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Heat transfer phenomena during thermal processing of liquid particulate mixtures – A Review

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Abstract:

During the past few decades, food industry is moving towards improved thermal and novel processing technologies as well as non-thermal processing techniques, to minimize the associated high quality loss involved in commercial thermal processing. Among these are the novel agitation systems which permit forced convection in canned particulate fluids to improve heat transfer, reduce process time and minimize heat damage to processed products. These include traditional rotary agitation systems involving end-over-end, axial or biaxial rotation of cans and the more recent reciprocating (lateral) agitation. The invention of thermal processing systems with induced container agitation has made heat transfer studies more difficult due to problems in tracking the particle temperatures of due to their dynamic motion during processing and complexities resulting from the effects of forced convection currents within the container. This has prompted active research on modeling and characterization of heat transfer phenomena in such systems. This review brings to perspective, the current status on thermal processing of particulate foods, within the constraints of lethality requirements from safety view point, and discusses available

techniques of data collection, heat transfer coefficient evaluation and the critical processing parameters which affect these heat transfer coefficients, especially under agitation processing conditions.

Keywords: canning; temperature measurement; heat transfer coefficient; liquid particulate mixtures; minimal process.

1. Introduction

Invented at the beginning of 1800 and scientifically established by early 1900, thermal processing technology has come a long way and is considered to be one of the very mature and scientifically based processes. Up until the 1950s, emphasis of thermal processing has largely remained on food safety, with quality loss barely registering an alarm. Common thermal processing techniques are still considered effective for preserving foods and ensure that the product is safe, shelf-stable and devoid of any harmful bacteria. Recently, however, due to increased consumer awareness about importance of food quality, technology has been gearing up to improve better quality retention without compromising safety. Several commercially adapted thermal processes like high-temperature short-time processing, ultra-high temperature processing, aseptic processing, agitation thermal processing, retort pouch / thin profile processing etc., have largely emerged to meet consumer demands for high quality products.

Thermal processing involves heating foods in hermetically sealed containers for specific time and temperature combinations in order to destroy pathogenic and spoilage micro-organisms (Ramaswamy and Marcotte, 2005). In order to properly predict the time and temperature combinations necessary to deliver a safe canning process, it is important to be able to predict the transient temperatures using models based on a thorough understanding of the mechanisms involved in heat transfer process. Further, the applied thermal treatment not only destroys microorganisms, but also simultaneously results in loss of many nutrients and degrades quality of products appreciably (Awuah *et al.*, 2007a). With the advancement of knowledge and advent of affordable technological concepts, there is a considerable interest in food industry for adaptation of technologies to satisfy consumer demand for producing high-quality products without

compromising safety and shelf stability. Thus, it is necessary to study and understand the heat transfer phenomenon associated with thermal processing for devising a process which not only imparts a minimal thermal treatment for ensuring safety and stability, but also result in minimal overcooking. Additionally, with many new products, new packages, and new processes appearing on the market each day, it is invariable to have appropriate methodologies for studying these systems for assuring minimal thermal treatments.

Convection is the dominant mechanism for heating of thermally processed foods. In static or batch processes, the phenomenon of natural convection comes into play. Convection heating is beneficial in terms of reducing process times, increasing production efficiency, and in some instances minimizing the ruinous effects of heat (Tucker, 2004). However, it has been observed that the natural convection involved in thermal processing is too slow to be of any benefit, except when dealing with liquid foods, and therefore needs to be supplemented with forced convection. This is achieved by agitating the containers by rotational (axial, biaxial or end-over-end) or reciprocation (lateral) agitation, and in doing so, forced convection current is induced that helps to mix and heat the food more effectively. Rotary retorts have tremendous advantages over still retorts for processing of viscous foods in large containers (Sablani and Ramaswamy, 1996; Tattiyakul *et al.*, 2002). Such retorts can increase convection in containers containing liquid–particle mixtures such as high-quality peas, corn, asparagus, mushrooms, and a variety of semisolid or viscous foods such as sauces or soups containing meat chunks or vegetables (Smout *et al.*, 2000). Most research on rotary agitation has been carried out with end-over-end (EOE) and axial modes of rotation. Apart from these two modes, biaxial mode of rotary agitation of containers (as is common in continuous turbo retorts) was studied by Dwivedi and Ramaswamy

(2010a) and was found to be better than other modes of rotation for reduction of process times and improvement in quality of products. Recently, a new concept of imparting agitation by using high frequency reciprocating (lateral) agitations has been proposed by Walden and Emanuel (2010). High frequency back-and-forth motion of containers during reciprocating mode of agitation has the potential to drastically reduce process times and to yield very high quality thermally processed food products. However, there exists a paucity of scientific data on the heat transfer phenomenon involved in this mode of agitation.

Validation of a thermal process by processing authorities requires gathering of time-temperature data to ensure that the minimum required lethality is achieved at slowest heating point of the system (particle-center for liquid-particulate foods). Time-temperature data are also required for evaluation of heat transfer coefficients associated with the process. However, with development of newer and faster agitation techniques, the task of collecting these data have become complex. It is difficult to gather center temperature of agitating particles without affecting their free motion. Various techniques have been developed for monitoring particle temperatures during agitation. Depending on the purpose of collection of heat penetration data, these techniques may range from the use of normal thermocouples (Deniston et al, 1987; Meng, 2006; Sablani, 1996) to the use of other technologies like wireless sensors (Awuah *et al.*, 2007b; Dwivedi and Ramaswamy, 2010b; Lesley, 1987; Wang *et al.*, 2006), time-temperature indicators like liquid crystals (Balasubramaniam and Sastry, 1995; Stoforos and Merson, 1991; Zitoun and Sastry, 1994) and, chemical (Van Loey *et al.*, 1995; Hendrickx *et al.*, 1995; Weng *et al.*, 1992) and biochemical indicators (Pflug *et al.*, 1980a, 1980b; Silva *et al.*, 1994).

Most of the earlier heat transfer studies on thermal processing (Berry and Bradshaw, 1980; Berry and Kohnhorst, 1983, 1985; Clifcorn *et al.*, 1950; Conley *et al.*, 1951) dealt with effect of agitation on specific heat penetration parameters of various products. However, the more recent ones have focused on evaluation of heat transfer coefficients associated with thermal processing. During thermal processing of liquid-particulate mixtures, two heat transfer coefficients are of interest: i) the overall heat transfer coefficient (U) from heating medium to can liquid through can wall, and ii) the fluid-to-particle heat transfer coefficient (h_{fp}) from liquid to particle-surface. Both U and h_{fp} are important parameters influencing the heating rate of liquid-particulate mixtures. Calculation of these heat transfer coefficients is also beneficial, because they can be used in predictive mathematical models and simulation programs for prediction and optimization of the thermal process, and thus reduce time and cost of experiments involved. A number of studies have been published to evaluate physical parameters that influence these heat transfer coefficients, and it has been recognized that agitation speed, retort temperature, headspace volume, system geometry, liquid viscosity, rotation radius, particle size, and particle density were key factors in influencing U and h_{fp} (Dwivedi, 2008; Lekwauwa and Hayakawa, 1986; Lenz and Lund, 1978; Meng and Ramaswamy, 2005, 2007a, 2007b; Naveh and Kopelman, 1980; Rao and Anantheswaran, 1988; Sablani and Ramaswamy, 1995, 1999).

Gathering of time-temperature data after the development of a new process is a primary task, both for the purpose of validation and for studying associated heat transfer. In this paper, various methods available in literature for gathering of time-temperature data during thermal processing of liquid-particulate mixture are reviewed. Further, different models used by researchers, to model the heat transfer associated with liquid-particulate mixture during canning, are

highlighted. Influence of system and product parameters on associated heat transfer coefficients during different modes of agitation is reviewed. In addition to this, this paper also presents the necessary requirements to impart a minimal process for safe sterilization during thermal processing.

2. Requirements for a minimal thermal process

Thermal processing primarily deals with application of heat for eliminating microorganisms which are of public health concern and for extension of shelf-life of foods. Although safety is the paramount consideration for designing a thermal process, yet it is important to preserve the quality of products as well. Hence, it is desirable for canners to be able to predict the amount of heat transferred which would be sufficient to eliminate most microorganisms while minimizing nutrient destruction. Therefore, understanding of process evaluation and process calculation methodologies is very important. The phrase “*minimal thermal process*” was introduced by the US Food and Drugs Administration in 1977 and is defined as “*the application of heat to food, either before or after sealing in a hermetically sealed container, for a period of time and at temperature scientifically determined to be adequate to ensure the destruction of microorganisms of public health concern*”. Calculating or determining the process schedule required to kill or inactivate heat resistant and pathogenic bacteria in food forms the basis of thermal process calculations. Determination of proper thermal process time and temperature for a can is a complex procedure with numerous factors involved like pH of the food, type and resistance of the target microorganism, storage conditions after the process, heating conditions, thermo-physical properties of the food, container shape and size etc. (Stumbo, 1973).

The operating temperature and pH are the most important factors as thermal resistance of microorganisms in low-acid foods is appreciably higher than the resistance of those found in high-acid foods and in both cases it is highly temperature dependent. Selection of a reference temperature for thermal processing is primarily based on the heat resistance of a target pathogenic microorganism that could be present in the product. Table 1 lists the heat resistance of common microorganisms in food at different levels of pH. From Table 1, it is clear that operating temperatures (around 250 °F) and decimal reduction times are highest for thermophilic and mesophilic spores in low-acid foods, and lower for mesophilic spores in pH range 4.0 – 4.6 (around 200-212 °F), and lowest for yeasts, molds, and non-spore-forming mesophilic bacteria in high-acid foods (around 150 °F). Thus, for a minimal process, heating low-acid foods at high temperatures under sterilization conditions is necessary, while a lower level of thermal treatment at pasteurization conditions is enough for high-acid foods. Additional details about heat resistance of microbes, level of pH and reference temperatures involved are available in literature (Ball and Olson, 1957; Breidt *et al.*, 2007; Brown, 2000; Larousse and Brown, 1996; Montville and Sapers, 1981; Odlaug and Pflug, 1997; Stumbo, 1973).

In order to determine the processing time, it is important to understand the concept of process lethality (F_0). Process lethality can be defined as the equivalent time of heating at reference temperature (250 °F for sterilization) which shall produce the same amount of microbial kill as the process. The desired degree of lethality (F_0) 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 preset value at slowest heating location of the product. Other than the basic mathematical model (Equation 1) used for lethality calculations, based on the original work

of Ball and Olson (1957), researchers have proposed many other methods to evaluate process lethality and process times for delivering a safe process, ranging from general and improved general methods, to various formula methods like Ball (1923), Stumbo (1973), Steele and Board (1979), Gillespy (1953), Hayakawa (1964), Pham (1989) methods etc.

$$F_0 = \int_0^t 10^{\frac{(T-T_0)}{z}} dt \quad (1)$$

Various researchers (Atherton and Thorpe, 1980; Barrett *et al.*, 2004; Brown, 2000; Montville and Sapers, 1981; Nelson and Tressler, 1980; Stumbo *et al.*, 1975) and trade associations (Institut-Appert, 1979; National Canners Association Research Laboratories, 1968; NFPA, 1971, 1982, 1985) have provided guidelines on minimal lethality requirements for the thermal processing of canned foods. In general, for low-acid foods, a minimum $F_0 = 3$ min (to achieve book-cook or 12-D reduction of *Clostridium botulinum* spores) is necessary; although, conservatively, a higher lethality between 5-15 min is usually applied. Different products like fish, meat, milk and dairy products etc. require different level of minimal treatments due to specific statutory laws in place for them. Some of the general principles on which these values must be selected have been highlighted for low-acid foods in Table 2.

On the other hand, for high-acid foods, a less severe heat treatment is necessary as it is very unlikely that *C. botulinum* spores will survive in a high-acid environment. A process of $F_0 = 0.7$ min is recommended by Hersom and Hullah (1980) for this purpose. For high-acid products, temperatures below 100 °C are usually adequate; however, heat-resistant fungi may survive and germinate to cause structural breakdown in some food products. The target lethality requirements at different operating temperatures for acidified foods at different operating temperatures and

temperature-sensitivity (z-value) of target microorganism have been given by various researchers and are presented in Table 3.

3. Methodologies for heat penetration data gathering

3.1. Use of thermocouples

Generation of heat penetration profile is important for establishing a thermal process. This involves carrying out experiments for measuring the time-temperature profiles at slowest point of heating for retort, liquid and food particles. Traditionally thermocouples are used for measuring temperature data. The most common types of thermocouples are duplex T-Type (copper/constantan with Teflon insulation) and K-type (chromel-constantan). Common configurations are flexible wires (20, 22 or 24-gauge) and rigid needle types. There are several other types of rigid and flexible thermocouples available: (a) molded thermocouples made of rigid Bakelite insulation, which are available to suit specific can sizes; (b) stainless-steel needle thermocouples, which are relatively thin and rigid; (c) flexible thin wire thermocouples which can be used for measuring the heat penetration at the center of particulate materials; and (d) custom-made thermocouples, rigid rod-in-tube type, which can be made to any suitable length (Holdsworth and Simpson, 2007). Literature exists that provide more details on the types of thermocouples which could be used for thermal processing operations (Bee and Park, 1978; Pflug, 2003). Table 4 details different types of thermocouple-based methodologies used for liquid and particle temperature measurements in agitating retorts.

Thermocouple-based approaches for data gathering generally use various thermocouple connectors and extension wires to transport temperature signals to a data logger or data

acquisition system. Caution must be exercised to avoid certain sources of error which may be associated with the use of these connectors and extension wires. These include: disparity in thermal emf between thermocouples, connectors and extension wires; temperature differences between two wire junctions; and reversed polarity at thermocouple-extension wire junction. Inaccuracies in temperature measurements may result in errors in process evaluation; hence, frequent calibration is essential to provide reliable data. Thermocouples should be calibrated in place as part of the complete data acquisition system against a traceable calibration standard (thermometer, RTD, thermistor). Factors affecting calibration include: improper junctions, metal oxidation, multiple connectors on single lead, worn or dirty slip-rings, and inadequate data-logger cold junction compensation. Some precautions when using thermocouple-based data acquisition systems include: minimizing multiple connections on the same wire; cleaning all connections; grounding the thermocouples and recording device; slitting thermocouple outer insulation outside the retort to prevent flooding of data-logger or data recording device; and using properly insulated thermocouple wires (Holdsworth and Simpson, 2007). Guidelines for use of thermocouples and thermocouple connection and calibration are detailed in literature (CFPRA, 1977; IFTPS, 1992; May, 1997).

3.2. Data collection via wireless sensors.

Data collection in the biaxial mode of rotation in a Steritort or data collection in hydrostatic sterilizers or rotary tunnels produces great challenges as it is almost impractical to use conventional wired thermocouples because of the haphazard/random motion of particles (Ex. helical motion in biaxial retorts or mixed and variable motions in hydrostatic or continuous

sterilizers). For data gathering under such situations, researchers have used self-contained wireless sensors which gather data continuously and transmit signals to a computer. Dwivedi and Ramaswamy (2010b) showed that there were no statistical differences between the performance of wireless systems compared to conventional systems and in fact, the wireless sensors were reported to be relatively more stable for rotary autoclaves especially for free axially rotating cans. By adopting wireless technology, 20-80% of the wiring costs in industrial application can be eliminated and also gives a chance of real time tracking of the free movement of particles (Wang *et al.*, 2006). Some other researchers (Francesco and Vittorio, 2003; Lesley, 1987) have also used wireless sensors and found them useful as a reliable temperature gathering technique.

However, it is not possible to realistically use data from these wireless sensors, even for determining local temperatures in cans, as these sensors are relatively large and occupy significant space resulting in deviations from actual process (Khurana *et al.*, 2009). Even with miniature wireless sensors, Awuah *et al.* (2007b) and Khurana *et al.* (2009) observed that process time for a target cumulative lethality was underestimated due to the combined effects of can contents, rotation speed, remote sensor configuration and sensor-mounting fixtures. Also, due to the sensor heat sink effect, recorded temperatures are not accurate. Hence, correction factors are required when wireless sensors (even miniature ones) are used. Ecklund (1965) provided correction factors for heat penetration thermocouples. However, results of Britt *et al.* (1997) revealed that Ellab Tracksense and Datatrace modules did not support Ecklund's correction factor (i.e., multiplying j_h value by 1.1) for 300x407 can size. Hence, use of wireless sensor always requires additional work for calculating appropriate correction factors to be used in process calculations and process validation. Some researchers are reluctant to use wireless

sensors in heat transfer studies as these correction factors frequently change with variation of process variables affecting thermal processing (discussed in detail in Section 5). For temperature measurement of moving particles, these would be of limited use because of the motion restrictions imposed on particle when such devices are attached. Although, in static retorts, attaching the largest particle to the tip of the wireless sensor of appropriate length might provide reasonable data, and could be used by determining the proper correction factor.

3.3. Use of thermochromic liquid crystals (TLCs)

Although particle-surface temperatures can be used to solve simple energy balance models (Equations 2 and 3) to predict heat transfer coefficients, these temperatures cannot be measured using conventional thermocouple techniques due to difficulties in fixing the thermocouple tip to the surface of a moving particle. In order to solve this problem, thermochromic liquid crystals (TLCs) have been used by different researchers (Balasubramaniam and Sastry, 1994a, 1994b, 1994c, 1995; Stoforos and Merson, 1991; Zitoun and Sastry, 1994). Liquid crystal is a chemical compound which changes its color in response to surface temperature changes due to their unique molecular structure rearrangement. They are coated on a free moving particle and the particle temperature history is generated by image analysis of videotaped color changes of liquid crystal during heating. Balasubramaniam and Sastry (1995) reviewed the use of thermochromic liquid crystals (TLCs) in heat transfer studies and their applications to food processing research.

Some researchers (Gadonna *et al.*, 1996; Ochoa *et al.*, 2005) have further improved this technique by using a transducer particle made up of two concentric spheres (core of the particle was a polypropylene sphere pre-coated with a black paint and then coated with a liquid-crystal

and subsequently coated with a layer of transparent epoxy resin of known thermal diffusivity) or by using a surface with a low thermal mass (a thin tissue embedded with TLCs). Image analysis of hue of liquid crystals reveals surface temperature distributions with positions and time and shows how the local heat transfer coefficients change with time. Dynamic flow behavior, such as vortex shedding etc., can be used to quantify the effect of flow behavior on heat transfer phenomenon occurring during the process. Basics involved in the calibration and the use of TLCs are described in detail in literature (Abdullah *et al.*, 2010; Kakade *et al.*, 2009)

The liquid crystal technique can provide useful information, if properly used, but does have limitations. Color calibration is difficult; and it must be emphasized that each calibration curve between temperature and hue value is unique as color determination depends both on equipment and environment conditions during the experiment (different lightening chambers, light bulbs, dust formation on light bulbs, image processing system etc.). Furthermore, the method can only be used with optically transparent liquids [Balasubramaniam and Sastry, 1995; Kakade *et al.*, 2009; Abdullah *et al.*, 2010).

3.4. Use of melting point indicators (MPIs)

The melting point approach is similar to use of TLCs, as both involve optical methods, for particle-surface temperature measurements. In this method, a melting point indicator (MPI), made of a polymeric material and which changes color at a specific temperature, is used. Hollow spherical particles are used in this method and they are filled with several MPIs with different melting points. Time taken by the surface of indicator (placed inside the particle) to reach its melting point is measured and their color changes are visually recorded. This method has been

used by several researchers (Damay and Pain, 1993; Mwangi *et al.*, 1993) for monitoring flow behavior and spatial heat transfer coefficient distribution during thermal processing. Like other optical methods, the melting point indicator method is also limited to transparent fluids and, therefore, can only be used with model systems.

3.5. Use of biological indicator units (BIUs)

Biological indicators have been used to monitor temperature and lethality achieved inside a can simultaneously. This was done by development of biological indicator units (BIUs), by Pflug *et al.* (1980a, 1980b), containing a sample tube filled with spores of *Bacillus stearothermophilus*. This BIU tube is then inserted into the cans at slowest point of heating. These tubes are generally made of plastic. For calibration of BIUs for lethality measurements, a representative sample from a large batch is calibrated by submitting them to a known thermal process, and F_0 value can be determined from heat penetration. Apart from *B. stearothermophilus*, other spores have been used recently. Rönner (2002) developed a technique for studying sterilization systems using BIUs made of *B. stearothermophilus* and *B. subtilis* in an 8 mm diameter microporous bead of modified polyacramide gel with 90% water. They also developed a similar system using ascospores for studying pasteurization. However, because of the associated thermal lag due to the containment material, this technique produces a lag in temperature outputs. Other researchers have also used aluminum containers to improve heat transfer and eliminate thermal lag during temperature measurements (Rodriguez and Teixeira, 1988). These techniques show good agreement between F_0 values from heat penetration compared with F -values from the BIUs, yet, the biological values tend to be slightly lower than

the F_0 from heat penetration measurements. In a more recent study on biaxial rotation processing of liquid particulates cans, Hassan and Ramaswamy (2013) used the biological validation technique to calculate process lethality. In this study, carrot and meat alginate spherical particles, inoculated with spores of *C. sporogenes* and *Geobacillus stearothermophilus*, were filled into cans along with a non-Newtonian liquid (carboxymethyl cellulose). Process times were calculated to achieve an accumulated lethality of 3 and 15 min for carrot and meat alginate fabricated particles were calculated. Using spore counts of the particles before and after the given process, the number of log reductions of the bacterial spores and hence the process lethality were determined. They showed that F_0 values obtained from biological validation were statistically same as those computed from numerical simulation thereby demonstrating that the standardized bio-validation technique can be effectively used for establishing/verification of thermal processing schedules

3.6. Use of chemical and biochemical indicators.

Apart from BIUs, several researchers have also developed many biochemical (Hendrickx *et al.*, 1995) and chemical indicators (Silva *et al.*, 1994) for collecting heat penetration data inside the can by correlating them with lethality measurements. These are collectively known as time-temperature integrators (TTIs) as they show the accumulated time-temperature history of a product and change properties (like color, degree of microbial kill etc.) when a particular time temperature combination has been reached. Thus, lethality measurements (using Equation 1) can be made. Chemical indicators have also been used to determine heat transfer coefficients between food particles and liquids (Maesmans *et al.*, 1993, 1994; Van Loey *et al.*, 1995; Weng *et*

al., 1992). Weng *et al.* (1992) used immobilized peroxidase as a chemical TTI to gather the temperature history of particles in the pasteurization process. Anthocyanins, thiamins, methylmethionine sulfonium and sucrose inversion are some other chemical TTIs which have been used. Ramaswamy *et al.* (1996) also used a chemical marker for process lethality measurement at 110°C in a continuous medium flow holding tube.

Apart from these, α -amylase has been used extensively as a biochemical TTI for studying inactivation kinetics during sterilization and pasteurization processes (Tucker, 1999; Van Loey, 1996; Van Loey *et al.*, 1997). However, α -amylase is unstable at very high temperatures and is hence treated with *Bacillus amyloliquefactens* (Maesmans *et al.*, 1994), *B. licheniformis* (De Cordt *et al.*, 1992) or polyols (De Cordt *et al.*, 1994) to improve its stability. Some researchers (Haentjens *et al.*, 1998; Guiavarc'h *et al.*, 2002a, 2002b; Van Loey *et al.*, 2004) have also developed enzyme systems such as using α -amylase at reduced water content as TTIs and residual denaturation enthalpy as response. A comprehensive list of potential TTIs which can be used for thermal processing is available in literature (Holdsworth and Simpson, 2007; Pflug, 2003; Van Loey *et al.*, 1995).

3.7. Use of magnetic thermometry and magnetic resonance imaging techniques

All previous experimental techniques are limited by the need to measure temperature directly by thermocouples or indirectly by using BIU, TTI etc. or by an optical method. But none of them can track the temperature of a freely-moving particle non-invasively. Some progress has been made with 'temperature pills' which use a particle with a quartz crystal inside acting as the temperature sensing element. The element resonates at a temperature-dependent frequency and

invokes a coil circuit and thus generates a magnetic signal (Balasubramaniam and Sastry, 1994c). An external receiver converts the magnetic signal into a temperature reading. Bhamidipati and Singh (1994, 1995) were the first to exploit this technique in detail. They used a cylindrical particle (22.6 mm length, 10.7 mm diameter) of known density and specific heat capacity, which broadcasts its temperature to an external antenna coil following the motion of particle sensor inside the tube, allowing temperature-time profile of the particle to be obtained. The test fluids were aqueous CMC solutions heated to 82 °C. Another novel non-invasive technique (Ghiron and Litchfield, 1997) involved measurement of temperature using magnetic resonance technique: the change in temperature of a magnetic particle was detected by change in its magnetization. More recently, researchers have used magnetic resonance imaging to measure temperature and subsequently derive the thermal diffusivity (Gultekin and Gore, 2006) and heat transfer coefficients (Gultekin and Gore, 2008) of products. Being non-invasive, these techniques do not interfere with particle trajectory as temperature is being measured, and has added advantage of yielding local h_{fp} values. However, the large size and density of the broadcasting particle is severely limiting. It is also crucial that sensing particle behaves the same way as the food. This type of technique offers scope for real process measurement, but it is again not yet fully developed.

4. Methodologies for evaluating heat transfer coefficients during thermal processing of particulate liquid foods

The heat transfer in a can is characterized by assuming a model in which heat is transferred in two stages: first from the heating medium (steam or water) to can liquid and then from can liquid

to the particle. The intensity of heat transfer is characterized by heat transfer coefficients existing at these two stages: overall heat transfer coefficient, U (from heating medium to can liquid through the can wall) and fluid to particle heat transfer coefficient h_{fp} (from liquid to particle surface). The values of U and h_{fp} are used to estimate efficiency of associated heat transfer.

The governing equation for heat transfer in such systems is given by the energy balance equation for a liquid-particulate canned system [Equation (2) (Deniston *et al.*, 1987)] according to which total heat coming inside the can is equal to the heat energy required for increasing the temperature of liquid and particle.

$$UA_c(T_R - T_f) = m_f c_{pf} \frac{dT_f}{dt} + m_p c_{pp} \frac{dT_p}{dt} \quad (2)$$

Second term on the right side of Equation (2) is equal to heat transferred to particles across liquid-particle interface, and can be represented as Equation (3). Simultaneous solution of these two equations yields the value of U and h_{fp} .

$$m_p \cdot c_{pp} \cdot \frac{dT_p}{dt} = h_{fp} A_p (T_f - T_{ps}) \quad (3)$$

4.1. From direct particle temperature measurements

Equations (2-3) form the basic framework which needs to be solved to calculate U and h_{fp} . This evaluation can be independently solved by collecting the time-temperature data of retort, liquid and particle-surface to estimate U and h_{fp} . However, it is difficult to collect particle-surface temperatures and instead, many researchers find it easier to collect particle-center temperatures using thermocouples fixed to the particle-center (as described in Section 3.1.).

Particle-center temperature collection is also preferred, as cold-spot temperatures are used to determine process lethality (Equation 1). On measuring particle-center temperatures, heat transfer coefficients (U and h_{fp}) are back-calculated by comparing experimental lethality (Equation 1) to that determined by solving Equations (2-3) together with conduction heat transfer equation inside the particle. For a spherical particle, heat flow is described by the following partial differential Equation (4) and initial and boundary conditions given by Equations (5-7) (Sablani and Ramaswamy, 1996):

$$\frac{\partial T}{\partial t} = \alpha_p \left(\frac{\partial^2 T}{\partial r^2} + \frac{2}{r} \frac{\partial T}{\partial r} \right) \quad (4)$$

$$T(r, 0) = 0 \quad \text{at } r=0 \quad (5)$$

$$\frac{\partial T(r, t)}{\partial r} = 0 \quad \text{at } r=0 \quad (6)$$

$$k_p \frac{\partial T(r, t)}{\partial r} = h_{fp} (T_f - T_{ps}) \quad \text{at } r=a \quad (7)$$

The value of h_{fp} can be back-calculated by solving Equation (4) (subject to initial boundary conditions defined by Equations 5-7), and using this h_{fp} , U values can be obtained by solving Equations (2-3). This technique describes the basic methodology for evaluating U and h_{fp} . Most researchers have used either a similar or a slight modification of this technique for measuring U and h_{fp} in liquid-particulate food mixtures in can. There are many numerical, computational and analytical techniques for solving these solutions (Table 5). On comparing analytical and numerical methodologies, analytical approach was found to show more accuracy (Cariño-Sarabia and Vélez-Ruiz, 2013).

4.2. From indirect particle temperature measurement

Apart from traditional thermocouple-based direct temperature measurement techniques, particle temperature can also be measured by indirect technique like the use of BIU and other TTI and TLC etc. For these types of techniques, researchers (De Cordt *et al.*, 1992, 1994; Guiavarc'h *et al.*, 2002a, 2002b; Hassan and Ramaswamy, 2013; Haentjens *et al.*, 1998; Hendrickx *et al.*, 1995; Maesmans *et al.*, 1993, 1994; Pflug *et al.*, 1980a, 1980b; Silva *et al.*, 1994, Tucker, 1999; Van Loey, 1996; Van Loey *et al.*, 1995, 1997, 2004; Weng *et al.*, 1992) have used lethality or other time-temperature integration models. The model gives lethality as output (F_m) using Equations 1-7 for different values of U and h_{fp} . Lethality of experimental TTI (F_{TTI}) is calculated using calibration charts or by reading the TTI's initial (N_0) and final (N) status in accordance with Equation (8).

$$F_{TTI} = D \log(N_0/N) \quad (8)$$

Values of F_{TTI} and F_m are matched by iteration of values of U and h_{fp} to yield the heat transfer coefficient (U and h_{fp}) of the thermal process. Weng *et al.* (1992) named this approach a *Least Absolute Lethality Difference* (LALD) method and also compared it with another common approach called *Least Square temperature difference* (LSTD) in which sum of the squares of temperature differences of the model output is matched with that of the experimental particle. They showed that using the LALD-approach, weight of higher temperatures (more lethal) is more pronounced than that of lower temperatures in their respective contribution to the final processing value, which is in accordance to the concept of higher microbial kill at higher temperatures. However in case of LSTD, weight of all temperatures (low or high) are same.

Thus, LALD procedures can be used in agitated conditions where it is difficult to satisfy the Fourier number stability criterion ($\tau < 0.2$) in the initial temperature range (ex. agitation processing or HTST methodologies), and thus accuracy of lower temperatures is limited.

4.3. When collection of particle temperature is difficult

Under certain conditions, temperature monitoring using traditional optical or thermocouple-based technologies is difficult. Dwivedi and Ramaswamy (2010b) developed an innovative methodology to measure U and h_{fp} under conditions where temperature monitoring is not possible. This methodology is particularly useful when an alternate mode of processing is available where particle-temperatures can be measured. This methodology is based on the assumption that ratio of U/h_{fp} fall within a statistically similar range, even if the mode of agitation is changed, because factors influencing U will also influence h_{fp} in a somewhat similar manner. This method involved developing correlations between h_{fp} and U using real time-temperature data gathered from the processing mode in which particle temperatures are measurable. This correlation was then used to calculate h_{fp} from U -value obtained from low Biot-number model described in Table 5. Dwivedi and Ramaswamy (2010b) coupled experimentally evaluated U from fluid temperatures (measured using wireless sensors) in the biaxial mode with values of ratio of U/h_{fp} in fixed axial mode to compute h_{fp} for the biaxial mode. This approach gave consistent results with the traditional approach of measuring both liquid and particular temperature. It is to be noted that h_{fp} values using this methodology are representative values and may deviate from the h_{fp} values computed using other methods, yet

they can be used to understand the effect of various process variables on the heat transfer happening during the thermal process.

4.4. Using heat-flux instead of temperatures

Apart from techniques involving direct and indirect temperature measurement, other innovative techniques have also been used to predict h_{fp} . In such approaches, heat conduction errors are considered to be negligible. Here, h_{fp} is determined by monitoring the heat flux, e.g., from a heating wire, needed to maintain particle-surface temperature constant. Generally, a good agreement between predicted and experimental data is obtained for fluid temperatures, however, significant variation are observed for particle-surface temperatures using these approaches (Chang and Toledo, 1989, 1990; Stoforos *et al.*, 1997).

5. Factors affecting heat transfer coefficients (U and h_{fp}) during thermal processing

There are many studies in which heat transfer coefficients (U and h_{fp}) have been evaluated by researchers. These studies can be clearly divided on the basis of mode of agitation. Some of these have been summarized for non-agitated and agitated systems in Tables 6 and 7, respectively. These values have been gathered under various operating conditions and with different particles and liquid. Some researchers have used model food particles for data collection, while others have used real food systems. An analysis of these values gives an idea of the intensity of heat transfer under various modes of operation. In general, any factor which increases agitation or decreases thickness of boundary layers leads to increase in values of U and h_{fp} . Some of these

factors affecting heat transfer coefficients during thermal processing of liquid-particulate mixtures have been discussed here.

5.1. Mode of agitation

Ultimate objective of agitation thermal processing is to improve the values of these heat transfer coefficients. In general, it is observed that increased agitation leads to increase in heat transfer occurring in the system. It can be seen that non-agitated mode of thermal processing has the least values of heat transfer coefficients (Table 6). Amongst agitated systems, biaxial mode of agitation has the highest heat transfer coefficient, followed by EOE mode and then by fixed axial mode of rotation (Dwivedi and Ramaswamy, 2010a). For oscillatory mode of rotation, 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 rotation and axial rotation. There is a possibility to increase these values further by using new modes of agitation like reciprocating or lateral agitations, which promises to increase the heat transfer tremendously (Walden and Emanuel, 2010). However, there is no published heat transfer coefficient data available for this mode of agitation. The reason for this increase is that magnitude of U and h_{fp} is governed by particle-to-fluid relative velocity (Chandarana and Gavin, 1989), which is increased by lateral or reciprocating agitation.

5.2. Speed of Agitation

As early as 1951, Conley *et al.* (1951) documented the impact of speed of agitation on heat transfer rates and resulting process times. Heat transfer and lethality of canned liquid foods

containing particles processed in Steritort have been studied (Lenz and Lund, 1978), and it was found that changing reel speed from 3.5 to 8 rpm resulted in an average increase in U by 33% and in values of h_{fp} by 44%. The effect of rotational speed is more evident at lower speeds. At higher speeds, effect of rotation generally dies down, and after a certain speed, heat transfer coefficients do not improve much. Hassan (1984) measured convective heat transfer coefficients of Teflon, aluminum and potato spheres in end-over-end rotation, and found that varying rotation speed from 9.3 to 101 rpm had more of an effect on U than on h_{fp} . Other researchers have also found similar trends of inhibited increase in heat transfer coefficients beyond a certain agitation speed (Sablani, 1996; Meng, 2006). This trend has been attributed to smaller relative particle-to-liquid velocity at higher rotational speeds. Under larger centrifugal force at higher speed, particles are forced to clump to one edge of the wall resulting in lower relative velocities. Researchers have confirmed these trends using particle motion analysis (Dwivedi, 2008; Meng, 2006; Sablani, 1996; Stoforos, 1988) by videotaping motion of particles inside transparent cans. Reciprocating agitations can be used to overcome this drawback associated with rotary agitation, as reciprocating motion is free of the effects of centrifugal forces. Dwivedi (2008) reported in his study that U and h_{fp} in free axial mode and biaxial mode increased from 448-907 and 477-1075 W/m²K, respectively, with increase in revolutions per minute from 4 to 24. Thus, in general, increase in speed of agitations increases heat transfer coefficients in all modes of agitation, however, this increase is more prominent at lower speeds.

5.3. Fluid viscosity

With an increase in liquid viscosity, level of turbulence inside the can decreases as Reynold's number is inversely proportional to viscosity. Hence, there is a lower mixing and thereby lower heat transfer coefficients. Various researchers have evaluated U and h_{fp} with liquids of varying viscosities. Lenz and Lund (1978) evaluated the effect of viscosity by comparing water and 60% aqueous sucrose solutions. They found that water had higher heat transfer coefficients, than a 60% aqueous sucrose solution, indicating a role of fluid viscosity in the values of U and h_{fp} . Other researchers (Hassan, 1984; Sablani, 1996; Stoforos and Merson, 1991) also noticed that with an increase in liquid viscosity, heat transfer coefficients decreased, and vice-versa. These trends were later confirmed by particle motion studies (Dwivedi, 2008; Meng, 2006; Sablani, 1996; Stoforos, 1988), which showed that for a more viscous liquid, flow was less turbulent, leading to lower particle mixing and lower liquid-particle velocities.

For non-Newtonian fluids, whose viscosities are dependent on temperature, some researchers have used an alternate methodology (discussed in Table 5) to predict U_a and h_{ap} (Meng, 2006, Meng and Ramaswamy, 2007a) instead of U and h_{fp} . Meng and Ramaswamy (2007a) found that for CMC solutions associated h_{ap} values ranged from 215 to 376 W/m²K and U_a values ranged from 112 to 293 W/m²K. Their U_a decreased with an increase in liquid viscosity, which could be explained by thickness of associated boundary layers. Higher thickness of momentum boundary layer leads to larger higher heat transfer resistance resulting in lower U and h_{fp} . With high viscosity glycerin concentration, researchers (Dwivedi, 2008; Dwivedi and Ramaswamy, 2010a, 2010b; Meng, 2006; Sablani, 1996) have reported that 20-40% lower values under various rotary modes of agitation.

5.4. Container headspace

It is generally expected that headspace might act as an insulator in natural convection scenarios (Mohamed, 2007). However, Ramaswamy and Grabowski (1996) noted that complex nature of heat transfer in headspace makes theoretical prediction difficult. Heat transfer in headspace is governed by evaporation resulting in significant mass transfer effects leading to more rapid heating of water surface inside the can right just below the headspace, compared to no headspace situation. Joseph et al. (1996) reported faster heating rates with an increase in headspace volume in canned conduction-heated materials. James et al. (2006) also reported rapid heating effect in cans with 10% headspace compared to no headspace situation during off-axial rotation of cans. Erdogdu and Tutar (2011), through their computational fluid dynamics analysis, observed that headspace temperature increased rapidly due to lower heat capacity and viscosity of air, and heating rates were faster in cans containing headspace compared to no headspace situation. Thus, it is seen that presence of headspace invariably increases the amount of heat transfer. However, after sufficient headspace (around 7-14 mm), the headspace bubble starts acting as an insulator (Mohamed, 2007; Ramaswamy and Grabowski, 1996), retarding further heat transfer. Mohamed (2007) evaluated the effect on heat transfer coefficients for three levels of headspace (10%, 14% and 20% v/v) and found that calculated average effective heat transfer coefficients to be 54.2, 53.2 and 50.8 W/m²K for the three headspaces, respectively. Other researchers (Chandarana and Gavin, 1989; Lekwauwa and Hayakawa, 1986; Sablani and Ramaswamy, 1995, 1999; Sastry *et al.*, 1985) have also seen that increase of headspace first increases heat transfer coefficients; however after an optimum headspace, U and h_{fp} start decreasing. On neglecting headspace, insulating effects will inevitably lead to either over-

processing or under-processing both of which may have negative effects on consumer's health and safety.

5.5. Particle concentration

Particle concentration (ϵ) is generally defined as percentage of particle volume to that total liquid-particulate volume. Researchers (Deniston *et al.*, 1987; Hassan, 1984) have studied effect of particle concentration on heat transfer rates and found that with increase in the particle concentration, heat transfer coefficients increase up to a maximum value and then start decreasing. This phenomenon is explained by the fact that the presence of additional particulates causes secondary agitation of the liquid due to collisions amongst the particles and affects heat transfer coefficients. This results in better mixing and distribution of heat inside the can, leading to increase in U and h_{fp} values. Hence, U and h_{fp} increase till an optimum concentration is reached. Beyond this concentration, particles become tightly packed in the can and are no longer free to move (Deniston *et al.*, 1987). Hence, functional dependency of U and h_{fp} on particle concentration is different for tightly packed particle as compared to loosely packed particles. Sablani (1996) found that U increased 20% for oil and 5% for water at 30% concentration when compared to a single particle, but a further increase in concentration to 40% resulted in decrease of h_{fp} by 12% for oil and 7% for water. Dwivedi (2008) also reported that particle concentration has a significant influence ($p < 0.05$) on U and h_{fp} . For a particle size of 25 mm, U increased with increase in particle concentration from 20% to 30%, while further increasing this concentration to 40% decreased U and h_{fp} .

5.6. Particle size

Hassan (1984) found that increase in diameter of potato spheres processed in water from 2.22-3.49 cm, resulted in greater process times for fluid and particles. It was observed that the effect of rotational speed on particle heating times was higher than for smaller particles than larger particles. The overall heat transfer coefficient, however, was reported to have highest values at all rotational speeds when particles of an intermediate size (2.86 cm) were used. Deniston *et al.* (1987) 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 (1996) 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 U_a and h_{ap} . Dwivedi (2008) reported that U values in free axial mode were found to decrease by 29-33% as size of particles increased from 19.05 to 25 mm. Under the same increasing size condition, h_{fp} also decreased, probably due to thicker boundary layers associated with larger particle diameters. They also noticed that increase in diameter had greater influence on h_{fp} than U (h_{fp} decreased by 32-41% for 19.05 to 25 particles). Thus, we may conclude that, like other parameters, U and h_{fp} increases to a maximum value with increase in size of the particle, due to increased turbulence created by larger particles, and then starts decreasing possibly due to restricted movements.

5.7. Particle shape

Different shapes like spheres, cubes, cylinders etc. contribute to varying void fraction between particles and hence liquid fill would be different which also creates differences in the severity of

agitation. Thus, heat transfer coefficients could very well depend on particle shapes. Sablani and Ramaswamy (1995, 1998) found that U-values for the cube-shaped single-particle in oil were about 6% lower than those of the cylinder, and 8% lower than that of the sphere. h_{fp} values for cube were 6% higher than cylinder and 23% higher than sphere. They concluded that the influence of shape is more noticeable on U than h_{fp} . Similar results have also been observed by Meng (2006) and Dwivedi (2008). The particle shape also influences the associated surface area as well as surface to volume ratios. These can also influence the associated heat transfer coefficients during thermal processing under container agitation conditions.

5.8. Particle density

Particle density can affect particle fluid motion pattern inside can, thereby affecting heat transfer coefficients. It has been observed that U and h_{fp} , generally, increase with increase in density. Particles of greater density settle in the can faster, resulting in more motion inside the can due to higher particle-liquid relative velocity, resulting in a higher h_{fp} (Sablani, 1996). Meng and Ramaswamy (2007b) also reported that h_{fp} values increased with increase in 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 (lighter than water) have lowest h_{fp} (495 W/ m²K) and Teflon particles (heavier than water) have the highest (922 W/m²K), while nylon particles (density almost equal to water) fell in between with h_{fp} of 764 W/ m²K. However, in a single-particle scenario, Sablani (1996) had found that Teflon particles had lower h_{fp} than nylon as Teflon settled at bottom of the can.

6. Conclusions

Thermal processing of liquid particulate mixtures is complex and understanding the associated heat transfer phenomenon requires extensive research. The choice of minimal lethality to be imparted during a thermal process is governed by many factors, product pH being the most important. Ample scientific literature is available to select the value of minimal lethality. However, calculation of time-temperature combinations required to deliver this lethality requires substantial experimental work involving collection of heat penetration data. Researchers have used both thermocouple and non-thermocouple-based methods to collect the heat transfer data, in both still mode of operation and in agitating containers. However, data collection in agitating containers is still troublesome and provides opportunities for improvement. Many numerical and analytical methods have been used by researchers to analyze these data for evaluating U and h_{fp} . All these methods involve solving the same energy balance equation; however, exact treatment differs distinctively for direct and indirect temperature measurement techniques. Various innovative and simpler methodologies are also available in certain conditions of processing. Although recent studies have attempted to understand the phenomenal behavior of heat transfer in liquid-particle mixtures in still and agitating conditions, several conflicting trends have been reported that need further clarification. It has generally been observed that any configuration which leads to increase in the intensity of agitation yields higher values of U and h_{fp} . However with increased agitation, role of the centrifugal forces in rotating systems limit the amount of heat transfer and thus, increase in U and h_{fp} is limited with rotary agitation. This prompts the need for improving thermal processing technology further through constant innovations in the design of retorts and data collection and analysis methodologies.

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Table 1. Comparative heat resistance of bacteria important to thermally processed foods
(adapted from the works of Larousse and Brown, 1996; Odlaug and Pflug, 1997).

Class of food	Microbial class	Microorganism(s)	Reference temperature (°F)	D value (min)	z value (°F)
Low-acid food (pH > 4.6)	Thermophiles	Flat-sour group (<i>B. stearothermophilus</i>)	250	4.0 – 5.0	14-22
	Mesophiles	Putrefactive anaerobes (<i>C. botulinum</i> - A and B)	250	0.1–0.2	14-18
		<i>C. sporogenes</i> group (including P.A. 3679)	250	0.1 - 1.5	14-18
Acid food and acidified food (pH 4.0 – 4.6)	Thermophiles	<i>B. coagulans</i> (facultatively mesophilic)	250	0.01–0.07	14-18
	Mesophiles	<i>B. polymyxa</i> and <i>B. macerans</i>	212	0.10 – 0.50	12-16
		Butyric anaerobes (<i>C. pasteurianum</i>)	212	0.10 – 0.50	12-16
		<i>B. licheniformis</i>	200	4.5	27
Acid food and acidified food (pH < 4.0)	Yeasts, molds, and mesophilic bacteria	<i>Lactobacillus</i> species, <i>Leuconostoc</i> species	150	0.50 – 1.00	8-10

Table 2. Principles for minimum lethality requirements for low-acid foods (pH>4.5)

Reference	Minimum Lethality Requirement	Comments
Stumbo <i>et al.</i> (1975)	<u>Small cans</u> (54.0 mm x 57.2 mm): $F_{10/121.1} \geq 2.8$ min	Food safety: To achieve 12 log reduction in <i>C. botulinum</i> spores Spoilage still possible.
	<u>Big cans</u> (157.2 mm x 177.8 mm): $F_{10/121.1} \geq 3.1$ min	
	<u>Small cans</u> (54.0 mm x 57.2 mm): $F_{10/121.1} \geq 9$ min.	Shelf life: To achieve 4 log reduction in <i>C. sporogenes</i> spores Organism of concern is spoilage spore <i>C. sporogenes</i>
	<u>Big cans</u> (157.2 mm x 177.8 mm): $F_{10/121.1} \geq 11$ min.	
Holdsworth and Simpson (2007)	$F_{10/121.1} \geq 3.0$ min	Food safety: To achieve 12-D reduction in <i>C. botulinum</i> spores. Spoilage still possible.
	$F_{10/121.1} \geq 6.0$ min	Shelf life: To be safe, and to prevent spoilage
	$F_{10/121.1} = 0.5 - 1.5$ min	For cured meat products: Curing controls initial spore load & subsequent spore growth
Sielaff (1996)	$F_{10/121.1} = 3 - 8$ min	Shelf life in moderate climate: 4 months to 4 years if storage temperature $T \leq 25$ °C. Microbial stable; 2-log reduction in thermophilic spoilage spores, which germinate at $T > 35$ °C
	$F_{10/121.1} = 16 - 20$ min	Shelf life in tropical climate: 1 year at storage temperature $T \geq 35-40$ °C. Food is microbial stable. 6-log reduction in thermophilic spoilage spores, which germinate at $T > 35$ °C
	$F_{10/121.1} = 0.65 - 0.85$ min	For cured meat products: 1 year storage life, if the product is cured and given this minimal treatment.
Heinz and Hautzinger (2007)	$F_{10/121.1} > 2.52$ min	Food Safety: To eliminate most heat resistant spores for pathogens (<i>C. botulinum</i>)
	$F_{10/121.1} > 2.58$ min	Shelf life: To eliminate most heat resistant spores for pathogens (<i>C. botulinum</i>)
	$F_{10/121.1} = 4 - 5.5$ min	For moderate climate: Shelf life of up to 4 years at temperature < 25 °C can be achieved
	$F_{10/121.1} = 12 - 15$ min	For tropical climate: In tropical countries, F-value have to be increase for safe storage of the finished products under storage temperatures up to 40°C

^a Minimal Lethality expressed as F_{zT} where z is the z-value concerned and T is the reference temperature of heating

Table 3. Minimum lethality requirements for acid and acidified foods

pH-range	Minimal Lethality ^a	Reference
3.1 – 3.2	$F_{10/195} = 0.1 \text{ min}$	Odlaug and Pflug (1997)
3.3 – 3.5	$F_{10/195} = 1.0 \text{ min}$	Nelson and Tressler (1980)
3.5 – 4.0	$F_{10/195} = 16 - 23 \text{ min}$	Barrett <i>et al.</i> (2004)
4.0 – 4.3	$F_{15/200} = F_{8.3/93.3} = 5 \text{ min}$	National Canners Association Research Laboratories (1968)
4.3 – 4.4	$F_{15/200} = 23 \text{ min}$	Larousse and Brown (1996)
	$F_{15/212} = 10 \text{ min}$	
4.5 – 4.6	$F_{15/200} = F_{8.3/93.3} = 10 \text{ min}$	National Canners Association Research Laboratories (1968)
	$F_{15/212} = 10 \text{ min}$	Montville and Sapers (1981)
	$F_{18/230} = 1.6 \text{ min}$	
	$F_{8.3/93.3} = 10 \text{ min}$	National Canners Association Research Laboratories (1968)

^a Minimal Lethality expressed as $F_{Z/T}$ where z is the z-value concerned and T is the reference temperature of heating

Table 4. Thermocouple-based temperature measurement techniques.

Liquid ^a	Agitating retorts ^b
<ul style="list-style-type: none"> • Rigid needle type thermocouples are generally used. • Some researchers have also used wireless sensors. • More than one thermocouple or thermocouples of different lengths (Ex: In No.2 cans, 3 thermocouples of 43 mm, 32 mm and 21 mm length and fixed respectively at 20mm, 56mm and 85mm from the bottom) can be used for temperature uniformity studies. • Procedures for attaching a needle type thermocouple are detailed in literature. 	<ul style="list-style-type: none"> • A slip-ring assembly is used to EOE and axial mode of agitation as there is a possibility of entangling of the thermocouple wires. • The slip-ring detaches the static side of the thermocouple wires outside the retort from the thermocouples inside the rotating cage, thus preventing the twisting of the wires during cage rotation. • A second slip-ring is attached during biaxial agitation to allow the twisting and turning of the thermocouple wires in opposing directions during the biaxial rotation of cage
Fixed particles ^c	Moving particles ^b
<ul style="list-style-type: none"> • A rigid thermocouple is attached to the particle-center or the slowest point of heating. • In agitation processing, this does not simulate the real free motion due to agitation. • Results may have inaccuracies due to restricted motion of the particles. 	<ul style="list-style-type: none"> • A flexible thin wire thermocouple attached to the particle-center is used. • To collect the particle-center temperature, for spheres, a hole slightly greater than its radius is drilled and the thermocouple tip (commonly of a duplex Type T copper/constantan thermocouple made of 20, 22 or 24-gauge flexible wires) is fixed at center with epoxy glue.

^a Lesley (1987); Meng (2006); Pflug (2003); Ramaswamy and Abbatemarco (1996); Sablani (1996); Sablani and Ramaswamy (1996); Wang *et al.* (2006);

^b Dwivedi and Ramaswamy (2010a, 2010b); Meng (2006); Meng and Ramaswamy (2005, 2007a, 2007b); Sablani (1996); Sablani and Ramaswamy (1995, 1999).

^c Deniston *et al.* (1987); Fernandez *et al.* (1988); Hassan (1984); Lenz and Lund (1978);.

Table 5. Methodologies for measuring U and h_{fp} using direct temperature measurement techniques.

From particle-center temperature measurements	
<p>Analytical method ^a</p> <ul style="list-style-type: none"> Dimensionless temperatures at a specific point of particle is calculated using the following equation and h_{fp} is calculated using iteration: $\frac{T - T_i}{T_f - T_i} = \sum_{n=1}^{\infty} C_n [\cos(\gamma_n r/a)]$ <p>where, $C_n = (4\sin\gamma_n \exp(-\gamma_n^2 \alpha T/a^2))/(\gamma_n + \sin(2\gamma_n))$,</p> <p>$\gamma_n$ = positive roots of $\gamma_n \tan \gamma_n = (h_{fp})/k_p$</p> <p>Semi-Analytical Method ^c</p> <ul style="list-style-type: none"> Some researchers used a semi-analytical approach for fitting the particle temperature and a semi-numerical for fitting the fluid temperature solution using Duhamel's theorem and a numerical Runge-Kutta scheme. 	<p>Numerical method ^b</p> <ul style="list-style-type: none"> These methods are based on finite element and difference approximations of Equations (2-7). U and h_{fp} are evaluated using numerical Runge Kutta scheme. They work well in the range $0.02 < Bi < 200$ and $\tau > 0.2$. Numerical solutions are satisfactory & less restrictive. <p>Low biot-number scenario ^d</p> <ul style="list-style-type: none"> When $Bi < 0.1$ (aluminum or metal particles), a lumped approach is used and U is evaluated as: $U = \frac{2.303 m_f c_{pf}}{f_h A_c}$ <ul style="list-style-type: none"> h_{fp} is evaluated from slope of the line: $\ln(T_f - T) = \ln(T_f - T_i) - \frac{h_{fp} A_p}{m_p c_{pp}} t$ <ul style="list-style-type: none"> Simple method but calculated h_{fp} doesn't represent the true h_{fp} value as it assumes a linear temperature profile.
From particle-surface temperature measurements ^e	
<ul style="list-style-type: none"> It is based on equation (2-3) from which h_{fp} can be calculated as: $h_{fp} = \frac{m_p c_{pp} (T_f - T_i)}{A_p \int_0^t (T_f - T_{ps}) dt}$ <ul style="list-style-type: none"> For spheres in rotating cans, the following simplification is available: $h_{fp} = \frac{a \rho_p c_{pp} (T_p - T_i)}{3 \int_0^t (T_f - T_{ps}) dt}$ <ul style="list-style-type: none"> Only average final and initial particle temperatures and fluid and particle-surface temperature required. 	
For non-Newtonian and viscous fluids ^f	
<ul style="list-style-type: none"> Changes in viscosity make traditional modeling difficult. U_{ap} and h_{ap} (apparent fluid-to-particle heat transfer coefficient) are calculated using a lumped model. Equations (2, 4-7) remain the same but Equation (3) changes as: 	

$$m_p \cdot c_{pp} \cdot \frac{dT_p}{dt} = h_{ap} A_p (T_R - T_{ps})$$

- While h_{fp} is based on the transient temperature difference between the canned fluid and the particle, h_{ap} is based on that between the retort medium and the particle.

^a Chang and Toledo (1989), Chandarana and Gavin (1989, 1990); Stoforos and Merson (1991).

^b Deniston et al. (1987); Dwivedi and Ramaswamy (2010a); Fernandez et al. (1988); Hassan (1984); Lenz and Lund (1978); Meng (2006); Meng and Ramaswamy (2005, 2007a, 2007b); Sablani and Ramaswamy (1995, 1999);

^c Stoforos *et al.* (1997).

^d Hassan (1984); Stoforos and Merson (1991); Fernandez *et al.* (1988); Tattiyakul *et al.* (2002); Sastry *et al.* (1985).

^e Chau and Snyder (1988); Rodriguez and Teixeira (1988); Sastry et al. (1989b); Alhamdan et al. (1990); Stoforos and Reid (1992).

^f Meng and Ramaswamy (2005, 2007b).

Table 6. Heat transfer studies on thermal processing of liquid-particulates in non-agitated systems.

Reference	Liquid	Particle	h_{fp} (W/m ² K)
Chau and Snyder (1988)	Water	Shrimp	1276
		Aluminum	1073
Chang and Toledo (1989, 1990)	Water	Potato Cubes	239-303
	Sucrose solution		146
	Sucrose solution (0 m/s)	Carrot Cubes	600-1533
	Sucrose solution (1.58 m/s)		356-735
Chandarana and Gavin (1989), Chandarana <i>et al.</i> (1990)	Starch	Silicone Rubber Cube	8.1-86.5
	Water		65.7-107.1
Alhamdan <i>et al.</i> (1990)	Water	Mushroom shaped aluminum	652-850
Awuah <i>et al.</i> (1995)	CMC	Potato Cylindrical	80 -450
		Carrot Cylindrical	100 -550
Cariño-Sarabia and Vélez-Ruiz (2013)	Water	-	57-248
	CMC	-	28-169
	-	Mushroom	28-132
	-	Tomato Puree	30-183
	-	Potato	31-248

Table 7. Heat transfer studies on thermal processing of liquid-particulates in agitated systems.

References	Liquid	Particle	U (W/m ² K)	h _{fp} (W/m ² K)	Mode
Lekwauwa and Hayakawa (1986)	Water	Potato (spheroidal)	113-1704	60-2613	Rotary retort
Deniston et al (1987)	Water	Potato Spherical	95-2300	127-200	Rotary retort
	Starch solution	Potato Cube	--	99	
	Water	Mushroom	--	850	
Lenz and Lund (1978)	Water	Lead Particle (Single)	732-970	522-1811	Rotary retort
		Lead Particles (Multiple)	210-330	341-1550	
	Water & Sucrose	Lead Particle (Single)	562-709	539-1175	
		Lead Particle (Multiple)	170-244	431-1198	
Hassan (1984)	Water	Potato Spheres	--	98-165	Rotary retort
		Teflon Spheres	--	32-108	
		Aluminium Spheres	--	139-735	
Stoforos and Merson (1991)	Water	Aluminium (spheres)	142-201	128.7-1296	Rotary retort
		Teflon(sphere)	83-192	410-2071	
		Potato	--	233	
Sablani and Ramaswamy (1995, 1996)	Water	Propylene Sphere	480- 880	65-120	EOE
	Oil	Nylon spheres cylinders & cubes	130 -200	170 - 1130	EOE
	Water		520 - 800	190 - 1430	
Meng and Ramaswamy (2005, 2007a, 2007b)	CMC	Nylon sphere	U _{ap} : 36-79	h _{ap} : 210- 380	EOE
	Glycerin	Nylon & Teflon (Single)	U _{ap} : 266- 347	h _{ap} : 50-65	EOE
		Nylon & Teflon (Multiple)	U _{ap} : 45-61	h _{ap} : 163- 276	

Dwivedi and Ramaswamy (2010a)	Glycerin	Propylene, Nylon & Teflon spheres	210-350	315-585	Fixed axial
			345-580	495-945	Biaxial
Hassan <i>et al.</i> (2012)	CMC	Nylon Sphere	265-455	335-630	Fixed axial
			430-720	605-1010	Biaxial

Appendix A*A.1 Nomenclature:*

α	thermal diffusivity, m^2/s
a	radius of sphere, m
A	total external surface area, m^2
Bi	Biot number
c_p	specific heat capacity, $\text{J/kg } ^\circ\text{C}$
c_{pf}	specific heat capacity of liquid, $\text{J/kg } ^\circ\text{C}$
c_{pp}	specific heat capacity of particle, $\text{J/kg } ^\circ\text{C}$
D	negative reciprocal of slope of the thermal destruction curve (D-value), min
f_h	heating rate index, min
F	lethality, min
h_{fp}	fluid to particle heat transfer coefficient, $\text{W/m}^2\text{ } ^\circ\text{C}$
k	thermal conductivity, $\text{W/m } ^\circ\text{C}$
m	mass, kg
N	population density
ρ	density, kgm^{-3}

r	radius, m
t	time, s
τ	Fourier number
T	temperature, °C
U	overall heat transfer coefficient, W/m ² °C
z	temperature sensitivity (z-value), °C

Subscripts

c	can
o	reference
i	initial condition
f	fluid
p	particle
ps	particle surface
R	retort
s	surface
m	model
TTI	time-temperature integrator