ABSTRACT

BATMAZ, EDIZ. Enhancement of Heat Transfer Rate and Minimization of Process Time by ResonantAcoustic® Mixing During In-Container Sterilization of Foods. (Under the direction of K. P. Sandeep.)

ResonantAcoustic® Mixing (RAM) is a novel in-container mixing technology that has found many applications in industries where it is desired to mix highly viscous materials in sealed containers. The food industry might also be able to benefit from RAM during thermal processing of foods in containers. Inducing mixing in food containers during thermal processing has been applied successfully in the last two centuries to provide high quality food products to the consumer. RAM is believed to have the potential to extend the viscosity range of products that can be properly mixed in containers during thermal processing. Therefore, one objective of this study was to quantify the mixing achieved in a lab scale RAM unit (LabRAM) for products of different apparent viscosities. To do so, fluid to particle heat transfer coefficients (h_{fp}) were measured between a copper ball and banana puree samples at different viscosities. Results showed that the h_{fp} values ranged between 337 and 5554 W/m²·K. In general, these values are much higher than those reported in literature for other mixing technologies.

Another objective of the current study was to use planar laser induced fluorescence (PLIF) to observe the progression of mixing in a LabRAM and quantify the mixing time to attain a well mixed state when two miscible liquid streams were placed in a container and mixed at different mixing intensities. Uniformity of mixing increased with increasing mixing intensity. The results showed that the time to attain a well mixed product ranged from 3.0 s to 35.6 s throughout the product and process parameters studied.
Another objective of this study was to compare the thermal process times for three different processing technologies, namely still retort, Shaka® process, and RAM integrated thermal process. Samples of different apparent viscosities were used to cover a wide spectrum of viscosities that are encountered in the food industry. Based on the results obtained, while the effectiveness of the Shaka® process varied (thermal process times were reduced by 16% to 91% with respect to the still retort process) within the viscosity range studied, RAM integrated thermal process was capable of shortening the thermal process times significantly (ranged between 83% and 94%) for all tested samples.

These results suggest that RAM integrated thermal processing could be pursued further from a commercial point of view, to minimize thermal process times and improve the quality of products for existing products, and to design new products of higher apparent viscosities which were not possible to retort successfully previously due to prohibitively long thermal process times.
Enhancement of Heat Transfer Rate and Minimization of Process Time by ResonantAcoustic® Mixing During In-Container Sterilization of Foods

by

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A dissertation submitted to the Graduate Faculty of North Carolina State University in partial fulfillment of the requirements for the degree of Doctor of Philosophy

Food Science

Raleigh, North Carolina

2013

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DEDICATION

To my family and my parents.
BIOGRAPHY

Ediz Batmaz was born in Adana, Turkey on June 19, 1977. He went to elementary, middle, and high school in his home town, Mersin, then moved to Ankara, Turkey in 1995 to pursue a Bachelor of Science degree in the Department of Food Engineering at Hacettepe University. Upon graduation in 2000, he moved to the United States of America and earned his Master of Science degree in Food Science in 2003 from North Carolina State University. In 2004, he started working towards a Doctor of Philosophy degree in the same department and continued his academic program as a part-time endeavor when he began working in the food industry in 2005. He is still employed in the industry as an R&D Process Engineer. He is married to Selda Ulukoylu Batmaz, and is a father of two beautiful children, Ali Nafiz and Azra.
ACKNOWLEDGMENTS

This work would not have been possible without the support and prayers of my parents. I have always felt their support in my heart regardless of the distance between us. I hope completion of this work helps with them being pleased with me.

The encouragement and support of my wife, Selda, has also been instrumental for completion of this work. She kept motivating me when I was not sure of how to make progress with this work. She also had to put up with not having me around when I had to conduct all the experiments in this work. Thank you for your patience and your understanding.

My children also supported this work indirectly, as they have let me concentrate and study at home, while they were keeping themselves busy without having their father around to play with.

I think it was a blessing from God to have Dr. K. P. Sandeep as my advisor. It took me 10 years to complete this work and I am not sure how many advisors would be as patient with their graduate students as he was with me. Despite the long delays in this work, I do not remember a time when I felt that he was not hopeful of completion of this work. I am grateful for it. I don’t think I would have had the courage to continue my program with a full time job and a family, if I had not felt his support.
I would like to thank each and every one of my committee members, Drs. Brian Farkas, Josip Simunovic, Saad Khan, and Curt Emenhiser. Their valuable input and guidance helped me shape this dissertation.

Finally, I would like to thank my colleagues at work for supporting this work in many different ways. Without their full support, I would have not been able to complete my research and write my dissertation while working full time. Thank you very much.
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INTRODUCTION

History of thermal processing of foods for preservation purposes dates back to 1804, when Nicolas Appert, a French confectioner, sealed glass jars and discovered a way of keeping foods such as mutton, vegetables, and milk from spoiling (Holland 2008). At that time, heat treatment was carried out at atmospheric pressure by heating the jars or cans in boiling water for approximately six hours. Chevalier and Appert invented the pressure cooker in 1851, which made it possible to heat the jars or cans to temperatures greater than 100 °C.

In the second half of the 19th century, the range of canned foods available to urban populations greatly increased, as canners competed with one another by offering food products with highly decorated printed labels and at lower prices. Though the temperature of the product was raised above 100 °C to shorten the cook time, canneries had to hold the product in the retort long enough due to a lag in raising the temperature of the cold spot in the container. This lag time was due to conduction heating within the container, which created a temperature gradient in the product being thermally processed. Efforts to reduce this lag time by inducing mixing in the container dates back to the late 19th century. In 1898, F.W. Smith patented an apparatus for treating or sterilizing canned food. In his invention, Smith used reciprocating movement of a crate filled with food containers to induce agitation in the containers. Since then, efforts to advance the process of retorting continuously increased, as the demand for canned food products increased globally. To demonstrate the magnitude of the canned food business globally, it should be noted that, in 2013, worldwide demand for
canned food was estimated at US$77.2 billion, with a forecast of US$79.6 billion in 2014 (Research and Markets 2013).

Today, several retort designs exist that make use of agitation to shorten the thermal process time with respect to the time required in still (non-agitating) retorts for proper sterilization. Despite the high number of unique retort designs that involve agitation, the type of agitating motion applied to food products in retorts can be categorized into three main groups: end-over-end agitation, axial rotation, and reciprocating agitation.

In an end-over-end agitation retort, containers are loaded into a retort crate in vertical orientation, and the crate is rotated around a central horizontal axis during the thermal process (Tucker 2004). In this mode of agitation, during the rotation of the crate, the headspace bubble moves through the food mixture, causing a continuous stirring action. With increasing speed of rotation, the time taken for the contents of the can to reach the required temperature was found to decrease. There is, however, a limiting speed, beyond which no further reduction was observed (Holdsworth and Simpson 2008). A widely used end-over-end retort in the industry is the Rotomat, developed by Hermann Stock of Neumünster, Germany, in 1952. Retorts of this type are usually operated at about 6-8 rpm during heating and cooling. However, a faster speed of ~15 rpm is used for the come-up period (Holdsworth and Simpson 2008).

In an axial rotation retort, containers rotate in a horizontal plane. There are batch and continuous retorts that make use of axial rotation. In some axial rotation retorts, the cans roll
in addition to the rotation around the periphery of the retort, while in others, cans are secured in a reel, which does not allow the cans to roll. FMC Sterilmatic (JBT FoodTech, Madera, CA) is a continuous reel-and-spiral cooker, which uses pressure valves to allow the passage of cans into and out of the various pressurized chambers. Within a chamber, the reel-and-spiral arrangement moves the cans along the internal periphery of a cylindrical vessel, allowing them to rotate while keeping them apart. The container rotation in this type of retorts can be divided into three phases, consisting of fixed reel (carried by reel over the top), sliding rotation and free rotation. The fixed reel and sliding rotation do not provide significant product agitation. However, during the free rotation phase, the containers roll freely along the inside bottom wall of the shell and the main product agitation occurs during this phase (Weddig 2007). The rotation speed in this type of retort is usually between 10 and 42 rpm. The Orbitort (JBT FoodTech, Madera, CA), which is a batch type axial rotation retort, does not allow cans to roll during processing. The conventional speed of rotation for an Orbitort is 35 rpm (Holdsworth and Simpson 2008). There is yet another type of retort (ACB Hydrolock, Stock America, Inc. Raleigh, NC) that provides agitation with axial rotation, where the containers are loaded into carriers, and the carriers are pulled by chains to progress the containers through the retort, while a stationary belt underneath the carriers causes rotation of carriers and containers. Contrary to the FMC Sterilmatic, the rolling action of containers in an ACB Hydrolock unit is continuous.

As the name suggests, reciprocating agitation retorts move a crate holding the food containers back and forth continuously to induce movement of the headspace in the
container, and hence, agitation of the product during thermal processing. Shaka® retort from Zinetec (Hereford, England) that uses reciprocating agitation claims better flavor, texture, and color, and process times are said to be cut by a factor of 10 (for products that are suitable, such as béchamel sauce) compared to traditional static retorts (Holland 2008). This technology has been heavily promoted in the last 10 years. However, commercial applications began in 2011, with a total of 9 units installed since then (PRWEB 2013).

The amount of literature on reciprocating retorts is few, compared to the open literature available for end-over-end and axial-rotation retorts. Many studies since the mid-1950s have dealt with understanding the influence of operating parameters such as product viscosity, rotation speed, amount of headspace, container size and shape, and retort temperature on the amount of mixing induced in containers during either an end-over-end or axial rotation in a retort (Holdsworth and Simpson 2008). Most of these parameters can be controlled during production of a specific food product. However, this might mean that a food manufacturer has to limit its operation to a certain range of product viscosities, beyond which the agitation method is found to be ineffective. In some studies conducted with Shaka® retorts (Walden 2010), it was shown that processing times could be reduced significantly for selected products compared to still or rotary agitation processes. However, no data was presented for the range of viscosities covered in the study. While it is reported that Shaka® retorts perform much better than retorts that use traditional agitation methods for inducing mixing during retorting, information on limitations of the Shaka® retort as it relates to the product viscosity, is not available. Like in any other non-contact agitation method, it is
suspected that there is a viscosity threshold, beyond which the Shaka® retort will lose a considerable amount of its effectiveness in inducing mixing.

There is yet another in-container mixing technology, ResonantAcoustic® Mixing (RAM), and to the best of my knowledge, it has not been used during thermal processing of in-container products. RAM induces mixing in in-container products by vibrating the container at a frequency in the acoustic range (~60 Hz). The intensity of mixing in a RAM unit can be controlled by controlling the amplitude of the vibration. While no data exists today for its effectiveness in inducing mixing for containers undergoing a thermal process, due to the high acceleration that can be achieved in a RAM unit (up to 981 m/s²), it is believed that this technology can extend the viscosity range of products that can benefit from in-container mixing during thermal processing. In light of these findings, the goals of this study were set to:

- Quantify the fluid to particle heat transfer coefficients achieved in a RAM unit at different mixing intensities and for products of different apparent viscosities
- Visualize the mixing process and quantify the time to mix two visually different constituents by using planar laser induced fluorescence (PLIF)
- Conduct thermal processing trials using three different setups to compare simulated versions of still retort, Shaka® retort, and RAM integrated retort
for the total thermal processing time and appearance of post-process samples for products of different apparent viscosities
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Effect of Mixing Intensity and Fluid Viscosity on the Fluid-to-Particle Heat Transfer Coefficient in a Sealed Container Exposed to ResonantAcoustic® Mixing

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Abstract

The effectiveness of a novel non-contact mixing technology known as ResonantAcoustic® Mixing (RAM) for agitation of in-container products was tested, with the ultimate goal of using the technology in retort applications. The tests were setup to determine the convective heat transfer coefficient between a particle and a liquid medium ($h_{fp}$) for liquids of various viscosities and at various mixing intensities. 41.5 Brix banana puree and water were mixed at different percentages to obtain products of different apparent viscosities (from 176 to 2670 cP at 25°C and at a shear rate of 100 s$^{-1}$). The mixing intensities ranged from 20% to 100%. A copper ball with a diameter of 0.0127 m was used as the particle and $h_{fp}$ values were calculated using the lumped capacitance method. The calculated $h_{fp}$ values ranged between 337 and 5554 W/m$^2$ K. With its effectiveness in inducing agitation for a wide range of viscosities and the ease in controlling the mixing intensity, the technology can be a promising alternative to currently used technologies in agitating retorts.
**Introduction**

Most foods need to be thermally processed to render them safe for consumption and/or to preserve their nutritional and organoleptic properties. To establish a thermal process, the thermal processing specialist should have data not only on the heat resistance of pathogenic and spoilage microorganisms, but also on the rate of heat transfer into the product during well controlled processing conditions. The heat resistance of microorganisms has been studied extensively and is readily available in the literature. The rate of heat transfer into a specific product, however, depends on many factors that can be grouped into two categories: product related factors and process related factors. Both categories have been the topic of many research studies. With advancements in thermal process technologies, researchers identified the need to investigate the effect of certain processing parameters unique to these process technologies to aid the industry in establishing an adequate thermal process. The most prominent examples of these processing technologies are the end-over-end (EOE), and the axial rotation retorts. A number of studies have been published evaluating the factors that influence the overall heat transfer coefficient (U) and/or the fluid-to-particle heat transfer coefficient (h_{fp}) for canned liquid foods exposed to either of these modes of agitation. These factors include process related factors such as rotational speed, retort temperature, rotational radius, system geometry, headspace volume, off-center axis of rotation; and product related factors such as product viscosity, particle density, particle size, and particle loading. These studies cover both the EOE mode (Naveh and Kopelman 1980; Anantheswaran and Rao 1985a,b; Lekwauwa and Hayakawa 1986; Rao and Anantheswaran 1988; Stoforos and Merson 1992; Sablani and Ramaswamy 1995, 1996, 1997, 1998; Sablani *et al.* 1997; Meng
and Ramaswamy 2007a,b,c) and the axial mode of agitation (Lenz and Lund 1978; Hassan 1984; Deniston et al. 1987; Fernandez et al. 1988; Stoforos and Merson 1990, 1992; Dwivedi and Ramaswamy 2010; Ramaswamy and Dwivedi 2011).

In the last decade, a linear reciprocating movement technology has gained traction in the food industry due to its increased effectiveness in inducing mixing in the product when compared to aforementioned agitation technologies. This technology is known as Shaka® and it is claimed that this process can reduce the processing times by up to 10 or 20 folds compared to a rotary retort process or a still retort process, respectively (Angalet 2011). This reduction in processing times is due to enhanced heat transfer coefficients, which is achieved by the acceleration that is applied to the product basket placed into the retort shell. Similar to the EOE and axial rotation technologies, for the Shaka® technology, the product movement in the retort is dependent on the amount of headspace in the container. As the containers are subjected to reciprocating movement, the relative movement of the headspace and the product induces agitation. The two most important processing parameters unique to the Shaka® process are the revolutions per minute (rpm) of the crank reciprocating the retort basket and the magnitude of the linear movement (stroke length) of the basket. Several combinations of these parameters have been tested. Rpm values of 40, 60, 80, 100, 120, 140, 160, 180, 200, 220 and 230 rpm; stroke lengths of 25, 50, 75, 150, 225 and 300 mm have been used (Walden 2010). To simplify comparison of different operating conditions, it has been suggested that the combination of rpm and stroke length be expressed as a single value by calculating the maximum acceleration for a combination of stroke length and frequency of
reciprocation (Walden 2010). In general, an acceleration of 2-3g (acceleration due to gravity) was found to be sufficient to have a significant impact on the thermal processing time for the tested samples. Relating this back to the rpm and stroke length, 180 rpm and 150 mm peak to peak stroke length have been accepted (Higgins 2012) as a good starting point for testing new products with the Shaka® technology.

All the technologies cited above have certain limitations. The first and foremost of these limitations is the product viscosity. While these technologies can produce highly effective results within a certain viscosity range when compared to a still retort process, beyond a certain viscosity, a rapid decrease in effectiveness can be observed (Walden 2010; Batmaz 2013). Recent advancements in in-container (non-contact) mixing technologies can be utilized to broaden the range of product viscosities that can benefit from significant reductions in thermal process times compared to still retort processing. The most prominent one of these advancements is known as ‘ResonantAcoustic® Mixing’ (RAM), which is similar to the Shaka® technology in the sense that both technologies utilize reciprocating movement to induce in-container mixing. Beyond this similarity, there are major differences between the two technologies. While the Shaka® process utilizes a nominal stroke length of 6” (150 mm) and a nominal operating frequency of 3 Hz (180 rpm), the corresponding parameters for the RAM technology are nominally in the range of 0.5” (0.0127 m) and 60 Hz, respectively. This mode of operation would result in a maximum acceleration of 100g for the RAM technology, compared to the 2-3g observed in the Shaka® process. To achieve such a high acceleration in a reciprocating movement, the RAM technology utilizes a special
spring-mass arrangement that is brought to resonance with the product for optimum performance. While operating at the resonance frequency, the amplitude of the reciprocating movement (stroke length equivalent for the Shaka® technology) can be easily controlled by controlling the amount of force exerted onto the system. Details of the RAM technology have been outlined by Howe et al. (2007). To the best of our knowledge, this technology has not yet been utilized in the food industry. However, due to its highly controllable nature of operation and effectiveness in a wide range of product viscosities, the technology deserves attention for its potential benefits when integrated into a food thermal process operation such as retorting. The objective of this study was to quantify the $h_{eq}$ values in a RAM unit for products of different apparent viscosities exposed to different mixing intensities.
Materials and Methods

Sample Preparation:

Banana puree that is concentrated to 41.5 Brix (commercially available product) through an evaporation process and subsequently frozen to extend its shelf life was used to prepare all the samples. First, the banana puree was thawed out in a refrigerator. Then, this concentrated banana puree was mixed with water at different ratios to obtain samples of different apparent viscosities. A whisk was used to mix the water and the puree until a uniform mixture was obtained. All samples were left at room temperature to equilibrate their temperature prior to conducting the experiments. 6 samples were made by utilizing the above procedure. Table 1 shows the % of banana puree in these samples along with their apparent viscosities at 24 °C and a shear rate of 100 s⁻¹. Since all the samples were non-Newtonian, a graphical representation of shear rate dependence of all 6 samples is shown in Figure 1. The high R² values in the fitted lines confirm a shear thinning power-law behavior for all samples. Considering the commercially available viscous food products that are subject to either a pasteurization or a sterilization process (concentrated milk, soups, sauces, meals), the six samples used for this study should cover most, if not all, of these products of interest in terms of their viscosity range.

Copper Ball Preparation:

For all experiments, a single, spherical (0.0127 m diameter) copper ball (Bal-tec, Los Angeles, CA) was used to measure the h₀ between the sample fluid and the copper ball. The reported thermal conductivity, density and specific heat of the copper ball were 386 W/m K,
8960 kg/m³, and 385.1 J/kg K, respectively. Copper was chosen as the material of the ball due to its high thermal conductivity, which is essential for the validity of the lumped capacitance method (N_Bi < 0.1). This method was used to determine h_fp and is described later in this section. The copper ball was drilled along its radius to roughly the center. Special care was not taken to drill exactly to the center, due to the assumption that there is no temperature gradient in the ball volume. A thermocouple wire (model number 5TC-TT-K-30-72, Omega Engineering, Stamford, CT) was inserted into the copper ball through the drilled hole and then the hole was sealed at the surface of the ball using a two part Loctite epoxy gel (Item # 1405602, Henkel Corporation, Rocky Hill, CT), that is stable at high temperatures to ensure that fluid did not leak into the center of the copper ball. Before each experiment, the copper ball was dipped into a beaker that was filled with boiling water on a hot plate. This procedure ensured creation of a temperature difference between the copper ball and the sample, which was at room temperature, prior to starting an experiment.

Lid Preparation:

For all the experiments, the sample was filled into a 236.5 ml (8 oz) capacity transparent plastic container with an internal diameter of 63 mm and a height of 86 mm (Taral Plastics, Union City, CA). 6 mm headspace was left at the top of the container. This headspace was within the range of what has been tested previously for Shaka® retorts (Walden 2010). A special lid (Part # 100826, Resodyn Acoustic Mixers, Inc., Butte, MT) was used to seal the container. Instead of using a screw-on type of sealing mechanism, the lid sealed the container by compression at the top of the container throughout its perimeter by
use of a circular rubber gasket. What provided the compression between the lid and the container was a standard hold-down fixture that was also supplied by Resodyn Acoustic Mixers, Inc. (Part # 100312). On the top surface of the lid, two compression fittings were placed to be able to insert two thermocouple wires into the container.

Setup for Data Acquisition:

One thermocouple wire that was sealed in the copper ball was used to monitor and record the temperature of the ball throughout an experiment. The second thermocouple wire was used to monitor and record the temperature of the sample throughout an experiment. As mentioned in the previous section, both thermocouples were first inserted and sealed through the lid by use of compression fittings. Both of these thermocouples were connected to a data acquisition system (Model # NI 9213, National Instruments, Austin, TX) that was connected to a PC through a USB port. The data acquisition system was configured to record temperature data from both thermocouples at a frequency of 5 data points per second.

Mixer Setup and Operation:

The RAM unit that was used for the experiments was a LabRAM (Resodyn Acoustic Mixers, Inc., Butte, MT), which is a bench-top scale mixing unit (Figure 2). At the top of the LabRAM, a payload plate is used to mount a hold-down fixture. The function of the hold-down fixture is to secure the sample container during operation of the machine. Once the sample is loaded into the mixer, a cover needs to be put around the payload plate to engage a pin, the status of which is constantly monitored by the mixer’s PLC. If the pin is not engaged,
the mixer cannot be operated. To eliminate the interference of the thermocouple wires with
the lid, a small port was opened at the side of the LabRAM to exit the wires from the mixer.

The control of the LabRAM was accomplished through a PC software called Ramware
(Resodyn Acoustic Mixers, Inc., Butte, MT). Through this software interface, the mixer can
be operated in either auto or manual mode. In auto mode, the mixer automatically dials into
the resonance frequency of the system and keeps track of the resonance frequency throughout
the run time. In the manual mode, the user can determine the frequency of operation.

However, since a fundamental principal of this technology is to bring the system to its
resonance frequency during operation, all experiments were run in auto mode. Another
parameter that can be adjusted is the ‘% intensity’. This parameter controls the amount of
reciprocating force exerted onto the system. The % intensity has a direct influence on the
amplitude of the reciprocating movement, and hence, on the maximum acceleration that the
payload plate and payload itself is exposed to. In general terms, 10% intensity corresponds
roughly to 10g maximum acceleration, and 100% intensity corresponds to approximately
100g acceleration.

Sequence of Operation:

Once a sample was prepared and the copper ball was heated to a temperature of ~100
°C, the lid was put on the sample container, the container was loaded into the hold-down
fixture, the mixer lid was put around the payload plate and the mixing was started through
the Ramware software. The time spent between removing the copper ball from the beaker
filled and starting the experiment was critical; as during this stage, the copper ball started
cooling off, and the temperature difference between the sample and the copper ball decreased. Therefore, special care was taken to minimize this time period to the extent possible (ranged between 15 s and 22 s). Once the mixing was started, the temperature change in the copper ball was observed through the PC monitor and the mixing was stopped after the temperature of the copper ball reached a plateau value.

Calculation of the fluid-to-particle heat transfer coefficient (h$_{fp}$):

Based on the lumped capacitance method, all h$_{fp}$ calculations were performed using the following equation:

$$\ln \left[ \frac{(T_c - T_{\infty})}{(T_{c(t)} - T_{\infty})} \right] = -\frac{h_{fp} A}{m c_p}$$

(1)

The left side of this equation can be determined by plotting $\ln(T_c - T_{\infty})$ with respect to time, fitting the data points to a linear line and computing the slope of this line. Then, h$_{fp}$ can be calculated using the following equation:

$$h_{fp} = -\frac{m c_p}{A} \times \text{slope}$$

(2)

This method makes use of multiple data points rather than utilizing only two data points (at the beginning and end of the experiment) and using equation 2 directly to calculate h$_{fp}$. 
In addition to calculating the $h_{fp}$ values due to forced convection (mixing), $h_{fp}$ values due to natural (free) convection were also determined. Instead of running separate experiments where essentially no forced mixing was taking place (0% mixing intensity), data from the mixing experiments were used, since, for all experiments, there was an elapsed time between insertion of the copper ball into the sample (putting the lid on the sample container) and start of the mixing process. A similar procedure as outlined above was carried out to compute $h_{fp}$ due to natural convection. In addition to calculating the $h_{fp}$ due to natural convection using the experimental data, a theoretical calculation was also carried out for comparison purposes. The following correlation was used for computing the theoretical Nusselt number:

$$\text{Nu} = 2 + \frac{0.589 \text{Ra}^{1/4}}{\left[1 + \left(\frac{0.469}{\text{Pr}}\right)^{9/16}\right]^{4/9}}$$

(3)

where,

$$\text{Nu} = \frac{h_{fp} D}{k}$$

(4)

$$\text{Gr} = \frac{D^3 \rho^2 g \Delta T \beta}{\mu^2}$$

(5)

$$\text{Pr} = \frac{\mu c_p}{k}$$

(6)

and,

$$\text{Ra} = \text{Gr} \text{Pr}$$

(7)
Equation 3 is valid for $\text{Ra} \leq 10^{11}$ and $\text{Pr} \geq 0.7$ and $\beta$ is assumed to be equal to $1/T_{\text{fl}}$, where $T_{\text{fl}}$ is the film temperature (the average of the bulk fluid temperature and the ball surface temperature). For all theoretical calculations, the average temperature of the banana puree was used for the bulk fluid temperature, and the average between the starting temperature and final temperature of the copper ball during the period of natural convection was used for ball surface temperature. Table 2 shows the values of the fluid properties used in the theoretical calculations.

Design of Experiments:

The mixer utilized for the experiments can be controlled to any mixing intensity between 1% and 100%. During preliminary trials, it was noticed that even for a 40% banana puree sample, a mixing intensity below 10% did not induce a significant amount of mixing. Therefore, the minimum mixing intensity used was 10%. For samples with higher apparent viscosities, the minimum mixing intensity was gradually raised until an appreciable amount of mixing was observed. At the high end, the mixing intensities ranged from 60% to 100%. As an example, for a 40% banana puree sample, at 60% mixing intensity, the mixing observed was fairly intense and chaotic. Moreover, as the amount of mixing increased, the amount of data points for analysis decreased, which can have an effect on the robustness of the data. Therefore, for this particular sample, it was determined not to test higher mixing intensities. The same arguments were considered when determining the mixing intensity range for all samples. Table 3 summarizes the intensities tested for each sample.
Results and Discussion

During the experiments, the temperature of the bulk fluid and the center of the copper ball were monitored as a function of time. A sample data for 40% banana puree mixed at a mixing intensity of 50% is shown in Figure 3. A similar graph was plotted for every experiment. The maximum acceleration that the sample was exposed to has also been plotted in the same graph to help with understanding the source of changes in the curvature of the graph of copper ball temperature as a function of time. For this experiment, the effective mixing time (the time from the start of the mixing to the point where the temperature of the copper ball reached a plateau) was 5 s. Considering all the experiments in the study, the effective mixing time ranged from 5 s to 48 s. In Figure 4, a plot of ln(T_c − T_∞) with respect to time can be seen corresponding to the experimental data in Figure 3. A high R^2 value for the slope confirms a steady h_{fp} value for the time span analyzed. To be able to compute the h_{fp} value for this experiment, the constant term in equation 2 also needed to be known. This constant term was calculated to be 7303 J/m^2 K based on the material properties of the copper ball. Multiplying (-7303) by (-0.7059), the h_{fp} value for this experiment was calculated to be 5155 W/m^2 K. A similar procedure was followed to calculate the h_{fp} value for each experiment.

In Figures 5-10, the change in the h_{fp} with respect to the change in the mixing intensity can be seen for each sample. In general, a linear relationship exists between these two parameters within the range of mixing intensities plotted. Furthermore, it should be noted that the obtained h_{fp} values were high compared to what was found in literature. The
highest $h_{fp}$ value found to be reported in open literature for an in-container mixing application is by Garrote et al. (2006), where the highest $h_{fp}$ value obtained was 1950 W/m$^2$ K. In the current study, $h_{fp}$ values ranged between 337 and 5554 W/m$^2$ K. Even for the most viscous sample, it was possible to obtain an $h_{fp}$ value above 2500 W/m$^2$ K when exposed to a mixing intensity of 100%. This can be attributed to the capability of RAM mixer to induce effective and intense mixing in a wide range of viscosities.

Figure 11 shows a graph similar to Figure 4, but for the part of the experiment before mixing was started. The time span covers the time from dipping the copper ball into the sample to the time when mixing was started. It is evident from this graph that the slope of this curve is decreasing for the first part of the graph. This can be interpreted as a slightly decreasing $h_{fp}$ value over time, since, based on equation 2, $h_{fp}$ is the product of the slope of this curve and a constant. This is due to the fact that dipping the copper ball into the sample causes some amount of forced convection, the effect of which disappears when the ball comes to rest within the sample. Therefore, to eliminate the effect of dipping the ball into the sample on calculation of $h_{fp}$ due to natural convection, only last 5 seconds of time-temperature data prior to starting mixing was used.

Figure 12 shows the theoretical and experimental values of $h_{fp}$ due to natural convection for all the tested samples. The experimental $h_{fp}$ values shown in this figure are the mean of all data points calculated for each and every experiment for a specific sample. The error bars depict the standard deviation for these mean $h_{fp}$ values. In general, the
experimental values are higher than the calculated theoretical values. However, for 40%, 50%, 60% and 90% banana puree samples, experimental and theoretical values are close to each other, suggesting that the experimental values obtained are representative of the actual $h_{fp}$ values due to natural convection for these specific samples. Moreover, the trend of the experimental $h_{fp}$ values due to natural convection for 40%, 50%, 60%, and 90% samples follow the expected decreasing trend with increasing banana puree concentration, which is also the case in the theoretically calculated values. For 70% and 80% banana puree samples, not only the experimentally calculated $h_{fp}$ values are significantly different from the theoretically calculated values, they also break the decreasing trend with increasing banana puree concentration. This can be attributed to the fact that for these two samples, the copper ball may not have come to rest within the timeframe where data is used to calculate $h_{fp}$ due to natural convection. For these two concentrations of banana puree (70% and 80%), the sample is viscous enough to slow down the copper ball once it is inserted into the sample compared to the less viscous samples (50%, 50%, and 60% banana puree samples), but it is not viscous enough, like the 90% banana puree sample, to completely stop it once dipped into the sample. Therefore, it is believed that the $h_{fp}$ values in Figure 12 for 70% and 80% banana puree samples are not representing a puree natural convection scenario.

The $h_{fp}$ values in Figure 12 can also be incorporated into Figures 5-10 as values for the case of 0% mixing intensity. In such a case, the linearity of the trendlines in these graphs would not hold anymore. Therefore, the linear trendlines in Figures 5-10 should only be used for the data range presented in these graphs. In other words, extrapolation should not be
attempted. The loss of linearity between the $h_{fp}$ for the 0% mixing intensity and the first reported value in Figures 5-10 can be interpreted as ineffective mixing at that range of mixing intensities.

In most research studies dealing with determination of fluid to particle heat transfer coefficients, it is assumed that the particle is completely in contact with a liquid that is exchanging heat with the particle. For this study, it should be noted that a headspace of ~6 mm was left in the container for all the experiments. During the mixing process, the air in this headspace broke down into small air bubbles (visually observed), which were homogenously distributed within the container until mixing was stopped. Therefore, it should be noted that the surface of the copper ball was covered with a liquid and air throughout the mixing process. However, since the volume of air in the container was much smaller than the volume of liquid (banana puree), the presence of air within the surface of the copper ball was neglected for all calculations.

In addition to presenting the influence of mixing intensity and sample viscosity on the fluid to particle heat transfer coefficients, one might attempt to fit the data into an empirical correlation. In most cases where forced convection is present, a convective heat transfer coefficient correlation would include a term that is a function of Reynolds number. Since Reynolds number is a function of both the velocity and the apparent viscosity of the fluid that is subjected to forced convection, these two parameters should easily be determined as a prerequisite to data fitting. In our case, determining the velocity and apparent viscosity of the
samples at a given mixing intensity is not trivial. Therefore, curve fitting was carried out using brix of banana puree sample and the mixing intensity as the independent variables.

Four different types of equations were considered for curve fitting. These were, a first order polynomial, a second order polynomial, an equation in the form of $h_{fp} = c_1 + c_2 \text{ (Brix)}^{c_3} + c_4 \text{ (Mixing Intensity)}^{c_5}$, and $h_{fp} = c_1 \text{ (Brix)}^{c_2} \text{ (Mixing Intensity)}^{c_3}$. Considering all the experimental data points for forced convection, the best fit was obtained with a second order polynomial. The equation was of the form $h_{fp} = c_1 + c_2 \text{ (Brix)} + c_3 \text{ (Mixing Intensity)} + c_4 \text{ (Brix)}^2 + c_5 \text{ (Brix)(Mixing Intensity)} + c_6 \text{ (Mixing Intensity)}^2$. The constants obtained by data fitting were, 5334, -409.1, 126.1, 6.77, -2.63, and 0.0482 for $c_1$, $c_2$, $c_3$, $c_4$, $c_5$, and $c_6$, respectively. The $R^2$ value for fitting the experimental data into this second order polynomial equation was 0.97.
Conclusions

Fluid to particle heat transfer coefficients were obtained for non-Newtonian food samples of different apparent viscosities subjected to ResonantAcoustic® Mixing at different mixing intensities. A lumped capacitance method was used to determine $h_{fp}$. Within the range of analysis, increasing the mixing intensity increased the $h_{fp}$ values in a linear fashion. The $h_{fp}$ values obtained were relatively high compared to the $h_{fp}$ values presented in relevant literature (337-5554 W/m$^2$ K for the current study versus 480-1950 W/m$^2$ K reported by Garrote et al. (2006)). This is due to the high effectiveness of the mixing technology utilized in these experiments. It may be possible to couple this technology with in-container thermal process applications, such as retorting, to create a viable alternative to end-over-end, axial or Shaka® retort systems, which are all currently employed in the food industry. Despite the promising results obtained in this study, due to the novelty of the ResonantAcoustic® Mixing technology, more studies should be carried out to assess the suitability of this technology for use in thermal processing of foods.
Symbols

A  surface area (m$^2$)  
c  constant  
c$_p$  Specific heat (J/kg K)  
D  Diameter (m)  
g  acceleration due to gravity (9.81 m/s$^2$)  
Gr  Grashof number  
h  heat transfer coefficient (W/m$^2$K)  
k  Thermal conductivity (W/m K)  
m  mass (kg)  
Nu  Nusselt number  
Pr  Prandtl number  
Ra  Rayleigh number  
t  time (s)  
T  temperature (°C)  
U  overall heat transfer coefficient (W/m$^2$K)  

Greek symbols

β  Fluid thermal expansion coefficient (K$^{-1}$)  
Δ  Delta  
μ  Fluid viscosity (Pa s)
\( \rho \)  
Fluid density (kg/m\(^3\))

**Subscripts**

c  copper ball
fp  fluid-to-particle
f  final
fi  film
i  initial
\( \infty \)  bulk fluid

**Abbreviations**

EOE  End-over-end
PC  Personal computer
PLC  Programmable logic controller
RAM  ResonantAcoustic\(^{\circledR}\) Mixing
rpm  Revolutions per minute
References


to canned particulate Newtonian fluids during free bi-axial rotary processing. *Food and Bioprocess Technology*, 4(1), 61-78.


Table 1.
% Banana puree and apparent viscosity of samples.

<table>
<thead>
<tr>
<th>% of 41.5 Brix banana puree</th>
<th>40%</th>
<th>50%</th>
<th>60%</th>
<th>70%</th>
<th>80%</th>
<th>90%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apparent viscosity (cP) at room temperature and a shear rate of 100 s⁻¹</td>
<td>278</td>
<td>507</td>
<td>770</td>
<td>1215</td>
<td>1548</td>
<td>2668</td>
</tr>
</tbody>
</table>

Table 2.
Properties of banana puree samples used for theoretical natural convection calculations.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thermal conductivity (W/m K)</th>
<th>Specific heat (J/kg K)</th>
<th>Density (kg/m³)</th>
<th>Apparent viscosity (Pa s) @ 0.1 s⁻¹ shear rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>40% Banana Puree</td>
<td>0.551</td>
<td>3662</td>
<td>1287</td>
<td>47</td>
</tr>
<tr>
<td>50% Banana Puree</td>
<td>0.540</td>
<td>3531</td>
<td>1359</td>
<td>277</td>
</tr>
<tr>
<td>60% Banana Puree</td>
<td>0.528</td>
<td>3399</td>
<td>1431</td>
<td>553</td>
</tr>
<tr>
<td>70% Banana Puree</td>
<td>0.517</td>
<td>3268</td>
<td>1504</td>
<td>695</td>
</tr>
<tr>
<td>80% Banana Puree</td>
<td>0.506</td>
<td>3137</td>
<td>1576</td>
<td>730</td>
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<tr>
<td>90% Banana Puree</td>
<td>0.494</td>
<td>3006</td>
<td>1649</td>
<td>960</td>
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</tbody>
</table>

Table 3.
Design of experiments.

<table>
<thead>
<tr>
<th>% 41.5 Brix banana puree in the sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixing intensity</td>
</tr>
<tr>
<td>------------------</td>
</tr>
<tr>
<td>10%</td>
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<tr>
<td>20%</td>
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<td>30%</td>
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<td>40%</td>
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<td>70%</td>
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<td>80%</td>
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<tr>
<td>90%</td>
</tr>
<tr>
<td>100%</td>
</tr>
</tbody>
</table>
Figure 1. Shear rate vs. apparent viscosity graph for all 6 samples.
Figure 2. Setup of the LabRAM.
Figure 3. Time-temperature history of 50% banana puree sample and copper ball exposed to mixing at an intensity of 40%.
Figure 4. Determination of slope to be used in Equation 2 to calculate $h_f$ due to forced convection.
Figure 5. Effect of mixing intensity on $h_{fp}$ (40% banana puree).
Figure 6. Effect of mixing intensity on $h_{fp}$ (50% banana puree).
Figure 7. Effect of mixing intensity on $h_p$ (60% banana puree).
Figure 8. Effect of mixing intensity on $h_{fp}$ (70% banana puree).
Figure 9. Effect of mixing intensity on $h_{fp}$ (80% banana puree).

$y = 42.306x - 783.83$

$R^2 = 0.9721$
Figure 10. Effect of mixing intensity on $h_f$ (90% banana puree).
Figure 11. Slope of $\ln(T_c - T_\infty)$ prior to starting mixing.
Figure 12. Theoretical and experimental $h_{fp}$ values due to natural convection.
Visualization and Quantification of Mixing in a ResonantAcoustic® Mixer

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Abstract

In-container mixing is used in the food industry to enhance heat transfer during retorting of in-container products. Planar laser induced fluorescence was used to visualize and quantify mixing by a novel in-container mixing technology, ResonantAcoustic® mixing (RAM). A lab scale RAM unit was used to mix 5 ml of Rhodamine 6G into 420 ml of 0.5%, 1.0% and 1.5% CMC solutions at various mixing intensities (50%, 75% and 100%). Results showed that the time to attain a well mixed state ranged from 3.0 s to 35.6 s for different product and process conditions. Mixing intensity was found to be the most influential factor in determining the mixing time. Variability in the mixing time decreased with increasing mixing intensity. RAM was found to be a technology that can be beneficial in reducing the overall sterilization times during in-container sterilization applications for a range of product viscosities.
**Introduction**

Mixing is an essential process utilized in many different manufacturing industries, including food and pharmaceutical industries. The purpose of mixing is different for different applications. It can be for the creation of a homogenous product such as a juice blend or to speed up the process of heat transfer to the center of the vessel in a steam jacketed vessel.

The need to predict and control results of mixing processes has prompted researchers to visualize and quantify mixing processes of various scales. Many techniques have been developed to carry out such studies, such as laser Doppler, phase Doppler and hot-wire anemometry; particle image velocimetry (PIV); planar laser induced fluorescence (PLIF); and near infrared (NIR) spectroscopy. Anemometry techniques are used for single point measurements, while PIV, PLIF, and NIR spectroscopy techniques are capable of whole-field measurements (Cullen 2009). The PLIF technique, which is used in this study, relies on using a camera to image local fluorescence caused by excitation of a sample seeded with a fluorescent organic substance. Details of the fluorescence theory, dye, laser, camera and sheet optics selection, and image processing methods for PLIF analysis in aqueous flows have been described by Crimaldi (2008). The PLIF technique has been successfully utilized for visualization of flow in stirred vessels (Gaskey *et al.* 1990; Houcine *et al.* 1996; Guillard *et al.* 2000a,b,c; Fall *et al.* 2001; Arratia and Muzzio 2004; Szalai *et al.* 2004; Arratia *et al.* 2006; Zadghaffari *et al.* 2009; Hu *et al.* 2010, 2012; Zhang *et al.* 2013), in jet nozzles (van Cruyningen *et al.* 1990; Guillard *et al.* 1998; Hu *et al.* 2009) and in static mixers (Jaffer and Wood 1998; Wadley and Dawson 2005; Lehwald *et al.* 2010).
Aforementioned mixing methods are all relevant to the food industry. However, in-container mixing is yet another method utilized extensively in the food industry, especially while containers filled with product and subsequently sealed are going through a retorting process. The purpose of in-container mixing during this thermal process step is to shorten the thermal process time, by inducing forced convection within the container. To date, in-container mixing has been successfully commercialized in a few different ways: axial rotation, end-over-end rotation, and reciprocating movement. Axial rotation refers to axial rotation of cans in a horizontal plane and also in a spiral motion along the perimeter of a horizontal retort. FMC Sterilmatic is an example of such a retort. End-over-end (EOE) rotation refers to rotation of food containers about a central horizontal axis. Stock Rotomat retorts make use of EOE agitation to increase the heat transfer rate within the container. Reciprocating movement refers to linear motion of containers in a horizontal plane. The amplitude and frequency of this linear motion can be adjusted based on the product characteristics. Shaka® is a retort system that makes use of reciprocating movement. The frequency used in Shaka® retorts usually range between 140 and 180 cycles per minute, while the stroke length is nominally 15 cm, which can also be adjusted based on the product properties (Higgins 2012). The intensity of agitation in a Shaka® system can be expressed as a single value by calculating the maximum acceleration for a combination of stroke length and frequency of reciprocating motion (Walden 2010). This maximum acceleration can then be expressed as multiples of \( g \) (acceleration due to gravity – 9.81 m/s\(^2\)). Based on the operating range quoted above, Shaka® systems would generate maximum acceleration values in the range of 2-3g, although tests have been performed at maximum accelerations.
approaching 4.5g (Walden 2010). In one study (Walden 2010), the Shaka® retort was compared to a rotary EOE and a static retort system with results presented as heat up and cool down times for a 5% bentonite solution undergoing thermal sterilization in a 73x110 mm can with 8 mm headspace. When heat up and cool down times were combined, the total processing time (excluding hold time) were reported as 132 min, 25 min, and 10 min, for a static retort, EOE rotary retort, and Shaka® retort, respectively. The dramatic reduction in the total processing times for the EOE and Shaka® retort systems is a result of effective mixing in the container during the thermal process. Although employing a certain mixing technology might have a high impact on overall thermal processing times, prior to employing such a technology, it is essential to study and understand the effect of certain product and process parameters on the effectiveness of mixing. For example, in a patent granted to Clifcorn et al. (1950), the inventors studied the most optimum processing parameters for EOE mixing and have concluded that the frequency of rotation should be set to result in a centrifugal force one half to one and one half times the weight of the product to achieve maximum mixing. Since then, many studies have been published that have dealt with testing effectiveness of different mixing parameters on different products and in different containers. Holdsworth and Simpson (2008) have compiled most of these studies with the respective processing conditions and presented them in a table format.

In the current study, the effectiveness of a novel in-container mixing technology, ResonantAcoustic® Mixing (RAM), was studied. Similar to the Shaka® technology, RAM utilizes reciprocating movement to induce mixing in a container. However, compared to the
Shaka® technology, RAM can be considered a short amplitude and high frequency reciprocating movement in the vertical direction. RAM mixers operate at a nominal frequency of 60 Hz, which can vary slightly depending on the resonance frequency of the whole system, including the product being mixed. The amplitude of the movement is 0.5” (1.27 cm) or less. Such a reciprocating movement would result in a maximum acceleration in the order of 100g. Compared to the Shaka® process, this corresponds to a maximum acceleration 30-50 times higher. RAM technology achieves such high acceleration values by utilizing a special spring-mass arrangement that is brought to resonance for optimum performance. The system continuously tracks the resonance frequency by use of an accelerometer mounted on the payload plate and automatically dials into that frequency. As the system operates at its resonance frequency, the force, and hence maximum acceleration exerted onto the system can be adjusted to control the mixing intensity. Details of the RAM technology have been outlined by Howe et al. (2007).

As an effective in-container mixing technology, RAM has a high potential for use in in-container sterilization processes. Unfortunately, to the best of our knowledge, there is no open literature available to aid the understanding of mixing taking place in a RAM unit, other than what is shared by Resodyne Acoustic Mixers, Inc. on their website (www.resodynmixers.com). Therefore, the objective of this study was to visualize mixing patterns in a RAM unit by utilizing PLIF and to quantify the time it takes to achieve a mixed state for products of different viscosities and at different processing conditions.
Materials and Methods

Fluorescent Dye Preparation:

The fluorescent dye used was prepared from powder form of Rhodamine 6G with ~95% dye content (Sigma-Aldrich, St. Louis, MO). To prepare desired solutions of Rhodamine 6G, ~0.5 g of dye powder was dissolved in 1000 ml de-ionized water to prepare an initial concentration of 500,000 µg/l. This solution was then used to prepare all of the concentrations of interest (20,000, 10,000, 5,000, 2,500, 1,500, 1,000, and 500 µg/l). The choice of the range was based on concentrations of dye expected to be observed throughout the mixing process and is explained further in the following section.

Sample Preparation:

Carboxymethylcellulose (Hercules Chemical Co, Ltd., Guangdong, China) was used as a thickening agent to obtain samples of different apparent viscosities. 0.5%, 1.0%, and 1.5% (w/w) carboxymethylcellulose (CMC) solutions in water were prepared to cover the viscosity range of the food products typically dealt with in the food industry. Apparent viscosity information for 0.5% CMC solution as a function of shear rate (5 - 125 s⁻¹) was obtained using a Haake RheoStress 600 with a PZ36 sensor. For 1.0% and 1.5% CMC solutions, apparent viscosity for the same shear rate range was measured using a Haake Viscotester 550 with a FL10 sensor.

473 ml capacity polypropylene containers with an internal diameter of 83 mm and a height of 92 mm (Taral Plastics, Union City, CA) were used to contain the samples for both
calibration and mixing experiments. In PLIF studies, calibration experiments are conducted for two main purposes:

- to ensure a linear response of grey scale value to concentration
- to account for variability due to experimental setup in pixel to pixel grey scale values by obtaining a unique calibration curve for each pixel in the region of interest (ROI)

For calibration experiments, a uniform mixture of CMC solution and dye solution was prepared by adding 5 ml of dye solution to 495 ml of CMC solution in a 1000 ml beaker and mixing it manually with a spoon. 425 g of this mixture was then transferred into a container and sealed with a lid in order to measure fluorescence of the sample. A total of 24 samples were prepared for calibration experiments, by uniformly mixing each CMC solution (0.5, 1.0, and 1.5%) with each dye solution (0, 500, 1,000, 1,500, 2,500, 5,000, 10,000, 20,000 µg/l). Mixing the dye solution into the CMC solution essentially diluted the dye concentration by 100 folds, resulting in final dye concentrations of 0, 5, 10, 15, 25, 50, 100, and 200 µg/l. The intent was to test final dye concentrations up to 500 µg/l, which was the dye concentration added to CMC solutions for the mixing experiments. However, a significant amount of saturation was observed beyond 200 µg/l, which is why the calibration curves were capped to a maximum of 200 µg/l.

For the mixing experiments, 210 g of CMC solution was filled into a container, followed by 5 ml of 500 µg/l dye solution, which was then followed by 210 g of CMC solution. With the amount of CMC and dye solutions added to the container, the headspace at
the top of the container was measured to be ~6 mm. The intent of adding the dye solution between the two parts of the CMC solution was to create a concentrated layer of dye solution in the middle of the container prior to starting an experiment. During the last step (addition of 210 g of CMC solution), some unintentional mixing occurred between the dye and CMC solution. The extent of this mixing was more for the samples prepared with 0.5% CMC solution compared to the ones prepared with 1.0% or 1.5% CMC solution. The design of experiments was setup as a full factorial of three CMC concentrations (0.5, 1.0, and 1.5% CMC solutions) and three mixing intensities (50%, 75%, and 100%). All mixing experiments were run in triplicates, resulting in a total of 27 experiments.

Experimental Setup:

A depiction of the experimental setup can be seen in Figure 2. A Nd:YAG laser (RayPower 450) operating in the continuous wave (CW) mode was used for the experiments. Classified as a Class 3B laser source, the maximum power of the laser at 532 nm is listed as 450 mW. The laser operated in a near TEM00 mode, which resulted in a laser beam output with a Gaussian cross-section. Three light sheet optics (Light sheet base module, angle modules 1 and 4) were placed in front of the laser beam to convert the beam into a sheet. The laser source and all related components were obtained from Dantec Dynamics Inc. (Holtsville, NY). For all experiments, the laser was operated at the maximum power setting (450 mW). The laser light sheet path was set such that it crossed the sample container through its side, following a path to the center of the cross section and out through the other
side. This setup provided maximum planar coverage within the container. The thickness of the light sheet was measured as 1 mm at the center of the light sheet.

A lab scale RAM unit (LabRAM) from Resodyn Acoustic Mixers, Inc. (Butte, MT) was used as the mixing source (Figure 2). A payload plate at the top of the unit was used to mount a hold-down fixture. The function of the hold-down fixture was to secure the sample container during operation of the mixer. Once a sample was loaded into the mixer, a cast acrylic, transparent cover was placed around the sample and the payload plate to engage a pin. The status of the pin was constantly monitored by the mixer’s PLC to ensure that the cover was present while the mixer was in operation. If the pin was not engaged, the mixer could not be operated. This meant that the laser sheet had to pass through the LabRAM cover and the container wall prior to reaching the sample. The control of the LabRAM was accomplished by use of an OEM software interface. Through this software interface, the mixer could be operated in either auto or manual mode. In auto mode, the mixer automatically dials into the resonance frequency of the system and keeps track of this resonance frequency throughout the run time. In the manual mode, the user can input the frequency of operation. However, since a fundamental principle of this technology is to bring the system to its resonance frequency during operation, all mixing experiments were conducted in auto mode. Another parameter used for control of the mixer was the ‘% intensity’. This parameter controls the amount of reciprocating force exerted onto the system. The ‘% intensity’ has a direct influence on the amplitude of the reciprocating movement, and hence, on the maximum acceleration that the payload plate and payload itself (container with
its constituents) is exposed to. 10% intensity corresponds to approximately 10g maximum acceleration, and 100% intensity corresponds to approximately 100g maximum acceleration.

A high-speed camera (Model # TM-6740GE, JAI Inc., San Jose, CA) was placed at a 90° angle from the line connecting the sample container to the laser source. The camera was connected to a laptop for configuration and operation. A filter (Item # 9080C0541, Dantec Dynamics Inc., Holtsville, NY) was placed in front of the camera to suppress scattered laser light and transmit the light at the fluorescent wavelength. For calibration studies, a single image was captured for each sample. For mixing studies, the frequency of images captured was adjusted based on the mixing intensity used for a particular mixing experiment. If the mixing intensity was 50%, then the camera was set to capture 30 frames per second (fps), and if the mixing intensity was 75% or 100%, the camera was set to capture 60 fps. All images were captured as 8-bit grey scale (values ranging from 0 to 255), with a size of 640x480 pixels.

Image Processing and Quantification of Mixedness:

Captured images were analyzed using the Matlab Image Processing Toolbox and Computer Vision System Toolbox (MathWorks, Inc., Natick, MA). During the mixing experiments, the location of the container with reference to a fixed 640x480 background frame changed due to the vertical reciprocating movement of the container. Therefore, for analyzing the images, a program was written to capture the inside of the container as the
ROI, crop this ROI, and compute the standard deviation and mean from each captured image. Standard deviation and mean values were then used for quantifying the mixedness over time.

Wadley and Dawson (2005) used ‘Coefficient of Variation’ (CoV) as a measure of mixedness, where CoV is defined as the ratio of standard deviation to mean. In an equation form:

$$\text{CoV} = \frac{\sigma}{\bar{x}}$$

(8)

where, $\sigma$ is the standard deviation and $\bar{x}$ is the mean. Though not named as CoV, other researchers (Masuda et al. 2008; Carroll and Hidrovo 2012) also used the same equation for quantification of mixedness. Wadley and Dawson (2005) noted that a CoV value of 0.05 is considered well mixed, and a value of 0.01 very well mixed. These values assume a CoV value of 0.00 for the case of uniform solutions. Obtaining a CoV of 0.00 for uniform solutions is nearly impossible without implementing any post-process correction, as some amount of difference in the grey scale intensity will be observed from pixel to pixel due to many factors, including a non-uniform laser sheet thickness and absorption of light along its pathway while it crosses the sample container. Therefore, correction methods have been developed to eliminate this variability (Crimaldi 2008). The correction methods assume a fixed relative location between the sample container (ROI), camera, and the laser sheet. In this study, the camera and the laser sheet were stationary, but the ROI moved up and down with a peak amplitude of ~0.0127 m and at a frequency of ~60 Hz. Therefore, it is not
feasible to apply such a correction and the reader should keep this in mind when interpreting
the final CoV values obtained in the mixing experiments.
Results and Discussion

CMC Solutions:

Apparent viscosity values of CMC solutions as a function of shear rate and the fitted power-law equations can be seen in Figure 1. Balasubramaniam (1993) has observed a shear thinning behavior for CMC solutions in water. In this study, a similar shear thinning behavior was observed for all three CMC solutions.

Calibration Experiments:

Images captured for the calibration experiments were cropped to extract the ROIs, and the mean grey scale values from the ROIs were plotted against the corresponding dye concentration in the samples. Figures 3-5 show these calibration curves for the three CMC concentrations. For all three curves, a linear relationship within the range of analysis (0-200 µg/l) is evident from the high $R^2$ values. However, a slight deviation from linearity can be seen beyond a dye concentration of 100 µg/l. It is typical to observe a deviation from linearity at high dye concentrations due to saturation. If the goal of this study was to determine absolute concentrations of the dye at different segments of the ROI, even slight deviations from linearity would have been significant. However, the goal of this study was to visualize the mixing process in a RAM unit and to quantify the time it takes to get to a mixed state in the container. Therefore, while it is important to ensure that variations in the dye concentration result in variations in the grey scale value, it is not critical to have a perfectly linear relationship between these two variables. Comparing the three calibration graphs, it can be seen that the slope of the line decreases with increasing CMC concentration. This can
be attributed to the less translucent nature of the solution with increase in the amount of CMC, which causes more attenuation in the emitted fluorescent light.

It is important to note the CoV values from the calibration images, as these are images taken from uniform mixtures and these CoV values reflect the inherent variability in the pixel to pixel grey scale values. For low dye concentrations (0-15 µg/l) CoV was low (between 0.07 and 0.08). For higher dye concentrations (25-200 µg/l), higher CoV values were obtained (0.09-0.14), possibly due to saturation in the images. CoV values for the dye concentration range of 0-10 µg/l is particularly important because for the mixing experiments, when a state of complete mixedness is achieved, the dye concentration in the solution should be 5.9 µg/l (mixing 5 ml of 500 µg/l dye solution into 420 ml of CMC solution). Therefore, a CoV of 0.08 should indicate a very well mixed solution when analyzing the images from the mixing experiments.

Mixing Experiments:

Change in CoV as a function of mixing time for 0.5%, 1.0%, and 1.5% CMC solutions are depicted in Figures 6-8, respectively. Analyzing the trends in the graphs, a decline in CoV was observed over time down to a certain CoV value (CoV\textsubscript{m}), beyond which the CoV value stayed approximately constant. The minor oscillations in CoV can be attributed to the headspace in the containers (~6 mm). During the mixing process, the air in the headspace broke down into small air bubbles and got dispersed throughout the container. The amount of air bubbles that aligned with the laser sheet at any given time affected the
CoV value for the image captured at that instance. The elapsed time from the start of the mixing process to an instance where the container contents were well mixed ($t_m$) was quantified for each mixing experiment. The start of the mix time was determined by analyzing the images and detecting the first image where movement was observed in the sample. The first instance where the contents of the container were well mixed was detected by analyzing the time versus CoV graph for an experiment. The first step was to determine the $CoV_m$ value by analyzing the graph. Dividing $CoV_m$ by 0.95 and analyzing the CoV trend to capture when the CoV value of $CoV_m/0.95$ was achieved, was determined to be the first instance where the contents of the container were well mixed.

In Figures 6-8, it can be observed that for most experiments, the CoV value reached a plateau at around 0.10, though plateau values as high as 0.17 can be seen. The increase in the plateau values can be attributed to dispersion of air bubbles and their appearance with respect to the sample. In Figure 9, air bubbles can be seen in a sample during the mixing process. Clearly, the darker shades of the air bubbles would tend to increase the CoV value.

Table 1 shows the $t_m$ values for each mixing experiment. Mixing times ranged between 3.0 s and 35.6 s considering all the experiments conducted. These results were analyzed with a two factor with replication ANOVA (analysis of variance) test with the confidence level set to 95% ($\alpha = 0.05$). P-values obtained from the ANOVA analysis for % CMC in solution, mixing intensity, and interaction of these two factors were $1.5 \times 10^{-3}$, $4.8 \times 10^{-11}$, and $4.3 \times 10^{-2}$, respectively. These results can be interpreted as all three of these
factors having an impact on $t_m$ with a confidence level of 95%. The extremely low P-value for the mixing intensity can be interpreted as the mixing intensity being the most influential factor for $t_m$. For a similar scale setup, Hu et al. (2010) experimented with different concentrations of glycerol solutions (5-80% v/v) and used an impeller type agitator (100 rpm) in a lab scale tank to mix in a fluorescent dye into a glycerol solution. They determined the mixing time to range between 17.3 s and 89.2 s, where higher mix times were required for increased concentrations of glycerol.

Figure 10 depicts the mean of the triplicate repetitions for each mixing intensity and CMC concentration combination. This graph indicates the significant influence of mixing intensity on $t_m$. It also shows that as the mixing intensity increases, the influence of CMC concentration on $t_m$ goes down. In addition to these observations, it should be noted that at 50% mixing intensity, a higher $t_m$ value was observed for 0.5% CMC solution than for 1.0% CMC solution. This is not intuitive as longer mix times were expected with increase in CMC concentration.

Table 2 shows the standard deviation in the $t_m$ values for the three replicates. For experiments where 50% mixing intensity was used, an increase in standard deviation was observed with an increase in CMC concentration. This indicates that the results become more and more unpredictable as the mixing intensity decreases and product apparent viscosity increases. At 100% mixing intensity, the standard deviation values for all three CMC concentrations are relatively low ($<1.0$), with no obvious trend. This can be interpreted as
more uniform and predictable mixing taking place at high mixing intensities, regardless of the CMC concentration (within the concentration range studied). To expand on this observation, it can be concluded that at high mixing intensities, predictable and reproducible results can be obtained for products of a wide range of viscosities.

Figure 11 represents images during mixing of a 1.5% CMC solution with Rhodamine 6G at a mixing intensity of 50%. The first snapshot was taken before mixing was initiated. As it can be seen from the subsequent images, mixing occurred only at the top of the container initially, until a stream of liquid progressed further downwards to the bottom of the container. From that point on, mixing continued at the top and bottom of the container, however, some stagnant zones were observed at the sides of the container towards the middle section vertically. This stagnant volume got smaller and smaller over time and eventually a uniform mixture was obtained. To compare the progression of mixing in a low mixing intensity experiment to a high mixing intensity experiment, images from mixing of a 1.5% CMC solution with Rhodamine 6G at a mixing intensity of 100% is shown in Figure 12. Progression of mixing was found to be similar to that observed for the same viscosity sample mixed at 50% mixing intensity (Figure 11).
Conclusions

Planar laser induced fluorescence was used to visualize and quantify mixing in a RenonantAcoustic® mixer. The time to get to a well mixed state ranged from 3.0 s to 35.6 s. At low mixing intensities (~25% mixing intensity), mixing was observed only at the top of the container volume. For mixing intensities studied in this work (50%, 75% and 100%), progression of mixing appeared to be the same. Mixing started at the top of the container and channeled to the bottom of the container while creating zones in the middle, where stagnation was observed. Eventually, the mixing regimes at the top and bottom of the container progressed further inwards and eliminated the stagnant regimes. Less variability was observed in the time to achieve a uniform mixture as the mixing intensity was increased. The RAM technology shows promise for use in thermal sterilization of foodstuff in containers, although more research is needed to understand its limitations and to set operational boundaries to ensure reproducible results.
References


Table 1. Mixing time (in seconds) for all mixing experiments.

<table>
<thead>
<tr>
<th>% CMC in sample</th>
<th>0.5%</th>
<th>1.0%</th>
<th>1.5%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50%</td>
<td>75%</td>
<td>100%</td>
</tr>
<tr>
<td>0.5%</td>
<td>22.1</td>
<td>5.2</td>
<td>3.7</td>
</tr>
<tr>
<td>1.0%</td>
<td>21.9</td>
<td>6.7</td>
<td>3.0</td>
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<td>1.5%</td>
<td>25.4</td>
<td>6.3</td>
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</tr>
<tr>
<td>1.0%</td>
<td>13.9</td>
<td>3.9</td>
<td>4.7</td>
</tr>
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<td>1.0%</td>
<td>19.6</td>
<td>7.3</td>
<td>4.0</td>
</tr>
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<td>4.3</td>
</tr>
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<td>1.0%</td>
<td>31.2</td>
<td>7.7</td>
<td>6.0</td>
</tr>
<tr>
<td>1.5%</td>
<td>35.6</td>
<td>11.4</td>
<td>5.8</td>
</tr>
</tbody>
</table>

Table 2. Standard deviation (in seconds) of three replicate mixing times.

<table>
<thead>
<tr>
<th>% CMC in sample</th>
<th>0.5%</th>
<th>1.0%</th>
<th>1.5%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50%</td>
<td>75%</td>
<td>100%</td>
</tr>
<tr>
<td>0.5%</td>
<td>2.0</td>
<td>0.8</td>
<td>0.4</td>
</tr>
<tr>
<td>1.0%</td>
<td>3.3</td>
<td>2.1</td>
<td>0.4</td>
</tr>
<tr>
<td>1.5%</td>
<td>6.9</td>
<td>2.1</td>
<td>0.9</td>
</tr>
</tbody>
</table>
Figure 1. Shear rate vs. apparent viscosity for CMC samples.
Figure 2. Setup for the PLIF experiments.
Figure 3. Calibration curve for dye in 0.5% CMC solution.

\[ y = 1.0339x + 13.253 \]

\[ R^2 = 0.9942 \]
Figure 4. Calibration curve for dye in 1.0% CMC solution.

\[ y = 0.9989x + 12.562 \]

\[ R^2 = 0.9923 \]
Figure 5. Calibration curve for dye in 1.5% CMC solution.

\[ y = 0.8635x + 10.839 \]
\[ R^2 = 0.9968 \]
Figure 6. CoV vs. time for 0.5% CMC samples mixed at 3 different mixing intensities.
Figure 7. CoV vs. time for 1.0% CMC samples mixed at 3 different mixing intensities.
Figure 8. CoV vs. time for 1.5% CMC samples mixed at 3 different mixing intensities.
Figure 9. Air bubbles dispersed in a sample.
Figure 10. Mixing intensity vs. average mixing time for the samples.
Figure 11. Progression of mixing over time for a 1.5% CMC solution mixed at a mixing intensity of 50%.
Figure 12. Progression of mixing over time for a 1.5% CMC solution mixed at a mixing intensity of 100%.
MANUSCRIPT III

Integration of ResonantAcoustic® Mixing into Thermal Processing of Foods: A Comparison Study Against Other In-container Sterilization Technologies

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Abstract

A comparison study was carried out for thermal processing times in three different modes of retort operation. Still retort simulation, processing with a motion simulating that in a Shaka® retort, and ResonantAcoustic® Mixing (RAM) integrated processing were used to process banana puree samples at different brix values (ranging from 16.6 to 41.5). Thermal processing times ranged from 76.6 minutes to 90.7 minutes for still retort simulation, from 6.8 minutes to 76.5 minutes for processing with a motion simulating that in a Shaka® retort, and from 4.3 minutes to 15.4 minutes for RAM integrated processing. While a significant amount of reduction in thermal process times (up to 91%) was observed for processing with motion simulating that in a Shaka® retort for banana puree samples with a brix value between 16.6 and 29.1, this effectiveness dropped sharply (down to 16%) for samples with higher brix values (33.2 – 41.5). For the RAM integrated processing, the applied mixing technology was found to be effective in reducing the thermal process times throughout the tested range of product viscosities.
Introduction

Sterilization of food products is an important step in delivering safe food that is shelf-stable at ambient storage conditions. Most sterilization processes use high temperature to destroy pathogenic microorganisms. Due to possible detrimental effects of high temperature sterilization, food manufacturers and academicians have developed new processing technologies to minimize the heat load into a product while still rendering it safe for consumption. One of the most significant of these developments was the agitation of containers during a thermal process. Agitation of containers induces forced convection in the product, which facilitates faster heating and cooling of the product. Two of the most widely used agitation methods in the food industry are end-over-end (EOE) rotation, where containers are loaded vertically into a retort crate and the crate rotates around a central horizontal axis; and axial rotation, where cans are rotated about its axis (Tucker 2004). The effect of these agitation methods on the heating times, cooling times, and/or overall thermal process times of different products has been the subject of many studies (Holdsworth and Simpson 2008). Factors such as product viscosity, solid to liquid ratio (for particulate products), headspace, retort temperature, and agitation speed have been tested to understand how the operational parameters in these retorts need to be set to induce maximum amount of mixing within the container. Clifcorn et al. (1950) showed that for EOE retorts, the product quality improved with increasing retort temperature and rotation speeds. Conley et al. (1951) also performed studies with an EOE retort at various rotation speeds and identified that there appears to be an optimum rotational speed, beyond which the amount of mixing in the container decreased. Naveh and Kopelman (1980) performed comparison studies between an
EOE retort and a retort in which cans are subjected to axial rotation, testing the effect of processing factors such as headspace and rotation speed. They concluded that the heat transfer coefficient values were 2 to 3 times greater in the EOE retort. They also concluded that increasing the headspace from 0% to 3% increased the heat transfer coefficient three times as much as that attained when further increasing the headspace from 3% to 11%. In terms of rotation speed, they observed different results for the heating part of the process and cooling part of the process. While, the heat transfer coefficient increased with increasing rotation speed (0-120 rpm) during heating, asymptotic values were reached at 40-70 rpm during the cooling process. In addition to the literature cited here, several more research studies were carried out, most of which were compiled in a table format by Holdsworth and Simpson (2008).

In the last decade, a new type of in-container agitation technology was developed for use in thermal processing of foods. This technology, known as Shaka®, makes use of linear reciprocating movement to agitate the contents of a container during thermal processing in a retort. The frequency of the reciprocating movement used in Shaka® retorts usually range between 140 and 180 cycles per minute, while the stroke length is nominally 15 cm. However, these values are adjusted based on the product properties (Higgins 2012). The reciprocating movement in a Shaka® retort is in the horizontal direction. The intensity of agitation in a Shaka® retort can be expressed as a single value by calculating the maximum acceleration for a combination of stroke length and frequency of reciprocating motion (Walden 2010). This maximum acceleration can then be expressed as multiples of g.
(acceleration due to gravity – 9.81 m/s²). Based on the operating range quoted above, Shaka® systems would generate maximum acceleration values in the range of 2-3g, although tests have been performed at maximum accelerations approaching 4.5g (Walden 2010). It has been claimed that by shaking cans and jars during sterilization, the Shaka® process can dramatically reduce the time to heat up and cool down the product, to the point where a 400 g product can be commercially sterilized in about six or seven minutes versus ninety minutes in a still retort (Dunn 2009). In another study reported by Angalet (2011), five different products were run in three different processing systems: Shaka®, EOE retort, and still retort. Four of these products were placed in 300x406 size cans and one product was placed in a pouch. In terms of retort temperature, 121 °C was used in both still and EOE retorts, and 130 °C was used in the Shaka® retort. The results were reported in terms of total processing times for each product in each retort system. For all products in metal cans, ~90% reduction in processing time was achieved for the Shaka® retort with respect to the EOE retort, and ~95% reduction was achieved for the Shaka® retort with respect to the still retort. For the product processed in pouches, these numbers were ~80% and ~85%, respectively. These findings suggest that Shaka® retort is capable of reducing thermal process times dramatically with respect to other existing retort technologies. However, the effect of product viscosity on the effectiveness of the Shaka® retort is not clear, due to the fact that only a small number of literature is available pertaining to this technology.

ResonantAcoustic® mixing (RAM), is another in-container mixing technology, which, to the best of our knowledge, has not yet been utilized in the food industry.
Developed by Resodyn Acoustic Mixers, Inc. (Butte, MT), this technology is similar to Shaka® in the sense that both technologies utilize reciprocating movement to induce mixing in a container. However, RAM is a short amplitude and high frequency reciprocating movement in the vertical direction. RAM mixers operate at a nominal frequency of 60 Hz, which can vary slightly depending on the resonance frequency of the whole system, including the product being mixed. The amplitude of the movement is 0.5” (1.27 cm) or less. Such a reciprocating movement would result in a maximum acceleration in the order of 100g. Compared to the Shaka® process, this corresponds to a 30-50 times higher acceleration.

RAM technology achieves such high acceleration values by utilizing a special spring-mass arrangement that is brought to resonance for optimum performance. The system continuously tracks the resonance frequency by use of an accelerometer mounted on the payload plate and automatically dials into that frequency. As the system operates at its resonance frequency, the force, and hence maximum acceleration exerted onto the system can be adjusted to control the mixing intensity. Details of the RAM technology have been outlined by Howe et al. (2007). As an effective in-container mixing technology, RAM has a high potential for use in in-container sterilization processes.

The objective of this study was to conduct a comparison study of thermal process times for products of different viscosities processed in the following three setups:

- A thermal process simulating that in a still retort
- A thermal process with motion simulating that in a Shaka® retort
- A thermal process with RAM integrated motion
Materials and Methods

Product Vessel and Connections:

A jacketed and sealable vessel made out of 316L stainless steel (Part # 900611, Resodyne Acoustic Mixers, Inc., Butte, MT) was used as the container in which thermal process studies were carried out for the three different modes of operation. Figure 1 shows a rendered and transparent view of this jacketed vessel from two different angles. The product vessel (8 oz.) within the jacketed vessel had a height of 3.5” (89 mm) and an internal diameter of 2.4” (61 mm). The lid was secured to the vessel with four bolts with wing nuts.

To provide liquid tight seals under pressure, two o-rings were placed in the assembly, one between the product vessel and the outer vessel, and one between the product vessel and the lid.

1/8” (3.175 mm) NPT ports were used for supply and exhaust of steam/cooling water. ¼” (6.35 mm) outer diameter stainless steel braided hoses with 1/8” (3.175 mm) NPT male connections were secured to these ports, which allowed movement of the vessel assembly during the thermal process experiments with motion.

The three ports on the lid were used for inserting thermocouples into the product vessel. Two of these ports were 1/8” (3.175 mm) compression fittings and one was a 1/8” NPT port. Two type K sheathed thermocouples (Model number M12MKIN-1/8-U-6, Omega Engineering, Stamford, CT) were inserted through the compression fittings and sealed by securing the nut on the fitting. One of these thermocouples measured the product temperature
close to the wall, while the other one measured the product temperature at the center of the product vessel. A gland assembly with two type K thermocouple wires (Model number MTG-24T(K)-A4-G,72/6BWT, Conax Technologies, Buffalo, NY) was inserted into the 1/8” NPT port on the lid to measure temperature at random locations within the product vessel.

Assemblies for Motion Supply:

A lab scale RAM unit (LabRAM) from Resodyn Acoustic Mixers, Inc. (Butte, MT) was used as the mixing source for RAM integrated thermal processing (Figure 2). The vessel assembly was mounted on top of the payload plate in the LabRAM through bolt holes on the base of the vessel assembly (Figure 1). The control of the LabRAM was accomplished by use of an OEM software interface. Through this software interface, the mixer could be operated in either auto or manual mode. In auto mode, the mixer automatically dials into the resonance frequency of the system and keeps track of this resonance frequency throughout the run time. In the manual mode, the user can input the frequency of operation. However, since a fundamental principle of this technology is to bring the system to its resonance frequency during operation, all mixing experiments were conducted in auto mode. Another parameter used for control of the mixer was the ‘% intensity’. This parameter controls the amount of reciprocating force exerted onto the system. The ‘% intensity’ has a direct influence on the amplitude of the reciprocating movement, and hence, on the maximum acceleration that the payload plate and payload itself (jacketed vessel with its constituents) is exposed to. 10% intensity corresponds to approximately 10g maximum acceleration, and 100% intensity corresponds to approximately 100g maximum acceleration.
To simulate the motion in a Shaka® retort during thermal processing, a linear motion machine (Model number XTB3810S-S-R03D, Dunkermotoren Linear Systems Limited, Essex, UK) was used (Figure 2). This linear motion machine (LMM) moves the forcer on a magnetic thrust rod to provide reciprocating movement to a payload mounted on the forcer. The payload is directly mounted onto the forcer. For the current study, a payload plate similar to the one on the LabRAM was fabricated and mounted onto the forcer. The vessel assembly was then mounted onto this payload plate. The control of the LMM was accomplished through a visual interface on a control box, where the operator was prompted to input a stroke length and a frequency of motion prior to starting the machine. For all experiments in this study that simulated motion in a Shaka® retort, these values were set to 6” (15 cm) and 180 cycles per minute, respectively.

Setup for Control of Heating/Cooling Media:

Steam and water were used for heating and cooling of the product, respectively. The control of these utilities was managed through manual valves as depicted in Figure 3. A three way valve was used to introduce either steam or cooling water into the jacket of the vessel. A pressure regulator was used to regulate the pressure of the incoming steam. For all experiments, the regulator was set to introduce steam into the jacket at a pressure of 15 psig (1 bar). For cooling water, both warm and cold water were used to temper the water going into the jacket. During start-up of cooling, only warm water was used for 30 seconds. Then, the cold water valve was also opened to mix warm and cold water going into the jacket. After another 30 seconds, the warm water valve was shut to continue cooling with cold water. A
needle valve (Model number EN20SS, Deltrol Fluid Products, Bellwood, IL) was used at the discharge side of the vessel jacket to control the amount of steam/condensate mixture or cooling water exiting from the jacket. During the heating phase, the needle valve was adjusted to graduation # 6 on the red band (almost completely closed) to be able to hold pressure in the jacket while discharging condensate through the valve. During the cooling phase, the valve was completely opened to allow maximum flow rate of water through the jacket.

Sample Preparation:

Banana puree that is concentrated to 41.5° brix (commercially available product) through an evaporation process and subsequently frozen to extend its shelf life was used to prepare all the samples. First, the banana puree was thawed in a refrigerator. Then, this concentrated banana puree was mixed with water at different ratios to obtain samples of different apparent viscosities. A whisk was used to mix the water and the puree until a uniform mixture was obtained. All samples were left at room temperature to equilibrate their temperature prior to conducting the experiments. Seven samples were prepared by utilizing the above procedure. Table 1 shows the % of 41.5° brix banana puree in these samples along with their K (consistency coefficient), n (flow behavior index), and apparent viscosity (at a shear rate of 100 s\(^{-1}\)) values for temperatures within the range of the thermal process. The samples were either poured or spooned into the product vessel for thermal processing of the sample. ~6 mm headspace was left in the product vessel when filled with a sample. For highly viscous samples (80%, 90% and 100% banana puree concentrations), the amount of
headspace in the product vessel was an approximation, since the product did not completely fill all the voids in the vessel and was not leveled at the top. The weight of product in the vessel was 235 g, 240 g, 245 g, 250 g, 255 g, 260 g, and 265 g for 40%, 50%, 60%, 70%, 80%, 90%, and 100% banana puree samples, respectively. Considering the commercially available viscous food products that are subject to either a pasteurization or a sterilization process (concentrated milk, soups, sauces, meals), the seven samples used for this study should cover most, if not all, of these products of interest in terms of their viscosity range.

Data Recording, Thermal Process Parameters, and Lethality Calculation:

All thermocouples were connected to a data acquisition system (Model # NI 9213, National Instruments, Austin, TX) that was connected to a PC through a USB port. The data acquisition system was configured to record temperature data from all thermocouples at a frequency of one data point per second. As the temperature values were being transmitted to the PC, accumulated lethality based on each thermocouple reading was also being calculated and displayed on the screen. The following equation was used for calculating incremental lethality:

\[ F = 10^{\frac{(T - T_{ref})}{z}} \Delta t \]

where, \( F \) is the incremental lethality (min), \( T \) is the instantaneous temperature (°C), \( T_{ref} \) is the reference temperature (°C), \( z \) is the temperature increase necessary to reduce decimal reduction time of the target microorganism by 10 fold (°C), and \( \Delta t \) is the time interval (min). In this equation, \( z \) and \( T_{ref} \) are constant values. A \( \Delta t \) of 0.167 min (1 s) was used for calculating \( F \), since thermocouple readings were recorded once per second. The calculated
incremental F values were then added for each thermocouple to display the accumulated lethality throughout the thermal process. For the current study, 93.3 °C (200 °F) and 8.9 °C (16 °C) were used for \( T_{\text{ref}} \) and \( z \), respectively. These values are commercially used values for thermal process calculation of acidified banana puree recipes. The target accumulated lethality was set to 40 min. Credit for lethality accumulation was given as soon as heating was started in an experiment, and continued till heating was stopped (as soon as an accumulated lethality value of 40 min was reached for the lowest of four accumulated lethality readings).

Visual Appearance of Samples:

A pre-process picture was taken for all seven samples. Post-process pictures were also taken at the end of each experiment to compare the three different processing modes against the pre-process sample and between themselves. All pictures were taken at the same spot in the same lighting conditions to avoid variability in images due to environmental factors.
**Results and Discussion**

For each experiment, temperature and lethality data based on the four thermocouples was plotted against elapsed time to depict the temperature and lethality profiles within the product vessel throughout the thermal process. Thermal process times were determined as the sum of the heating time for an F value of 40 min to be achieved, and the cooling time until the highest thermocouple reading was below 35 °C. Figures 4, 5 and 6 show the time-temperature profiles for the 40%, 90% and 100% banana puree samples processed in a still retort simulation process, respectively. Profiles from 50%, 60%, 70% and 80% banana puree samples are not shown for this method of processing, since only slight differences were observed for the respective temperature and lethality profiles for samples between 40% and 90% banana puree concentrations. For these samples, thermal process times varied between 75 and 80 minutes. For the 100% banana puree sample, the shape of the temperature curve for the thermocouple near the wall of the vessel (Figure 6) was significantly different from the ones for 40% to 90% banana puree samples. The thermal process time for this sample was also significantly higher (90 min). This can be attributed to the difference in the apparent viscosity of the samples (Table 1). Also evident from the observed consistency of the samples during sample preparation, the fluidity of the sample decreases significantly from 90% to 100% banana puree concentration. Based on the observed temperature profiles, it can be concluded that the 100% banana puree sample behaved more like a solid than a liquid, where no natural convection took place within the product vessel during the thermal process simulating that in a still retort.
Figures 7-13 show the temperature and lethality profiles for the seven samples processed with motion of the product vessel simulating that in a Shaka® retort. Comparing Figure 7 to Figure 4, a significant difference was observed in the temperature and lethality profiles of 40% banana puree sample processed in the two modes (still vs. Shaka® retort simulation). The temperature gradient within the product vessel (Figure 4) was eliminated to a large extent when exposed to a motion simulating that in a Shaka® retort (Figure 7). Sharp decreases and increases in temperature were observed in the readings of both loose thermocouples, which suggest that these thermocouples moved within the product vessel with the reciprocating movement of the vessel, and that some temperature variation existed within the product vessel. This temperature variation was more prominent up to an average product temperature of 80 °C, possibly because of the higher apparent viscosities at the lower temperatures. For 50% banana puree sample (Figure 8), a similar profile to that in Figure 7 was observed. The most significant difference between Figure 7 and Figure 8 was the lag in the temperature reading from the center thermocouple compared to the temperature reading from the other three thermocouples during heating and cooling of the sample within the temperature range of 25 °C to 80 °C. The magnitude of this lag, however, was not significant enough to cause an increase in the overall thermal process time of the sample. In Figures 9-13, the temperature and lethality profiles of 60%, 70%, 80%, 90% and 100% banana puree samples processed with motion simulating that in a Shaka® retort can be seen. Unlike the mostly uniform temperature profiles within the product vessel seen in Figures 7 and 8, a wider variability within the temperature profiles from the four thermocouples was observed with increasing banana puree concentration. This can be attributed to less effective mixing
occurring within the product vessel as the viscosity of the product increased with increasing banana puree concentration. For 40%, 50%, 60%, and 70% banana puree samples, while convective heat transfer within the product was the dominant mode of heat transfer, conduction heat transfer became more and more dominant as the concentration of banana puree further increased (80%, 90% and 100%). Temperature profiles observed in Figures 7-13 and comparison of these profiles to the ones in Figures 4-6 suggest that while the type of motion induced in Shaka® retorts could be highly effective in reducing the thermal process times, this effectiveness was found to be highly dependent on the viscosity of the sample being processed.

Figures 14-20 show the temperature and lethality profiles of all seven samples processed in a RAM integrated setup. For samples with up to 80% banana puree concentration (Figures 14-17), no significant variation was observed in the temperature profiles of the four thermocouples, suggesting that almost instantaneous mixing was occurring in the product vessel during the heating and cooling phases. For 80% and 90% banana puree samples (Figures 18-19), some fluctuations in thermocouple readings could be seen, although practically the same amount of heat load was delivered to the whole product volume, which is evident from the narrow F value range obtained based on the time-temperature history from the four thermocouples. For the 100% banana puree sample processed in a RAM integrated setup (Figure 20), a significant amount of lag in the center thermocouple reading was observed compared to the reading from the wall thermocouple. It can thus be concluded that the effective convective heat transfer within the product vessel
diminished for the 100% banana puree sample processed in a RAM integrated setup, compared to the convective heat transfer achieved in samples with lower concentrations of banana puree (40%-90%). Nevertheless, when Figure 20 was compared to Figures 6 and 13, 83% and 80% reduction in thermal process time was realized for the RAM integrated thermal process compared to the still retort simulation and to the thermal process with motion simulating that in a Shaka® retort, respectively. Therefore, it can be concluded that even for samples with high apparent viscosities (similar to a 100% banana puree sample), a RAM integrated thermal process is highly effective in reduction of thermal process time when compared to existing retort technologies.

Thermal process times for all experiments are presented in Table 2, along with the % reduction in thermal process times for Shaka® simulated process with respect to still retort simulation, and for RAM integrated thermal process with respect to Shaka® simulated process. With increasing product viscosity, a decreasing trend was observed for % reduction in thermal process times for Shaka® simulated process with respect to still retort simulation, and an increasing trend was observed for % reduction in thermal process time for RAM integrated thermal process with respect to Shaka® simulated process. This indicates that the effectiveness of the Shaka® simulated process decreased with increasing product viscosity when compared to a still retort process, whereas, the effectiveness of the RAM integrated thermal process increased with increasing product viscosity when compared to a Shaka® simulated process. Figure 21 shows a graphical representation of the thermal process times for all three modes of processing. For the still retort simulation, only a slight increase was
observed in processing times with increasing product viscosity. A similar trend was also observed for the RAM integrated thermal process. However, for the Shaka® simulated process, first two phases of a sigmoid type of curve, with a lag phase and an exponential phase was observed. A similar sigmoid curve can be expected for all methods of thermal process studied in this work, and other methods of thermal process, such as with EOE or axial agitation. Even for a still retort, if the product viscosity is low enough, natural (free) convection can have a significant influence in inducing mixing within a container. Therefore, the magnitude of the lag, exponential, and plateau phases, along with the viscosity range where these phases can be observed, is unique to each method of thermal process. Based on the trends in Figure 21, the points for the still retort simulation seemed to fall between the exponential phase and the plateau phase of the sigmoid curve. For the RAM integrated thermal process, most points seemed to fall within the lag phase of the sigmoid curve, with transition into the exponential phase at a brix of 41.5. For the Shaka® simulated thermal process, the points seemed to start at the lag phase and go through the exponential phase within the viscosity range presented. In other words, the lag phase was observed to be much shorter for the Shaka® simulated thermal process, when compared to the RAM integrated thermal process.

In Figures 22-28, pictures of the samples before and after processing are presented for each concentration of banana puree. In Figure 22, comparing the still retort simulation sample to the pre-process sample, a color change was observed. This color change was more prominent at the wall of the vessel and decreased in intensity towards the center of the
product vessel. This observation corresponds well to the lethality variation observed in Figure 4. For the Shaka® simulation and RAM integrated process samples in Figure 22, no noticeable color change was observed when compared to the pre-process sample.

Observations in Figures 23 and 24 (for 50% and 60% banana puree samples, respectively) were similar to the ones from Figure 22. Starting with Figure 25, significant color changes were observed for the samples processed with a motion simulating that in a Shaka® retort when compared to the respective pre-process samples. This color change was more prominent with increasing concentrations of banana puree and the Shaka® simulated post-process samples looked less and less like the respective pre-process samples, and more and more like the respective still retort simulated samples with increasing product viscosity (Figures 25, 26, 27 and 28). For the RAM integrated thermal process samples in Figures 25-28, no noticeable difference was observed in color when compared to the respective pre-process samples. All these visual observations correlated well to the thermal process times presented in Figure 21.
Conclusions

Thermal processing times and visual appearance of pre- and post-process samples from three different modes of retort processing were compared for a wide range of apparent viscosities (278-5728 mPa s at 24 °C and at a shear rate of 100 s⁻¹) of products. These three different modes of retort processing were still retort simulation, processing with a motion simulating that in a Shaka® retort, and RAM integrated thermal processing. Based on the obtained results, it can be concluded that it is possible to reduce the thermal process times within the studied range of viscosities by inducing mixing via integration of RAM into a retort system. For process mode with motion simulating that in a Shaka® retort, the magnitude of reduction in thermal process times with respect to thermal process times in a still retort simulated process fell sharply due to loss of effectiveness of mixing within the container as the viscosity of the product increased.
References


Conley, W., Kaap, L., & Schuhmann, L. (1951). The application of ‘end-over-end' agitation to the heating and cooling of canned food products. Food Technology, 5, 457-460.


Table 1.
K, n, and apparent viscosity (at a shear rate of 100 s$^{-1}$) values of samples throughout the thermal process temperature range.

<table>
<thead>
<tr>
<th>Sample</th>
<th>24 °C</th>
<th>50 °C</th>
<th>70 °C</th>
<th>90 °C</th>
<th>110 °C</th>
<th>130 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>K (Pa s$^n$)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100 % Banana puree</td>
<td>266.38</td>
<td>103.98</td>
<td>62.24</td>
<td>34.45</td>
<td>23.96</td>
<td>22.63</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>0.17</td>
<td>0.25</td>
<td>0.30</td>
<td>0.37</td>
<td>0.33</td>
</tr>
<tr>
<td></td>
<td>Viscosity (mPa s)</td>
<td>5728</td>
<td>3344</td>
<td>2505</td>
<td>1853</td>
<td>1102</td>
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<tr>
<td>90 % Banana puree</td>
<td>79.16</td>
<td>51.25</td>
<td>29.35</td>
<td>19.88</td>
<td>15.03</td>
<td>15.66</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>0.26</td>
<td>0.28</td>
<td>0.32</td>
<td>0.35</td>
<td>0.34</td>
</tr>
<tr>
<td></td>
<td>Viscosity (mPa s)</td>
<td>2668</td>
<td>1821</td>
<td>1298</td>
<td>991</td>
<td>725</td>
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<tr>
<td>80 % Banana puree</td>
<td>46.81</td>
<td>35.02</td>
<td>20.05</td>
<td>12.67</td>
<td>10.80</td>
<td>10.90</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>0.26</td>
<td>0.28</td>
<td>0.34</td>
<td>0.36</td>
<td>0.34</td>
</tr>
<tr>
<td></td>
<td>Viscosity (mPa s)</td>
<td>1548</td>
<td>1249</td>
<td>967</td>
<td>655</td>
<td>521</td>
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<tr>
<td>70 % Banana puree</td>
<td>39.12</td>
<td>25.01</td>
<td>13.91</td>
<td>8.91</td>
<td>8.13</td>
<td>6.74</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>0.25</td>
<td>0.27</td>
<td>0.33</td>
<td>0.35</td>
<td>0.33</td>
</tr>
<tr>
<td></td>
<td>Viscosity (mPa s)</td>
<td>1216</td>
<td>865</td>
<td>626</td>
<td>441</td>
<td>368</td>
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<tr>
<td>60 % Banana puree</td>
<td>20.91</td>
<td>15.92</td>
<td>8.87</td>
<td>5.75</td>
<td>5.16</td>
<td>4.45</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>0.28</td>
<td>0.29</td>
<td>0.34</td>
<td>0.35</td>
<td>0.30</td>
</tr>
<tr>
<td></td>
<td>Viscosity (mPa s)</td>
<td>770</td>
<td>604</td>
<td>421</td>
<td>292</td>
<td>208</td>
</tr>
<tr>
<td>50 % Banana puree</td>
<td>14.38</td>
<td>10.49</td>
<td>5.72</td>
<td>3.97</td>
<td>3.48</td>
<td>3.07</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>0.27</td>
<td>0.29</td>
<td>0.35</td>
<td>0.34</td>
<td>0.30</td>
</tr>
<tr>
<td></td>
<td>Viscosity (mPa s)</td>
<td>507</td>
<td>404</td>
<td>288</td>
<td>194</td>
<td>139</td>
</tr>
<tr>
<td>40 % Banana puree</td>
<td>6.25</td>
<td>3.57</td>
<td>3.65</td>
<td>3.15</td>
<td>1.76</td>
<td>0.94</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>0.32</td>
<td>0.33</td>
<td>0.31</td>
<td>0.33</td>
<td>0.36</td>
</tr>
<tr>
<td></td>
<td>Viscosity (mPa s)</td>
<td>278</td>
<td>160</td>
<td>151</td>
<td>141</td>
<td>92</td>
</tr>
</tbody>
</table>
Table 2.
Thermal process times for three modes of retort processing.

<table>
<thead>
<tr>
<th>Sample</th>
<th>(A) Still retort simulation (min)</th>
<th>(B) Thermal process with motion simulating that in a Shaka retort (min)</th>
<th>(C) RAM integrated thermal process (min)</th>
<th>Reduction in thermal process time for (B) with respect to (A) (%)</th>
<th>Reduction in thermal process time for (C) with respect to (B) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40% Banana Puree</td>
<td>76.6</td>
<td>6.8</td>
<td>4.3</td>
<td>91%</td>
<td>37%</td>
</tr>
<tr>
<td>50% Banana Puree</td>
<td>76.6</td>
<td>7.1</td>
<td>4.6</td>
<td>91%</td>
<td>35%</td>
</tr>
<tr>
<td>60% Banana Puree</td>
<td>75.6</td>
<td>10.3</td>
<td>5.7</td>
<td>86%</td>
<td>45%</td>
</tr>
<tr>
<td>70% Banana Puree</td>
<td>78.8</td>
<td>21.7</td>
<td>6.3</td>
<td>72%</td>
<td>71%</td>
</tr>
<tr>
<td>80% Banana Puree</td>
<td>78.3</td>
<td>45.9</td>
<td>6.2</td>
<td>41%</td>
<td>87%</td>
</tr>
<tr>
<td>90% Banana Puree</td>
<td>79.8</td>
<td>62.6</td>
<td>8.2</td>
<td>22%</td>
<td>87%</td>
</tr>
<tr>
<td>90% Banana Puree</td>
<td>90.7</td>
<td>76.5</td>
<td>15.4</td>
<td>16%</td>
<td>80%</td>
</tr>
</tbody>
</table>
Figure 1. Vessel assembly for thermal processing of products.
Figure 2. Assemblies for motion supply.
Figure 3. Control of heating/cooling media.
Figure 4. Temperature and F value curves for still retort thermal process simulation (40% banana puree).
Figure 5. Temperature and F value curves for still retort thermal process simulation (90% banana puree).
Figure 6. Temperature and F value curves for still retort thermal process simulation (100% banana puree).
Figure 7. Temperature and F value curves for Shaka® retort thermal process simulation (40% banana puree).
Figure 8. Temperature and F value curves for Shaka® retort thermal process simulation (50% banana puree).
Figure 9. Temperature and F value curves for Shaka® retort thermal process simulation (60% banana puree).
Figure 10. Temperature and F value curves for Shaka® retort thermal process simulation (70% banana puree).
Figure 11. Temperature and F value curves for Shaka® retort thermal process simulation (80% banana puree).
Figure 12. Temperature and F value curves for Shaka® retort thermal process simulation (90% banana puree).
Figure 13. Temperature and F value curves for Shaka® retort thermal process simulation (100% banana puree).
Figure 14. Temperature and F value curves for RAM integrated thermal processing (40% banana puree).
Figure 15. Temperature and F value curves for RAM integrated thermal processing (50% banana puree).
Figure 16. Temperature and F value curves for RAM integrated thermal processing (60% banana puree).
Figure 17. Temperature and F value curves for RAM integrated thermal processing (70% banana puree).
Figure 18. Temperature and F value curves for RAM integrated thermal processing (80% banana puree).
Figure 19. Temperature and F value curves for RAM integrated thermal processing (90% banana puree).
Figure 20. Temperature and F value curves for RAM integrated thermal processing (100% banana puree).
Figure 21. Thermal process time comparison for all three modes of operation.
Figure 22. Pre and post-process samples for 40% banana puree.
Figure 23. Pre and post-process samples for 50% banana puree.
Figure 24. Pre and post-process samples for 60% banana puree.
Figure 25. Pre and post-process samples for 70% banana puree.
Figure 26. Pre and post-process samples for 80% banana puree.
Figure 27. Pre and post-process samples for 90% banana puree.
Figure 28. Pre and post-process samples for 100% banana puree.
CONCLUDING REMARKS

A new in-container mixing technology, ResonantAcoustic® Mixing (RAM), was assessed for use in in-container thermal process applications in the food industry. Product viscosity and mixing intensity were the two factors considered during this assessment. For most experiments, different concentrations of banana puree were used to obtain products of different apparent viscosities. Where transparency was needed in the sample, different concentrations of CMC were prepared. In addition to assessing the RAM technology, experiments were carried out to compare the performance of RAM to other existing retorting techniques (still retort process and Shaka® process) in terms of thermal process times.

Results showed that fluid to particle heat transfer coefficient values of over 2500 W/m² K can be achieved even for a highly viscous product (apparent viscosity of 2670 cP at 25 °C and at a shear rate of 100 s⁻¹). Based on the literature available for heat transfer coefficient values obtained in agitating retorts, such high numbers were not found, even for products of lower viscosities.

Visualization experiments were carried out using the planar laser induced fluorescence technique (PLIF) to understand the progression of mixing in a RAM unit. It was found that mixing starts at the top of the container, progresses to the bottom without covering the middle of the container, and finally progresses to the middle through expansion of the top and bottom mixing regimes. In terms of mixing times required to properly mix two miscible
fluids, values ranged between 3.0 and 35.6 s, depending on the viscosity of the product and the mixing intensity used.

Comparison studies for thermal process times revealed that at a wide range of product viscosities, a RAM integrated thermal process can reduce the processing times anywhere from 83% to 94% with respect to the processing times in a still retort, indicating that the mixing is effective throughout the testing range. The corresponding numbers ranged from 16% to 91% for a Shaka® simulation process with respect to processing times in a still retort, indicating that Shaka® can be highly effective within a certain viscosity range, with decreasing effectiveness at higher viscosities.

The results obtained in this study revealed that RAM integrated thermal processing can potentially be superior to the current agitation technologies used in retort applications. However, these results should only be taken as a preliminary assessment for this technology, due to the novelty of the technology and the limited number of experiments carried out in this study. Recommendations are listed in the next section for future work that would be valuable in further assessing the capabilities and limitations of this technology for use in thermal process applications.
RECOMMENDATIONS FOR FUTURE WORK

This study has only covered product viscosity and mixing intensity as process variables for assessing the use of RAM technology in in-container food thermal process application. There are many other process variables that should be varied and tested with this technology.

The effect of amount of headspace in a container on the effectiveness of mixing is an important aspect that should be considered for future studies. This study could be carried in two dimensions, where the product viscosity is also varied in addition to the headspace to understand whether or not interaction effects exist.

The thermal process time comparison studies were carried out at the same steam pressure (1 bar) for all three technologies. Since, it was demonstrated in this study that, it is possible to heat up and cool down products rapidly during thermal processing with an integrated RAM unit, the effect of higher steam temperatures on thermal process times and final product quality could be studied.

Rotary agitation technologies, such as end-over-end and axial rotation could be used in a study to compare with the RAM integrated thermal processing. The viscosity range for such a study might need to be adjusted based on viscosity values reported in literature where these rotary agitation technologies perform well.
The current study has dealt with homogenous products when assessing the RAM technology. Its effectiveness on particulate products could be assessed in a similar fashion, paying close attention to the integrity of particles after thermal processing. Optimization of experiments could also be carried out for particulate products based on achieved thermal process time reduction and integrity of particles at different mixing intensities.