AQL_{norm}$ . A linear model (Equation 4.7) was used to estimate QLI for different kind of products.

$$QLI = f(TQL_{norm}, AQL_{norm}) = a.TQL_{norm} + (1 - a).AQL_{norm}$$
(4.7)

where, 'a' is the fractional contribution of *thermal quality loss* to the *agitation quality loss*. QLI for different values of 'a' was plotted to obtain critical reciprocation intensity to compromise between thermal and agitation induced losses.

### 4.4 Results and discussion

### 4.4.1 Rheological properties of CMC solutions

Carboxymethyl cellulose (CMC) is a commercial thickener used in many industrial applications. It is a derivative of cellulose, an insoluble homopolymer of repeating,  $\beta$ -D-glucopyranosyl units which are connected through 1-4-glycosidic linkages. The long rigid structure of CMC consisted of anionic molecules (Ionized carbonyl group) which are responsible for non-

Newtonian behaviour of CMC solution. BeMiller and Whistler (1996) have described the stability and high viscous nature of CMC solutions as due to electrostatic repulsion the ionized groups tend to be extended in solutions, and adjacent chains repel each other.

The evaluated rheological properties of CMC solutions are summarized in Table 4.2. Power law model was found best to describe the rheological characteristics of CMC aqueous solution which is also evident from literature (Abdelrahim *et al.*, 1994; Vais *et al.*, 2002). It was observed that by increasing the CMC concentration the value of K (consistency coefficient) increases and n (flow behaviour index) decreases. By varying the CMC concentration 0.2 to 1 (%w/w) the value of 'K' increased from 0.75 to 4.15 and 'n' decreased from 0.75 to 0.45. Thus, CMC solution showed shear thinning nature which is also well explained in previous finding (Abdelrahim *et al.*, 1994; Vais *et al.*, 2002). Abdelrahim *et al.* (1994) also found that at higher concentration of CMC, there were dramatic increases in apparent viscosity (increase in the shear resistance and/or consistency) of the solution.

Material	Consistency Coefficient (K) Pa s <sup>n</sup>	Flow Behavior Index (n)
	CMC Solutions (g/100g) <sup>a</sup>	
0.2	$0.75(\pm 0.02)$	$0.75(\pm 0.04)$
0.6	$1.55(\pm 0.03)$	$0.56(\pm 0.03)$
0.8	$2.17(\pm 0.09)$	$0.51(\pm 0.05)$
1.0	4.15 (± 0.15)	$0.45(\pm 0.04)$
	<b>Commercial Soups</b>	
Tomato soup <sup>b</sup>	4.2	0.42
Vegetable soup <sup>c</sup>	1	0.49
Exotic fruit soup <sup>c</sup>	1.04	0.42
Tarhana Soup <sup>d</sup>	1.6	0.52
	<b>Commercial Thickeners</b>	
Guarcol <sup>e</sup>	1.11	0.23
Keltrol <sup>e</sup>	4.09	0.33

	<b>Table 4.2:</b>	<b>Properties</b>	of CMC	solutions
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<sup>a</sup> Values of experimental results indicated as mean values (± standard deviation in mean value),

<sup>b</sup> Sanchez *et al.*, (2003),

<sup>c</sup> Wendin *et al.* (2010),

<sup>d</sup> İbanoğlu and İbanoğlu (1998),

<sup>e</sup> Sopade *et a*l., (2007).

The values of CMC at different temperatures and concentrations were compared with other values of real liquid soups. It was seen that CMC concentrations between 0.20 to 1.00 % showed rheological properties in the range of commercial soups and thickeners (Table 4.2). Due to this proximity of rheological properties to real fluids, CMC has many applications in foodstuff formulations, product development and processing due to its specific rheological properties. This is also one of the main reason why CMC solutions were used in this study.

# 4.4.2 Experimental design (I)

U and  $h_{fp}$  values computed at different experimental conditions using the CCRD experimental design are tabulated in Table 4.2 and its analysis of variance is presented in Table 4.4. Using the Design Expert software, a second-order polynomial response surface model was fitted to both the response variables Y (overall heat transfer coefficient, U and fluid to particle heat transfer coefficient,  $h_{fp}$ ) (U – Equation 4.8 and  $h_{fp}$  - Equation 4.9) as functions of temperature (T), CMC concentration (C) and reciprocation frequency (R):

$$U = 4980.74 - 69.19T - 1219.65C + 6.77R + 4.88T^*C - 0.68TR + 63.07RC + 0.29T^2 + 219.88C^2 + 36.43R^2$$

$$h_{fp} = -11253.80 + 187.32T - 964.63C + 274.79R + 7.00T^*C + 0.77TR - 91.10RC - 0.75T^2 + 97.40C^2 - 27.34R^2$$
(4.8)

High  $R^2$ , adj- $R^2$  and low coefficient of variation (Table 4.4) demonstrated that the models fitted the experimental data well. Results showed that the observed values compared well to the values calculated from the regression model (Figure 4.2) and hence, the models could be used to represent heat transfer coefficients in reciprocation container agitation.



Figure 4.2: Comparison between predicted and actual values of U and h<sub>fp</sub> with 95% confidence intervals during reciprocating container agitation (blue diamond – plot points; black solid line – diagonal y=x line; black broken line – 95% confidence intervals)
	Factors		Response		
Retort	СМС	Reciprocation	U <sup>a</sup>	h <sub>fp</sub> <sup>a</sup>	
Temperature	Concentration	Frequency			
			(W/m <sup>2</sup> °C)	$(W/m^2 \circ C)$	
(°C)	(%)	(Hz)			
110	0.50	2.5	770(±20)	850(±22)	
120	0.00	2.5	990(±16)	1260(±13)	
120	1.00	2.5	710(±12)	990(±10)	
126	0.80	3.4	920(±08)	1185(±09)	
114	0.20	3.4	1035(±11)	1240(±31)	
120	0.50	2.5	780(±21)	1090(±23)	
120	0.50	1	675(±21)	760(±30)	
114	0.80	3.4	890(±20)	990(±30)	
120	0.50	2.5	770(±15)	1015(±19)	
126	0.80	1.6	620(±17)	855(±24)	
120	0.50	2.5	835(±24)	1110(±25)	
120	0.50	4	1080(±25)	1310(±35)	
120	0.50	2.5	765(±10)	1055(±24)	
126	0.20	1.6	800(±18)	960(±31)	
114	0.80	1.6	610(±23)	740(±33)	
120	0.50	2.5	770(±10)	1080(±17)	
130	0.50	2.5	875(±14)	1200(±26)	
120	0.50	2.5	770(±22)	1080(±26)	
114	0.20	1.6	790(±15)	830(±21)	
126	0.20	3.4	995(±11)	1320(±19)	

 Table 4.3: CCRD experimental design for evaluating U and h<sub>fp</sub> during reciprocation agitation

 of can as affected by retort temperature, CMC concentration and reciprocation frequency

<sup>a</sup> values in brackets denote the standard deviation from the mean value

Table 4.4: ANOVA and regression coefficients of the second-order polynomial model for U and
h <sub>fp</sub> in reciprocating agitation thermal processing of cans demonstrating effect of retort
temperature, CMC concentration and reciprocation frequency

Source	df	U				$\mathbf{h_{fp}}$			
	_	Sum of	<b>F-value</b>	<i>p</i> -value	Sum of	<b>F-value</b>	<i>p</i> -value		
		Squares			Squares				
Model	9	316400	39	< 0.0001	570500	32	< 0.0001		
Т	1	2522	2	0.1236	89185	45	< 0.0001		
С	1	79488	89	< 0.0001	78451	40	< 0.0001		
R	1	214300	240	< 0.0001	378600	191	< 0.0001		
TXC	1	595	1	0.4331	1225	1	0.4491		
TXR	1	105	1	0.7385	136	1	0.7982		
CXR	1	2278	3	0.1411	4753	3	0.1518		
T2	1	1564	2	0.2149	10038	5	0.0478		
C <sup>2</sup>	1	5443	6	0.0331	1068	1	0.4789		
R <sup>2</sup>	1	12551	14	0.0038	7070	4	0.0877		
Residual	10	8920			19738				
Total	19	325300			590200				
<b>R</b> <sup>2</sup>		0.98			0.97				
Adj-R <sup>2</sup>		0.95			0.94				
CV		3.64			4.26				

T, C, R = Linear effects of Retort temperature, CMC Concentration, and Reciprocation frequency respectively; T2 C2 R2 = Quardretic effects of Retort temperature, CMC Concentration, and Reciprocation frequency respectively, TXC, CXR, TXR =Interaction effects of Retort temperature, CMC Concentration, and Reciprocation frequency respectively

Analysis of variance (Table 4.4) was used to evaluate the significance of effect of reciprocation frequency, CMC concentration and retort temperature on the associated heat transfer coefficients. Linear and quadratic effects of CMC concentration and reciprocation frequency on U were significant (p < 0.001), while both the linear and quadratic effects of retort temperature were not (p > 0.05). The mutual interactions between CMC concentration, temperature and reciprocation frequency were also found to be non-significant (p > 0.05) on U. Based on the sum of squares, the

importance of the independent variables on U in reciprocation agitation could be ranked in the following order: reciprocation frequency > CMC concentration > temperature. Ramaswamy and Dwivedi (2011) used a Newtonian fluid (glycerin) and reported the same order of process variables on associated heat transfer in free axial agitation. Similar order was also reported for non-Newtonian fluids (Hassan *et al.*, 2012).

In case of  $h_{fp}$  calculation linear effects of temperature, CMC concentration and reciprocation frequency and quadratic effect of retort temperature were significant (p < 0.05), while the quadratic effect of CMC concentration and reciprocation frequency were not (p > 0.05). The mutual interactions between CMC concentration, temperature and reciprocation frequency were also found to be non-significant (p > 0.05) on  $h_{fp}$ . Again, based on the sum of squares, the importance of the independent variables on  $h_{fp}$  in reciprocation agitation could be ranked in the following order: reciprocation frequency > CMC concentration > retort temperature. Hassan *et al.* (2012) also showed the same trends for  $h_{fp}$  with CMC under axial processing. Figures 4.3 and 4.4 show 3D surface plots demonstrating the effect of these three parameters on U and  $h_{fp}$  in reciprocation container agitation.

#### 4.4.2.1 Effect of retort temperature

Retort temperature was found to be least effective amongst the selected variables, however; it had a positive effect on the associated heat transfer coefficients and both U and  $h_{fp}$  increased at higher temperatures (Figures 4.3 and 4.4). For the same levels of CMC concentration and reciprocation frequency, heat transfer coefficients at 126 °C were ~ 6-9 % higher that at 114 °C (Table 4.3). This positive effect can be attributed to the reduction in viscosity of CMC at higher temperature due to its shear thinning nature resulting in better liquid convection at higher temperatures. This is evident from previous works on rheological evaluation of CMC dispersions (Abdelrahim and Ramaswamy, 1995; Gomez-Diaz and Navaza, 2003). They reported that with an increase in temperature, the consistency coefficient decreases and flow behavior index increases reaching close to 1.0 and net result of these two is a significant decrease in apparent viscosity of CMC dispersions at higher temperatures. This trend was also reported by Sablani and Ramaswamy (1996), Meng and Ramaswamy (2007a, 2007b), and Dwivedi and Ramaswamy (2010a, 2010b) in previous studies with other modes of agitation.



Figure 4.3: Response surface plot for effect of reciprocation frequency, retort temperature and CMC concentration on overall heat transfer coefficient U (W/m<sup>2</sup> °C)



Figure 4.4: Response surface plot for effect of reciprocation frequency, retort temperature and CMC concentration on fluid to particle heat transfer coefficient h<sub>fp</sub> (W/m<sup>2</sup> °C)

Similar effects of operating temperature on U and  $h_{fp}$  were also presented by Singh *et al.* (2015b) with Newtonian fluids, although the magnitudes of the effect were lower. Thus, it is seen that effect of temperature was comparatively more significant with non-Newtonian fluid (used in this study) than with Newtonian fluid when subjected to reciprocating agitation. Also, Singh *et al.* (2015b) used a single particle suspended in a Newtonian fluid, while the current study included multiple particles (30% v/v). At higher temperature, under agitation and higher particle concentration, the associated shear rates would be higher and therefore the CMC solution would become less viscous leading to increased particle motion which leads to a higher turbulence and more effective heat transfer.

#### 4.4.2.2 Effect of CMC concentration (liquid viscosity)

Heat transfer coefficients (U and  $h_{fp}$ ) decreased significantly (p < 0.001) with increasing CMC concentration for all conditions within the range of experiments. It was observed that for the same levels of temperature and reciprocation frequency, increasing CMC concentration from 0.2 to 0.8% resulted in an average decrease of ~10-22% in the values of corresponding heat transfer coefficients (Table 4.3). Results showed an increase in both liquid (Figure 4.3) and particle-side (Figure 4.4) heat transfer resistance with increasing CMC concentration. This is due to the dramatic increase in the shear resistance of the fluid at higher CMC concentrations (Abdelrahim et al., 1994). As fluid viscosity increases with CMC concentrations, thickness of the boundary layer around the can wall and particle surface becomes larger, resulting in lower heat transfer rates. Previous studies with other rotary retorts have also shown similar trends of reduced heat transfer rates at higher viscosity levels (Meng and Ramaswamy, 2007a, 2007b; Hassan et al., 2012). Lenz and Lund (1978) found that heat transfer coefficients were higher in water than in 60% aqueous sucrose solution. Sablani and Ramaswamy (1996) compared the heat transfer coefficients to Nylon particles in oil and water and observed that U and h<sub>fp</sub> values were higher with water than oil. The present findings on the effect of CMC concentration on the heat transfer were consistent, in general, with the results from previous studies. However, the relative values of U were found to be greater during reciprocating agitation thermal processing than other works on non-Newtonian fluids involving rotary modes of agitation (Meng and Ramaswamy, 2005, 2007b; Hassan et al., 2012). Higher agitation intensity not only increased the turbulence but also made the shear thinning can liquid (CMC) less viscous.

Further, the fluid-side heat transfer resistance also decreases considerably, resulting in higher values of U. Stoforos and Merson (1992) evaluated  $h_{fp}$  with Teflon particles in Newtonian fluids subjected to axial rotation and reported that  $h_{fp}$  values increased with increasing fluid viscosity; however, they explained this observation with their particle motion study, which showed that in low viscosity liquids, the high density Teflon settled at the bottom of the can due to its weight. However, as viscosity increased, buoyancy forces also increased and hence Teflon spheres did not settle down resulting in increase in particle to fluid relative velocity. Yet, for particles close to the density of water like nylon, higher viscosity will lead to slowing down of particle due to viscous forces, resulting in lower U an  $h_{fp}$ .



Figure 4.5: Typical experimental time temperature profiles of liquid and particle centers for cans processed at 120 °C retort temperature, 0.5 % CMC concentration, 10 cm headspace and 15cm amplitude.

# 4.4.2.3 Effect of reciprocation frequency

Heat transfer coefficients, U and  $h_{fp}$ , increased significantly (p < 0.001) with increasing reciprocation frequency. For the same levels of CMC concentration and retort temperature, increasing the reciprocation frequency from 1 to 4 Hz almost doubled the value of corresponding heat transfer coefficients. This can be explained by the improved mixing due to higher degree of turbulence by rapid back and forth motion of container, resulting in increased relative particle to fluid velocity (Anantheswaran and Rao, 1985; Sablani, 1996; Hassan *et al.*, 2012). In Figure 4.5, the experimental time temperature profile of liquid and particle centers are shown.

It can be seen that the liquid and particle centers temperature rose more rapidly at higher reciprocation frequencies. Rapid heat transfer rates at higher agitation speeds have also been documented by other researchers. Lenz and Lund (1978) found that increasing the rotation speed from 3.5 to 8.0 rpm significantly increased the heat transfer. Stoforos and Merson (1992) found that increasing rotational speed from 9.3 to 101 rpm resulted in increasing U by 1.2-2.0 times. Sablani and Ramaswamy (1996) observed that increasing rotational speed from 10 to 20 rpm resulted in increases in h<sub>fp</sub> by 56% for oil and 53% for water and increase of U by 24% for oil and 13% for water. Meng and Ramaswamy (2007b) and Ramaswamy and Dwivedi (2011) reported a significant effect of the rotational speed on the associated heat transfer coefficients. These observations conform to the findings of the present study.

# 4.4.3 Experimental design (II)

Values of U and  $h_{fp}$  as affected by reciprocation amplitude and can headspace at various levels of reciprocation frequency are presented in Table 4.5 and ANOVA results for fitting a twofactor interaction (2FI) model are summarized in Table 4.6. The factors used to ensure adequacy of model fitting are same as those for CCRD design and discussed in Section 3.2a. Based on the sum of squares (Table 4.6), the importance of the independent variables on U and  $h_{fp}$  in reciprocation agitation could be ranked in the following order: reciprocation frequency > reciprocation amplitude > headspace. Response surface models (Equation 4.10, 4.11) were developed for response surface analysis of U and  $h_{fp}$  as influenced by the selected process variables.  $R^2$  values for U and  $h_{fp}$  were found to be 0.99, and 0.98 respectively, which indicated that the developed regression models were suitable to explain the heat transfer phenomena in the present system.

$$U = 225.36 + 203.12 R + 13.05A + 5.47H - 0.675RA - 2.39RH + 0.06AH$$
(4.10)

$$h_{fp} = 101.15 + 245.48R + 8.71A + 18.42H + 3.31RA + 1.42RH - 0.07AH$$
(4.11)

where, U and  $h_{fp}$  are the overall heat transfer coefficient and fluid-to-particle heat transfer coefficients (W/m<sup>2</sup> °C); and, R, A and H are the values for reciprocation frequency (Hz), reciprocation amplitude (cm) and can headspace (mm) respectively.

# 4.4.3.1 Effect of reciprocation amplitude

It was observed that an increase in reciprocation amplitude increased U and  $h_{fp}$  to a larger extent. At any fixed set of all other process variables, changing reciprocation amplitude from 5 to 25 cm, resulted in 30-35% increase in the values of U and  $h_{fp}$  (Figures 4.6). This is because increasing amplitude actually increases the can velocity, as the container has to cover more distance in the same time to maintain the same reciprocation frequency. Hence, due to higher cage velocity, heat transfer at wall is increased. Also, the particles experience a greater acceleration due to the motion of the can, resulting in higher liquid-to-particle relative velocity as well. After reciprocation frequency, the reciprocation amplitude was the second most significant factor affecting both U and  $h_{fp}$ .

Figure 4.6 clearly shows that reciprocation amplitude has positive effect on increasing the heat transfer coefficients U. These trends conform to previous works on reciprocation agitation thermal processing (Singh *et al.*, 2015a, 2015d). This also conforms to findings in works on rotary agitation (Stoforos and Merson, 1992; Meng and Ramaswamy, 2007a, 2007b; Hassan *et al.*, 2012) and oscillatory agitation (Sablani and Ramaswamy, 1999), if we consider reciprocation frequency analogous to rotation speed and reciprocation amplitude analogous to diameter of rotation.

Table 4.5: 3X3X2 Full factorial experimental design for evaluating U and h<sub>fp</sub> during reciprocation agitation of can as affected by reciprocation frequency, reciprocation amplitude, and can headspace.

	Factors	Response		
Reciprocation Frequency	Reciprocation Amplitude	Can Headspace	U <sup>a</sup>	$\mathbf{h_{fp}}^{\mathbf{a}}$
(Hz)	(Hz) (cm)		(W/m <sup>2</sup> °C)	(W/m <sup>2</sup> °C)
1	5	6	525(±12)	550(±31)
1	5	12	535(±14)	675(±27)
1	15	6	645(±10)	655(±19)
1	15	12	670(±11)	765(±28)
1	25	6	790(±17)	810(±31)
1	25	12	800(±14)	890(±26)
2	5	12	690(±11)	885(±32)
2	5	6	680(±27)	755(±34)
2	15	12	830(±28)	1000(±30)
2	15	6	795(±17)	865(±24)
2	25	6	900(±10)	1025(±24)
2	25	12	940(±26)	1165(±34)
3	5	12	890(±41)	1235(±30)
3	5	6	885(±18)	1100(±30)
3	15	12	980(±26)	1360(±31)
3	15	6	1025(±16)	1265(±21)
3	25	6	1125(±18)	1470(±21)
3	25	12	1120(±21)	1610(±30)

<sup>a</sup>values in brackets denote the standard deviation from the mean value

df			U			h <sub>fp</sub>			
Source		Sum of Squares	F-value	<i>p</i> -value	Sum of Squares	F-value	<i>p</i> -value		
Model	6	534500	189	< 0.0001	147100	80	< 0.0001		
R	1	352900	750	< 0.0001	113800	371	< 0.0001		
Α	1	180100	382	< 0.0001	258700	84	< 0.0001		
Н	1	420	1	0.3647	65884	21	0.0007		
RXA	1	364	1	0.0375	8778	2	0.0114		
RXH	1	616	2	0.2767	216	1	0.7951		
AXH	1	40	0.086	0.7751	56	1	0.0445		
Residual	11	5174			33662.15				
Total	17	539600			1.505E+006				
R <sup>2</sup>		0.99			0.98				
Adj-R <sup>2</sup>		0.98			0.97				
C.V.%		2.64%			5.51%				

Table 4.6: Analysis of variance showing influence of reciprocation frequency, reciprocation amplitude, and can headspace on U and h<sub>fp</sub>.

R, A, H = Linear effects of Reciprocation frequency, Reciprocation amplitude and Headspace respectively, RXA, RXH, AXH =Interaction effects of Reciprocation frequency, Reciprocation amplitude and Headspace respectively



Figure 4.6: a). Effect of amplitude on 'U' at various reciprocation frequency and 12 mm can headspace (W/m<sup>2</sup> °C); b). Effect of reciprocation amplitude on 'h<sub>fp</sub>' at various reciprocation frequency and 12 mm headspace (W/m<sup>2</sup> °C)



# Figure 4.7: Effect of can headspace on fluid to particle heat transfer coefficient h<sub>fp</sub> at 2Hz reciprocation frequency and various reciprocation amplitude (W/m<sup>2</sup> °C)

# 4.4.3.2 Effect of can headspace

Can headspace significantly affected  $h_{fp}$  (p<0.001) but did not significantly affect U (p>0.05), although U values also increased slightly with increasing headspace. Increasing values of  $h_{fp}$  at higher headspace (Figure 4.7) can be attributed to higher extent of mixing produced on account of movement of larger headspace bubble. However, bubble size beyond a critical level may have a dampening effect on the extent of mixing, due to disturbance in bubble-liquid phenomena created by large bubble size (Naveh and Kopelman, 1980). Further, Ramaswamy and Grabowski (1996) noted that an insulation effect due to air present in the headspace may increase heat transfer resistance. Yet, various researchers (Sablani and Ramaswamy, 1999; Dwivedi and Ramaswamy, 2010c) have found  $h_{fp}$  to increase with increase in headspace in the range 6-12 mm in other modes of agitation, which is consistent with the observations in this study. This is mainly due to the evaporation effect of boiling liquid at the top of the can which enhances the heat transfer from top wall (Singh and Ramaswamy, 2015a). Erdogdu and Tutar (2011) validated this trend through their CFD analysis and

observed that headspace temperature increased rapidly due to lower heat capacity and viscosity of air, resulting in better heat transfer coefficients.

# 4.4.4 Optimization of reciprocation intensity for minimizing quality losses

There are two types of quality damage that might occur in canned foods subjected to reciprocating agitation thermal processing. The first is the thermal damage due to the severity of temperature-time effect and can be designated as *thermal quality loss*. This loss, present in other modes of agitation as well, is generally minimized appreciably under rapid heating conditions such as reciprocating agitation due to enhanced heat transfer and lower process time. Since the *thermal quality loss* is enhanced at situation involving poor heat transfer, this loss can be considered to be proportional to the reduction in the overall heat transfer resistance represented by reciprocal of overall heat transfer coefficient (1/U). Within the range of the heat transfer conditions studied, the thermal quality loss can be normalized using Equation 4.4.

In addition to thermal loss, quality of food products like canned vegetables (peas, mushrooms, beans, sliced tomatoes etc.) and fruits (mango pulp, strawberries, avocados etc.) may degrade on rapid agitation due to higher impact intensity and collision frequency. These impact induced quality losses may result in texture degradation, nutrient leaching etc. and hence, food products processed using reciprocating agitation may not be acceptable to consumers. These agitation quality losses are accelerated at higher reciprocation intensity (frequency or amplitude), and it is imperative that we try to reduce this *agitation quality loss*. Thus we see a contradiction - thermal quality losses are reduced at higher agitation intensity while the agitation quality losses are enhanced. Agitation quality loss is directly proportional to the agitation force (and hence acceleration) felt by the can during reciprocation and thus can be considered directly proportional to reciprocation intensity. Reciprocation intensity is derived (Equation 4.6) from position analysis of reciprocating cage (Walden and Emanuel, 2010). This can also be normalized within the range of conditions employed in this study as shown in Equation 4.5.

Total quality loss in any product is a sum of these two losses. For some products with low temperature sensitivity (z) of quality attributes (ex. soups which may change color quickly but will

not degrade due to agitation), thermal loss may be considered the dominant factor, while for others high z (ex. soft solids like green beans, which may incur high texture degradation on shaking), agitation induced losses may be dominant. Hence, final quality loss indicator (QLI) was considered to be a function of both  $TQL_{norm}$  and  $AQL_{norm}$ . A linear model (Equation 4.12) was used to estimate QLI for different kinds of products. QLI for different values of 'a' is plotted in Figure 4.8.



Figure 4.8: Effect of reciprocation intensity on quality loss during reciprocation thermal processing.

It is seen from Figure 4.8 that thermal (a=1) and agitation (a=0) induced losses follow an inverse trend i.e. thermal loss decreased while agitation loss increased on increasing reciprocation intensity. For products with high fractional contribution of thermal quality loss (i.e. 'a' close to 1), operating near highest agitation intensity would be desirable, while for products with lower fractional contribution of thermal loss, lower reciprocation intensities would be more desirable to minimize quality loss (QLI). On fitting a polynomial, it is seen that for all values of 'a', the fitted polynomials intersected at a reciprocation intensity of 18.0 ms<sup>-2</sup> (=1.8g). At a higher reciprocation

intensity than 18.0 ms<sup>-2</sup>, the contribution of agitation induced losses is more dominant, while at lower intensity, contribution of thermal quality losses are more dominant. This value of reciprocation intensity can be obtained by multiple combinations of reciprocation frequency and amplitude (Equation 4.10). The critical value of reciprocation intensity obtained in our study is lower than the recommended reciprocation intensity of 3-4g (Walden and Emanuel, 2010) for reciprocating cans containing béchamel sauce, as béchamel sauce is not affected much by agitation. Walden and Emanuel (2010) also pointed that the recommended intensity will be lower for other products whose quality may degrade due to agitation.

#### 4.5 Conclusions

The effect of operating temperature, reciprocation frequency, reciprocation amplitude, can headspace and liquid viscosity on the coefficients U and hfp related to a food simulating liquidparticulate mixture (Nylon particles in a non-Newtonian solution of CMC) was studied during thermal processing with reciprocation container agitation. It was found that all the process variables were significant for the coefficient h<sub>fp</sub> and only frequency, amplitude and liquid viscosity were significant process variables affecting coefficient U. U and h<sub>fp</sub> increased with increase in frequency, amplitude, headspace and temperature and decreased with increase in CMC concentration. Reciprocation frequency followed by reciprocation amplitude were the most significant factors and increased heat transfer tremendously due to increase in liquid-to-particle relative velocity. Increase in headspace also resulted in higher U and hfp due to increase in mixing on movement of headspace bubble. Despite decreasing thermal quality losses, in some products, rapid agitation may incur agitation induced quality loss leading to texture and other degradations. A reciprocation intensity of 18.0 ms<sup>-2</sup> (1.8g) which may translate to various combinations of reciprocation frequency and amplitude such as 3 Hz-10 cm, 2 Hz-20 cm etc., was found to be the critical for thermal and agitation induced quality losses. Reciprocation intensity greater than 18.0 ms<sup>-2</sup> are desirable for products which are not susceptible to agitation induced quality losses, while a lower intensity is desirable for products which are susceptible to agitation losses. This study swill be useful to understand the phenomenon of heat transfer in non-Newtonian fluids and the optimized conditions reported will be valuable for designing a reciprocation thermal process.

#### **PREFACE TO CHAPTER 5**

It was demonstrated in Chapter 3 that,  $h_{fp}$  calculation problems could result primarily due to very fast heat transfer resulted from rapid motion of the container. To solve this problem a methodology was established in Chapter 3 and U and  $h_{fp}$  were calculated for heat transfer to canned particulate non-Newtonian fluids during reciprocation agitation thermal process. It was found that the refined method was appropriate for both slow and rapid heating conditions.

Further in Chapter 4, the effect of process variables on U and  $h_{fp}$  was discussed in detail. It was demonstrated that rapid agitation may also trigger agitation induced quality loss to the particulate product. To solve this problem, optimisation was carried out in order to make a compromise between thermal and agitation induced quality losses to canned mixture.

Chapter 5 elucidates the effect of various particle characteristics on heat transfer coefficients in canned liquid particulates subjected to reciprocating agitation. Four most influencing particulate variables were selected by performing preliminarily experiments and their effect of U and  $h_{fp}$  were analysed. This study shall be useful to establish the reciprocation agitation thermal process for particulates of different physical characteristics.

Part of this research was presented in 2015 at the 12th International Congress on Engineering and Food, ICEF12, June 14-18, Québec City, Canada.

Parts of this Chapter have been published as an original research article entitled "*Effect of Product Related Parameters on Heat Transfer Rates to Canned Particulate non-Newtonian Fluids (CMC) During Reciprocating Agitation Thermal Processing*" in Journal of Food Engineering, 165, 1–12.

The experimental work and data analysis were carried out by the candidate under the supervision of Dr. H. S. Ramaswamy

The full manuscript as presented in Chapter 5 does contain some sections already presented in previous chapters. Rather than deleting them, they have been left in place to provide continuity

# Chapter 5. EFFECT OF PARTICULATE CHARACTERISTICS ON HEAT TRANSFER COEFFICIENTS IN CANNED NON-NEWTONIAN FLUID PARTICULATES FOODS DURING RECIPROCATING AGITATION THERMAL PROCESSING

# 5.1 Abstract

Overall (U) and fluid-to-particle heat transfer coefficients ( $h_{\rm fp}$ ) in canned particulates suspended in non-Newtonian fluid undergoing reciprocation agitation was evaluated in a pilot-scale vertical steam retort. The retort was modified to provide reciprocating agitation of cans during thermal processing. Four influencing product parameters were identified and their effect on the heat transfer during reciprocating container agitation was studied. For this, 307x409 cans were filled with Nylon particles in a non-Newtonian fluid (1% CMC), processed at required operating conditions. Two full factorial experiment designs (3x3x3) were used for the study. Reciprocation frequency (1, 2, 3 Hz), being the most influential system parameter, was selected as common factor in both the design. Apart from reciprocation frequency, particle size (15, 19, 25 mm) and particle density (Polypropylene (830kg/m<sup>3</sup>), Nylon (1128kg/m<sup>3</sup>), and Teflon (2210kg/m<sup>3</sup>)) were varied in a first design, while particle concentration (20, 30, and 40% (v/v)), and particle shape (cube, sphere, and cylinder) were used to relate U and  $h_{\rm fp}$  to these product variables in the second design.

Analysis of variance showed that the particle concentration, shape (contributing to sphericity), size and density were highly significant factors (p < 0.001) for calculation of both the heat transfer coefficients (U and  $h_{fp}$ ). It was noted that, with an increase in particle size, the associated U and  $h_{fp}$  values tend to decrease. Also, in terms of density, Nylon particles showed the maximum and polypropylene showed the minimum values of heat transfer coefficients. Among the three shapes of the particle both the heat transfer coefficients followed the trend; Cube > Sphere > Cylinder. An increase in particle concentration showed an initial increasing trend in U and  $h_{fp}$  values to certain limit (20%) and (30%) respectively, which subsequently, decreased with a further increase in particle concentration.

# 5.2 Introduction

Thermal processing is a well-recognized technique for long term extension of shelf life and is recently being used to even obtain high product quality, through use of agitation processing and thinprofile packages (Singh et al., 2015b). Axial and end-over-end modes of agitation are generally used in industries to impart agitation while thermal processing. In end-over-end mode of agitation, a retort crate containing vertically placed cans rotates around a central horizontal axis, and is common in batch processing systems. In axial mode of agitation, cans are rotated in the horizontal plane, and this mode of rotation is encountered with continuous systems such as reel and spiral cookers. As compared to still/stationary mode, agitating retorts provide a uniform heat distribution and promote enhanced quality retention of thermally processed food product (Singh et al., 2015d). This is because agitation helps in increasing the convection current inside the food container and hence, promotes high heat transfer rate providing opportunity for employing high temperature short-time (HTST) processing. HTST techniques provide same level of sterility as conventional processes but offer better quality retention (Ramaswamy et al., 1993, Awuah et al., 2007). The HTST conditions can be realized in convection heating foods under normal commercial thermal processing (canning) conditions. They are also possible in conduction heating foods if processed in thin profile forms or independently processed and packaged under aseptic processing conditions.

Various modes of agitation such as end-over-end, axial and bi-axial agitation processing have been studied by researchers to enhance the heating rate of liquid particulate mixtures (Sablani and Ramaswamy, 1999; Meng and Ramaswamy, 2007a, 2007b and Ramaswamy and Dwivedi, 2011). Heat transfer to liquid particulate foods is accelerated through the use of agitation processing which induces forced convection in these products. It also increases the mixing inside the package and establishes a more uniform temperature distribution inside which helps to promote better quality retention. Apart from rotary agitation, recently, reciprocating agitation in the form of rapid back and forth motion of containers has been proposed to increase heating rates in canned foods (Walden and Emanuel, 2010). Recently, Singh *et al.* (2015b) found that reciprocating agitation was effective in reducing process time by 46-62% resulting in better quality retention as compared to conventional still mode of processing. Hence, the system has potential to preserve the quality of the thermally processed food. Thermal processing is a process which relies on mathematical models for process calculations and optimization. Heat penetration parameters are commonly used in combination with the process calculation methods like Ball, Stumbo and Pham formula for the establishment and optimization of the thermal process. Theoretical models are useful tools for the design, optimization and validation of such food systems; however the usefulness of the theoretical models depends upon the accuracy of the input physical parameters. In the canned liquid particulate system, the resulting heat transfer is a combination of the overall heat transfer coefficient (U) and the fluid-to-particle heat transfer coefficient ( $h_{fp}$ ) (Stoforos and Merson, 1992). The values of U and  $h_{fp}$  are necessary parameters, besides the thermo physical properties of the product, required for the modeling programs (Sablani and Ramaswamy, 1996).

To optimize the processing conditions in such a system, there is a need to understand the influence of various physical properties of the product such as particle shape, density, concentration, and size on the heat transfer coefficients. These factors have been found to be important in end-overend agitation processing (Naveh and Kopelman, 1980; Anantheswaran and Rao, 1985; Sablani and Ramaswamy, 1996, 1997; Ramaswamy and Sablani 1997a; Meng and Ramaswamy, 2007a, 2007b) as well as axially rotating cans (Lenz and Lund, 1978; Deniston *et al.*, 1987 and Ramaswamy and Dwivedi, 2011). On the other hand there is a lack of similar studies for understanding the influence of product parameters on U and h<sub>fp</sub> under reciprocation mode of agitation processing. Based on the available literature, it can therefore be realized that the product parameters influence the heat transfer rates is important for the optimized thermal process. Although, Singh *et al.* (2015b and 2015d) investigated reciprocating agitation thermal processing, yet, these works were more focused on system parameters.

In Chapter 3, a methodology was developed for analysing the heat transfer to canned particulate non-Newtonian fluids during reciprocating agitation thermal processing. Nylon spheres in CMC aqueous solutions were used and it was shown that at high reciprocation intensity the conventional method of calculation of  $h_{fp}$  will yield uncertain or indefinite values of heat transfer coefficients. In order to overcome this problem a new approach to compute U and  $h_{fp}$  was proposed. Furthermore, the effect of system related/operational variables on associated heat transfer

coefficients was studied (Chapter 4) in order to test the validity of the developed methodology. It was found that the retort temperature, reciprocation speed, amplitude, headspace and CMC concentration were significant factors for calculation of heat transfer coefficients.

This Chapter shall investigate the effect of product related variables such as particle concentration, particle shape, particle size, and particle density on U and h<sub>fp</sub> in canned particulates in non-Newtonian fluids. The results shall be useful in understanding how the heat transfer associated with reciprocation agitation can be optimized for thermal processing of canned non-Newtonian fluid particulate mixtures containing particles of different physical and thermal properties.

#### 5.3 Material and methods

#### 5.3.1 Materials used

A pilot scale reciprocating retort (Singh *et al.*, 2015b), developed by modifying a vertical static steam retort (Loveless Manufacturing Co., Tulsa, OK), was used in this study. This retort consisted of a reciprocating cage powered by a  $\frac{1}{2}$  hp direct current magnetic motor. The reciprocating cage was capable of holding 4 vertical cans (No. 2) along the diameter of the retort. The speed of reciprocation was controlled through an external voltage controller.

Carboxymethyl cellulose (CMC, Sigma, St. Louis, MO) at 1% aqueous concentration was used as the non-Newtonian fluid. Because of the low concentrations of CMC used in the tests, the heat capacity of the fluid was assumed to be the same as that of water (Anantheswaran and Rao, 1985). To overcome the uncertainty in the thermo-physical property data associated using real foods, Nylon, Teflon, and Polypropylene particles (Small Parts Inc., Miami, FL) were used as model food particles. For studying the effect of particle shape (sphericity), Nylon particles of three different shapes were used, viz. sphere, cube and a finite cylinder. All particles were of similar significant dimension (sphere = 19 mm diameter, cylinder = 19 mm diameter and 19 mm length, and cube = 19 mm length). Sphericity was defined as the ratio of the surface area of a sphere (with the same volume as the given particle) to the surface area of the particle as given by:

$$S = \frac{\pi^{1/3} (6V_p)^{2/3}}{A_p}$$
(5.1)

where,  $V_p$  and  $A_p$  are the volume and surface area of the particle, respectively. The sphericity of the sphere was 1.00 which reduced to 0.874 and 0.806, respectively, for the cube and cylinder. To study the effect of size, three different diameters (15, 19, and 25 mm) of Nylon particles were used. Thermo-physical properties of liquid and particles were obtained from available literature (Sablani and Ramaswamy, 1997 and Meng and Ramaswamy, 2005).

# 5.3.2 Experimental design and statistical analysis

Based on some preliminary experiments and existing literature (Ramaswamy and Sablani, 1997b, Meng and Ramaswamy, 2007a; 2007b and Sablani and Ramaswamy, 1996; 1997), four product parameters with potential to affect the heat transfer rate in liquid particulate foods were selected: particle shape, particle size, particle density and particle concentration. The influence of selected product parameters on U and  $h_{fp}$  were studied using two full factorial designs, in combination with one of the most prominent system variable; reciprocation frequency. Table 5.1 provides the details of these experimental designs with independent variables at selected levels. Apart from the two designs, 9 separate test runs were carried out with a single particle of different shapes (cube, sphere, and cylinder) in can at different reciprocation frequency (1, 2 and 3 Hz) providing uninhibited heat transfer conditions for the particle. All test runs were repeated twice, each with two cans placed equidistant from the axis of reciprocation, providing 4 repetitions in each run.

The experimental data was fitted to a quadratic model. Analysis of variance (ANOVA) was used to find significant terms in the model for each response. The adequacy of model was checked by comparing the  $R^2$  and adjusted- $R^2$  values and by ensuring that the lack of fit was not significant and coefficient of variation was below 10%.

Table 5.1: Experiment design to study the effect of product parameters on heat transfer rates to non-Newtonian fluid particulates in cans subjected to reciprocating agitation thermal processing

Design	Independent variables	Selected level of independent variable	Fixed parameters	
Full Factorial, <sup>-</sup> 3X3X3 (27exp.)	Reciprocation frequency (Hz)	1, 2, 3 Hz	Headspace = 12mm. Reciprocation amplitude =	
	Particle size (mm)	15, 19, 25 mm	15cm.	
			Particle shape = Sphere	
	Particle density $(l_{1/2}/m^3)$	Polypropylene (830), Nylon (1128) Teflon (2210)	CMC concentration = $1\%$ .	
	(kg/m)	(1120), 101011 (2210)	Particle concentration = $30\%$ (v/v)	
	Particle concentration (%v/v)	20, 30, 40 %		
Full	Pagipropotion		Can headspace = $12$ mm.	
Factorial, 3X3X3	frequency (Hz)	1, 2, 3 Hz	Reciprocation amplitude =	
(2/exp.)	Particle shape	Cube (0.874), Sphere (1),	150111.	
	(Sphericity)	Cylinder (0.806)	CMC concentration = 1%.	
Single Particle in Can (9exp.)	Reciprocation frequency (Hz)	1, 2, 3 Hz	Size = 19mm (see section 2.1)	
	Particle shape (Sphericity)	Cube (0.874), Sphere (1), Cylinder (0.806)		

#### 5.3.3 Temperature collection and thermal processing

Temperature measurements of the liquid inside the cans were gathered using CNS copperconstantan needle-type thermocouples (diameter 1.6 mm, Ecklund-Harrison Technologies Inc., Fort Myers, FL) with tips located at the geometric center of the can. Particle center temperature was measured using flexible CNS copper-constantan wire thermocouples (diameter 0.0762 mm, Omega Engineering Corp., Stamford, CT) introduced into the particle center through a fine hole and fixed by a small amount of epoxy glue (Sablani and Ramaswamy, 1996). For thermal processing, 307x409 metallic cans were filled with liquid (1% CMC solution) and particle (as outlined in the experimental design) mixture leaving a 12 mm headspace and were held vertically in the reciprocating cage inside the retort (shown in Chapter 4, Figure 4.1). The empty space in the cage was filled with dummy cans to provide for some ballast load. Retort was closed from the top and locked. Motor was turned on and set at the required reciprocation frequency. Steam was turned on and retort was heated up to operating temperature (121.1 °C) and then the temperature was held constant for 30 min or till all thermocouples read 121.1 °C, whichever was later. After completing the process, steam was turned off and cold water inlet was opened while the reciprocating agitation was continued until the completion of the cooling cycle.

# 5.3.4 Methodology for evaluation of U and h<sub>fp</sub>

Heat transfer coefficients were calculated using a methodology developed in chapter 3. For this purpose, the heating rate indices of the liquid and particle were used to compute U and  $h_{fp}$  in reciprocating agitation. Values of  $h_{fp}$  were estimated by fitting the predicted temperature profile of the cold-spot (particle-center) with simulated temperature profile of the cold-spot under constant retort temperature conditions ( $T_{crt}$ ) for Fourier number greater than 0.2. Predicted temperature profile of liquid ( $T_{pred-1}$ ) and particle-center ( $T_{pred-p}$ ) under constant retort operating conditions was generated from heating rate indices of liquid and particle (Equation 5.2).

$$T_{\text{pred}} = T_{\text{crt}} - (T_{\text{crt}} - T_i) * 10^{\left(-\frac{t-t_0}{f_h}\right)} \qquad \tau > 0.2$$
 (5.2)

$$= T_i \qquad \forall \tau < 0.2$$

where,  $T_{pred}$ ,  $T_{crt}$  and  $T_i$  are the predicted temperature profile, constant retort temperature and initial temperatures (°C) respectively;  $f_h$  is the heating rate index (s); t is the time instant (s);  $t_o$  is the time (s) at which Fourier number  $\tau=0.2$ .

The simulated temperature profile of the particle-center was generated from the equilibrated liquid profile ( $T_{pred-L}$ ) using a finite difference simulation of the unsteady state heat conduction equation for particles of different shapes immersed in liquid with prescribed initial and boundary conditions (Ramaswamy and Sablani, 1997b). Instead of actual experimental profile the 'predicted' liquid ( $T_{pred-1}$ ) and particle ( $T_{pred-p}$ ) temperature profiles, was used to subsequently calculate U and  $h_{fp}$ . In Chapters 3 and 4, only spherical shaped particles were used and hence, using equation 3.1 (Chapter 3) was sufficient. However for cube and cylindrical shaped particles appropriate equations describing unsteady heat conduction through the respective geometry must be used (5.3 and 5.4).

For cube shaped particles : 
$$\frac{\partial T}{\partial t} = \alpha_p \left( \frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right)$$
 (5.3)

For cylindrical shaped particles: 
$$\frac{\partial T}{\partial t} = \alpha_{p} \left( \frac{\partial^{2} T}{\partial r^{2}} + \frac{2}{r} \frac{\partial T}{\partial r} + \frac{\partial^{2} T}{\partial z^{2}} \right)$$
 (5.4)

These equations and numerical procedures involved with the finite difference method where described in detail in Ramaswamy and Sablani (1997b) for the three particle shapes. For fitting the predicted and simulated profiles, an initial guess of  $h_{fp}$  was assumed and then difference between the  $F_0$  values of the two profiles ( $T_{pred-L}$  and  $T_{pred-P}$ ) were minimized by sequentially changing the value of  $h_{fp}$ . Further details about methodology to calculate heat transfer coefficients are explained in Chapter 3.

#### 5.4 Results and discussion

# 5.4.1 Experimental design (I): Effect of particle size, particle density and reciprocation frequency on U and h<sub>fp</sub>

# 5.4.1.1 Model fitting and analysis of variance

To analyze the effect of particle size and density on heat transfer, calculated values of U and  $h_{fp}$  presented in Table 5.2 were subjected to ANOVA analysis to test significance of the effect of each variable. Based on model summary statistical analysis (Table 5.3), quadratic models (Equation 5.5 and 5.6) were developed for U and  $h_{fp}$  as influenced by the selected product variables (reciprocation frequency, F; particle size, Z; particle density, D):

$$U = -87.192 + 217.3142F + 1.145D - 10.8Z + 0.005FD - 3.6FZ - 0.0007DZ - 3.1F^{2}$$
(5.5)  
- 0.0003D<sup>2</sup>  
$$h_{fp} = 619.931 + 271.32F + 2.005D - 0.9Z + 0.0105FD - 10.325FZ - 0.001D + 41.45F^{2}$$
(5.6)  
- 0.0006D<sup>2</sup> + 0.3919Z<sup>2</sup>

where, U and  $h_{fp}$  are the overall heat transfer coefficient and fluid-to-particle heat transfer coefficients; and, F, Z and D are the actual values for reciprocation frequency, particle size and particle density, respectively. For the corresponding fitting of the explanatory models and the variation of U and  $h_{fp}$  values, the sum of squares of the suggested model was analyzed. These analyses indicated that adding terms up to quadratic significantly improved the model for estimating both the heat transfer coefficients. The estimated regression coefficients of the models for the response variables, along with the corresponding coefficients of determination (R<sup>2</sup>) are given in Table 5.4. Coefficient of variation (CV) and adjusted-R<sup>2</sup> were also calculated to check the model adequacy. Coefficient of determination, R<sup>2</sup>, is the proportion of variation in the response attributed to the model rather than to random error. The R<sup>2</sup> values for U and  $h_{fp}$  in the present study were found to be 0.98 and 0.99 (Table 5.4), respectively, indicating that the regression models were suitable to explain the behavior. Higher adj-R<sup>2</sup> obtained for both responses indicated that non-significant terms have not been included in the model. Coefficient of variation (CV) should always be within 10% for a model to be precise and reliable (Montgomery, 2001).

Table 5.2: 3X3X3 Full factorial experimental design for evaluation of U and h<sub>fp</sub> during reciprocating agitation of canned liquid particulates as affected by reciprocation frequency, particle size and particle density.

		Density (kg/m <sup>3</sup> )					
Reciprocation Frequency (Hz)	Particle	Polypropylene (830)		Nylon	(1120)	<b>Teflon (2110)</b>	
	Size (mm)	U <sup>a</sup> (W/m <sup>2</sup> °C)	h <sub>fp</sub> <sup>a</sup> (W/m <sup>2</sup> °C)	<sup>a</sup> U <sup>a</sup> <sup>2</sup> °C) (W/m <sup>2</sup> °C)	h <sub>fp</sub> <sup>a</sup> (W/m <sup>2</sup> °C)	U <sup>a</sup> (W/m <sup>2</sup> °C)	h <sub>fp</sub> <sup>a</sup> (W/m <sup>2</sup> °C)
1	15	580(±12)	660(±17)	735(±17)	900(±19)	690(±09)	810(±05)
2	15	700(±11)	895(±11)	910(±12)	1170(±13)	855(±14)	1060(±14)
3	15	880(±14)	1190(±11)	1080(±18)	1565(±20)	1010(±17)	1400(±20)
1	19	565(±15)	610(±17)	670(±10)	785(±04)	630(±11)	705(±13)
2	19	695(±18)	8007(±17)	825(±14)	1025(±14)	780(±17)	920(±14)
3	19	825(±27)	1060(±22)	980(±19)	1360(±32)	920(±21)	1225(±19)
1	25	480(±12)	460(±09)	570(±07)	585(±11)	535(±03)	530(±12)
2	25	590(±17)	600(±14)	705(±13)	765(±13)	660(±14)	690(±17)
3	25	700(±21)	795(±22)	835(±19)	1020(±28)	785(±10)	920(±21)

<sup>a</sup>values in brackets denote the standard deviation from the mean value

Our results showed that the coefficients of variation for U and  $h_{fp}$  were 1.48 and 3.53%, respectively, representing a better precision and reliability of the conducted experiments. For checking the model adequacy we have also verified the normality assumption (that the combination of variables follows a multivariate normal distribution). The normal probability plot of the residual

for U and  $h_{fp}$  is shown in Figure 5.1. It is clear from the curve that there is no indication of nonnormality, nor is there any evidence pointing to possible outliers and the equality of variance assumption does not seem to be violated. The actual and predicted plots of U and  $h_{fp}$  is shown in Figure 5.2. The graph indicates the models are adequate to be used to navigate within the design space.

Table 5.3. Model Summary Statistics for 3X3X3 Full factorial experimental design for evaluation of U and h<sub>fp</sub> during reciprocating agitation of liquid particulate cans as affected by reciprocation frequency, particle density and particle size.

Model Summary Statistics (Reciprocation Frequency-Particle Size-Particle Concentration)								
Source	Std. Dev	<b>R</b> <sup>2</sup>	Adjusted- R <sup>2</sup>	Predicted- R <sup>2</sup>	PRESS			
(U) Model								
Linear	157	0.8572	0.8612	0.8361	100200			
2FI	159	0.8541	0.8494	0.8073	117800			
Quadratic	<u>12</u>	<u>0.9861</u>	<u>0.9900</u>	<u>0.9801</u>	<u>6032</u>	Suggested		
			(h <sub>fp</sub> ) M	lodel				
Linear	109	0.8664	0.8490	0.8202	368800			
2FI	109	0.8828	0.8476	0.8097	390400			
Quadratic	<u>32</u>	<u>0.9915</u>	<u>0.9869</u>	<u>0.9787</u>	<u>43636</u>	Suggested		

It is clear from ANOVA (Table 5.4) that effects of all three independent variables were significant (p < 0.0001) for predicting U and  $h_{fp}$  in reciprocating agitation thermal processing of cans. The linear term of all the selected factors; the interaction term between reciprocation frequency and particle size and quadratic term of particle density were significant (p < 0.0001) for both U and  $h_{fp}$ . Apart from this, quadratic term of reciprocation frequency was significant (p < 0.001) for  $h_{fp}$ . Based on the sum of squares (Table 5.4), the importance of the independent variables on U and  $h_{fp}$  could be ranked in the following order: reciprocation frequency > particle size > particle density. With other modes of agitation, researchers have also reported that by varying the density and size of the particle used, the heat transfer could be varied to a large extent (Stoforos and Merson, 1992; Meng and Ramaswamy, 2007a; Hassan, 1984; Lenz and Lund, 1978; Sablani, 1996).

Table 5.4: Analysis of variance (ANOVA) results for U and h<sub>fp</sub>, demonstrating the effect of reciprocation frequency, particle size and particle density in reciprocating agitation thermal processing of cans.

Source		U		h <sub>fp</sub>		
-	Sum of Squares	F-value	p-value	Sum of Squares	F-value	p-value
Model	635700	480	< 0.0001	203400	220	< 0.0001
F	456800	2415	< 0.0001	103600	1005	< 0.0001
D	87139	206	< 0.0001	65942	64	< 0.0001
Z	78787	1121	< 0.0001	613900	595	< 0.0001
FD	7052	2	0.2402	707	1	0.4189
FZ	4165	28	< 0.0001	32408	31	< 0.0001
DZ	478	1	0.4208	412	1	0.5352
F <sup>2</sup>	1	1	0.5190	10308	10	0.0057
D <sup>2</sup>	1235	485.93	< 0.0001	212100	205	< 0.0001
R <sup>2</sup>	0.98			0.99		
Adj-R <sup>2</sup>	0.99	-		0.98	_	
CV	1.48	_		3.53	_	

F, D, Z = Linear effects of Reciprocation frequency, Particle density, and Particle size respectively;  $F^2$ ,  $D^2$ ,  $Z^2$  = Quadratic effects of Reciprocation frequency, Particle density, and Particle size respectively; FXD, FXZ, DXZ =Interaction effects of Reciprocation frequency, Particle density, and Particle size respectively



Figure 5.1: Normal plot of residuals for U and h<sub>fp</sub> (Design factors: frequency, particle size and particle density)



Figure 5.2: The predicted vs. actual plot for U and h<sub>fp</sub> (Design factors: frequency, particle size and particle density)

### 5.4.1.2 Effect of particle size

Particle size had significant effect on U and  $h_{fp}$  (Table 5.2). A decrease in the particle size resulted in an increase in the values of both heat transfer coefficients. Lowering the particle size from 25 to 15 mm has increased the U value by  $\approx 25\%$ . Similarly, by lowering the value of particle size from 25 mm to 19 mm,  $h_{fp}$  value increased  $\approx$ 30-35% and from 19 mm to 15 mm,  $h_{fp}$  value increased  $\approx 10-15\%$  (Table 5.2). This trend can be better observed from the contour plot (reciprocation frequency vs particle size) shown in Figure 5.3. The reason for the increase in heat transfer coefficients with a decrease in solid particle diameter could be due to the relative surface area to volume ratio which increases slightly with a decrease in the particle diameter. The ratio is 1.67 time higher for the particle with 15 mm diameter as compared with the same particle of 25 mm diameter. Moreover, at the same concentration by volume or weight, there will be more of smaller particles than the larger ones there by providing better turbulence (by increasing number of collisions) and hence, U and hfp values. In fact also in natural convection heat transfer conditions (still mode) the heat transfer may increase as we increase the number of particles in can as a result of gravitational forces. Dimou and Yanniotis (2011) showed that in still mode of process by increasing the number of asparaguses in the can, the brine velocity increased and remained higher at the beginning of the heat-up and cool-down phases. Similar findings were reported by other researchers on the influence of particle size in other modes of agitation (Hassan, 1984; Stoforos and Merson, 1992; Sablani, 1996; Meng and Ramaswamy, 2007a; Ramaswamy and Dwivedi, 2011). They explained the phenomena by the fact that a thicker boundary layer may be associated with larger particle diameters. Sablani and Ramaswamy (1997) noted that when the particle diameter in their experiment were increased from 19.05 to 22.25 mm (Nylon spheres), the fluid-to-particle heat transfer coefficient  $h_{fp}$  in oil decreased by 13%, and further increasing the size to 25 mm reduced the h<sub>fp</sub> by 24%. This trend of decreasing h<sub>fp</sub> was less pronounced when the particles were in water; when the particles were increased in size from 19 mm to 25 mm, the decrease was only around 9%. Since glycerin at 100% is more viscous than water, the drop in the value of  $h_{fp}$  will be even higher than in oil, as indicated in this study. Lenz and Lund (1978) observed that value of overall heat transfer coefficient U increases with increasing particle diameter; they also reported higher values for fluid to particle heat transfer coefficients when increasing particle diameters with water as a fluid.

# 5.4.1.3 Effect of particle density

Maesmans *et al.* (1992) showed that not all particle properties may necessarily affect fluid to particle heat transfer coefficient ( $h_{fp}$ ) as long as the relative motion between particle and liquid interface is not altered. In earlier studies, measurement of  $h_{fp}$  was mostly done with restricted particle motion and hence, particle density as not a significant parameter to affect  $h_{fp}$  values. This phenomena is verified by Sablani and Ramaswamy (1997) by comparing  $h_{fp}$  values for different particle densities, in the still mode of thermal processing. They found that particle density was not significant at 0 rpm, however, when particle motion was included, the effect of density on  $h_{fp}$  was found to be highly significant.

The influence of particle density on U and h<sub>fp</sub> values is presented in Figure 5.4 which was highly significant (p < 0.001) for U and  $h_{fp}$  values (Table 5.3). Overall at all reciprocation frequencies, h<sub>fp</sub> values showed the followed trend: Nylon > Teflon > PP. At maximum heat transfer scenario (3Hz of reciprocation frequency and 15 mm particle diameter), PP particle (lowest density) had the lowest  $h_{fp}$  (1190 W/m<sup>2</sup> °C), followed by Teflon (1400 W/m<sup>2</sup> °C) and Nylon (1560 W/m<sup>2</sup> °C). It can be explained by the density difference between particulates and fluid. Nylon has a median density closer to 1% CMC solution hence, could allow better mixing between liquid and particulates. While PP being the lightest particle stays on top, and Teflon being the heavier particle tends to settle down more on the bottom. This could restrict the particle motion (relative motion between fluid and particle) and reduce the heat transfer. The particles with different densities will result in different velocity profiles (boundary layer) when subjected to rapid back and forth motion (instead of rotary agitation), thereby, affecting the heat transfer coefficient to a different degree. From Figure 5.4 it can also be seen that the effect of particle density on U values and  $h_{fp}$  values are similar. The trends of U and h<sub>fp</sub> with particle density under reciprocating agitation was observed to be slightly different from those observed in studies on rotary agitation (Stoforos and Merson, 1992; Meng and Ramaswamy, 2007a; Ramaswamy and Dwivedi, 2011). A majority of these have shown h<sub>fp</sub> to be highest for Teflon and lowest for PP particles. This difference may be attributed to the fact that during rotary agitation, heavier particles continuously fall down due to gravity during rotation cycle of cage, thus improving heat transfer, as compared to lower density particles. However, during reciprocating agitation, the agitation is due to back and forth motion and is similar for all densities.



Figure 5.3: Contour plot for Nylon spheres of different diameters depicting the effect of particle size on: a) overall heat transfer coefficient (U), b) fluid to particle heat transfer coefficient (h<sub>fp</sub>)



Figure 5.4: Contour plot for spheres (19mm) of Nylon, Teflon, and Polypropylene depicting the effect of particle density on: a) overall heat transfer coefficient (U), b) fluid to particle heat transfer coefficient (h<sub>fp</sub>)

# 5.4.1.4 Effect of reciprocation frequency

Results of the present study (Table 5.2) indicated that increasing the reciprocation frequency resulted in large increase in the heat transfer coefficient values. Higher reciprocation frequencies helped to create greater turbulence, inducing greater motion to the particles and hence, was expected to enhance the rate of heat transfer. It can be visualized from Figure 5.7 that time taken by cold-spot to reach the operating temperature increases tremendously by using reciprocation agitation of containers. In the first detailed work on reciprocating agitation thermal processing, Singh *et al.*, (2015c) reported that the reciprocation frequency is the most significant factor for influencing the heat transfer coefficient with U and h<sub>fp</sub> increased by 3 and 8 times, respectively when the reciprocation frequency increased from 0 to 3 Hz. In a general sense, other studies on rotary mode of agitation have found similar results (Lenz and Lund 1978, Anantheswaran and Rao, 1985; Meng and Ramaswamy, 2007b; Dwivedi and Ramaswamy, 2010b; Ramaswamy and Dwivedi, 2011) because of the improved mixing in the can and reduction in the viscous forces associated with the fluids. Sablani and Ramaswamy (1996) used single Nylon particle in bath oil and reported that U and hfp values increased by 38% when the rotation speed increased from 10 to 20 rpm. Overall, reciprocation agitation had the most pronounced influence on heat transfer in liquid particulate canned systems. It should also be noted that in rotary agitation it is not possible to increase the rotation speed beyond a certain limit, as the centrifugal forces may start acting on the particle and they tend to stick in the can wall.

In the present study, it was observed that even for heavier particle (Teflon) the heat transfer increased (both the U and  $h_{fp}$ ) with reciprocation frequency (Figure 5.4). For 19 mm Teflon spheres, U increased from 630 to 920 W/m<sup>2</sup> °C and  $h_{fp}$  from 705 to 1225 W/m<sup>2</sup> °C, with an increase in reciprocation frequency from 1 to 3 Hz. However, some earlier reports observed that heat transfer coefficient associated with heavy particles (Teflon and Derlin) actually decreased with increasing speed of agitation (rotation) (Sablani, 1996). Stoforos and Merson (1992) also found decreasing  $h_{fp}$  with increasing rotational speed, for Teflon particles. The discrepancy between these trends could be explained by the fact that heavier particle have more kinetic momentum associated with them and higher agitation as reciprocating agitation causes greater amount of turbulence in the can fluid and hence, higher rate of heat transfer. Also in reciprocating agitation there is no limiting factor such as
centrifugal force acting upon particles at the high agitation scenario. For axial and bi-axial mode of agitation, Ramaswamy and Dwivedi (2011) reported that values of U and  $h_{fp}$  increased with an increase in rotation speed. They explained the phenomena by enhanced mixing at high rotation (24 rpm) caused by the movement of the headspace bubble.

## 5.4.2 Experimental design (II): Effect of particle concentration and particle shape on U and h<sub>fp</sub>

#### 5.4.2.1 Model fitting and analysis of variance

To analyze the effect of particle concentration and particle shape on heat transfer, coefficients calculated values of U and  $h_{fp}$  presented in Table 5.5 were subjected to ANOVA analysis to test significance of the effect of the each product variable. Based on model summary statistical analysis (Table 5.6), quadratic models (Equations 5.7 and 5.8) were developed for U and  $h_{fp}$  as influenced by the selected product variables.

$$U = 11721.14 + 290.21F + 4.93C - 25023.31S - 2.42FC - 64.90FS + 1.71CS - 0.07F^{2}$$
(5.7)  
$$- 0.14C^{2} + 13754S^{2}$$
$$h_{fp} = 33607.33 + 205.55F + 67.70C - 74354.9S + 1.56FC - 141.85FS + 22.30CS$$
(5.8)  
$$- 31.9F^{2} - 1.61C^{2} + 40221.31C^{2}$$

where, U and  $h_{fp}$  are the overall heat transfer coefficient and fluid-to-particle heat transfer coefficients; and, F, C and S are the actual values for reciprocation frequency, particle concentration and particle shape (Sphericity), respectively.

The estimated regression coefficients of the models for the response variables, along with the corresponding coefficients of determination ( $R^2$ ) are given in Table 5.7. Coefficient of variation (CV) and adjusted- $R^2$  were also calculated to check the model adequacy. The  $R^2$  values for U and  $h_{fp}$  in the study were found to be 0.99 and 0.97 (Table 5.7), respectively, indicating that the regression models were suitable to explain the behavior. Higher adj- $R^2$  obtained for both responses indicated that non-significant terms have not been included in the model. Our results showed that the coefficients of variation for U and  $h_{fp}$  were 2.30 and 7.18%, respectively, representing a better

precision and reliability of the conducted experiments. For checking the model adequacy we have also verified the normality assumption of ANOVA. The normal probability plot of the residual for U and  $h_{fp}$  is shown in Figure 5.5. It is clear from the curve that there is no severe indication of non-normality, nor is there any evidence pointing to possible outliers and the equality of variance assumption does not seem to be violated. The actual and predicted plots of U and  $h_{fp}$  are shown in Figure 5.6. The graph indicates the models are adequate to be used to navigate within the design space.

Table 5.5: 3X3X3 Full factorial experimental design for evaluation of U and h<sub>fp</sub> during reciprocating agitation of liquid particulate cans as affected by reciprocation frequency, particle concentration and particle shape.

		Particle shape (Sphericity)						
Reciprocati on Frequency (Hz)	Particle Conc. (%v/v)	Cube (0.874)		Sphere (1.00)		Cylinde	Cylinder (0.806)	
		U <sup>a</sup> (W/m <sup>2</sup> °C)	h <sub>fp</sub> <sup>a</sup> (W/m <sup>2</sup> °C)	U <sup>a</sup> (W/m <sup>2</sup> °C)	h <sub>fp</sub> <sup>a</sup> (W/m <sup>2</sup> °C)	U <sup>a</sup> (W/m <sup>2</sup> °C)	h <sub>fp</sub> <sup>a</sup> (W/m <sup>2</sup> °C)	
1	20	735(±17)	1020(±19)	700(±21)	735(±11)	630(±09)	635(±11)	
2	20	960(±15)	1200(±17)	880(±11)	950(±17)	795(±17)	850(±17)	
3	20	1120(±16)	1500(±21)	1070(±16)	1220(±17)	970(±17)	990(±17)	
1	30	700(±18)	1080(±15)	670(±17)	785(±17)	615(±17)	655(±12)	
2	30	875(±07)	1320(±17)	830(±18)	1020(±17)	745(±17)	930(±16)	
3	30	1080(±27)	1800(±16)	980(±22)	1350(±36)	880(±17)	1100(±23)	
1	40	635(±17)	760(±11)	615(±12)	675(±27)	550(±11)	550(±11)	
2	40	790(±10)	1050(±17)	745(±14)	850(±25)	670(±17)	715(±15)	
3	40	935(±32)	1400(±37)	880(±24)	1160(±25)	795(±13)	925(±20)	

<sup>a</sup>values in brackets denote the standard deviation from the mean value

ANOVA results (Table 5.7) revealed that all factors had a significant (p < 0.001) effect on U and  $h_{fp}$  (Table 5.7). The linear term of all factors and quadratic term of particle shape (Sphericity) were significant (p < 0.001) for U and  $h_{fp}$ . Apart from these the interaction terms of reciprocation frequency and particle concentration were significant (p < 0.001) on U and quadratic effect of particle concentration was significant (p < 0.001) on  $h_{fp}$ . Based on the sum of squares, the importance of independent variables on U and  $h_{fp}$  was in the following order: reciprocation frequency > particle shape > particle concentration. Other researchers have also reported that particle concentration and particle shape are highly significant factors for heat transfer happening in the system (Stoforos and Merson, 1992, Meng and Ramaswamy, 2007a; 2007b, Ramaswamy and Dwivedi, 2011, Ramaswamy and Sablani 1997b).

Table 5.6: Model Summary Statistics for 3X3X3 Full factorial experimental design for evaluation of U and hfp during reciprocating agitation of liquid particulate cans as affected by reciprocation frequency, particle concentration and particle shape.

Model Summary Statistics (Reciprocation Frequency-Particle Concentration-Particle Shape)									
Source	Std. Dev	R <sup>2</sup>	Adjusted- R <sup>2</sup>	Predicted- R <sup>2</sup>	PRESS				
(U) Model									
Linear	62	0.8583	0.8398	0.8106	120800				
2FI	64	0.8702	0.8312	0.7878	135200				
<u>Quadratic</u>	<u>18</u>	<u>0.9908</u>	<u>0.9859</u>	<u>0.9776</u>	<u>14264</u>	Suggested			
			(h <sub>fp</sub> ) Mode						
Linear	198	0.6045	0.5530	0.4727	121000				
2FI	211	0.6094	0.4922	0.3741	143600				
Quadratic	<u>72</u>	<u>0.9712</u>	<u>0.9507</u>	<u>0.9182</u>	<u>210600</u>	Suggested			

Table 5.7: Analysis of variance (ANOVA) results for U and h<sub>fp</sub>, demonstrating the effect of reciprocation frequency, particle shape and particle concentration in reciprocating agitation thermal processing of cans.

Source	U			h <sub>fp</sub>			
	Sum of Squares	F-value	p-value	Sum of Squares	F-value	p-value	
Model	631500	200	< 0.0001	220500	46	< 0.0001	
F	445000	1290	< 0.0001	112300	214	< 0.0001	
С	85090	245	< 0.0001	51170	10	0.0061	
S	11650	35	< 0.0001	315900	60	< 0.0001	
FC	7050	20	0.0003	2940	1	0.4635	
FS	500	2	0.2487	2360	1	0.5108	
CS	35	1	0.7564	5835	2	0.3057	
F <sup>2</sup>	1	0.1	0.9925	6100	2	0.2950	
C <sup>2</sup>	1230	4	0.0766	154700	30	< 0.0001	
$S^2$	75670	220	< 0.0001	646300	130	< 0.0001	
R <sup>2</sup>	0.99			0.97			
Adj-R <sup>2</sup>	0.98			0.95	-		
CV	2.30			7.26	-		

F, C, S = Linear effects of Reciprocation frequency, Particle concentration and Particle shape respectively;  $F^2$ ,  $C^2$ ,  $S^2$  = Quadratic effects of Reciprocation frequency, Particle concentration and Particle shape respectively; FXC, FXS, CXS =Interaction effects of Reciprocation frequency, Particle concentration and Particle shape respectively



Internally Studentized Results

# Figure 5.5: Normal plot of residuals for U and h<sub>fp</sub> (Design Factors: reciprocation frequency, particle concentration and particle shape.)



Figure 5.6: The predicted vs. actual plot for U and h<sub>fp</sub> (Design factors: frequency, particle concentration and particle shape)

#### 5.4.2.2 Effect of particle concentration

As explained by Sablani and Ramaswamy (1997), in the presence of high concentrations of particles, the velocity gradient surrounding each particle may be affected. Thus, the magnitude of heat transfer coefficients can be expected to depend upon the particle concentration. Table 5.7 summarizes the mean values and standard deviations of overall and fluid to particle heat transfer coefficients obtained at various particle concentrations. The U values ranged from 260 to 1120 W/m<sup>2</sup> °C and h<sub>fp</sub> values ranged from 320 to 1800 W/m<sup>2</sup>°C. The response plot of the influence of particle concentration on the U and h<sub>fp</sub> at different reciprocation frequency is presented in Figure 5.7. It was expected that the number of particles present inside the can would affect the mixing behavior and hence, the heat transfer coefficient. The particle causes secondary agitation contributing to the mixing of can contents due to their motion (Sablani and Ramaswamy, 1997). This phenomenon helps in increasing the turbulence and hence, the heat transfer coefficients.

From Figure 5.7, it can be seen that up to a certain limit, increasing the particle concentration (from single particle to 20%) helps to increase the overall heat transfer coefficient by ~3 times. However, beyond that limit the U values actually start decreasing. Similarly hfp value increased from 320 to 1320 W/m<sup>2</sup> °C up till 30% of particle concentration and then started to decrease. The increase in the respective heat transfer coefficient could be explained by the increased turbulence by the particle motion in the can, and hence, better heat transfer. On the other hand, a further increase in concentration beyond 30 to 40% level, tends to restrict the particle motion because of lack of space for mobility resulting in deterioration of heat transfer rates. As a result to this, the conduction mode of heating becomes more dominant. These results with reciprocation agitation are similar to the study of particle concentration with other modes of agitation. Rao and Anantheswaran (1988) reported in their study that the flow pattern of a pure liquid could be altered by the presence of particulate matter during agitated processing, thus, affecting the heat transfer coefficient. Lenz and Lund (1978) reported that increasing the particle concentration by adding real food particles of different sizes to the fluid also decreased the U and h<sub>fp</sub> values. During the processing of potato spheres in water, Deniston et al. (1987) found that the U and h<sub>fp</sub> increased with increasing particle concentration from 10.7% to 40%, but decreased at higher particle concentrations. Hassan (1984) observed that for 2.54 cm diameter Teflon spheres h<sub>fp</sub>, improved with increased particle

concentration from 20% to 31%. Ramaswamy and Dwivedi (2011) also observed that particle concentration influenced the heat transfer coefficients, U and  $h_{fp}$ . They found that the influence of particle concentration was greater in free axial mode than in fixed axial. According to their results U and  $h_{fp}$  increased with particle concentration ranging from 20 to 30%, while further increase in concentration to 40% decreased both U and  $h_{fp}$ .



Figure 5.7: Effect of particle concentration %(v/v) for 19mm Nylon spheres on: a) overall heat transfer coefficient (U), b) fluid to particle heat transfer coefficient (h<sub>fp</sub>).



Figure 5.8: Contour plot for nylon particles of different Sphericity depicting the effect of particle shape on: a) overall heat transfer coefficient (U), b) fluid to particle heat transfer coefficient (h<sub>fp</sub>)

#### 5.4.2.3 Effect of particle shape

Three different particle shapes namely cube, sphere and cylinder were used in the study. Sphericity factor was used to relate the shape effects of these particles using Equation 5.1, which was 1.0 for the sphere and reduced to 0.874 and 0.806, respectively, for cube and cylinder. From the preceding sections, it was observed that the optimal particle concentration is 30%. The number of particles required to constitute 30% volume concentration varied with change in shape: 25 for cubes, 32 for cylinders and 48 for spheres. Also, different shapes affect the settlement of the particle in the can. Furthermore, based on these observation it is expected that particle of different shape will affects the flow pattern (boundary layer near the particle surface) of the mixture in the can.

Table 5.5 summarizes the mean values and standard deviations of U and h<sub>fp</sub> obtained with particles of different shapes. The influence of particle shape on the U and h<sub>fp</sub> for various reciprocation frequency is graphically represented in Figure 5.8. Our results show that at 3 Hz reciprocation frequency and 30% particle concentration, U value was lowest (880 W/m<sup>2</sup> °C) for cylindrical particle and highest (1080 W/m<sup>2</sup> °C) for cube shaped particles. Similarly, in case of h<sub>fb</sub>, cylindrical particle showed lowest (1100 W/m<sup>2</sup> °C) and cube shaped particles showed highest (1800 W/m<sup>2</sup> °C) values. However, the observed trend was not very significant in case of U values at lower agitation condition (1 Hz reciprocation frequency). From the results we observed that U and h<sub>fp</sub> values for three shapes could be ranked in following order: cube > sphere > cylinder. Analysis of variance of data revealed that particle shape influence was more dominant for h<sub>fp</sub> than U. At any frequency,  $h_{fp}$  reduced by ~29% when shape changed from cube to sphere and ~14% on changing it from sphere to cylinder. The possible reason for this trend could be due to higher secondary agitation caused by cubes, as they have more sharp edges and hence, contribute to higher turbulence in the can. Ramaswamy and Sablani (1997b) also reported that the cube-shaped particle behaved as an extreme case of a particle with a rough surface. However, they reported a different trend of h<sub>fp</sub> with particle shape in end-over-end agitation, giving highest values for spherical particles, and lowest values for cube-shaped particles. They explained their finding by the packing of the particles in the can, and the extent of void spaces between different shaped particles. A can filled with cubical particles would have negligible void spaces compared to other geometry, resulting in a very low liquid velocity around the particles and hence, lower h<sub>fp</sub>. However, in case of reciprocating agitation,

high extent of packing of cubes is disturbed tremendously due to rapid back and forth motion of containers. As a result, the turbulence caused by the sharp edges of cubes predominantly dominates the effect of high extent of packing, and thus, cubes show higher heat transfer coefficients. It should be noted that apart from the particle parameters discussed in this chapter, rheological behavior of covering fluid used may also affect heat transfer to a certain extent. For example, particle shape, size, and density may induce different shear rates in the system, which can induce modification of the viscosity of CMC and the particle movement may be affected. Depending of the shear rates, temperature increase at the particle surface due to friction factor may also occur. This change in particle motion may be very less and can be neglected specially in vary rapid agitation conditions.

#### 5.5 Conclusions

A detailed study has been made to analyze the influence of significant product related parameters on heat transfer coefficients associated with reciprocating agitation thermal processing of canned non-Newtonian fluid particulate mixture. Overall, for evaluation of both U and h<sub>fp</sub>, selected product parameters (particle size, shape, density and concentration) were found to be significant. Two full-factorial experiment designs involving these factors showed that with the decrease of particle diameter and increase in the concentration of particle until an optimal particle concentration was noted. Overall U and h<sub>fp</sub> values increased due to greater turbulence produced by movement of larger number of particles. Any further increase in the particle concentration after this, led to a decrease in the heat transfer coefficients due to onset of a conduction-dominant heat transfer regime as a result of high particle packing. Among the three particles, Nylon particles have shown maximum values of both heat transfer coefficients, while Teflon particles showed maximum values of rate of temperature increase. In terms of particle shape, the cube shaped particles had faster heat transfer as compared to sphere and cylinder shaped particle due to higher turbulence created by its sharp edges. This study shall be useful to establish the thermal processing of liquid particulate mixtures associated with reciprocating mode of agitation.

#### **PREFACE TO CHAPTER 6**

Although very effective in terms of maximizing heat transfer rates (Chapters 3-5), and a promising technology to get high quality canned food products (Walden and Emanuel, 2010) reciprocating retorts have not been fully explored to their full potential. There is of lack of full length scientific work demonstrating and validating their usefulness for processing particulate fluids. In previous chapters (Chapters 3-5) model food (Nylon in CMC solution) was used to evaluate heat transfer coefficients during reciprocating agitation thermal processing. Reciprocation agitation was found to be very effective for increasing heat transfer and hence, has potential to reduce the total process time. Many commercially available canned products such as vegetable soups, vegetable pieces and meat chunks suspended in gravy etc. are best characterized by particulate canned foods in non-Newtonian fluids. For such foods, most quality evaluation studies under agitation processing have been focused on rotary (end-over-end and axial) modes of processing. Hence, in order to fully investigate and establish the reciprocating mode of agitation thermal processing in similar food systems. The quality of processed food can be best described by its appearance, texture and the health promoting compounds such as antioxidants.

In this chapter, the quality parameters (color, texture, chlorophyll and antioxidant activity) were studied during processing of canned vegetable (green beans suspended in Carboxymethyl cellulose solution), which were subjected to reciprocation agitation.

Parts of this chapter won the first prize in **Charles Stumbo Graduate Student Paper Competition** and an oral presentation was made in the Annual Meeting of the Institute for Thermal Processing Specialists (IFTPS) held on Mar 3-6, 2015, San Antonio, Taxes, USA.

Parts of this chapter have been published as an original research article entitled "*Effect of processing conditions on quality of green beans subjected to reciprocating agitation thermal processing*." in Food Research International, 78, 424-432.

The experimental work and data analysis was carried out by the candidate under the supervision of Dr. H. S. Ramaswamy.

### Chapter 6. EFFECT OF RECIPROCATING AGITATION THERMAL PROCESSING ON QUALITY OF CANNED VEGETABLES

#### 6.1 Abstract

This is a study characterizing the effect of reciprocating agitation thermal processing on quality of canned liquid-particulate food products. Canned green beans were selected due to their high antioxidant activity (indicator of thermal loss) and soft texture (indicator of agitation losses). No. 2 (307x409) cans, filled with green beans (2.5 cm length X 0.8 cm diameter) in Carboxymethyl cellulose (0-2%) solutions, were processed in a lab-scale reciprocating retort at different operating temperatures (110-130°C) and reciprocation frequency (1-3 Hz) for predetermined heating times to achieve a minimal process lethality (F<sub>T/z=121.1/10</sub>) of 10 min. Products processed at higher temperatures and reciprocation frequency resulted in greener green beans with a high chlorophyll content and better antioxidant activity retention. Thus, efficiency of reciprocating agitation in preserving quality attributes susceptible to thermal degradation was demonstrated. However, very high reciprocation frequency also resulted in texture losses, higher %breakage of beans, increased turbidity, more %solids and higher leaching of chlorophyll in CMC solutions. There was a complete product loss under very high agitation speed, particularly for low viscosity covering solutions. These results suggest that, although reciprocating agitation would improve the quality attributes of thermally processed food, it may also result in higher particle breakage, leaching of nutrients and other non-desirable effects. Thus, for getting best quality products, particularly for canned liquid particulate foods with soft particulates and other products susceptible to losses on agitation, a gentle reciprocating motion (~1 Hz) might be preferable.

#### 6.2 Introduction

The Canning food industry relies predominantly on conventional sterilization in order to destroy microorganisms of public health concern and to provide shelf stable products (Abbatemarco and Ramaswamy, 1994). Although thermal processing is a well-established process, it also has a detrimental effect on the nutrients and other important quality attributes (Stumbo *et al.*, 1975; Rattan and Ramaswamy, 2014a). Newer concepts like agitation processing, thin-profile processing and aseptic processing have evolved as a result of rapidly changing consumer demand to obtain shelf-

stable, safe and high quality canned food products (Singh et al., 2015d). Agitation processing has been found very effective in achieving the desired objective of reducing quality degradation in various studies evaluating the quality of canned vegetables processed in various types of agitation retorts, like hydrostatic cookers, continuous and semi-continuous agitating retorts, and end-over-end processing, to name a few (Lee and Ammerman, 1974; Hayakawa and Timbers, 1977; Abbatemarco and Ramaswamy, 1993, 1994; Lebovka et al., 2004; Moyano et al., 2007; Garrote et al., 2008; Rattan and Ramaswamy, 2014a, 2014b). More recently, a new form of agitation processing involving reciprocating motion of containers (reciprocating agitation thermal processing) has gained popularity with introduction of Shaka and Gentle Motion retorts (Barbosa-Cánovas et al., 2014). Both Shaka and Gentle Motion involve reciprocating mechanism, albeit at different levels of frequency (frequency of 0-1 Hz for Gentle Motion and frequency of 1.5-3 Hz for Shaka). Since, reciprocating agitation technology is still relatively new, literature is scarce on this agitation mechanism. It was demonstrated that reciprocating agitation increases the heat transfer coefficients by 2-7 times and reduces process times by 46-62%, and hence, lowers the value of the quality deterioration index (Co/Fo) by 26-36% for model liquid-particulate canned foods (Singh et al., 2015b). For liquid food products, Batmaz (2013) and Walden and Emanuel (2010) demonstrated the effect of reciprocating agitation thermal processing through processed product images of bentonite solutions and banana puree respectively, and Jacob and Walden (2015) did the same for ham soup and white sauce in their white paper. However, none of these researchers conducted any analysis of quality attributes and most of the data available is still only for images of final processed products and heat transfer analysis. To the best of our knowledge, no work is available evaluating the quality of real liquid-particulate food products, where the chances of texture damage due to intense mixing, agitation and turbulence are also present.

Green bean is one of the most nutritive vegetable as it is a rich source of most of the essential vitamins (Byers and Perry, 1992). Color and texture are very important quality indicator for all vegetables, which can degrade very fast when exposed to heat. Plenty of research has been carried out analyzing the texture and color changes during thermal processing of canned vegetables (Abbatemarco and Ramaswamy, 1994; Garrote *et al.*, 2008; Rattan and Ramaswamy, 2014a, 2014b). Most of the practical research on canned vegetables has involved optimizing the various processing

conditions so as to achieve a greener product with a better texture (Greve et al., 1994). The CIE-L\*a\*b\* system is a versatile and reliable method to assess the color of fruit and vegetables and its changes during storage and processing, wherein the parameters a\* and b\* express the green-red and blue-yellow axis, respectively (Erge et al., 2008; Gnanasekharan et al., 1992). The -a value has been used as a physical parameter to represent greenness in color measurement (Tijskens *et al.*, 2001). Additionally, some other researchers (Abbatemarco and Ramaswamy, 1994) have monitored changes in chlorophyll content by the ratio of -a to b value (-a/b) for canned green beans. Apart from using these colorimetric parameters, chlorophyll content of green vegetables was studied by several researchers to investigate the quality degradation in terms of loss in green color of vegetables (López-Ayerra et al., 1998; Almela et al., 2000). During thermal processing, as chlorophylls converted to pheophytin, the color of green olives changed to brown (Minguez-Mosquera *et al.*, 1989). Green vegetables mainly contain chlorophylls in two forms, viz. chlorophyll A and chlorophyll B in the approximate ratio 3:1, of which Chlorophyll A was reported to be thermally less stable than chlorophyll B (López-Ayerra et al., 1998; Tan et al., 2000; Tenorio et al., 2004). Since color is a major sensory characteristic in determining product acceptability, it is important to prevent or at least minimize chlorophyll degradation during thermal processing in green vegetables. Moreover, thermal processing also induces structural and chemical variations to the tissue of green beans and other vegetables, changing their texture (Steele, 1987; Abbatemarco and Ramaswamy, 1994; Ávila et al., 2006; Taherian and Ramaswamy, 2009; Delchier et al., 2012; Iborra-Bernad et al., 2013). Minimizing texture loss is another objective in order to get a good quality product. Apart from color and texture, antioxidants (vitamins and phenolic compounds) have also been found to be significantly influenced by thermal processing. Thermal processing at high temperature is generally believed to be responsible for depletion of naturally occurring antioxidants in processed vegetables (Nicoli at al., 1999). Effect of thermal processing on antioxidant activity has been extensively investigated by various researchers (Hunter and Fletcher, 2002; Turkmen et al., 2005; Delchier et al., 2012). These nutritional components all together with sensory attributes, such as color and texture contributes to the final product quality in green beans. The objective of a thermal process must be to maximize the retention of each of the quality attributes, so as to deliver a product of better quality.

Based on available knowledge and recognizing the need to scrutinize the effect of reciprocating agitation thermal process on the overall quality of thermally processed liquid-particulate foods, the objective of this work was to determine the quality changes (color, texture, chlorophyll and antioxidant activity) in canned vegetables (Green bean) as influenced by reciprocating agitation thermal processing. Furthermore, turbidity, %solids and chlorophyll content of the covering liquid was also analyzed to determine the leaching of nutrients and green bean breakage as a possible demerit of reciprocating agitation thermal processing.

#### 6.3 Material and methods

#### 6.3.1 Sample preparation

Fresh green beans, obtained from a local supermarket (Metro Plus, Pincourt, QC, Canada), were washed and cut into cylinders (2.5 cm length X 0.8 cm diameter). In order to facilitate packing and eliminate dissolved gases within the tissues, beans were water-blanched for 1 min at 100°C in a water bath and then quickly cooled in cold water. Aqueous solutions of 0, 1 & 2% (w/w) Carboxymethyl cellulose (CMC) were prepared overnight by dissolving CMC slowly in water in an agitated vessel. These concentrations were chosen as they represent the viscosity of most commercially available soups containing vegetables or meat chunks (Meng and Ramaswamy, 2007a, 2007b). Approximately 175 g of green beans (30% w/w) were added to experimental cans (307x409) and were filled with prepared CMC solutions to a headspace of 10 mm. All cans were then sealed with the help of a manually operated seaming machine (Home Canning Co., Montreal, QC, Canada).

#### 6.3.2 Thermal processing

A vertical steam retort (Loveless Manufacturing Co., Tulsa, OK) was modified to facilitate reciprocation of cans during thermal processing (Singh *et al.*, 2015b). Duplicate experimental cans were placed in the reciprocating cage equidistant from axis of reciprocation and were subjected to thermal treatment. Details about various orientations possible during reciprocating agitations are discussed in Singh and Ramaswamy (2015a), although in this study, vertical orientation was used. Remaining spaces were filled with dummy cans containing water to provide ballast. A 3x3x3 full

factorial design was employed in this study with three processing temperatures (110, 120 and 130°C), reciprocation frequency (1, 2 or 3 Hz) and liquid viscosity (0, 1, 2 %w/w CMC concentrations), for a total of 27 experimental runs. Each experimental run containing duplicate cans was repeated thrice for calculating standard deviations. For determining the process time to impart a lethality of 10 min under all conditions, time-temperature data of the center of five green beans were collected for each experimental run and the lowest temperature-profile was considered representative of cold-spot conditions. Heat penetration parameters ( $f_h$  and  $j_{ch}$ ) for each experimental run were computed from cold-spot temperatures using procedure adopted by Hayakawa (1977). Operator's process time ( $P_t$ ) was then calculated from these heat penetration parameters using Ball's formula method (Ball and Olson, 1957) by employing a z value of 10 °C for most microorganisms and an average come up time (CUT) of 3 min (Singh *et al.*, 2015b). The experimental runs along with the processing time are detailed in Table 6.1.

For collecting the temperature profile, tip of a flexible wire thermocouple (wire diameter 0.0762 mm, Omega Engineering Corp., Stamford, CT) was inserted into the geometric center of green bean, and was held in place with the help of a cotton mesh. Thermocouple equipped particle was inserted inside the experimental can through a C-5.2 stuffing box (Ecklund-Harrison Technologies Inc., Fort Myers, FL). The thermocouple outputs were recorded at one second intervals using a data acquisition system (HP34970A, Hewlett, Packard, Loveland, CO). Small fluctuations during temperature collection were normalized and set-points of initial and retort temperature were fixed at 25 and 121.1°C respectively (Stumbo *et al.*, 1975). Data during cooling was not analyzed as Ball's formula method assumes cooling rate index to be equal to heating rate index ( $f_h=f_c$ ) and a constant cooling lag factor ( $j_{cc}=1.41$ ).

#### 6.3.3 Quality analysis

#### 6.3.3.1 Color

Particles of the can were subjected to color analysis using Tristimulus Minolta Colorimeter (Minolta Corporation, Ramsey, NJ). In the instrument, pulsed xenon arc lamp with D65 illumination was used as the light source, and the standard 10° observer was used for measuring the color

parameters. CIELab's color parameters for the processed particles were recorded by a computer software (SpectraMagic, Minolta Corporation, Ramsey, NJ). Apart from the L<sup>\*</sup>, a<sup>\*</sup> and b<sup>\*</sup> values from the software, the total color change ( $\Delta E$ ) was obtained using Equation 6.1, and colorimeter ratio (-a/b) was calculated from the ratio of  $-a^*$  and  $b^*$  to characterize green color.

$$\Delta E = \sqrt{[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]}$$
(6.1)

#### 6.3.3.2 Antioxidant activity

Total antioxidant capacities of raw and processed green beans were quantified using the DPPH (2, 2-diphenyl-1-picrylhydrazyl) assay (Brand-Williams *et al.*, 1995). DPPH, a stable radical deep purple in color, is reduced in the presence of antioxidants decolorizing the solution. The loss of color results in a decrease in the absorbance intensity, thus providing a basis for measurement of antioxidant activities in the extracts For this 4 gm of treated green bean samples were grounded by pestle and mortar and washed with 85% ethylene (85% ethanol, 15% methanol v/v) solution. The sample was then placed in centrifuge for 40min at 5000 rpm. An aliquot of 1ml of sample ethanoic extract was added to 2ml of 0.1nM DPPH in ethanol, and the decolourization of DPPH reaction was allowed to proceed for 30min in the dark. The absorbance was measured by spectrophotometer at 515 nm. Free radical scavenging activity (FRSA) of DPPH radicals was expressed as percent (100%) from the obtained absorbance for the sample (As) and the blank (Ao) using Equation 6.2:

$$FRSA = \frac{A_0 - A_s}{A_0} \times 100$$
 (6.2)

Where,  $A_0$  is the absorbance at 515 nm in absence of sample; As is the absorbance at 515 nm in presence of sample. Following the same procedure as described above the antioxidant activity in can liquid was also determined.

#### 6.3.3.3 Texture

The texture analysis was done using TA-XT Plus Texture Analyzer (Texture Technologies Corporation, Scarsdale, NY/ Stable Micro Systems, Godalming, Surrey, U.K.). The machine was calibrated for a maximum shear force of 2 kg. Triangular shear blade was used to record maximum

force necessary to shear break the green beans. In order to evaluate the effect of the reciprocation, CMC concentration and agitation time, the firmness and toughness of each samples were obtained from texture profiles using Texture Exponent software (Texture Technologies Corporation, Scarsdale, NY/ Stable Micro Systems, Godalming, Surrey, U.K.). The obtained firmness value was defined as the peak force (gm) required for irreversible crushing of green beans and toughness was defined as the total cross sectional area under the texture profile curve (gm-sec)

#### 6.3.3.4 Breakage of beans

The main quality loss during reciprocating agitation was due to breaking of green beans on high intensity shaking. Due to this breakage, considerable amount of green bean were broken completely and were dissolved in the liquid. The breakage of green beans were quantified by counting the number of whole green beans obtained after carefully separating them from the can. Breakage was expressed through Equation 6.3.

% Breakage = 
$$100*(N_{before} - N_{after})/N_{after}$$
 (6.3)

where,  $N_{\text{before}}$  and  $N_{\text{after}}$  is number of whole (unbroken green beans) green bean before and after thermal processing.

#### 6.3.3.5 Turbidity and % solids

Turbidity of the can liquid was measured to estimate its haziness or cloudiness after thermal treatment. It was measured as percentage transmittance of light through the can liquid. The absorbance of samples was measured at 650 nm using a UV-vis spectrophotometer at room temperature. A turbidity of 0% corresponds to the original CMC solution used, while that of 100% corresponded to opaque solution (transmittance = 0%). Thus, %turbidity was defined as Equation 6.4.

% Turbidity = 
$$100*(T_{\text{orig}} - T_{\text{sample}})/(T_{\text{orig}}) = 100* [10^{-\text{Aorig}} - 10^{-\text{Asample}}]/ 10^{-\text{Aorig}}$$
 (6.4)

where,  $T_{orig}$  and  $T_{sample}$  is transmittance of can liquid before and after processing;  $A_{orig}$  and  $A_{sample}$  is absorbance of can liquid before and after processing.

% solids in can liquid were also calculated to estimate the migration of solid green beans into the can liquid. For this, 50ml centrifuge tubes were weighed empty and after filling with collected liquid containing fines from green bean. Liquid was vigorously stirred on a vortex mixer and then filled quickly in the centrifuge tubes. After centrifugation for 45 min, the supernatant was separated and the precipitate was weighed carefully. The % solids (w/w) was expressed as Equation 6.5.

% Solids = mass of precipitate / (mass of filled tube - mass of empty tube) (6.5)

#### 6.3.3.6 Chlorophyll content

The chlorophyll content was determined spectrophotometrically in fresh and processed green beans. Green beans were cut into small pieces and weighed to 0.50g. Measured vegetable material was mixed with acetone and homogenized in a mortar. Very small amount of MgCO<sub>3</sub> was added before vegetable homogenization to prevent chlorophyll pheophytinization. Mixtures were filtered, mortar and pestle were washed several times with 10 ml acetone, and the content was quantitatively transferred to the filter (Whatman No.1). Filtrate was diluted with acetone to a total volume of 25 ml. For measuring the chlorophyll content in can liquid, 1ml of can liquid was added to 24ml of acetone. Absorbance of prepared mixtures was recorded at 662, 644 and 440 nm using acetone as blank and pigment content was calculated using Equations (6.6-6.8) (Holm, 1954; Von Wettstein, 1957).

Chlorophyll a = 
$$9.784 * A_{662} - 0.990 * A_{644}$$
 (6.6)

Chlorophyll b = 
$$21.426 * A_{644} - 4.650 * A_{662}$$
 (6.7)

Chlorophyll a + b = 
$$5.134 * A_{662} - 20.436 * A_{644}$$
 (6.8)

Where, A is absorbency at corresponding wave length, values 9.784, 0.990, 21.426, 4.650 and 0.288 is the molar absorptivity coefficient for acetone (Von Wettstein, 1957). After calculating the concentrations, the amounts of pigment per g of sample were calculated using Equation 6.9.

$$c\left(\frac{mg}{g}\right) = \frac{c_1 V_r}{m} \tag{6.9}$$

where,  $c = \text{content of pigment (mg/g) of sample; } c_1 = \text{the concentration of pigment calculated}$ by the previous formula (mg/l); v = the starting volume of extract (ml); m = the weighed fresh plant(g).

Chlorophyll content of can liquid was determined using the procedure described by Arnon, (1949) by employing Equation 6.10. The total concentration of chlorophyll in the plant whole homogenate and in the  $12,000 \times g$  fraction was determined by measuring the absorbance of the total chlorophyll extracted with 80% acetone at 652 nm. Each determination was conducted three times and the resultant average absorbance was used to calculate the final chlorophyll concentration.

$$c\left(\frac{mg}{ml}\right) = \frac{A_{652}}{34.5} \tag{6.10}$$

#### 6.4 Results and discussion

#### 6.4.1 Process time calculation

Ball process times to achieve a lethality ( $F_0$ ) of 10 min for the canned green beans are shown in Table 6.1. It is evident from our results that, process time reduces as the reciprocation frequency increases. An increase in reciprocation frequency from 1 to 3 Hz reduced the process time up to 35% (up to 33 mins). These trends are similar to the trends observed in earlier works for model food particles subjected to reciprocating agitation thermal processing (Singh and Ramaswamy, 2015b; Singh *et al.*, 2015b). Improvement in process time reduction have been attributed to the increase in heat transfer coefficients due to the high intensity turbulence created during reciprocating agitation. Moreover, Meng (2006) concluded that non-Newtonian fluids exhibit higher generalized Reynolds number (GRe) at higher shear rates due to the effects of changes in viscosity on reciprocating agitation. Thus, heat transfer rates improve, resulting in considerable reduction in process times as rotation speed was increased from 0 to 20 rpm (end-over-end agitation). However, they have also mentioned that this trend was more prominent at higher temperatures (130°C). Thus, higher intensities of reciprocating agitation was found to be much more effective than rotary agitation in terms of process time reduction. Also, these results imply that high frequency reciprocating agitation, in the ranges used in Shaka retorts, are definitely preferable if the sole objective is to minimize processing times, particularly if thermal degradation is the only factor.

Table 6.1: Experimental designmental	gn with calculate	d Ball process time	s (minutes) to achieve	e an
equivalent lethality of 10 min				

СМС	Temperature	<b>Reciprocation Frequency (Hz)</b>			
<b>Concentration (%)</b>	(°C)				
	<b>``</b> ,	1	2	3	
0	110	88.30	69.93	56.77	
-	120	17.66	15.54	14.94	
-	130	9.23	8.43	7.43	
1	110	98.34	80.73	68.50	
-	120	20.02	17.94	16.31	
-	130	10.09	9.52	8.45	
2	110	108.7	88.02	75.26	
-	120	21.74	19.56	17.92	
-	130	11.09	10.34	9.734	

#### 6.4.2 Visual analysis of product

Figure 6.1 shows images of canned green beans processed with water (0% CMC) at 3 Hz for different temperatures (110 °C, 120 °C and 130 °C) of processing. It was seen that visual quality of green beans processed at higher temperatures was much better than those processed at low temperatures. They were a lot greener and preserved most of their texture. Lower temperature resulted in higher thermal and agitation degradation due to longer process times. This is in accordance with the HTST principle, which suggests processing at higher temperatures to obtain better quality products. During reciprocating agitation, HTST principle seems to be more preferable, as lower process times also minimize the time of agitation.



Figure 6.1: Effect of operating temperature on visual quality of canned green beans processed with water (0% CMC) at 3 Hz reciprocation frequency.



Figure 6.2: Effect of liquid viscosity on visual quality of canned green beans processed at 1 Hz reciprocation frequency and 120°C.

Figure 6.2 shows images of canned green beans filled with liquids of different viscosities (0%, 1% and 2% CMC) and processed at 1 Hz and 120°C. Generally, higher viscosity products have been found to be poorer in quality as compared to lower viscosity products due to lower process times involved (Rattan and Ramaswamy, 2014b). In contrast to the expectation, higher viscosity products (2% CMC) demonstrated better visual quality during reciprocating agitation thermal processing. Similar results have also been reported by other researchers (Batmaz, 2013; Jacob and

Walden, 2015; Walden and Emanuel, 2010) for reciprocating agitation thermal processing. Better visual quality of high-viscous liquid-particulate products under reciprocating agitation may be attributed to the preservation of texture by high fluid viscosity due to lower levels of turbulence.



Figure 6.3: Effect of reciprocation frequency on visual quality of canned green beans processed with 2% CMC at 130°C

Figure 6.3 shows images of canned green beans processed with 2% CMC at 130 °C for different reciprocation frequencies (1 Hz, 2 Hz, 3 Hz). It was seen that, although green beans processed at 3 Hz exhibited better color than 1 Hz products due to lower process times, some of the green beans were broken. The breakage of green bean was higher with both high and low viscous CMC (0% & 1%) when processed at higher frequency (3 Hz), leading to significant product breakdown and discoloration. %breakage of green beans at different processing conditions is discussed later in this chapter. These results show that reciprocating agitation thermal processing at high frequencies may not be very suitable for canned green beans and other similar products, although the thermal degradation is lower in these cases. Thus, reciprocating agitation at levels of Shaka technology seems to be disadvantageous for visual quality of liquid particulate products. Detailed analysis of various quality attributes of the product are discussed henceforth, which shall be useful for studying the levels of degradation of individual quality attributes during various configurations of reciprocating agitation.

#### 6.4.3 Beneficial effects on quality of canned green beans during reciprocating agitation

#### 6.4.3.1 Lower color loss and greener products

Table 6.2 shows the tristimulus  $L^*$ ,  $a^*$  and  $b^*$  values for the processed green bean samples. It was noted that among the three color parameters ( $L^*$ ,  $a^*$  and  $b^*$ ), only  $L^*$  and  $a^*$  values were significantly (P < 0.001) affected by different processing variables. However, b values were found to be insignificant within the range of all the experimental conditions. It was observed that a values became more negative (i.e. product was greener) as the processing temperature increased from 110 to 130°C and frequency increased from 1 to 3 Hz. On the other side, the Lightness ( $L^*$ ) values tend to show an increasing trend with increasing temperature. The total color change ( $\Delta E$ ) in green beans as affected by process variables viz. reciprocation frequency, CMC concentration and operating temperature is graphically presented in Figure 6.4. It was noted that total color loss was significantly  $(P \le 0.001)$  less at lower viscosity levels due to shorter associated process times. At any combination of reciprocation frequency and temperature, changing the value of CMC concentration from 0 to 2 % increased the value of  $\Delta E$  by 25-75%, demonstrating that color change in green beans was higher in more viscous fluids. This may be attributed to the increased process times and resultantly higher thermal degradation of green beans. Moreover, it is well described by various researchers (Von-Elbe and LaBorde, 2009) that the color changes in green beans when thermally processed is associated to the Chlorophyll degradation (formation of olive-colored pheophytin and pyropheophytin). The reduced process time at higher reciprocating frequencies helps in reducing the thermal damage and hence, preserves the color of green beans. Operating temperature was also found significant (P < P0.001) for total color change. Varying retort temp from 120 to 130°C resulted in an average decrease of 30-40% in the values of corresponding total color loss ( $\Delta E$ ). This was mainly because of high susceptibility of green beans to the imparted cook time (Leadley *et al.*, 2008). Processing at high temperature reduces the total heating time and hence makes the process amenable to HTST operations. With regards to agitation frequency, Figure 6.4 shows that color change was lower at higher agitation frequencies. This is as expected and has been attributed to reduction of process time with increase in agitation (Singh et al., 2015b; Singh et al., 2015d).

Reciprocation Frequency (Hz)	CMC Concentration (%)	Temperature (°C)	L*	a*	b*	ΔΕ
	0	110	30.1 (0.4)	0.3 (1.2)	18.3 (1.3)	11.2 (1.1)
	0	120	37.1 (1.1)	-3.5 (0.6)	22.7 (0.1)	7.2 (0.4)
		130	38.4 (0.6)	-4.3 (0.4)	22.4 (0.7)	7.1 (1.3)
		110	36.4 (1.3)	1.1 (0.1)	23.4 (2.5)	11.2 (1.2)
1	1	120	37.1 (2.5)	-3.2 (1.6)	22.9 (1.3)	7.5 (1.4)
		130	38.1 (1.6)	-4.0 (0.1)	22.4 (0.6)	7.4 (0.9)
		110	36.5 (1.3)	2.7 (0.4)	23.4 (2.5)	12.8 (0.8)
	2	120	38.7 (1.9)	-2.7 (0.6)	23.9 (1.8)	9.0 (0.2)
		130	38.4 (3.4)	-3.7 (1.0)	22.4 (0.5)	7.9 (0.6)
	0	110	30.5 (0.6)	-1.4 (1.6)	22.1 (1.1)	8.6 (0.7)
	0	120	32.0 (0.3)	-5.2 (0.3)	22.3 (0.9)	4.5 (0.2)
		130	33.4 (1.6)	-5.2 (1.6)	22.3 (0.8)	4.4 (0.8)
2	1	110	36.3 (1.2)	-0.9 (0.7)	23.4 (1.2)	9.3 (0.5)
2		120	36.4 (1.3)	-3.7 (2.2)	22.2 (3.4)	6.7 (0.4)
		130	35.5 (1.5)	-4.1 (1.4)	22.3 (1.1)	5.8 (1.4)
	2	110	37.6 (0.9)	-0.1 (0.8)	23.4 (0.6)	10.1 (0.8)
		120	37.9 (1.7)	-3.15 (0.4)	22.4 (0.9)	8.0 (0.2)
		130	38.5 (0.4)	-3.9 (0.5)	23.7 (2.8)	7.9 (0.4)
	0	110	32.9 (1.3)	-1.6 (0.7)	22.5 (1.7)	7.9 (0.5)
		120	33.8 (1.1)	-5.2 (0.9)	22.0 (0.9)	4.4 (1.4)
		130	35.0 (0.3)	-5.4 (1.4)	22.1 (1.3)	4.6 (0.7)
2		110	33.4 (0.8)	-1.0 (2.8)	22.4 (0.8)	8.5 (0.4)
3	1	120	36.1 (1.7)	-4.1 (1.8)	22.1 (0.4)	6.2 (1.6)
		130	36.8 (0.8)	-4.5 (0.6)	23.6 (0.8)	5.9 (1.2)
	•	110	33.5 (0.6)	-0.3 (0.8)	22.4 (1.7)	9.3 (0.9)
	2	120	36.9 (1.2)	-3.7 (0.7)	22.1 (1.9)	7.0 (0.8)
		130	36.6 (0.4)	-4.1 (0.1)	23.6 (0.4)	6.3 (1.9)

Table 6.2: L\*, a\*, b\* values and total color change ( $\Delta E$ ) for green beans obtained after reciprocating agitation thermal processing under different experimental conditions.



# Figure 6.4: Total color change (△E) in green beans as affected by processing conditions; a). Effect of reciprocation frequency and concentration on △E at 120°C; b). Effect of temperature and concentration on △E at 2 Hz reciprocation frequency

The color change was mainly in the form of change in greenness of the product. The calorimetric ratio (-a/b) is often used to denote the 'greenness' of vegetables and has been calculated to determine the change in green color of various vegetables on subjecting to thermal processing (Abbatemarco and Ramaswamy, 1993, 1994; Erge *et al.*, 2008). Lee and Ammerman (1974) found that 'a\*' values were not a reliable measure of greenness in sweet potato and instead, '-a/b' values correlated better with the green color. Figure 6.5 shows the effect of increasing reciprocation frequency and CMC concentration on the (-a/b) ratio. As frequency was increased from 1 to 3 Hz, the (-a/b) ratio also increased about 35-55%. However, this trend was not very clear at the high temperature of 130°C. Steele (1987) found that the calorimetric ratio for canned green beans rotated during processing was significantly higher than for green beans subjected to a stationary process. Our results show further improvement on reciprocation, as compared to rotary processing. However, in another study, Abbatemarco and Ramaswamy (1994) reported that increasing rotation speed did not have a significant effect (P > 0.05) on colorimetric ratio. The ratio was found to increase slightly following 10 and 20 rpm; however, this change was not significant. Similar to this, we also observed

that change in greenness was not significant between 2 to 3 Hz for less viscous products. Abbatemarco and Ramaswamy (1994) also reported higher greenness in less viscous products due to lower process time requirements in higher reciprocation intensities. Our results show agreement to these findings as value of calorimetric ratio was higher at high frequency low viscosity combinations.



Figure 6.5: Effect of reciprocation frequency and CMC concentration on colorimetric ratio (-a/b) at 120 °C.

#### 6.4.3.2 Antioxidant activity

Figure 6.6 shows the effect of increasing reciprocating agitation and temperature combinations on the antioxidant activity of green beans canned with 1% CMC solution in terms of DPPH inhibition. While the DPPH inhibition of fresh green beans was close to 95%, the DPPH inhibition under 1 Hz-120 °C was only 30%. However, at 3 Hz-130 °C processing helped to increase DPPH inhibition to 55%. This implies that reciprocating agitation at high temperature has the ability to retain more than 50% of the antioxidants present in green bean. The amount of antioxidant retention was also found strongly influenced by the process time and temperature of the process. At higher temperatures and faster reciprocating agitation, process times were lower and hence, thermal process was less severe. This directly resulted in lower antioxidant inactivation, even under high temperature processes. At 110°C, antioxidant activity was lower, despite lower temperatures due to

very long process required to kill the microbes. These results are similar to the trends in antioxidant activity observed by other researchers on exposure to thermal treatment during canning (Nicoli *et al.*, 1999; Hunter and Fletcher, 2002; McGlynn *et al.*, 2003; Turkmen *et al.*, 2005; Delchier *et al.*, 2012). Thus, in terms of antioxidant activity, higher frequency of reciprocating agitation in the order of that of Shaka process is desirable, similar to results for color and greenness of product.







#### 6.4.3.3 Higher chlorophyll retention

Equations (6.6-6.9) were used to calculate chlorophyll content (mg/g) in fresh and processed green beans. The total chlorophyll content of fresh green beans was found as 1.96 mg/g, out of which 1.26 mg/g was chlorophyll A and, 0.7 mg/g was chlorophyll B. In the fresh sample, amount of chlorophyll A was higher than chlorophyll B, which is in agreement with the literature (López-Ayerra *et al.*, 1998). For green beans, Turkmen *et al.* (2005) reported the values of 1.11 mg/g for chlorophyll A, which is little less from our result (1.96 mg/g) and, 0.74 mg/g for chlorophyll B, which agrees with the concentration obtained from our study (0.7 mg/g). De La Cruz-García *et al.* (1997) reported that raw green bean contains 3.66–10.50 mg/100g of chlorophyll A, and 0.2–0.46 mg/100g of chlorophyll B, which is much higher than our findings. This may be because of differences in varieties, pre-processed nature of our samples and discrepancies in the analytical

method applied. The concentrations of chlorophyll A and B, after processing are presented in Table 6.3. All three process variables were found statistically significant (P < 0.05) for calculation of final concentration of chlorophyll A. For chlorophyll B, temperature and frequency was significant and CMC concentration was not significant within the range of experiments. Result showed that a large amount of chlorophyll was destroyed during the thermal process. The reduction in chlorophyll A and B content is attributed to degradation of both chlorophylls into their major derivatives pheophytin A and B, respectively. However, being more susceptible to pheophytinization, chlorophyll A degrades faster as compared to chlorophyll B, when subjected to heat. The heat sensitivity of chlorophyll A is also reported by other researchers (López-Ayerra et al., 1998; Tan et al., 2000; Tenorio et al., 2004). Similar results were obtained in present study as more chlorophyll A was destroyed than chlorophyll B in all the processed samples. It can be seen from Table 6.3 that chlorophyll content was maximum at 3 Hz reciprocation frequency. Total chlorophyll content increased up to 62-64% at high temperature (130°C) and frequency (3 Hz) scenarios. At any CMC concentration and temperature, increasing the reciprocation frequency from 1 to 3 Hz resulted in 55% increase in concentration of chlorophyll A and 50% increase in final concentration of chlorophyll B. Effect of operating temperature and reciprocation frequency on final chlorophyll (A and B) content in terms of % retention of chlorophyll is graphically presented in Figure 6.7.



Figure 6.7: % Retention of chlorophyll as affected by operating temperature and reciprocation frequency: a). % retention of chlorophyll A; b). % retention of chlorophyll B.

0     110     0.03 (0.01)     0.21 (0.06)     0.24 (0.04)       120     0.35 (0.04)     0.37 (0.08)     0.72 (0.07)       130     0.41 (0.08)     0.41 (0.02)     0.82 (0.05)       1     10     0.04 (0.01)     0.20 (0.05)     0.24 (0.06)       120     0.32 (0.02)     0.36 (0.04)     0.68 (0.03)       130     0.42 (0.08)     0.41 (0.09)     0.83 (0.05)       2     110     0.03 (0.02)     0.21 (0.05)     0.24 (0.04)       130     0.42 (0.08)     0.41 (0.09)     0.83 (0.05)     0.24 (0.04)       110     0.03 (0.02)     0.21 (0.05)     0.24 (0.04)     0.59 (0.07)       130     0.42 (0.08)     0.41 (0.09)     0.83 (0.04)     0.21 (0.04)       120     0.25 (0.06)     0.34 (0.07)     0.59 (0.07)     130 (0.25 (0.05)     0.34 (0.04)       120     0.45 (0.06)     0.50 (0.07)     0.93 (0.07)     130 (0.05)     0.91 (0.07)       130     0.52 (0.05)     0.57 (0.14)     1.08 (0.12)     130 (0.50 (0.05)     0.57 (0.03)       120     0.45 (0.04)	Reciprocation Frequency (Hz)	CMC Conc. (%)	Temp. (°C)	Chlorophyll a (mg/g)	Chlorophyll b (mg/g)	Total (a+b) (mg/g)
Image: constraint of the system of		0	110	0.03 (0.01)	0.21 (0.06)	0.24 (0.04)
Image: constraint of the system of		0 -	120	0.35 (0.04)	0.37 (0.08)	0.72 (0.07)
1     10     0.04 (0.01)     0.20 (0.05)     0.24 (0.06)       120     0.32 (0.02)     0.36 (0.04)     0.68 (0.03)       130     0.42 (0.08)     0.41 (0.09)     0.83 (0.05)       2     110     0.03 (0.02)     0.21 (0.05)     0.24 (0.04)       130     0.42 (0.08)     0.41 (0.09)     0.83 (0.05)     0.24 (0.04)       2     110     0.03 (0.02)     0.21 (0.05)     0.24 (0.04)       130     0.38 (0.04)     0.41 (0.13)     0.79 (0.09)       130     0.38 (0.04)     0.41 (0.13)     0.79 (0.09)       110     0.04 (0.02)     0.30 (0.05)     0.34 (0.04)       120     0.45 (0.06)     0.50 (0.07)     0.93 (0.07)       130     0.53 (0.09)     0.57 (0.14)     1.08 (0.12)       1     110     0.14 (0.03)     0.36 (0.03)     0.50 (0.03)       2     110     0.10 (0.02)     0.33 (0.04)     0.43 (0.03)       120     0.42 (0.04)     0.51 (0.05)     0.91 (0.07)       130     0.48 (0.07)     0.54 (0.01)     1.00 (0.06)		_	130	0.41 (0.08)	0.41 (0.02)	0.82 (0.05)
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	1	1	110	0.04 (0.01)	0.20 (0.05)	0.24 (0.06)
130     0.42 (0.08)     0.41 (0.09)     0.83 (0.05)       2     110     0.03 (0.02)     0.21 (0.05)     0.24 (0.04)       120     0.25 (0.06)     0.34 (0.07)     0.59 (0.07)       130     0.38 (0.04)     0.41 (0.13)     0.79 (0.09)       130     0.38 (0.04)     0.41 (0.13)     0.79 (0.09)       130     0.38 (0.04)     0.41 (0.13)     0.79 (0.09)       110     0.04 (0.02)     0.30 (0.05)     0.34 (0.04)       120     0.45 (0.06)     0.50 (0.07)     0.93 (0.07)       130     0.53 (0.09)     0.57 (0.14)     1.08 (0.12)       110     0.14 (0.03)     0.36 (0.03)     0.50 (0.03)       120     0.42 (0.04)     0.51 (0.05)     0.91 (0.07)       130     0.52 (0.05)     0.57 (0.09)     1.07 (0.08)       110     0.10 (0.02)     0.33 (0.04)     0.43 (0.03)       120     0.35 (0.04)     0.48 (0.02)     0.83 (0.04)       130     0.48 (0.07)     0.54 (0.01)     1.00 (0.06)       120     0.54 (0.04)     0.59 (0.04)     1.11 (0.04)	1	1 -	120	0.32 (0.02)	0.36 (0.04)	0.68 (0.03)
2     110     0.03 (0.02)     0.21 (0.05)     0.24 (0.04)       120     0.25 (0.06)     0.34 (0.07)     0.59 (0.07)       130     0.38 (0.04)     0.41 (0.13)     0.79 (0.09)       110     0.04 (0.02)     0.30 (0.05)     0.34 (0.04)       120     0.45 (0.06)     0.50 (0.07)     0.93 (0.07)       130     0.53 (0.09)     0.57 (0.14)     1.08 (0.12)       110     0.14 (0.03)     0.36 (0.03)     0.50 (0.03)       120     0.42 (0.04)     0.51 (0.05)     0.91 (0.07)       130     0.52 (0.05)     0.57 (0.09)     1.07 (0.08)       110     0.10 (0.02)     0.33 (0.04)     0.43 (0.03)       120     0.35 (0.04)     0.48 (0.02)     0.83 (0.04)       130     0.52 (0.05)     0.57 (0.09)     1.07 (0.08)       110     0.10 (0.02)     0.33 (0.04)     0.43 (0.03)       120     0.35 (0.04)     0.48 (0.02)     0.83 (0.04)       130     0.48 (0.07)     0.54 (0.01)     1.00 (0.06)       120     0.54 (0.04)     0.59 (0.04)     1.11 (0.04)		_	130	0.42 (0.08)	0.41 (0.09)	0.83 (0.05)
$3 \qquad \begin{array}{ c c c c c c c c c c c c c c c c c c c$		2	110	0.03 (0.02)	0.21 (0.05)	0.24 (0.04)
$3 \qquad \begin{array}{c} 130 & 0.38  (0.04) & 0.41  (0.13) & 0.79  (0.09) \\ \hline 130 & 0.04  (0.02) & 0.30  (0.05) & 0.34  (0.04) \\ \hline 120 & 0.45  (0.06) & 0.50  (0.07) & 0.93  (0.07) \\ \hline 130 & 0.53  (0.09) & 0.57  (0.14) & 1.08  (0.12) \\ \hline 110 & 0.14  (0.03) & 0.36  (0.03) & 0.50  (0.03) \\ \hline 120 & 0.42  (0.04) & 0.51  (0.05) & 0.91  (0.07) \\ \hline 130 & 0.52  (0.05) & 0.57  (0.09) & 1.07  (0.08) \\ \hline 2 & 110 & 0.10  (0.02) & 0.33  (0.04) & 0.43  (0.03) \\ \hline 120 & 0.35  (0.04) & 0.48  (0.02) & 0.83  (0.04) \\ \hline 130 & 0.48  (0.07) & 0.54  (0.01) & 1.00  (0.06) \\ \hline 120 & 0.54  (0.04) & 0.59  (0.04) & 1.11  (0.04) \\ \hline 130 & 0.65  (0.09) & 0.65  (0.06) & 1.25  (0.05) \\ \hline 120 & 0.50  (0.04) & 0.57  (0.03) & 1.04  (0.07) \\ \hline 130 & 0.61  (0.05) & 0.63  (0.05) & 1.22  (0.04) \\ \hline 2 & 110 & 0.18  (0.04) & 0.39  (0.04) & 0.57  (0.06) \\ \hline 120 & 0.45  (0.03) & 0.54  (0.07) & 0.96  (0.07) \\ \hline 130 & 0.61  (0.05) & 0.63  (0.05) & 1.15  (0.05) \\ \hline \end{array}$		2 -	120	0.25 (0.06)	0.34 (0.07)	0.59 (0.07)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		_	130	0.38 (0.04)	0.41 (0.13)	0.79 (0.09)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		0	110	0.04 (0.02)	0.30 (0.05)	0.34 (0.04)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		0 -	120	0.45 (0.06)	0.50 (0.07)	0.93 (0.07)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		_	130	0.53 (0.09)	0.57 (0.14)	1.08 (0.12)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	2	1	110	0.14 (0.03)	0.36 (0.03)	0.50 (0.03)
$3 \qquad \begin{array}{ c c c c c c c c c c c c c c c c c c c$	2	1 -	120	0.42 (0.04)	0.51 (0.05)	0.91 (0.07)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		_	130	0.52 (0.05)	0.57 (0.09)	1.07 (0.08)
$3 \qquad \begin{array}{c ccccccccccccccccccccccccccccccccccc$		2	110	0.10 (0.02)	0.33 (0.04)	0.43 (0.03)
$3 \qquad \begin{array}{c ccccccccccccccccccccccccccccccccccc$		2 -	120	0.35 (0.04)	0.48 (0.02)	0.83 (0.04)
$3 \qquad \begin{array}{c} 110 & 0.12 (0.04) & 0.36 (0.06) & 0.48 (0.05) \\ \hline 120 & 0.54 (0.04) & 0.59 (0.04) & 1.11 (0.04) \\ \hline 130 & 0.65 (0.09) & 0.65 (0.06) & 1.25 (0.05) \\ \hline 110 & 0.23 (0.03) & 0.42 (0.08) & 0.65 (0.06) \\ \hline 120 & 0.50 (0.04) & 0.57 (0.03) & 1.04 (0.07) \\ \hline 130 & 0.61 (0.05) & 0.63 (0.05) & 1.22 (0.04) \\ \hline 2 & \hline 110 & 0.18 (0.04) & 0.39 (0.04) & 0.57 (0.06) \\ \hline 120 & 0.45 (0.03) & 0.54 (0.07) & 0.96 (0.07) \\ \hline 130 & 0.56 (0.04) & 0.60 (0.05) & 1.15 (0.05) \end{array}$		_	130	0.48 (0.07)	0.54 (0.01)	1.00 (0.06)
$3 \qquad \begin{array}{ c c c c c c c c } \hline 120 & 0.54 (0.04) & 0.59 (0.04) & 1.11 (0.04) \\ \hline 130 & 0.65 (0.09) & 0.65 (0.06) & 1.25 (0.05) \\ \hline 130 & 0.23 (0.03) & 0.42 (0.08) & 0.65 (0.06) \\ \hline 120 & 0.50 (0.04) & 0.57 (0.03) & 1.04 (0.07) \\ \hline 130 & 0.61 (0.05) & 0.63 (0.05) & 1.22 (0.04) \\ \hline 2 & \hline 110 & 0.18 (0.04) & 0.39 (0.04) & 0.57 (0.06) \\ \hline 120 & 0.45 (0.03) & 0.54 (0.07) & 0.96 (0.07) \\ \hline 130 & 0.56 (0.04) & 0.60 (0.05) & 1.15 (0.05) \\ \hline \end{array}$		0	110	0.12 (0.04)	0.36 (0.06)	0.48 (0.05)
$3 \qquad \begin{array}{c ccccccccccccccccccccccccccccccccccc$		0 -	120	0.54 (0.04)	0.59 (0.04)	1.11 (0.04)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		_	130	0.65 (0.09)	0.65 (0.06)	1.25 (0.05)
3     1     120     0.50 (0.04)     0.57 (0.03)     1.04 (0.07)     1.05 (0.04)     1.05 (0.04)     0.39 (0.04)     0.57 (0.06)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)     1.15 (0.05)	2	1	110	0.23 (0.03)	0.42 (0.08)	0.65 (0.06)
130     0.61 (0.05)     0.63 (0.05)     1.22 (0.04)       110     0.18 (0.04)     0.39 (0.04)     0.57 (0.06)       120     0.45 (0.03)     0.54 (0.07)     0.96 (0.07)       130     0.56 (0.04)     0.60 (0.05)     1.15 (0.05)	5	1 -	120	0.50 (0.04)	0.57 (0.03)	1.04 (0.07)
110     0.18 (0.04)     0.39 (0.04)     0.57 (0.06)       120     0.45 (0.03)     0.54 (0.07)     0.96 (0.07)       130     0.56 (0.04)     0.60 (0.05)     1.15 (0.05)			130	0.61 (0.05)	0.63 (0.05)	1.22 (0.04)
120     0.45 (0.03)     0.54 (0.07)     0.96 (0.07)       130     0.56 (0.04)     0.60 (0.05)     1.15 (0.05)		2	110	0.18 (0.04)	0.39 (0.04)	0.57 (0.06)
<b>130</b> 0.56 (0.04) 0.60 (0.05) 1.15 (0.05)		2 -	120	0.45 (0.03)	0.54 (0.07)	0.96 (0.07)
			130	0.56 (0.04)	0.60 (0.05)	1.15 (0.05)

Table 6.3: Chlorophyll content (mg/g) for green beans obtained after reciprocating agitation thermal processing under different experimental conditions.

<sup>a</sup> values in brackets represent standard deviation from the mean value

Thus, it is seen that in terms of the results of the color analysis, antioxidant analysis and chlorophyll retention analysis, it was found that these factors were strongly dependent on the

intensity of thermal treatment. The change in these parameters was highly correlated to the process times involved. Any factor which decreased process time, including higher temperature and higher reciprocation frequency, resulted in a greener product (high –a/b value) with lower total color change ( $\Delta E$ ) and high antioxidant and chlorophyll retention. In this context, combining with the results produced in previous sections, it is apparent that higher agitation frequencies, in the order of those employed in Shaka retorts, is preferable to reduce color loss, antioxidant loss and chlorophyll loss of canned products.

## 6.4.4 Detrimental effects on quality of canned green beans during reciprocating agitation thermal processing

#### 6.4.4.1 Texture and % breakage

Table 6.4 shows the results for texture analysis at different processing conditions. It was observed that with increase in agitation intensity and decrease in CMC concentration, the texture of the product degraded immensely. In general, products treated at higher temperatures, lower reciprocation intensity and higher CMC concentration had higher toughness and hardness values closer to that of fresh green beans (Firmness-3548 g & Toughness-12042 g.sec). For some cases under high reciprocation intensity, texture value could not be determined as the green bean samples were extensively broken (Table 6.4). The cans containing more than 70% of broken green bean were considered as all broken and the texture and firmness values were not measured. Although, we could not perform analysis of variance for texture data, while looking at our results, we did notice that firmness and toughness values were preserved in high viscosity filling liquid and during higher temperature process. The firmness and toughness of the product at higher frequency degraded during reciprocating agitation despite reduction in thermal treatment. On the other hand, in studies on rotary mode of agitation, Abbatemarco and Ramaswamy (1994) observed that only processing at 20 rpm for green bean yielded a significantly firmer product as compared to still processing and 10 rpm. They also found that for carrot, potato and green bean, firmness after processing at 20 rpm was significantly higher than still processing. Similar results were observed by Rattan and Ramaswamy, (2014b).

Reciprocation	СМС	Temp.	Firmness	Toughness	%	Turbidity	Leaching
Frequency	Concentration	(°C)	<b>(g)</b>	(g.sec)	Breakage	(%)	of Solids
(Hz)	(%)						(%)
1	0	110	49 (3)	87 (9)	60 (7)	45 (2)	1.7 (0.2)
		120	87 (5)	176 (12)	46 (6)	40 (4)	1.8 (0.3)
		130	100 (12)	180 (11)	10 (2)	30 (3)	1.0 (0.2)
	1	110	57 (8)	101 (7)	55 (3)	58 (4)	2.4 (0.4)
		120	121 (11)	489 (12)	36 (1)	37 (2)	1.2 (0.1)
		130	130 (9)	497 (13)	0 (0)	4 (1)	0.2 (0.1)
	2	110	68 (5)	111 (9)	20 (7)	20 (3)	1.0 (0.3)
		120	189 (16)	521 (18)	13 (1)	16 (3)	0.6 (0.1)
		130	178 (11)	532 (23)	0 (0)	3 (1)	0.3 (0.2)
2	0	110	Ν	Ν	90 (7)	70 (4)	4.1 (0.1)
		120	Ν	Ν	100 (0)	85 (2)	7.2 (0.4)
		130	212 (14)	517 (12)	27 (5)	63 (3)	2.4 (0.3)
	1	110	Ν	Ν	71 (8)	68 (3)	3.1 (0.3)
		120	Ν	Ν	95 (4)	76 (4)	6.2 (0.5)
		130	198 (9)	543 (16)	19 (3)	54 (4)	2.3 (0.2)
	2	110	150 (11)	434 (14)	25 (6)	40 (2)	2.6 (0.3)
		120	178 (4)	523 (7)	29 (4)	46 (2)	0.9 (0.4)
		130	170 (7)	500 (8)	10 (4)	17 (3)	0.5 (0.3)
3	0	110	Ν	Ν	100 (0)	87 (5)	15.0(0.4)
		120	Ν	Ν	100 (0)	89 (3)	11.2(0.4)
		130	254 (12)	436 (21)	40 (8)	92 (4)	13.3 0.4)
	1	110	Ν	Ν	89 (6)	78 (2)	10.0 0.5)
		120	Ν	Ν	98 (4)	82 (4)	9.9 (0.6)
		130	202 (17)	493 (24)	32 (7)	79 (5)	9.2 (0.5)
	2	110	150 (13)	435 (12)	50 (6)	50 (6)	2.3 (0.4)
		120	167 (5)	456 (8)	39 (11)	40 (4)	1.5 (0.3)
		130	170 (9)	469 (11)	11 (6)	32 (1)	1.6 (0.3)

Table 6.4: Texture, % breakage, leaching and turbidity values for green beans obtained after reciprocating agitation thermal processing under different experimental conditions.

<sup>a</sup> values in brackets represent standard deviation from the mean value

N represents cases where texture measurement was not possible due to more than 70% broken green beans

The deviation in trends from studies on rotary agitation may mainly be attributed to the rapid shear forces of the order of 3-4g under high reciprocation intensities (Singh and Ramaswamy, 2015a). These shear forces made the texture weak and adversely affected the product characteristics and eventually resulted in breakage of the product. The amount of destruction of green beans was expressed as %breakage and was calculated using Equation 6.3 and presented in Table 6.4. It is clear that more than 50% breakage of green beans was observed for many processing conditions, which certainly can be considered as total quality loss. Also, the total breakage was less with higher concentration of CMC. For 2% CMC, maximum breakage was 50% for 3Hz reciprocation frequency. Contrary to expectation, toughness and firmness were better with 130 °C especially at high concentration of CMC. It was noted that from 110 to 120°C, % breakage increases because of softening of tissue at higher temperature, but any further increase in temperature (120 °C to 130 °C) decreases the %breakage (Figure 6.8). Although it is expected that high temperature (130 °C) soften the vegetable but, the effect may be nullified as the processing time is very less and the vegetables are subjected to that temperature for a shorter period of time. Overall, %breakage reduced with increase in CMC concentration and increased with increase in reciprocation speed.



Figure 6.8: a) % Breakage of green beans at 2 Hz frequency subjected to various temperature & concentrations. b) % Breakage of green beans at 130 °C subjected to various frequencies & concentrations.

#### 6.4.4.2 Turbidity and % solids

Breakage of green beans resulted in accumulation of fines in the liquid which was measured as percentage of insoluble solids (leaching) and turbidity of liquid. Turbidity and %solids in can liquid recorded for various processing conditions are presented in Table 6.4. Less viscous liquids became more turbid and had more %solids at higher agitation frequencies. 0% CMC at 3 Hz frequency became 87-92% turbid and contained 11-15% (w/w) solids after processing. This was happening due to excessive leaching of broken green beans in the liquid. Effect of temperature was also found significant for turbidity. Figure 6.9 demonstrates that at 2 Hz reciprocation frequency, turbidity of 1% CMC varied from 68 to 54% as temperature changed from 110 to 130 °C respectively. It was noticed that by increasing the temperature from 110 to 120 °C, %turbidity increased by 10%. This could be explained in terms of softening of tissues at higher temperature. Contrary to expectation, on further increase in temperature from 120 to 130°C, the turbidity reduced by 28%. The possible reason for this could be explained in terms of reduction in process time and hence, total exposure to the higher temperature (Table 6.1). Similar to turbidity, % solids was higher in less viscous liquids at higher agitation frequencies, which supports the argument that excessive leaching of broken green beans in the liquid occurs at high agitation frequency (Table 6.4). This leaching was lower under HTST conditions due to lower process times and was lower for more viscous liquids due to protective effect from high viscosity liquids.



Figure 6.9: Turbidity of canned liquid processed at 2 Hz frequency under various temperatures and CMC concentrations

#### 6.4.4.3 Leaching of nutrients

Some leaching of nutrients is unavoidable in all instances of thermal processing of liquidparticulate products, although extensive leaching of nutrients may be a cause of concern. The extent of leaching of chlorophyll into the canned liquid was analyzed by determining the levels of chlorophyll in the canned liquid using Equation 6.10 and presented in Figure 6.10. It is seen that with increase in frequency, the amount of chlorophyll leaching into the solution during reciprocating agitation also increased. The maximum chlorophyll content in the liquid was recorded as 0.093 mg/ml of solution at 120 °C for 3 Hz reciprocation frequency in 0% CMC. This is consistent with our earlier results, wherein the maximum turbidity and %solids were recorded for least viscous liquids and highest reciprocation frequency. However, as with turbidity and % solids, the effect of temperature was not very clear. For most of the cases, highest chlorophyll content was obtained in liquids for 120 °C. At 110°C, although the process time was too high, the longer process times resulted in higher degradation of chlorophyll, as discussed in Section 6.4.3. Also, the lower breakage at 110 °C, as compared to 120 °C, also resulted in lower leaching of chlorophyll. Low chlorophyll content at 130 °C signifies lower leaching of nutrients at higher temperature shorter time processing conditions due to lower process times.



Figure 6.10: Chlorophyll content of canned liquid signifying the amount of nutrient leaching during reciprocation agitation.
Thus, in terms of the results of the texture and breakage analysis of green beans, and analysis of the turbidity, % solids and chlorophyll content of canned liquid, it was found that these factors were influenced by both thermal and agitation effects. Higher reciprocation intensities generally resulted in deterioration of these parameters, despite the decrease of associated process times. High temperature processing in higher viscous liquids was found favorable to minimize the detrimental effects of reciprocating agitation on product quality. In this context, it was seen that although, some agitation was necessary, very high intensity agitation in the order of those employed in Shaka retorts, was not preferable for the overall quality of the product. Rather, a gentle motion agitation around 1 Hz, was found to produce the most optimal results.

## 6.5 Conclusions

The effect of reciprocating agitation on product quality of liquid particulate canned foods was tested in this study using green beans. It was observed that reciprocating agitation can preserve the overall quality of the product. Higher reciprocation frequency and temperatures significantly reduced the required process times for thermal processing of green beans. Increasing frequency of agitation was very effective to reduce the total color change and generally resulted in a greener product with a higher colorimetric ratio. Antioxidant activity and chlorophyll retention were also higher for green beans processed at higher levels of agitation. However, high reciprocating frequency also resulted in breakage of green beans in many scenarios with high % solids & turbidity in liquid. Reciprocating agitation also resulted in higher leaching of nutrients (chlorophyll) from green beans to canned liquid. These quality losses occurred due to excessive shear on food particles at high reciprocation intensity. These losses are a major cause of concern for soft textured vegetable, such as green bean, which are highly susceptible to rapid agitation. Generally, high temperature conditions (due to lower associated process times) and high viscosity liquid (providing protection on agitation) were found better to impart reciprocating agitation. Hence, it was seen that high frequency reciprocating agitation, similar to those employed in Shaka retorts, would be preferable for products where high frequency agitation would not be a cause of concern (ex. soups, sauces, pet foods etc.). However, for canned products containing soft-particulates, like canned green beans, or other products susceptible to rapid agitation, operating at frequencies similar to those involved in Gentle Motion retorts would be preferable and more advantageous to preserve the overall quality of the food products.

### **PREFACE TO CHAPTER 7**

As discussed previously (Chapters 3-6) reciprocating agitation is a very effective for increasing heat transfer rates in thermal processing of convection heated food. In Chapter 6, it was shown that higher reciprocation frequency significantly reduces the required process times for thermal processing of green beans. As a result, higher reciprocation was found very effective to reduce the total color change and to retain the antioxidant activity in green beans. Although very good in terms of overall quality and nutritional aspect of thermally processed food, reciprocating agitation was found ineffective for getting good texture food. It was discussed in previous chapters (Chapters 4 and 6) that high speed agitation can trigger impact induced losses (surface leaching, breakage of particulates etc.) to food particulates.

In this chapter a new approach called controlled agitation (stopping agitation after sufficient buildup of cold-spot temperature) was introduced and examined to improve the quality of canned liquid particulate foods when subjected to reciprocating agitation thermal processing.

Parts of this chapter won the first prize in the **Charles Stumbo Graduate Student Paper Competition** and an oral presentation was made in the Annual Meeting of the Institute for Thermal Processing Specialists (IFTPS) held on Mar 3-6, 2015, San Antonio, Texas, USA.

Parts of this chapter have been submitted for publication in Journal of Food Science as an original research article entitled "*A controlled agitation process for improving quality of canned green beans during agitation thermal processing*".

The experimental work and data analysis was carried out by the candidate under the supervision of Dr. H. S. Ramaswamy.

The full manuscript as presented in Chapter 7 contains some information previously presented in earlier sections (Chapter 6). Rather than deleting them, they have been left in place to provide continuity.

# Chapter 7. CONTROLLED AGITATION FOR IMPROVING QUALITY OF CANNED GREEN BEANS SUBJECTED TO RECIPROCATING AGITATION THERMAL PROCESSING

# 7.1 Abstract

This work presents the concept of controlled agitation during reciprocating agitation thermal processing to reduce the quality damage resulting because of rapid motion of cans filled with particulates. For this, green beans were processed in 307x409 cans filled with liquids of various viscosity levels (0-2% CMC). In order to reduce the impact of agitation, a new concept of stopping agitations mid-way (after sufficient development of cold-spot temperature) during the process was proposed. Three agitation times viz; full time agitation; equilibration time agitation and partial time agitation were considered and corresponding operator's process time and corresponding agitation time to impart a lethality of 10 min were calculated using heat penetration results. Processed products were photographed and tested for visual quality, color, texture, breakage of green beans, turbidity and percentage of insoluble solids in the can liquid.

Effect of agitation times on associated process time was established and discussed using heat penetration results. Results showed that stopping agitations after sufficient development of cold-spot temperatures is an effective way of reducing product damages caused by agitation (ex. breakage of beans and its leaching into liquid). Agitations till one-log temperature difference gave best color, texture and visual product quality for low-viscosity liquid-particulate mixture and extended agitations till equilibration time (time required to bring the cold spot temperature to operating temperature i.e 121.1°C) was best for high-viscosity products.

# 7.2 Introduction

The overall quality of processed vegetables is a function of combined effects of nutritive properties and sensory attributes, as the acceptance or rejection of these vegetables rely on their nutritive value, texture, color and external appearance (Berger *et al.*, 2008). These sensory and quality attributes are often affected by heat treatment and hence, may be lost during thermal processing (Abbatemarco and Ramaswamy, 1994). In order to fulfil the consumer demands for better

quality food, the thermal processing industry is seeking alternative approaches (like agitation processing, high temperature short time processing, thin-profile processing etc.) to the conventional heating methods (Singh *et al.*, 2015a). Rapid and uniform heat transfer is the key for achieving high quality products in a thermal process and is often realized by agitating the containers (Ramaswamy and Marcotte, 2005). This results in reduction of thermal process time and hence, overall better quality of canned vegetables.

Quality changes in various vegetables during thermal processing, using still and rotary agitation modes (end-over-end, axial and bi-axial), have been studied extensively by many researchers (Saguy, 1980; Rao *et al.*, 1981; Rao and Lund, 1986; Steele, 1987; Szczesniak, 1991; Gnanasekharan *et al.*, 1992; Abbatemarco and Ramaswamy, 1994; Shin and Bhowmik, 1995; Ahmed *et al.*, 2002a, 2002b; Garrote *et al.*, 2008, Rattan and Ramaswamy, 2014a). Most of these researches have involved optimizing various processing conditions so that more of the products' original/desired texture can be retained (Greve *et al.*, 1994). Some other work in this area has focused on the kinetics of color degradation and thermal softening (Lau *et al.*, 2000) in a variety of vegetables such as carrots, green beans, peas, asparagus and mushrooms. Many researchers have used green bean as a representative food particle for various studies on thermal processing (Steele, 1987; Abbatemarco and Ramaswamy, 1994; Leadley *et al.*, 2008). Steele (1987) studied the effect of can rotation on changes in the color of green beans. Results indicated that color retention was increased during agitation processing. In another study, Abbatemarco and Ramaswamy (1994) studied color and textural changes in green beans as influenced by rotational processing and also compared the quality with green beans processed in the still mode.

More recently, a new mode of container agitation, namely, reciprocating agitation, is being promoted to improve associated heat transfer rates (Walden and Emanuel, 2010). Singh *et al.* (2015b) showed that reciprocating agitation resulted in 2-7 times improvements in heat transfer coefficients, 46-62% reduction in process time and 26-36% lower quality degradation as compared to conventional still mode of processing. Despite the decrease in process time and quality enhancements, product damage associated with high frequency rapid back and forth motion during reciprocating agitation thermal processing has been a cause of concern for adopting this novel technology (Walden and Emanuel, 2010). These quality losses occur due to excessive shear on food

particles at high reciprocation intensity. These losses are a major cause of concern for soft textured vegetables, such as green beans, which are highly susceptible to rapid agitation. Moreover, no quality related work is available for reciprocating agitation thermal processing of liquid-particulates.

It has been shown in the previous chapter (Chapter 6) that, although very good in terms of overall quality and nutritional aspect of thermally processed food, high speed reciprocation can trigger impact induced losses such as surface leaching, breakage of particulates etc. to food particulates (Singh *et al.*, 2015e). Based on these observations, the specific objective of this chapter was to introduce and examine a new approach called controlled agitation (stopping agitation after sufficient buildup of cold-spot temperature) to improve the quality of canned liquid particulate foods when subjected to reciprocating agitation thermal processing.

#### 7.3 Materials and methods

#### 7.3.1 Sample preparation

Fresh green beans, obtained from a local supermarket (Metro Plus, Pincourt, QC, Canada), were washed and cut into cylinders (2.5 cm length X 0.8 cm diameter). In order to facilitate packing and eliminate dissolved gases within the tissues, the beans were water-blanched for 1 min at 100°C in a water bath and then quickly cooled in cold water. Aqueous solutions of 0, 1 & 2% (w/w) Carboxymethyl cellulose (CMC) were prepared overnight by dissolving CMC slowly in water in an agitated vessel. These concentrations were chosen as they represent the concentration of most commercially available soups containing vegetables or meat chunks (Meng and Ramaswamy, 2007b). Approximately 175 g of green beans (30% w/w) were added to experimental cans (307x409) and were filled with prepared CMC solutions to a headspace of 10 mm. All cans were then sealed with the help of a manually operated seaming machine (Home Canning Co., Montreal, QC, Canada).

# 7.3.2 Thermal processing

A vertical steam retort (Loveless Manufacturing Co., Tulsa, OK), which was earlier retrofitted to include reciprocating cage for holding experimental cans, was used in this study. The

reciprocating cage was powered by a 0.5 HP motor (through slider crank mechanism) and its frequency of reciprocation was controlled through an external voltage controller. Further details of the slider-crank assembly and retort modifications are detailed in Singh *et al.* (2015b). Duplicate experimental cans were placed vertically in the reciprocating cage equidistant from axis of reciprocation and were heated till 121.1°C. Remaining spaces were filled with dummy cans containing water to provide ballast. The experiments were conducted at various combinations of reciprocation frequency (1, 2 or 3 Hz) and liquid viscosity (0, 1, 2 %w/w CMC concentrations) using 3x3 full-factorial design for each agitation time. Separate experiments were conducted for still mode to compare the results with that observed under no agitation. Each experimental run containing duplicate cans was repeated twice for calculating standard deviations.

## 7.3.3 Heat penetration parameters, process time and agitation time

For process time calculations, time-temperature data of the cold-spot (particle-center) were first collected using thermocouples for each experimental run. For this, the tip of a flexible wire thermocouple (wire diameter 0.0762 mm, Omega Engineering Corp., Stamford, CT) was inserted into the geometric center of a green bean, and was held in place with the help of a cotton mesh. The thermocouple equipped particle was inserted inside the experimental can through a C-5.2 stuffing box (Ecklund-Harrison Technologies Inc., Fort Myers, FL). The thermocouple outputs were recorded at one second intervals using a data acquisition system (HP34970A, Hewlett, Packard, Loveland, CO). Small fluctuations during temperature collection were normalized and set-points of initial and retort temperature were fixed at 25 and 121.1 °C respectively, using a procedure detailed in Stumbo (1973). Heat penetration parameters ( $f_h$  and  $j_{ch}$ ) were computed from cold-spot temperatures using procedure adopted by Hayakawa (1977). Operator's process time ( $P_t$ ) was then calculated using Ball's formula method (1957). An average come up time (CUT) of 3 min (Singh *et al.*, 2015b) was employed in the calculation of operators' process time ( $P_t$ ). Data during cooling was not analyzed as Ball's formula method (1957) assumes cooling rate index to be equal to heating rate index ( $f_h=f_c$ ) and a constant cooling lag factor ( $j_{cc}=1.41$ ).

Agitation time (AT) and corresponding total cook time (TPT) for each experiment were determined using following considerations:

#### 7.3.3.1 Full time agitation (FTA)

Reciprocation was turned off at the same instant at which steam was turned off and hence, agitation time was equal to the total cook time (Equation 7.1). The cook time (time between 'steam on' and 'steam off') was set as sum of  $P_t$  and CUT (Equation 7.2).

$$AT_{FTA} = CUT + P_t \tag{7.1}$$

$$TPT_{FTA} = CUT + P_t \tag{7.2}$$

# 7.3.3.2 Equilibration time agitation (ETA)

Equilibration time was defined by Singh *et al.* (2015d) as the time required for the semilogarithmic temperature difference between cold-spot and retort temperature to become zero. EQT can be derived to be equal to the negative ratio of y-intercept and slope of the heat penetration curve (or simply the x-intercept) graphically and can be represented as Equation 7.3. Agitation was turned off when cold-spot temperature reached equilibration time. Since, any agitation beyond EQT does not help in reduction of process time, cook time was same as that in full time agitation (Equation 7.4).

$$AT_{ETA} = EQT = f_h * \log (j_{ch}(T_r - T_i))$$
(7.3)

$$TPT_{ETA} = TPT_{CTA} = CUT + P_t$$
(7.4)

# 7.3.3.3 Partial time agitation (PTA) till one log temperature difference

Partial agitations, till one log temperature difference, were imparted to the experimental cans. This time is basically one-log cycle earlier than EQT, and hence, can be expressed as Equation 7.5. Equation 7.5 can also be derived graphically from the heat penetration curve by calculating the time taken for log temperature difference to become one. Stopping agitations before EQT requires additional process time to achieve the same level of lethality (Singh *et al.*, 2015d), as in absence of agitation further decrease in logarithmic temperature difference is at a slower rate. Assuming that the time taken for last log-cycle decrement after stopping the agitation would be equal to that of the still mode, an additional process time (Equation 7.6) was added to the sum of Pt and CUT.

$$AT_{PTA} = EQT - f_h = f_h * \log (j_{ch}(T_r - T_i)/10)$$
(7.5)

$$TPT_{ETA} = TPT_{CTA} + PT_{additional} = CUT + P_t + (f_h)_{still} - f_h$$
(7.6)

# 7.3.4 Quality analysis

Processed cans were stored in a refrigerator overnight. Samples from a commercial can containing cut green beans were also used in the quality analysis for comparison. After opening the cans, liquid and particles were separated and photographed. The contents of the can were subjected to the quality analysis:

Color of treated samples were analysed using Tristimulus Minolta Colorimeter (Minolta Corporation, Ramsey, NJ). In order to characterize total color loss from the processed samples  $\Delta E$  value was calculated using equation 7.7.

$$\Delta \mathbf{E} = \sqrt{\left[ (\Delta \mathbf{L}^*)^2 + (\Delta \mathbf{a}^*)^2 + (\Delta \mathbf{b}^*)^2 \right]}$$
(7.7)

The texture analysis was done using TA-XT Plus Texture Analyzer (Texture Technologies Corporation, Scarsdale, NY/ Stable Micro Systems, Godalming, Surrey, U.K.). The triangular shear blade was used to record maximum force necessary to shear break the green beans. In order to evaluate the effect of the reciprocation, CMC concentration and agitation time, the firmness and toughness of each samples were obtained from the texture profiles using the Texture Exponent software.

Percent breakage of green beans was quantified by counting the number of whole green beans obtained after carefully separating them from the can. Breakage was expressed through Equation 7.8.

% Breakage = 
$$100*(N_{before} - N_{after})/N_{after}$$
 (7.8)

Turbidity and % solids in can liquid was measured in order to represent the amount of leaching which occurred in the can. Turbidity was measured as percentage transmittance of light

through the can liquid. The absorbance of samples was measured at 650 nm using a UV-vis spectrophotometer at room temperature. De-ionized water was used as the control. Percent solids in can liquid were also calculated to estimate the migration of solid green beans into the can liquid. For this, 50ml centrifuge tubes were weighed empty and filled with collected liquid containing fines from green bean. Liquid was vigorously stirred to ensure uniform concentration and then poured quickly in the centrifuge tubes. After centrifugation for 45 min, the supernatant was separated and the precipitate was weighed carefully. The % solid (w/w) was expressed as Equation 7.9.

% Solids = mass of precipitate / (mass of filled tube - mass of empty tube) (7.9)

## 7.4 Results and Discussions

#### 7.4.1 Heat penetration parameters

Ball's formula method (Ball, 1923) was used for calculating the process time to impart a lethality of 10 min in this study. Ball's formula method requires heat penetration parameter for prediction of process time. With the correction factor for converting Ball's process time (B) to Operator's process time (B=Pt+0.42\*CUT), this method gives a conservative estimate of the imparted lethality (Stoforos, 2010). Although, Improved General Method (Bigelow et al., 1920) is the most preferred method for process establishment studies as it gives most accurate results (Abbatemarco and Ramaswamy, 1994; Hassan et al., 2012; Ramaswamy and Marcotte 2005), Ball's process method was preferred in this study. It was already explained in Chapter 3 that the large fluctuations in gathered time temperature data exists while employing rapid reciprocating agitation. Fluctuations in data creates unwanted errors when using all the gathered time-temperature data for process calculations using Improved General Method. On the other hand, Ball's method does not require all data-points and instead uses only the average heat penetration parameters ( $f_h$  and  $j_{ch}$ ) for calculation of process time, and is hence, most suitable for studies on reciprocating agitation thermal processing (Singh et al., 2015a). Further, since the objective of this study was to compare various agitation times, heat penetration study (for finding EQT, f<sub>h</sub> and j<sub>ch</sub>) was much more desirable for generating agitation times of PTA and ETA processes and further aided the use of Ball's formula method over Improved General Method. Values of fh, jch, EQT and corresponding Operator's process

time (Pt) calculated after applying the correction factor on Ball's process time for each experimental points are detailed in Table 7.1.

Table 7.1: Heat Penetration parameters (fh, jch), equilibration time, and operator's process
time calculated using Ball's method for different experimental conditions.

Frequency (Hz)	CMC Concentration (%w/w)	f <sub>h</sub> (min)	jch (min)	EQT (min)	Pt (min)
0	0	5.87	1.33	12.38	16.94
1	0	3.78	1.45	8.10	14.16
2	0	2.48	1.51	5.36	12.34
3	0	2.01	1.48	4.32	11.64
0	1	8.39	1.74	18.65	21.43
1	1	5.31	1.76	11.83	16.80
2	1	3.94	1.68	8.69	14.64
3	1	2.80	1.64	6.17	12.91
0	2	10.55	1.92	23.92	25.16
1	2	6.24	1.98	14.22	18.53
2	2	4.74	1.87	10.68	16.06
3	2	3.94	1.86	8.87	14.81

Figure 7.1 shows the effect of liquid viscosity (in the form of CMC concentrations) and reciprocation frequency on  $f_h$ , EQT and  $P_t$ . Agitation by reciprocation of cans at 3 Hz frequency resulted in reduction of process time required for imparting 10 min lethality to canned green beans by 30-44%, as compared to the conventional still mode of heating. This reduction was higher when higher viscosity of liquid used, suggesting greater effectiveness of reciprocating agitation for more viscous products. This reduction was mainly due to 60-70% reduction in equilibration time due to faster heating rates exhibited under reciprocating agitation conditions. Singh *et al.* (2015b) also reported a 46-62% reduction in process time on using reciprocating agitation as compared to still mode in canned nylon particles in glycerin.



# Figure 7.1: Heating rate index (fh), equilibration time (EQT) and operator's process time (Pt) obtained for canned green beans under different processing conditions.

It was generally seen that with the use of more viscous fluids, heating was slower with higher f<sub>h</sub>. Consequently, time taken in rise of temperature of green bean from ambient conditions to operating temperature (EQT) was also higher, resulting in higher process times for more viscous fluids. These trends are similar to previous studies on reciprocating thermal processing (Singh *et al.*, 2015a, 2015b, 2015d; Walden and Emanuel, 2010). This phenomenon has been attributed to reduction in Reynold's number at higher viscosities due to higher thickness of boundary layer for high viscosity fluids (Sablani and Ramaswamy, 1996). As a result of this effect high intensity agitations (reciprocation frequency) is required for canned foods of higher viscosity. Moreover, using fluid of higher viscosity shall also reduce the impact of agitations on product quality.

# 7.4.2 Operator's process time as affected by agitation time

Stopping agitation mid-way effectively results in conditions existing under natural convection heating. Hence, the slower heat transfer after stopping agitation had to be compensated by allowing the heating time to be higher. Thus, the process time required to achieve the same level of lethality on stopping agitations during partial time agitations (PTA) would be higher as compared to full time agitation (FTA). It was assumed that the rate of temperature rise shall be equal to that of the still mode, after agitation was stopped. It must be also mentioned that, although PTA required additional process time as compared to FTA, ETA did not require any additional process time as agitations were stopped. Theoretically, even after zero log temperature difference (ETA), the particle center would require an infinite amount of time to reach operating temperature. However, in the context of this study, equilibration in heat transfer inside the system was assumed when difference between temperature of cold-spot and operating temperature reached zero log difference. The additional process time (PT<sub>additional</sub>) required for PTA has been plotted in Figure 7.2 for different reciprocation frequency. As expected, higher relaxation time were required at higher frequencies due to greater difference with heating rate in still mode. Additional process time requirements was also greater for more viscous fluids. This may be attributed to the higher effectiveness of forced convection in increasing heat transfer rates in thicker liquids than thinner liquids, as discussed in preceding section.



Figure 7.2: Additional process time requirements for green bean processed under partial time agitation (PTA) regime

Table 7.2: Process time and agitation times used to impart 10 min lethality for full time agitation (FTA), equilibration time agitation (ETA) and partial time agitation (PTA) calculated from heat penetration parameters.

Frequency	СМС	Full Time	Agitation	Equilibriur	n Time	Partial	Time
(Hz)	Concentration			Agitation		Agitation	
	(%w/w)	Agitation	Process	Agitation	Process	Agitation	Process
		time (min)	Time	time (min)	Time	time (min)	Time
			(min)		(min)		(min)
1	0	17.16	17.16	8.10	17.16	4.32	19.25
2	0	15.34	15.34	5.36	15.34	2.88	18.73
3	0	14.64	14.64	4.32	14.64	2.31	18.50
1	1	19.80	19.80	11.83	19.80	6.52	22.88
2	1	17.64	17.64	8.69	17.64	4.75	22.09
3	1	15.91	15.91	6.17	15.91	3.36	21.50
1	2	21.53	21.53	14.22	21.53	7.98	25.85
2	2	19.06	19.06	10.68	19.06	5.94	24.87
3	2	17.81	17.81	8.87	17.81	4.93	24.43

The total cook time requirements under each agitation regime to achieve a lethality of 10 min has been tabulated in Table 7.2. Agitation time decreased significantly from cook time agitation to equilibration time agitation. Stopping the agitations at EQT is highly desirable as it results in saving 8-10 minutes of unnecessary agitation impact (Table 7.1) as compared to FTA (without any reduction in process time). As mentioned earlier, forced convection is not required after equilibration is achieved and any excess agitation only damages the products without any benefit of reduced thermal losses. As compared to FTA and ETA, very short agitation time (only 2-8 min) was required for PTA which is preferable for products prone to breakage or leaching due to agitation. It is worth mentioning that by stopping the agitations very early in PTA regime, quality deterioration due to effects of heat will be greater on account of the additional process time. Yet, the extra process time required is between 2-6 min in most cases for PTA regimes (Figure 7.2), and the effect of this slightly higher process time may be countered by the consideration of higher quality loss on longer agitations for most liquid-particulate foods. However, if higher process times are highly undesirable, slightly longer agitation time till EQT can be imparted. Also, it is noteworthy that imparting agitations beyond EQT is unnecessary for all liquid-particulate foods.

# 7.4.3 Visual quality

The photographs of green beans for canned green beans processed for the entire cook period (FTA) under different agitation frequency and liquid viscosity are shown in Figure 7.3 and Figure 7.4 respectively. It is clear that imparting full time agitation is highly detrimental to product quality. At higher agitation frequencies, green beans were broken and leached into the liquid. In fact, for green beans processed in water at 3 Hz, there were no whole particles available and a suspension of fines was seen in the liquid (Figure 7.3). This was because of the greater impact on green beans collisions with each other and with the wall of the container.



1 Hz

2 Hz

3 Hz

# Figure 7.3: Green beans obtained after processing with 0% CMC for different reciprocation frequencies under full time agitation (FTA) regime.

High viscous liquids were seen to reduce the extent of breakage occurring due to agitation (Figure 7.4). With 2% CMC, even at 3 Hz frequency, non-broken green beans could be obtained, although the color of the product seemed darker due to the extra process time required under slower heating conditions. This is mainly because of the protection offered by higher viscosity liquids due to decrease in liquid-particle relative velocity and velocity of impacts.



Figure 7.4: Green beans obtained after processing with different CMC concentrations at 3 Hz frequency for full time agitation (FTA) regime.



2% CMC 3Hz - FTA

2% CMC 3Hz - ETA





1% CMC 2Hz - FTA

1% CMC 2Hz - ETA



1% CMC 2Hz - PTA

Figure 7.5: Green beans processed under different processing regimes; full time agitation (FTA), equilibrium time agitation (ETA) & partial time agitation (PTA).

Figure 7.5 shows the photographs of green beans obtained on processing at the three agitation times considered in this study. Extensive breakage of green beans for full time agitation (FTA), while partial time agitation (PTA) implicated least damage to products. However, green beans processed with PTA were darker than FTA and exhibited greater thermal loss, possibly due to long process time (thermal exposure).

Figure 7.6 shows the photograph of can liquids (1% CMC) obtained after processing at 3 Hz at the three agitation time. It is clear that maximum leaching of green beans occurred for full time agitation resulting in a higher concentration of particles in the liquid. On the other hand, on stopping the agitations at PTA or ETA, not much difference was observable in the two liquids.



# Figure 7.6: Canned liquids from green beans processed with 1% CMC under full time agitation (FTA), equilibrium time agitation (ETA) & partial time agitation (PTA) regimes.

From the visual observation results, it is clear that equilibrium time agitation presented a better compromise between FTA and PTA with best quality products in terms of visual observation. However, in certain cases, like slow heating rate conditions, ETA also resulted in some amount of breakage. This suggests stopping agitation at other points apart from one log temperature difference (PTA) and zero log temperature difference (ETA) used in this study.

# 7.4.4 Color and texture

Figure 7.7a shows the total color changes ( $\Delta E$ ) in green beans processed with 0, 1 and 2% CMC solutions at 2 Hz frequency for the 3 agitation regimes and compares it with that during still mode. It was seen that color loss was significantly lower at lower viscosity levels due to lower associated process times. Further, as compared to still mode, even a slight agitation with PTA regime was sufficient to reduce color losses by half. There was no statistically significant difference (p>0.05) between ETA and FTA regimes, as the associated process times were same, and hence, thermal losses were equal. However, green beans processed with PTA had higher levels of color losses as compared to ETA and FTA products. This was mainly because of high susceptibility of green beans to the imparted cook time (Leadley et al., 2008). Since, PTA had a higher cook time to compensate for the slower heat transfer rate after stopping the agitation, processed green beans were darker (lower L\* value) and less green (higher a) as observed in Table 7.3. Student's T-test on the data presented in Table 7.3 also revealed that L\* and a\* of PTA products were statistically different (p < 0.05) from ETA and FTA products, while there was no statistical difference (p > 0.05) between L\*, a\* and b\* values of ETA and FTA processed green beans. This is in agreement with the visual results presented in Figure 7.5, where green beans processed with PTA looked far more cooked and darker than ETA and FTA.



Figure 7.7: Total color change (△E) in green beans for full time agitation (FTA),
equilibrium time agitation (ETA) & partial time agitation (PTA) regimes as affected by:
a) liquid viscosity; b) reciprocation frequency.

Processing Mode	Frequency	CMC Concentration	Full Time Agitation				
	(112)	(% w/w)	La	a <sup>a</sup>	b <sup>a</sup>	$\Delta \mathbf{E}^{\mathbf{a}}$	
	1	0	37 (3.7)	-3 (0.01)	23 (0.1)	7 (1.5)	
	2	0	32 (0.1)	-5 (0.02)	22 (0.2)	4 (1.2)	
	3	0	33 (0.5)	-5 (0.04)	22 (2.1)	5 (0.8)	
	1	1	37 (0.1)	-3 (0.05)	23 (0.1)	8 (0.1)	
Full Time	2	1	36 (0.1)	-5 (0.05)	22 (0.08)	7 (1.8)	
	3	1	36 (1.1)	-4 (0.01)	22 (0.01)	6 (2.0)	
-	1	2	38 (0.3)	-3 (0.05)	24 (0.05)	8 (1.5)	
_	2	2	38 (0.2)	-3 (0.01)	22 (0.02)	8 (0.5)	
_	3	2	37 (1.2)	-4 (0.7)	22 (0.03)	7 (0.3)	
Equilibration Time Agitation	1	0	38 (1.5)	-4 (0.6)	23 (1.01)	7 (0.5)	
	2	0	32 (1.6)	-5 (0.5)	22 (0.04)	5 (0.1)	
	3	0	33 (0.5)	-7 (0.1)	22 (2.1)	3 (0.5)	
	1	1	36 (0.4)	-3 (0.02)	21 (0.04)	7 (1.1)	
	2	1	36 (1.4)	-3 (0.05)	23 (0.05)	7 (1.4)	
	3	1	37 (0.7)	-4 (0.01)	23 (0.2)	6 (2.2)	
	1	2	40 (0.3)	-2 (0.01)	23 (0.3)	10 (2.43)	
	2	2	38 (0.2)	-4 (0.1)	23 (0.1)	8 (1.6)	
	3	2	36 (1.1)	-3 (0.03)	22 (0.05)	7 (0.9)	
	1	0	35 (0.1)	-2 (0.09)	23 (0.9)	8 (1.8)	
	2	0	33 (1.0)	-4 (0.1)	22 (1.3)	5 (1.1)	
	3	0	31 (1.2)	-4 (0.03)	22 (1.3)	6 (1.9)	
	1	1	34 (0.3)	-2 (0.01)	17 (1.6)	10 (2.1)	
Partial Time	2	1	33 (0.9)	-2 (0.1)	18 (0.7)	9 (1.3)	
	3	1	33 (0.5)	-3 (0.02)	18 (0.9)	8 (0.7)	
_	1	2	40 (0.1)	-1 (0.01)	23 (1.1)	11 (0.4)	
-	2	2	39(0.2)	-2 (0.05)	23 (2.1)	10 (1.4)	
	3	2	38 (0.1)	-2 (0.02)	22 (25)	9 (2.3)	

Table 7.3: L\*, a\*, b\* values and total color change ( $\Delta E$ ) for green beans obtained after reciprocating agitation thermal processing under different experimental conditions.

<sup>a</sup>values in brackets denote the standard deviation from the mean value

With regards to agitation speed, Figure 7.7b shows that color change was lower at higher agitation frequencies for green beans processed with 1% CMC solutions. This is as expected and has been attributed to reduction of process time with increase in agitation (Singh and Ramaswamy, 2015b). The color change was mainly in the form of change in greenness. Lee and Ammerman (1974) found that a\* values were not a reliable measure of greenness in sweet potato and Weckel *et al.* (1962) and Abbatemarco and Ramaswamy (1994) found similar results in carrot and green beans respectively. Greenness has been measured as colorimetric ratio (–a/b) by these researchers (Lee and Ammerman, 1974; Abbatemarco and Ramaswamy, 1994). Consistent with visual observations (Figures 7.3-7.5), greener product was found by using ETA regime in a lower viscous fluid (Figure 7.8). Abbatemarco and Ramaswamy (1994) also reported higher greenness in less viscous products due to better Reynold's number situation. By using ETA regime, colorimetric ratio (–a\*/b\*) of green beans were higher than that found for canned green beans rotated during processing by Steele (1987). Thus, the effectiveness of controlled reciprocating agitation is demonstrated.



Figure 7.8: Colorimetric ratio (-a\*/b\*) as affected by CMC concentrations for green beans processed at 2 Hz reciprocation frequency under full time agitation (FTA), equilibrium time agitation (ETA) and partial time agitation (PTA) regimes.

Table 7.4 shows the results for texture analysis for different processing conditions and agitation time. It is seen that with increase in agitation intensity and decrease in CMC concentration, the texture of the product degraded immensely. For some case under full time agitation (2-3 Hz at 0-

1% CMC), texture value could not be determined as the green bean samples were broken completely (100% - Table 7.5) and no whole green bean was available for analysis (visualized in Figure 7.5). Reduction in agitation time by processing under ETA or PTA helped in decreasing the breakage and the readings of texture could be taken.

 Table 7.4: Texture properties (firmness and toughness) of green beans obtained after

 reciprocating agitation thermal processing under different experimental conditions.

CMC Frequency Conc.		Full Time Agitation		Equilibr Agi	ium Time tation	Partial Time Agitation	
(Hz)	(%	Firmness <sup>a</sup>	Toughness <sup>a</sup>	Firmness <sup>a</sup>	Toughness <sup>a</sup>	Firmness <sup>a</sup>	Toughness <sup>a</sup>
	w/w)	(g)	(g.sec)	(g)	(g.sec)	(g)	(g.sec)
0	0	101	167	101	167	101	167
U	U	(10)	(11)	(11)	(20)	(8)	(6)
1	0	87	176	159	478	172	571
1	U	(8)	(23)	(25)	(18)	(19)	(31)
2	0	N	N	166	634	162	578
2	U	1	IN	(25)	(108)	(22)	(41)
2	2 0	N	N	187	656	157	532
3	U	1	IN	(23)	(122)	(28)	(27)
0	1	189	265	189	265	189	265
	1	(13)	(28)	(30)	(102)	(10)	(17)
1	1	121	489	397	1389	443	1393
	1	(5)	(32)	(33)	(129)	(26)	(238)
2	1	N	N	378	1345	398	1346
	1	19	1	(31)	(158)	(31)	(156)
3 1	1	N	Ν	367	1367	369	1300
5	1	1		(28)	(167)	(33)	(157)
0	2	231	342	231	342	231	342
U		(23)	(13)	(19)	(145)	(25)	(98)
1	2	189	521	932	2091	1017	3027
1	2	(18)	(24)	(121)	(212)	(234)	(195)
2	2	178	500	900	2089	1009	3003
2	2	(21)	(35)	(115)	(234)	(167)	(201)
2	n	167	456	887	2089	986	2078
3	2	(18) (101)	(101)	(118)	(124)	(196)	(173)

<sup>a</sup>values in brackets denote the standard deviation from the mean value



# Figure 7.9: Texture of green beans processed with 2% CMC at 2 Hz reciprocation frequency under full time agitation (FTA), equilibrium time agitation (ETA) and partial time agitation (PTA) regimes.

Figure 7.9 compares the toughness and firmness of beans processed at 2 Hz frequency and with 2% CMC solution under various agitation times. It was seen that both toughness and firmness were significantly greater for PTA and ETA regimes than full time agitation, which is consistent with visual results presented in Figure 7.5 Also, toughness of PTA products was greater than ETA as they were agitated for a very less time. However, no significant difference (p>0.05) was observed for firmness of products between PTA and ETA regimes. This may be attributed to the extra cooking imparted during PTA regimes for compensating the stopping of agitation earlier.

#### 7.4.5 Breakage of green beans and turbidity and % solids in can liquid

As discussed earlier, in many scenarios, for full time agitation, all green beans were found broken and were not available for analysis. The amount of destruction of green beans was expressed as %breakage calculated using Equation 8. From Table 7.5, it is clear that 100% breakage was obtained for many scenarios when imparting agitations continuously throughout the thermal processing (FTA), which is very undesirable. This breakage was more predominant for lower viscosities of can liquid (0 and 1% CMC). For 2% CMC, maximum breakage was 37% for 3 Hz reciprocation speed for full time agitation (FTA).

Frequency (Hz)	CMC concentration (%)	Full Time Agitation	Equilibrium Time Agitation	Partial Time Agitation
1	0	44%	15%	4%
2	0	100%	21%	6%
3	0	100%	37%	10%
1	1	32%	9%	0%
2	1	100%	12%	3%
3	1	100%	19%	6%
1	2	11%	2%	0%
2	2	28%	5%	4%
3	2	37%	9%	3%

 Table 7.5: Percent breakage of green beans for different conditions under various agitation regimes.

However, by stopping agitations after sufficient temperature increase, breakage was severely reduced. On stopping it as early as one log temperature difference (PTA), % breakage was reduced to below 10% for all scenarios (Table 7.5). Even when the agitation time was increased till zero log temperature difference was reached (ETA), maximum %breakage was 35% for water at 3 Hz. This also suggests that most of the breakage was occurring after green beans became soft at higher temperatures. So, if agitations are imparted for the purpose of bringing temperatures to operating conditions and are subsequently stopped, most of the concerns of using high intensity reciprocating agitation may be eliminated.

Breakage of green beans resulted in accumulation of fines in the liquid which was measured as percentage of insoluble solids and turbidity of liquid. Figure 7.10 presents the turbidity and %solids in liquids recorded for various agitation regimes under different processing conditions. Less viscous liquids became more turbid and had more %solids at higher agitation frequencies. 0% CMC became 91% turbid and contained 11% (w/w) solids after processing at 3 Hz frequency for entire cook time. This was happening due to excessive leaching of broken green beans in the liquid in the latter half after heat transfer equilibration was established. Stopping agitations early (at ETA or

PTA) led to considerably less breakage of green beans and leaching of its fines. Corresponding 0% CMC solutions were only 13% turbid and contained 0.8% (w/w) of leached green beans at PTA regime and with 18% turbidity and 0.6% (w/w) solids under ETA regime. PTA regimes produced most clear liquids with lowest levels of turbidity and were most preferable due to lower agitation time. These observations of turbidity and %solids are consistent with visual results presented in Figure 7.10.



**Processing Conditions** 



Although PTA processing tends to produce best results in terms of breakage of green beans, it must be noted that for some scenarios (high viscosity liquid), ETA processing may be preferred over PTA for obtaining greener and better products (Figures 7.5 & 7.6) due to the associated additional process times (Figure 7.2). Percent breakage was below 10% when ETA processing was

used for high viscosity liquids (Table 7.5), which supports the argument that ETA processing is better for very high viscosity liquids, and PTA processing is better for low viscous liquids. Stopping the agitation between ETA and PTA may be desirable for medium viscosity products to minimize agitation losses.

## 7.5 Conclusions

The concept of stopping agitations mid-way during agitation thermal processing and its impact on product quality was tested in this study. It was seen that stopping agitations early was beneficial for product quality. Allowing agitations for the entire process (FTA), on the other hand, resulted in 100% breakage of green beans in many scenarios with high percentage of insoluble solids and high turbidity of liquid. Texture and color attributes, although considerably better than still mode of processing, in many cases, were unacceptable for processing. In this context, stopping agitations till equilibration time (ETA) was best for visual product quality (color and toughness), particularly for high viscous liquid particulates. Shorter agitation times (PTA) were best for encountering breakage of green beans and reducing the leaching of green bean fines in can liquid. However, stopping agitation earlier, when the temperature difference reaches 10 °C (PTA) may result in additional process time requirements for achieving the same level of lethality, in particular for viscous liquid-particulates. Although, textural changes, breakage, %solids and turbidity of liquid were lowest for PTA, associated higher process times may result in larger thermal losses like deterioration of toughness and color. In conclusion, stopping agitations after sufficient development of cold-spot temperatures is an effective way of reducing product damage associated with reciprocating agitation. Agitations can be stopped when the temperature difference falls below one log difference for low viscosity liquid-particulate mixture and these agitation may be extended till equilibration time for higher viscosity products.

Note: In this study, a process lethality of 10 min was employed which is a far severe process. Under controlled conditions, a Fo value of 5 min should be more than adequate to achieve commercial sterility and hence will provide further opportunity for quality retention.

### **PREFACE TO CHAPTER 8**

In Chapters 4 and 5, effect of various process and product variables on U and h<sub>fp</sub> were analysed using model food mixtures. Further, quality changes in real food (canned green beans) when subjected to reciprocation was evaluated in chapter 6. To counter the texture damage to canned particulates (green bean) due to high speed agitation, a novel concept of stopping agitation midway was proposed in Chapter 7 and found very effective in terms of reducing breakage and surface leaching of particulates. Conventional predictive models and optimization methods, which make use of various mathematical models, have been widely applied in the modeling and optimization of food thermal processing. However, with the development of computer technologies and artificial intelligent technologies such as artificial neural networks (ANN) have been found to outperform conventional models in modeling and optimization of processing systems (heat transfer coefficients, process time calculations etc.). Other researchers (Meng, 2006; Dwivedi, 2008) have reported that artificial neural networks (ANN) can give more accurate predictions of heat transfer coefficients than conventional modelling approaches.

In this chapter artificial neural network models were developed for the prediction of the associated heat transfer coefficients U and  $h_{fp}$ . These models were validated for real food particulate mixtures (U and  $h_{fp}$  was calculated for green beans in particulates). ANN models coupled with finite difference were also developed to calculate thermal process time and the calculated process time was compared with process time by Ball formula.

Parts of this chapter has been submitted for publication in Computers and Electronics in Agriculture as an original research article entitled "*Predictive modelling of heat transfer coefficients* and process time for reciprocating agitation thermal processing using artificial neural networks coupled with finite difference model (ANN-FD)".

The experimental work and data analysis was carried out by the candidate under the supervision of Dr. H. S. Ramaswamy.

The full manuscript as presented in Chapter 8 contains some sections already presented in previous Chapters. Rather than deleting them, they have been left in place to provide continuity.

# Chapter 8. PREDICTIVE MODELLING OF HEAT TRANSFER COEFFICIENTS AND PROCESS TIME FOR RECIPROCATING AGITATION THERMAL PROCESSING USING ARTIFICIAL NEURAL NETWORKS COUPLED WITH FINITE DIFFERENCE MODEL (ANN-FD)

# 8.1 Abstract

For design and efficient control over thermal processing, modelling of heat transfer using artificial intelligence has been proven as a promising tool. Artificial neural networks (ANNs) has shown potential to be used as an alternative to conventional mathematical correlation approaches. In this chapter, ANN models were developed for overall (U) and fluid to particle heat transfer (h<sub>fp</sub>) coefficients associated with canned particulates in non-Newtonian fluids during reciprocating agitation thermal processing in a lab-scale reciprocating retort. The experimental data obtained for the associated heat transfer coefficients for model fluid particulates over various range of process and product parameters were used for training and testing of the neural network. The model was fitted for optimal configuration parameters viz. learning rules, transfer function, number of learning runs, training function, number of hidden layers and number of neurons in each hidden layer. For the range of operating parameters used in our study (Chapter 3-6), the optimal conditions obtained were two hidden layers, eight neurons in each hidden layer, the delta learning rule, tangent sigmoid transfer function, Levenberg-Marquardt back-propagation training function and 60,000 learning runs. In order to examine the validity of the developed ANN model for real foods, the prediction accuracies of the ANN models were tested for calculation of U and h<sub>fp</sub> in real food (canned green beans). The ANN models were found able to provide a meaningful prediction of U and h<sub>fp</sub> especially at higher reciprocation frequencies. Furthermore, an ANN model coupled with finite difference was developed for prediction of process time to provide desired lethality to a thermally processed product. It was observed that the process times predicted by the ANN-FD model and the Ball's formula method matched for most of the processing scenarios.

## 8.2 Introduction

Thermal processing is one of the most effective approach used for achieving commercial sterilization in canned foods. Lowering the process time to minimize the nutrient degradation while,

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applying an appropriate level of microbial destruction during thermal processing, has always been the main focus of thermal processing specialists. In the past few decades, thermal processing has come a long way and so many advancements to the technology have been added. Now a days, to provide faster rates of heat transfer, most of the industrial retorts are designed to facilitate rotary motion of the can loaded cage (rotary retorts). There are two different modes of rotation used in rotary retorts viz. end-over-end mode, where the containers are loaded vertically into a retort crate and rotate around a central horizontal axis, and axial mode, where the cans are rotated in a horizontal plane (Dwivedi, 2008). Rotary retorts processing was studied by various researchers and found very effective in terms of reducing total process time and achieving better quality retention in thermally processed food.

Process modeling for thermal processing of liquid/particulate canned food requires data on the overall heat transfer coefficient, U and the fluid-to-particle heat transfer coefficient,  $h_{fp}$ . In real thermal processing operations the operating conditions such as operating temperature, agitation speed, food physical properties etc. may vary during the thermal process and it might be difficult to experimentally collect data for each processing condition (Sablani and Ramaswamy, 1996). To solve this problem, various researchers have widely used predictive models (dimensionless correlations, neural network models etc.). Traditionally, dimensionless correlations have been used for the predictive modeling of U and  $h_{fp}$  (Rao and Anantheswaran, 1988). Using the traditional approach of nonlinear multiple regression analysis, the heat transfer coefficient in the form of a Nusselt number is correlated to the other dimensionless numbers involving parameters which influence U and  $h_{fp}$ .

Moreover, use of artificial intelligence such as artificial neural networks (ANN) have been found very effective for prediction of U and h<sub>fp</sub> for a wide variety of processing conditions (Meng, 2006; Dwivedi, 2008). An artificial neural network (ANN) is a data processing system developed for predicting the relation and/or trends between input and output sets of data. ANN models are generally represented as a group of interconnected neurons which transmits the signal based on numeric weights to each neurons. Various researchers have used ANN in many disciplines of engineering and science because of its ability to correlate large and complex data sets. Thermal processing is a complex process that requires many types of information (such as required destruction of microorganisms, minimum loss of quality parameters, lethality requirement, can properties, food thermal and physical properties etc.) to be processed at the same time. ANN has been efficiently used by thermal processors and researchers for predictive modelling of the process (Sablani, 2007).

For conduction heated foods, Sablani et al. (1997) have used ANN models to optimize the process parameters and a predictive model was developed to predict heat transfer coefficients when the most influencing operational variables (optimal sterilization temperature, corresponding processing time and quality factor retention with the can dimension, food thermal diffusivity and kinetic parameters of the quality factors) are known. The developed neural network which had 8 neurons in hidden layers and 50,000 learning runs was found optimum for performance. The trained network was found to predict a response with less than 5% associated errors, with respect to optimal sterilization temperature, process times and quality factor retention. A neural network with 10 neurons in the hidden layers and 50,000 learning runs was found optimal for their data with convection heating systems. The trained network was found to predict U and h<sub>fp</sub> responses with less than 3% and 5% associated errors, respectively. Meng and Ramaswamy (2008) have developed ANN models for the apparent heat transfer coefficient hap and Ua associated with canned particulate high viscous Newtonian and non-Newtonian fluids during end-over-end rotation thermal processing. They found that these models were able to predict responses with mean relative error of 2.9-3.9 % in Newtonian fluids and 4.7 to 5.9 % in non-Newtonian fluids. They also compared the ANN models with dimensionless correlations, and observed that ANN modes gave more accurate prediction of heat transfer coefficients as compared to dimensionless correlations. Similarly, in end-over-end mode, Sablani et al. (1997) developed ANN models for estimation of U and h<sub>fp</sub> and reported that the prediction errors were about 50% better than those associated with dimensionless correlations; less than 3% for U and 5% for h<sub>fp</sub>. Dwivedi and Ramaswamy (2010d) developed ANN models to predict U and hfp associated with canned Newtonian fluids with and without particles during free axial, fixed axial and end-over-end mode agitation processing. The developed ANN models were able to predict the heat transfer coefficient with the mean relative error of 2.6 and 2.9% respectively in fixed axial mode, and 1.85 and 2.5% respectively in free axial mode. Similarly, mean relative error associated with U in Newtonian fluid (liquid only cans) in end-over-end mode was 3.8%, and was 2.06% in the case of free axial mode.

The objective of this chapter was to develop ANN models to predict the heat transfer coefficients (U and  $h_{fp}$ ) for canned non-Newtonian liquids with particulates during reciprocating agitation thermal processing. Subsequently, the developed ANN model was coupled with a finite difference model (FD) to predict process times for sterilization of liquid particulate food. The developed ANN-FD model was validated using the data of processing of canned green beans presented in Chapter 7.

# 8.3 Material and methods

## **8.3.1** Materials, physical properties and experimental range

A lab scale reciprocating retort (Singh *et al.* 2015b) was used in this study. The retort cage was modified to provide reciprocating motion of cans during thermal processing. Cans were positioned vertically (Singh and Ramaswamy, 2015a) in the retort cage. The cage was designed to hold four No. 2 cans (307x409) along the diameter of the retort. The cage was connected to a slider crank unit through a reciprocating rod inserted into the retort. This rod was connected to the rotating shaft on the slider crank assembly through a crank. The speed of rotation of the motor was controlled through an external voltage controller. In order to pivot the crank on the rotating shaft, 15 different positions were provided, through which amplitude of reciprocation could be varied.

Nylon, Teflon and polypropylene particles (Small Parts Inc., Miami, FL) were used as food particulate models. CMC aqueous solutions were used as non-Newtonian fluid food models. Details regarding can preparation, temperature measurement, data normalization and heat transfer coefficient calculation were given in Chapter 3. U and h<sub>fp</sub> data with respective to various input variables to develop ANN models were taken from Chapters 3, 4, and 5. In order to validate the developed model, green beans with different CMC concentration were chosen as the fluid particulate mixture. The overall range of parameters related to model food and real food mixtures (green beans in CMC solution) used in either development and/or validation of ANN models were summarized in Table 8.1)

Table 0.1 Mange of variables used for development and variation of Arviv models
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Parameter	Symbol	Experimental range
Operating temperature	Т	110 to 130 °C
<b>Reciprocation Frequency</b>	R	0- 4 Hz
Amplitude of reciprocation	А	5 -25 cm
Can headspace	Н	6 to 12 mm
Test Fluid (App. Viscosity)	V	CMC (non-Newtonian) concentration; 0 to 1 %
Particle Concentration (%)	С	Single particle (liquid only) 20, 30 and 40% (v/v)
Particle Density (kg/m <sup>3</sup> )	D	PP(830), N(1128), (TEF)2210 kg/m <sup>3</sup>
Particle Size (mm)	Z	15, 19 and 25 mm
Particle Shape	S	Sphericity; Cube (0.874), Sphere (1), Cylinder (0.806)

#### For development of ANN model

# For validation using real food

Parameter	Symbol	Experimental range
Operating temperature	Т	110,120,121.1,130 °C
<b>Reciprocation Frequency</b>	R	0,1,2,3 Hz
<b>Reciprocation Amplitude</b>	А	15 cm
Can headspace	Н	10 mm
Test Fluid (App. Viscosity)	V	CMC (non-Newtonian) concentration; 0, 1%
Particle Concentration (%)	С	30% (v/v)
Particle Density (kg/m <sup>3</sup> )	D	835 kg/m <sup>3</sup>
Particle Size (mm)	Ζ	0.025 m
Particle Shape	S	Cylinder (0.806)

R=reciprocation frequency, T= retort temp., A= amplitude of reciprocation H= can head space, V= App. Viscosity (CMC), D=particle density, PP=Polypropylene, N=Nylon, TEF = Teflon, S=Particle shape, Z = Particle size, C = Particle concentration

# 8.3.2 U and h<sub>fp</sub> in reciprocation agitation

Methodology developed in chapter 3 was used to evaluate U and  $h_{fp}$  for all the experimental conditions. For this, the heating rate index of the liquid and particle were calculated from the time experimental temperature curve and values of  $h_{fp}$  were estimated by fitting the predicted temperature profile of the cold-spot (particle center) with simulated temperature profile of the cold-spot under constant retort temperature conditions ( $T_{crt}$ ) for Fourier number greater than 0.2. Predicted temperature profile of liquid ( $T_{pred-l}$ ) and particle-center ( $T_{pred-p}$ ) under constant retort operating conditions was generated from heating rate indices of liquid and particle (Equation 8.1).

$$\mathbf{T}_{\text{pred}} = \mathbf{T}_{\text{crt}} - (\mathbf{T}_{\text{crt}} - \mathbf{T}_{i}) * \mathbf{10}^{\left(-\frac{\mathbf{t}-\mathbf{t}_{0}}{f_{h}}\right)} \text{ for } \forall \tau > \mathbf{0.2 \text{ or }} = \mathbf{T}_{i} \text{ for } \forall \tau < \mathbf{0.2}$$
(8.1)

where,  $T_{pred}$ ,  $T_{crt}$  and  $T_i$  are the predicted temperature profile, constant retort temperature and initial temperatures (°C) respectively;  $f_h$  is the heating rate index (s); t is the time instant (s);  $t_o$  is the time (s) at which Fourier number  $\tau=0.2$ .

The simulated temperature profile of the particle-center was generated from equilibrated liquid profile ( $T_{pred-L}$ ) using a finite difference simulation of the unsteady state heat conduction equation for particles of different shapes immersed in liquid with prescribed initial and boundary conditions (Ramaswamy and Sablani, 1997b). Instead of the actual experimental profile the 'predicted' liquid ( $T_{pred-l}$ ) and particle ( $T_{pred-p}$ ) temperature profiles, were used to subsequently calculate U and  $h_{fp}$  by employing the same procedures as described in Ramaswamy and Sablani (1997b). In order to develop the ANN models correlations, the heat transfer coefficients data from 107 set of experiments from previous chapters were used. To validate the developed ANN model, heat transfer coefficient for real food (green beans in CMC solutions) were calculated for all the experimental conditions used in Chapter 7. The range of parameters related to liquids, particles and retort operating conditions are summarized in Table 8.1.

## 8.3.3 Neural Network

Neural network tool box (NNtool) of MATLAB was used for the development of ANN models. Nine significant variables (retort temperature, liquid viscosity (CMC concentrations),

reciprocation frequency, reciprocation amplitude, can headspace, particle density, particle size, particle concentration and particle shape) corresponded to 9 neurons which made up the input layer for the test data. The output layer consisted of 2 neurons each for U and  $h_{\rm fp}$ . For learning and training the network, the standard back-propagation algorithm was used.

To develop the neural network model for canned liquids with particles under reciprocating agitation, the neural network parameters such as the transfer function, learning rule, number of hidden layers, number of neurons in each hidden layer, training function and number of learning runs needed to be optimized. A full factorial design was created by varying the learning rules and transfer functions in different combinations, all the while keeping two hidden layers with 10 neurons in each, and 60,000 learning runs. In order to optimize the number of hidden layers and the number of neurons in each of them, another full factorial design was developed, while using the same optimal combination of learning rules and transfer functions as obtained from the first design. One to three hidden layers, with 2, 4, 6, 8 and 10 neurons in each hidden layer was tested to optimize the neural network configuration. Finally to optimize the training function and number of learning runs, a full factorial design was created by varying the same as was optimized in the first two designs. Root mean square (RMS) error was used to represent the difference between the predicted and the desired output. The parameters were optimized for minimum RMS error.

# 8.4 Results and Discussion

## 8.4.1 Learning/training of ANN

Neurons are highly interconnected processing segments, which are arranged in layers and connected by different weights (w) which represent the strength of these connections. In order to generate a network output, each neuron receives signals from other neurons in proportion to its connection weight, which it sends to the transfer function. The behavior of an ANN depends on both the weights and the input-output function called transfer function that is specified for the units. The transfer functions are generally non-linear and are of three types viz. linear, threshold and sigmoid. During the learning of the neural network, the connection weights are adjusted explicitly (utilizing a

priori knowledge) to minimize the error between model and desired output. The increment or decrement by which the weights of the processing elements are changed during the learning phase are determined by the learning rule. The transfer functions used in this study were log sigmoid, tan sigmoid, positive linear and linear. The learning rules were Gradient descent (learngd), Gradient descent w/momentum weight/bias learning (learngdm), Hebb weight (learnh), Hebb with decay weight (learnhd) and Self-organizing map weight learning (learnsom).



# Figure 8.1: Effect of learn rule on R<sup>2</sup> and root mean square error in reciprocating agitation (Gradient descent or delta = <u>learngd</u>, Gradient descent w/momentum = <u>learngdm</u>, Hebb weight = <u>learnh</u>, Hebb decay weight = <u>learnhd</u> and Self-organizing map weight = <u>learnsom</u>)

Figures 8.1 and 8.2 shows the average error (RMS) associated with experimental and predicted U and  $h_{fp}$  values in reciprocating agitation as a function of the transfer function and learning rule. It was observed that the combination of Delta as a learning rule and Tan sigmoid as a transfer function showed smallest RMS and the highest R<sup>2</sup> values. It was also reported by Rai *et al.* (2005) and Ochoa-Martinez *et al.* (2007) that the TanH (tan hyperbolic) transfer function usually behaves better than the log sigmoid transfer function. Dwivedi (2010d) has also obtained highest value of R<sup>2</sup> with combination of Delta as a learning rule and Tan hyperbolic (TanH) as a transfer function. The learn rule caused the RMS to vary slightly, but did not cause any significant

differences. Therefore, in this chapter the combination of Delta as a learning rule and Tan sigmoid as a transfer function were selected as the optimal combination for modeling reciprocation agitation thermal process of fluid particulate cans.



Figure 8.2: Effect of transfer function on  $\mathbb{R}^2$  & root mean square error in reciprocation agitation (log sigmoid = <u>logsig</u>, tan sigmoid = <u>tansig</u>, positive linear = <u>poslin</u> and linear = <u>purelin</u>)

Table 8.2 represents the root mean square (RMS) and  $R^2$  obtained as a function of the number of hidden layers and the number of neurons in each. It was found that, eight is the optimum number of neurons. The number of hidden layers and neurons in each hidden layers resulted in very large variations in  $R^2$  values. It was noted that  $R^2$  values increased by increasing the hidden layer from 1 to 2, which further decreases when the hidden layer changed from 2 to 3. Similarly,  $R^2$  was increased when number of neurons were varied from 2 to 8, which further starts decreasing when neurons were changed to 10. Contrary to our results Dwivedi and Ramaswamy (2010d) observed that axial mode of agitation hidden layer did not cause any significant effect when raised from one to

three. Eight neurons also gave the highest  $R^2$  values, which reduces remarkably when increased beyond 8. This may be happening because with less nodes the model would not be sufficient to recognize a pattern between variables and on the other side with too many nodes, computation takes more time and an ANN may 'memorize' the input training sample (Rai *et al.*, 2005). Hence, we have selected two hidden layer with eight neurons which resulted in maximum  $R^2$  value and minimum value of RMSE.

Using the optimized parameters of neural network architecture from the first two designs i.e. Delta as a transfer function, Gradient decent learning rule, 2 hidden layers with 8 neurons in each layer, the number of learning runs and training function were optimized in the third design. For this, three training functions viz. Levenberg-Marquardt back-propagation, Gradient descent back-propagation and Bayesian Regulation back-propagation and number of learning runs (20000 to 120000) were selected in a full factorial design. It can be seen in Figure 8.3 that the RMS error decreased continuously when the learning runs increased, however, no additional benefit was realized when the learning runs were increased beyond 60,000. Hence, 60000 learning runs were used for modeling U and  $h_{\rm fp}$  in reciprocation mode.

Table 8.2: Effect of hidden layers and number of neurons in each hidden layer on RMSE an	d
<b>R<sup>2</sup></b> in reciprocation agitation mode	

	No. of hidden layer								
		1		2		3			
		RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>	RMSE	R <sup>2</sup>		
No. of	2	137	0.60	490	0.71	453	0.68		
neurons	4	620	0.65	114	0.76	608	0.30		
	6	93	0.82	614	0.90	531	0.15		
	8	104	0.84	56	0.98	545	0.17		
	10	563	0.54	127	0.85	519	0.52		



# Figure 8.3: Effect of number of learning runs on R<sup>2</sup> in reciprocation agitation

Training a neural network model essentially means selecting one model from the set of allowed models that minimizes the cost criterion. There are numerous algorithms available for training neural network models; most of them can be viewed as a straightforward application of optimization theory and/or statistical estimation. Training functions are functions that basically update weight and bias values which further produces output from that junction. Levenberg-Marquardt back-propagation function is used in large data handling problems and is often the fastest back-propagation algorithm in the toolbox, and is highly recommended as a first-choice supervised algorithm, although it does require more memory than other algorithms. Bayesian Regulation backpropagation also updates the weight and bias values according to Levenberg-Marquardt optimization. However, it minimizes a combination of squared errors and weights, and then determines the correct combination of neural network configurations which generalizes well. Gradient descent back-propagation updates weight and bias values according to gradient descent. We observed that Bayesian Regulation back-propagation decreases the RMS error to minimum value but takes long time for developing the model. Although very less RMSE errors are associated to Bayesian Regulation back-propagation, Levenberg-Marquardt back-propagation model takes less time and results in higher R<sup>2</sup> value. Based on these observations Levenberg-Marquardt backpropagation was selected as a training function in the developed ANN model (Table 8.3). Figure 8.4 shows the schematic of a developed multilayer neural network developed in the present study.
Table 8.3	: Selection	of	training	function	for	developing	ANN	models	for	U	and	h <sub>fp</sub>	in
reciprocating agitation (learning runs = 60,000)													

Training function	R <sup>2</sup>	RMSE
Levenberg-Marquardt back-propagation	0.98	56
Gradient descent back-propagation	0.21	398
Bayesian Regulation back-propagation	0.97	13



Figure 8.4: General scheme of an ANN with 2 hidden layers with 8 neurons in each

### 8.4.2 Performance of the ANN model

The performance of the developed neural network was evaluated after the completion of the learning and training phase. A full set of data was used to optimize the parameters. The 107 set of experimental data were divided into different proportions for training, testing and cross-validation. The combination of data sets included 60 % for Training, 20% for Testing, 20 % for Validation; 70 % for Training, 20% for Testing, 10 % for Validation; 70 % for Training, 10% for Testing, 20 % for Validation; 75 % for Training, 15% for Testing, 10 % for Testing, 10 % for Validation; 80 % for Training, 10% for Testing, 10% for Training, 15% for Testing, 5% for Testing, 5% for Validation; 70 % for Training, 15% for Testing, 15% for Validation. Compared in Table 8.4 are the associated errors with the neural networks output. The excellent agreement between experimental and predicted % and U values for the neural networks can be seen by the high R<sup>2</sup> and corresponding low mean

relative error (MRE). For both modes of rotation, the highest  $R^2$  was achieved for the data set with 70% used for training, 15% for testing and 15% for the cross validation. Therefore this combination was used for the development of neural network models. Figure 8.5 shows the prediction performances of the neural networks for the optimum combinations as plots of neural network predicted values vs. experimental values for both U and  $h_{fp}$ .

		U	h <sub>fp</sub>		
Different combinations of learning and test data	R <sup>2</sup>	MRE (%)	R <sup>2</sup>	MRE (%)	
A: 60 % for Training, 20% for Testing, 20 % for Validation	0.89	3.7	0.86	6.7	
<b>B:</b> 70 % for Training, 20% for Testing, 10 % for Validation	0.75	2.4	0.78	5.4	
C: 70 % for Training, 10% for Testing, 20 % for Validation	0.97	2.8	0.95	7.8	
<b>D:</b> 75 % for Training, 15% for Testing, 10 % for Validation	0.98	3.0	0.91	8.0	
E: 80 % for Training, 10% for Testing, 10 % for Validation	0.98	4.9	0.92	8.9	
<b>F:</b> 90 % for Training, 5% for Testing, 5 % for Validation	0.98	6.3	0.95	9.3	
G: 70 % for Training, 15% for Testing, 15 % for Validation	0.98	1.6	0.98	3.2	



Figure 8.5: Plots of neural network predicted values vs. experimental values

## 8.4.3 ANN output represented in algebraic equations

A trained neural network consists of an input layer with all the input variables, hidden layers, connections weights, bias values at each synaptic joints, a transfer functions forming a complex calculation process and a output layer. Equation 8.2 is the algebraic representation of an output neural network with a single hidden layer (Hernandez-Perez *et al.*, 2004).

$$Y = f_2 [W_2 f_1 (W_1 X + B_1) + B_2]$$
(8.2)

where, X is input matrix and Y is output the matrix,  $f_1$  and  $f_2$  are the transfer functions for the hidden layer and output layer,  $W_1$  and  $W_2$  are the matrices of connection weights in the hidden and output layers, and  $B_1$  and  $B_2$  are the matrices of biases in the hidden and output layers. A complex neural network output can be easily presented as in the form of Equation 8.2. In the present study, Equation 8.3 was established for the trained neural network output.

$$Y = [W_2 TanSig (W_1 X + B_1) + B_2]$$
(8.3)

where, *TanSig* is the transfer function used for training of neural network and X is the input matrix expressed in equation 8.4

$$X = \begin{vmatrix} X_{1} \\ X_{2} \\ X_{3} \\ X_{4} \\ X_{5} \\ X_{6} \\ X_{7} \\ X_{8} \\ X_{9} \end{vmatrix}$$
(8.4)

In Matrix X,  $X_1$  is the operating temperature,  $X_2$  is reciprocation frequency,  $X_3$  is amplitude of reciprocation,  $X_4$  is can headspace,  $X_5$  is CMC concentration (apparent viscosity),  $X_6$  is particle concentration,  $X_7$  is particle density,  $X_8$  is particle diameter, and  $X_9$  is Sphericity of particle.

Y is the output matrix expressed in Equation 8.5, in which  $Y_1$  is overall heat transfer coefficient (U) and  $Y_2$  is fluid to particle heat transfer coefficient ( $h_{fp}$ ). In the training process of neural network, all the input and output data were normalized according to Equation 8.6 (NeuralWare, 1996).

$$\boldsymbol{Y} = \begin{bmatrix} \boldsymbol{Y}_1 \\ \boldsymbol{Y}_2 \end{bmatrix} \tag{8.5}$$

Norm. data = 
$$\left[\frac{Upper bound - Lower bound}{Max - Min}\right] * [Real data - Max] + Upper bound$$
 (8.6)

The upper bound, lower bound, Max and Min values for input and output variables can be easily read through ANN output Matrix. The actual value of neural network output can be get by denormalizing the output from Equation 8.3. The bias and weight matrix of developed model are represented in following equations (Equations 8.7-8.10). Where,  $b_1$  is 8 x 1,  $W_1$  is 8 x 9,  $b_2$  is 2 x 1 and  $W_2$  is 2 x 8 matrix listed in Equations 8.7-8.10.

$$\boldsymbol{b}_{1} = \begin{vmatrix} +1.816 \\ -1.212 \\ +0.642 \\ +0.229 \\ -0.449 \\ -0.879 \\ -0.888 \\ +2.151 \end{vmatrix}$$
(8.7)

 $W_1$ 

$$= \begin{bmatrix} -0.179 + 0.853 - 0.044 + 0.258 + 0.8457 + 1.201 - 0.392 + 0.074 - 0.737 \\ +0.690 + 0.621 + 0.589 + 1.154 - 0.433 + 0.445 - 1.343 - 0.464 + 0.081 \\ -1.689 - 0.492 - 1.256 - 0.447 - 0.909 - 0.556 - 1.195 + 0.457 + 0.630 \\ -1.596 + 0.248 + 0.421 - 1.345 - 0.394 + 0.107 + 0.351 + 0.002 - 0.132 \\ -0.710 - 0.194 + 0.263 - 0.385 - 0.653 - 1.346 - 0.660 + 0.364 - 0.036 \\ -2.111 + 0.398 + 0.123 + 0.874 + 1.553 + 0.347 + 0.0603 - 0.007 + 0.267 \\ -1.137 - 0.448 + 0.633 + 0.089 + 0.264 + 1.006 - 0.335 - 0.064 - 0.0046 \\ -0.323 + 0.419 + 0.200 - 0.178 + 0.254 + 0.081 - 0.908 + 0.268 - 1.157 \end{bmatrix}$$
(8.8)

$$\boldsymbol{b}_2 = \begin{bmatrix} +0.980\\ -1.494 \end{bmatrix} \tag{8.9}$$

$$W_{2} = \begin{bmatrix} +0.766 + 0.340 - 0.512 - 1.597 - 1.431 + 0.518 + 0.571 - 0.110 \\ +0.181 + 0.883 - 0.644 - 1.484 - 1.096 + 0.975 - 0.533 + 0.604 \end{bmatrix}$$
(8.10)

Using the above matrix, U and  $h_{fp}$  can also be expressed as simplified algebraic system of equations (Equations 8.11-8.12).

$$U = 0.766a + 0.340b - 0.512c - 1.597d - 1.431e + 0.518f + 0.571g - 0.110h$$

$$+ 0.980$$

$$h_{fp} = 0.181a + 0.883b - 0.644c - 1.484d - 1.096e + 0.975f - 0.533g + 0.604h$$
(8.12)

- 1.494

where, **a-h** are the constants which can represented as:

$$a = \tan Sig \left(-0.179X_1 + 0.853X_2 - 0.044X_3 + 0.258X_4 + 0.845X_5 + 1.201X_6 \right)$$
(8.13)  
- 0.392X\_7 + 0.074X\_8 - 0.737X\_9 + 1.816

$$b = \tan Sig (0.690X_1 + 0.621X_2 + 0.589X_3 + 1.154X_4 - 0.433X_5 + 0.445X_6$$
(8.14)  
- 1.343X\_7 - 0.464X\_8 + 0.08X\_9 - 1.212)

$$c = \tan Sig \left(-1.689X_1 - 0.492X_2 - 1.256X_3 - 0.447X_4 - 0.909X_5 - 0.556X_6 \right)$$
(8.15)  
- 1.195X<sub>7</sub> + 0.457X<sub>8</sub> + 0.63X<sub>9</sub> + 0.642)

$$d = \tan Sig (-1.596X_1 + 0.248X_2 + 0.421X_3 - 1.345X_4 - 0.394X_5 + 0.107X_6$$
(8.16)  
+ 0.351X<sub>7</sub> + 0.002X<sub>8</sub> - 0.132X<sub>9</sub> + 0.229)

$$e = \tan Sig (-0.710X_1 - 0.194X_2 + 0.263X_3 - 0.385X_4 - 0.653X_5 - 1.346X_6$$
(8.17)  
- 0.660X<sub>7</sub> + 0.364X<sub>8</sub> - 0.036X<sub>9</sub> - 0.449)

$$f = \tan Sig \left(-1.137X_1 - 0.448X_2 + 0.633X_3 + 0.089X_4 + 0.264X_5 + 1.006X_6 \right)$$
(8.18)  
- 0.335X\_7 - 0.064X\_8 - 0.0046X\_9 - 0.888)

$$g = \tan Sig \left(-0.323X_1 + 0.419X_2 + 0.200X_3 - 0.178X_4 + 0.254X_5 + 0.081X_6 \right)$$
  
- 0.908X<sub>7</sub> + 0.268X<sub>8</sub> - 1.157X<sub>9</sub> + 2.151

Once the normalized output matrix (Y) is generated, the values need to be deformalized again using the upper and lower bound values from ANN output matrix. This can be easily done by using standard spreadsheet programs. Although, neural network can give very precise and reasonable predictions for the output variable, but it has to be always kept n mind that the output can be unsatisfactory if represented data was not used for training purpose.

### 8.4.4 Validation of ANN models using data from real foods

ANN Correlations generated in the previous section were developed using data from a mixture of model food particles and non-Newtonian fluids. However, real canned foods are of complex nature. Properties of real food particles like density and other thermal properties tend to vary during processing and as a result of which prediction of U and  $h_{fp}$  is never straightforward. However, the aim of every modeling scenario, mathematical or evolutionary, is to predict the phenomenon happening during processing of real food mixtures.

In order to examine the validity of the developed ANN model for real foods, developed correlations were tested on data from experiments involving canned green beans in various concentrations of CMC solutions (0-2% w/w). Using the ANN model developed in preceding section 8.4.3, U and  $h_{fp}$  values were predicted for canned green beans processed using reciprocating agitation thermal processing. U and  $h_{fp}$  values were also calculated using the methodology developed in Chapter 3 and modified for cylindrical green bean particles, as discussed in Section 8.3.2. The corresponding values of U and  $h_{fp}$ , predicted using the refined methodology and that predicted using the developed ANN correlations, are compared in Figure 8.6.



Figure 8.6: Comparison between the values of U and h<sub>fp</sub> for canned green beans obtained by using ANN model and methodology developed in chapter 3

It is seen from Figure 8.6 that the developed ANN model were able to provide a meaningful prediction of U and  $h_{fp}$ . It is seen that the error from ANN model was more prominent for lower values of U and  $h_{fp}$ . This might be attributed to the fact that these low values are associated with lower reciprocation speeds where the process time is quite high. Hence, the properties of the green beans would have changed more resulting in deviation from that predicted using the ANN model, which was based on model particles whose properties do not change. However, the ANN model, even under these deviation conditions, gave a lower value of U and  $h_{fp}$  than the real U and  $h_{fp}$  calculated using refined methodology. This represents a desirable conservative scenario as the temperatures predicted using these values would be lower than the real values and hence, the predicted process times would be higher.

### 8.4.5 Prediction of process time using ANN-FD model

The use of ANN and mathematical models for prediction of U and  $h_{fp}$  is aimed at developing the ability to predict process times for new processes. The prediction of process time is the reverse process of what has been described in this chapter. These process times can be evaluated from the time-temperature profile of the liquid and particle center in a can undergoing the reciprocating agitation, which can be predicted by established mathematical models once U and  $h_{fp}$  values, boundary conditions and thermo-physical properties of the product are available.

U and  $h_{fp}$  were obtained from the developed ANN model (Section 8.4.3) for different processing conditions highlighted in Table 8.1 for canned green beans processed using reciprocating agitation thermal processing. Once U and  $h_{fp}$  were available, time-temperature profile of the liquid & green-beans can be solved by simultaneous solution of the energy balance equation at can wall (Equation 8.6) and partial differential equation (Ramaswamy and Sablani, 1997b) which describes heat flow in a cylindrical particle immersed in liquid (Equation 8.7).

For cylindrical shaped particles: 
$$\frac{\partial T}{\partial t} = \alpha_p (\frac{\partial^2 T}{\partial r^2} + \frac{2}{r} \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2})$$
 (8.11)

The initial and boundary conditions are:

$$T(r, z, 0) = T_i \text{ at } t=0$$
 (8.12)

$$\frac{\partial \mathbf{T}(\mathbf{0}, \mathbf{z}, \mathbf{t})}{\partial \mathbf{r}} = \mathbf{0} \text{ at } \mathbf{t} > 1 \text{ and } 0 < \mathbf{z} < +\mathbf{L}$$
(8.13)

$$\frac{\partial \mathbf{T}(\mathbf{r}, +\mathbf{L}, \mathbf{t})}{\partial \mathbf{r}} = \mathbf{0} \text{ at } \mathbf{t} > 0 \text{ and } 0 < \mathbf{r} < + a$$
(8.14)

$$\mathbf{k_p} \frac{\partial \mathbf{T}}{\partial \mathbf{r}} = \mathbf{h_{fp}}(\mathbf{T_l} - \mathbf{T_{ps}}) \text{ at } \mathbf{r} = \mathbf{a}; \ 0 < \mathbf{z} < +L \text{ and } \mathbf{t} > 0$$
(8.15)

$$\mathbf{k_p} \frac{\partial \mathbf{T}}{\partial \mathbf{z}} = \mathbf{h_{fp}}(\mathbf{T_l} - \mathbf{T_{ps}}) \text{ at } \mathbf{z} = +\text{L}; \ 0 < r < +a \text{ and } t > 0$$
(8.16)

The process time was then predicted by calculating the accumulated lethality over the various instants of time using equation described in Chapter 3 (Section 3.3.4). The process time obtained for each processing conditions using the coupled ANN-FD model and that evaluated using the Ball process time from the heat penetration parameters (Table 6.1 and Table 7.1) are compared in Figure 8.7.



Figure 8.7: Comparison of Process time calculated by Ball formula and ANN-FD model.

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It was observed that the process times predicted by the ANN-FD model and the Ball's formula method matched for most of the processing scenarios. However, there was over-prediction of process time under lower frequencies of reciprocation. The sources of deviation may be attributed to the changes in the physical properties of the materials at high temperatures and due to inherent error associated with prediction of U and  $h_{fp}$  from ANN model, as discussed in Section 8.4.4. However, as the error resulted in over-prediction of process times, it can be considered conservative and the developed ANN model may be used to predict process times without any danger to the product safety.

Here, it must also be mentioned that despite the high accuracy of neural network models, it is definitely a 'black box' without any insight into the realities of the process. Certainly, ANN models should not be considered complete alternatives to a traditional mathematical modeling approach, which give useful insight into the physical phenomena and might benefit in scale up considerations. However, the mathematical models are computationally slow and require many different physical properties, high mathematical skills, good understanding of the problem and usually high computational resources for accurate prediction of complex phenomenon. On the other hand, implementation of an ANN model is rather straightforward and simple. In order to use the ANN models effectively, representative data from the actual experimental conditions must be used for training. Proper care must be taken in terms of selection of variables and optimization of parameters of the ANN model. Yet, if implemented carefully and under strict supervision, after initial training and testing, ANN models can help the process industries to simplify many complex processes with quick output time.

### 8.5 Conclusions

Artificial neural network models were developed to predict overall and fluid to particle heat transfer coefficient associated to canned non-Newtonian fluid particulate mixtures subjected to reciprocating agitation. The best performance of neural network was observed with 70% of data used for training the network while, 15% data were used for testing and 15% for the cross validation. Neural network parameters such as combination of hidden layers, learning runs, learning rules, transfer function and number of neurons were optimized. The developed ANN models were found to

accurately predict the heat transfer coefficients with mean relative error of 1.6 and 3.2% for U and  $h_{\rm fp}$ . ANN models coupled with finite difference were also developed to calculate thermal process time. The process time calculated from ANN-FD model and by using Ball formula were compared. Results shown that the process times predicted by the ANN-FD model and the Ball's formula method matched for most of the processing scenarios. However, there was over-prediction of process time under lower frequencies of reciprocation but, it can be considered conservative and the developed ANN model may be used to predict process times without any danger to the product safety.

# Chapter 9. GENERAL CONCLUSIONS, CONTRIBUTION TO THE KNOWLEDGE AND RECOMMENDATIONS

### 9.1 General conclusions

1. A methodology for evaluation of U and h<sub>fp</sub> in thermal processing of fluid particulate mixture subjected to agitation was developed based on the average heating rate index and operating temperature. The developed method was found free from fluctuations during come up period and was reliable and reproducible under rapid heating conditions. This could be used as a standard to compare the heat transfer of different retort processing systems.

2. On studying the effect of system parameters (i.e. temperature, frequency, amplitude, headspace and liquid viscosity) during thermal processing with reciprocation container agitation, both heat transfer coefficients (U and  $h_{fp}$ ) were found to increase with frequency, amplitude, headspace and temperature and decrease with increase in CMC concentration. Furthermore, a critical reciprocation intensity of  $18 \text{ms}^{-2}$  was identified to make a compromise between thermal and agitation induced quality losses to canned food.

3. On studying the effect of product parameter (particle size, shape, density and concentration) during thermal processing with reciprocating container agitation, it was observed that increasing the particle concentration up to certain limit is desirable for increasing the heat transfer. However, after certain limit any further increase in particle concentration restricts the particle motion and hence, reduces the heat transfer coefficients (U &  $h_{fp}$ ). Among the three particles, Nylon particles (density closer to covering fluid) have shown to have highest values of heat transfer coefficients, followed by Polypropylene and Teflon. Similarly, among the three particle shapes, the cube shaped particles had faster heat transfer followed by sphere and cylinder.

4. After investigating the heat transfer of reciprocating agitation thermal processing of non-Newtonian liquid-particulate mixtures, quality of real foods was investigated in this study. For this green beans were selected as they are more sensitive to thermal processing. Higher reciprocation frequency and temperatures significantly reduced the required process times and hence, reduced the total color change and resulted in a greener product with a higher colorimetric ratio, antioxidant activity and chlorophyll retention. However, due to excessive

shear on food particles breakage of green beans and leaching of nutrients (chlorophyll) to canned liquid was observed at high reciprocation intensity. It was concluded that canned products containing soft-particulates, like canned green beans, should be processed at lower frequencies to preserve the overall quality.

5. To minimize the impact induced losses to canned particulate foods, the concept of stopping agitations mid-way during agitation thermal processing was tested. For this, green beans were processed by imparting reciprocating agitation for various lengths of time *viz*. Full time, Partial time and Equilibration time agitation. It was concluded that stopping agitations after sufficient development of cold-spot temperatures could be an effective way of reducing product damages caused by agitation.

6. Artificial neural network models were developed to predict associated heat transfer coefficients and the ANN model was coupled with a finite difference model (ANN-FD model) to predict process time for a given lethality to canned non-Newtonian fluid particulate mixtures subjected to reciprocating agitation. Both ANN and ANN-FD models were found to accurately predict heat transfer coefficients and process time conservatively. It was also concluded that error associated to the ANN models were low at high frequencies.

## 9.2 Contribution to knowledge

- There was scarcity of literature available investigating the heat transfer and quality aspects of canned food subjected to reciprocating mode of agitation. This research is the first scientific investigation of the heat transfer and quality of canned non-Newtonian fluid particulate foods as affected by reciprocating agitation thermal processing.
- 2. The reciprocation agitation was found very effective in terms of increasing the heat transfer coefficients, lowering the process time and establishing uniform heating conditions inside the retort and within the can. Further, values of heat transfer coefficients under different processing conditions were provided which would be useful for process designing and mathematical modeling of the thermal processing of particulate foods of different thermal and physical properties.
- 3. Based on the severity of thermal treatment and reciprocation intensity, a critical reciprocation intensity was identified to make a compromise between thermal and agitation induced

quality losses to canned particulate food. This concept of critical reciprocation intensity can be used to divide foods into different categories, with each category requiring its own reciprocation intensities.

- 4. A comprehensive study was carried out to analyse the quality of canned fluid particulates subjected to reciprocating agitation and it was suggested that higher reciprocation intensities may cause texture damage to the particulates in the can.
- 5. A new concept of controlled agitation was investigated and was found very effective for controlling the texture damage to particulate during reciprocation agitation. This new concept can be used not only by reciprocating agitation, but in other agitation modes as well, to treat fragile products and to decrease energy requirements.
- 6. Artificial Neural Network models/correlations were established to predict process time and heat transfer coefficients (U and h<sub>fp</sub>) for liquid-particulate canned foods subjected to reciprocating agitation processing. These correlations and the ANN-FD model can be used by industry and academia alike to predict the relevant processing scenarios.

# 9.3 Recommendations for future research

This research work has demonstrated several important findings. Meanwhile it also showed some areas of interests for future research and development, which could be summarized as follows:

- Optimisation of process and product variables for thermal processing of other categories of canned food like fruits, pet foods, baby foods, sauces, liquid foods, food containing very small particulates etc. so that faster heat transfer will be achieved without compromising the quality of final product.
- Comparison of reciprocation mode of agitation with other novel processing methods like high pressure processing, microwave processing, ohmic heating, irradiation, pulsed light treatment etc. in terms of sterilizing/pasteurizing the food and preserving the end-quality of the processed product.
- 3. Extending this study to design optimal processing schedules and quantifying the effect of reciprocating agitation on other non-Newtonian fluids which might be shear thickening,

dilatant, thixotropic etc. These studies often are more complex and the objectives are specific for different categories of fluid.

- 4. Investigation of other possible modes of agitation (such as oscillatory agitation) which can provide gentle agitation rather than high impact agitation to cans and still can enhance heat transfer.
- Investigation of reciprocating mode of agitation during processing of semi-rigid and plastic food containers and minimize the damage to the containers during reciprocation processing.
- 6. Investigation for scale up the reciprocation processing with possible modifications for making the system semi-continuous to be integrated as a part of an industrial process.
- 7. Conducting biological validation of reciprocating agitation thermal processes.

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