ABSTRACT

KUMAR, PRABHAT. Aseptic processing of a low-acid multiphase food product using a continuous flow microwave system. (Under the direction of K.P. Sandeep.)

Continuous flow microwave heating is an emerging technology in the food industry with a potential to replace the conventional retort process for viscous and pumpable food products. Aseptic processing of a low-acid multiphase food product using continuous flow microwave heating system can combine the advantages of an aseptic process along with those of microwave heating.

The main objective of this research was to develop a systematic approach for biological validation of aseptic processing of *salsa con queso* products using a continuous flow microwave system operating at 915 MHz. Dielectric properties of pumpable food products were measured by a new approach (under continuous flow conditions) and compared with the dielectric properties measured by the conventional approach (under static conditions). The results suggested that, for a multiphase product, dielectric properties measured under continuous flow conditions should be used for designing a continuous flow microwave heating system.

Thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients (tomatoes, bell peppers, jalapeno peppers, and onions) were measured at a temperature range of 20 to 130 EC. The results were used to fabricate design particles from PP (polypropylene) and PMP (polymethylpentene) using a custom developed CPD (Conservative Particle Design) software. These particles could be used as thermo-sensitive implant carriers for bacterial spores in biological validation of a multiphase aseptic process.

Salsa con queso was processed in a 5 kW microwave unit with a specially designed focused applicator. The temperature profiles at the outlet during processing of *salsa con*

queso in the 5 kW microwave unit showed a narrow temperature distribution between the center and the wall of the tube. Thus, continuous flow microwave heating could overcome the problems (degradation of color, flavor, texture, and nutrients) associated with the wider temperature distribution between the center and the wall in a conventional heating system.

The results from this study will assist processors in designing a safe process for aseptic processing of *salsa con queso* using a continuous flow microwave system. However, further research is required to biologically validate such a process as the final step in establishing an aseptic process for *salsa con queso* using a continuous flow microwave system.

ASEPTIC PROCESSING OF A LOW-ACID MULTIPHASE FOOD PRODUCT USING A CONTINUOUS FLOW MICROWAVE SYSTEM

by

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DEDICATION

To the most important lady

in my life

my mother

BIOGRAPHY

Prabhat Kumar was born on June 9, 1982 in Patna, India. He graduated from Indian Institute of Technology, Kharagpur in 2003, with a Bachelor of Technology (Honors) degree in Agricultural and Food Engineering. After his graduation, he joined Coca Cola India as a Graduate Engineer Trainee in July 2003. During his tenure at Coca Cola, he had the opportunity to manage bottling lines and to implement projects for improving efficiency of the plants. In the fall of 2004, he began his Master of Science degree program in the Department of Food Science at North Carolina State University and currently works with Dr. K.P. Sandeep and Dr. Josip Simunovic as a graduate research assistant. His research focuses on aseptic processing of low-acid multiphase foods. He has decided to stay at North Carolina State University for his PhD under the supervision of Dr. K.P. Sandeep.

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Chapter 1

INTRODUCTION

Thermal processing of food materials is the most widely used method of food preservation. The extent of thermal treatment given to a food product depends on whether the food product is a high-acid product or a low-acid product. A high-acid food product is one with a natural equilibrium pH of less than 4.6 and a water activity equal to or greater than 0.85. These food products include jams, jellies, tomato-based sauces, and pickled products. High-acid food products are typically treated at 90-95 EC for a period of 30-90 s to inactivate yeasts, molds, and bacteria (*Lactobacillus* species). A low-acid food product is one with a natural equilibrium pH of greater than 4.6 and a water activity equal to or greater than 0.85. These food products include butter, cheese, fresh eggs, pears, papaya, sweet apples, and raisins (Skudder, 1993).

Low-acid food products are capable of sustaining the growth of *Clostridium botulinum* spores. *Clostridium botulinum* is an anaerobic, gram-positive, heat-resistant spore-forming bacteria which produces a potent neurotoxin. Foodborne botulism is a severe type of food poisoning caused by the ingestion of foods containing the potent neurotoxin formed during the growth of *Clostridium botulinum*. The spores of *Clostridium botulinum* must be destroyed or effectively inhibited to avoid germination and subsequent production of the deadly toxin which causes botulism. Traditionally, conventional canning has been used to process low-acid food products to ensure the destruction of *Clostridium botulinum* spores (U.S. Food and Drug Administration, 1992).

Conventional canning involves filling of the product in metal cans, glass jars, retortable semirigid plastic containers or pouches, doubles seaming or heat sealing, followed by heating and cooling in a pressurized batch or continuous retort. Conventional canning produces commercially sterile and shelf stable product. Commercial sterility refers to absence of disease causing microorganisms, absence of toxic substances, and absence of spoilage causing microorganisms capable of multiplying under normal nonrefrigerated conditions of storage and distribution. Canning usually involves excessive thermal treatment of the product because heat transfer from the wall of the can to the center of the can is mainly by conduction and convection. Excessive thermal treatment of the product in conventional canning results in degradation of color, flavor, texture, and nutrients (David *et al.*, 1996).

With consumers becoming more health conscious and educated, the demand for convenient and high quality foods has increased over time. Aseptic processing offers a potential option from conventional canning to meet these demands. As opposed to conventional canning, aseptic processing is a thermal process in which the product and container are sterilized separately and brought together in a sterile environment. It involves pumping, deaeration, and sterilization of a food product, followed by holding it for a specified period of time in a holding tube, cooling it, and finally packaging it in a sterile container. The use of high temperature for a short period of time in aseptic processing yields a high quality product with the same level of microbiological safety as that in a conventional canning system. In addition, it is also possible to fill the finished product into flexible packages of different sizes and shapes. Some of the other advantages of aseptic processing include longer shelf life (1 to 2 years) at ambient temperatures, less energy consumption, less space requirement, eliminating the need for refrigeration, and easy adaptability to automation (David *et al.*, 1996; Sastry and Cornelius, 2002).

Aseptic processing of liquid foods such as milk, fruit juices, salad dressings, and liquid eggs has been in place for several decades. Foods containing small particles (smaller than 3.2 mm) such as baby foods, cottage cheese, soups, and rice desserts have also been aseptically processed. However, aseptic processing of low-acid multiphase foods containing large particulates (larger than 3.2 mm) such as soups containing meatballs or vegetables has not been a commercial reality in the U.S. even though it has been in place in the European market for several years (Morris-Lee, 2004). In recent years, there has been an increasing interest in aseptic processing of low-acid multiphase food products. One such product is *salsa con queso* which is a commercially successful product with continued expansion. Currently, *salsa con queso* products are sterilized by the conventional retort process which is associated with degradation of color, flavor, texture, and nutrients.

Continuous flow microwave heating is an emerging technology in the food industry with a potential to replace the conventional retort process for viscous and pumpable food products. Instant start-up and rapid heating of food products make microwave heating suitable for aseptic processing. In the U.S., only four microwave frequencies (915 ± 13, 2,450 ± 50, 5,800 ± 75, and 24,150 ± 125 MHz) are permitted by Federal Communications Commission (FCC) for industrial, scientific, and medical applications (47 CFR18.301, 2005). Continuous flow microwave sterilization is also associated with improved color, flavor, texture, and nutrient retention. Thus, aseptic processing of a low-acid multiphase food product using continuous flow microwave heating system can combine the advantages of an aseptic process along with those of microwave heating.

However, aseptic processing of low-acid multiphase foods containing large particles (larger than 3.2 mm) such as soups containing meatballs or vegetables has not yet been a commercial reality in the U.S. even though it has been in place in the European market for several years (Morris-Lee, 2004). The main reason for this is that unlike in European countries, where approval of a process is based on spoilage tests, in the U.S., the FDA requires that the processor demonstrate by means of experiments and mathematical modeling that every part of the food product receives adequate heat treatment to ensure commercial sterility.

The time-temperature data of the critical point within the product is needed to show that the product receives adequate heat treatment. The critical point in a multiphase product is usually the center of the particle that receives the least heat treatment (critical particle). The critical particle in a system containing only one type and size of particle is the fastest moving particle in the holding tube. In a multiphase (multi particle) product, the fastest moving particle may not be the critical particle because the fastest moving particle with a high thermal diffusivity can receive more heat treatment that a slower moving particle with a low thermal diffusivity. The main problem in determining the critical particle in a multiphase product has been the inability to measure the temperature of particles suspended in a carrier fluid and flowing in a continuous system.

In an attempt to address the problem of validation of aseptic processing of multiphase foods, a workshop was organized by the Center for Advanced Processing and Packaging Studies (CAPPS) and the National Center for Food Safety and Technology (NCFST) in 1995 and 1996. The workshop participants included industry & university personnel and the FDA. The conclusions from the workshop were that determination of the residence time distribution (RTD) of at least 299 particulates in the products, determination of the heat transfer coefficient between the particulates and the fluid, and mathematical modeling are vital in process validation (CAPPS and NCFST, 1995, 1996). Tetra Pak, Inc. (IL, USA) made use of results from this study to successfully develop an aseptic process for a diced potato soup in a modified starch suspension and received a “no-objection letter” from the FDA for the process (Palaniappan and Sizer, 1997). Despite interest in the food industry, cost and time involved in the validation didn’t encourage Tetra Pak to market such a product. Campbell Soup Co. brought aseptic Gold Label soups in market in 2005, but small size and number of particles in the soup limit its widespread use among consumers.

Based on the results of the NCFST-CAPPS workshop, identification of the critical particle was essential for successful validation of a low-acid multiphase food product. Conservative (slowest heating) simulated particles can be designed and further used to validate aseptic processing of a food product. Conservative design means that the thermal protection provided by the design particles to its center is at least equivalent or greater than the thermal protection provided by the critical particle to its center under identical heating conditions.

The main objective of this research was to develop a systematic approach for biological validation of aseptic processing of *salsa con queso* products in a continuous flow microwave system operating at 915 MHz.

Chapter 2

REVIEW OF LITERATURE

2.1 Thermal processing of food materials

Thermal processing of food materials is the most widely used method of food preservation. The extent of thermal treatment given to a food product depends on whether the food product is a high-acid product or a low-acid product. A high-acid food product is one with a natural equilibrium pH of less than 4.6 and a water activity equal to or greater than 0.85. These food products include jams, jellies, tomato-based sauces, and pickled products. High-acid food products are typically treated at 90-95 EC for a period of 30-90 s to inactivate yeasts, molds, and bacteria (*Lactobacillus* species). A low-acid food product is one with a natural equilibrium pH of greater than 4.6 and a water activity equal to or greater than 0.85. These food products include butter, cheese, fresh eggs, pears, papaya, sweet apples, and raisins (Skudder, 1993).

Low-acid food products are capable of sustaining the growth of *Clostridium botulinum* spores. *Clostridium botulinum* is an anaerobic, gram-positive, heat-resistant spore-forming bacteria which produces a potent neurotoxin. Foodborne botulism is a severe type of food poisoning caused by the ingestion of foods containing the potent neurotoxin formed during the growth of *Clostridium botulinum*. The spores of *Clostridium botulinum* must be destroyed or effectively inhibited to avoid germination and subsequent production of the deadly toxin which causes botulism. Traditionally, conventional canning has been used to process low-acid food products to ensure the destruction of *Clostridium botulinum* spores

(U.S. Food and Drug Administration, 1992). Low-acid food products come under regulatory authority of either the FDA or the USDA depending on the proportion of meat or poultry in the food product. The general thermal process requirements of both regulatory agencies are similar and they are compiled in 21 CFR Parts 108 and 113 (FDA) and 9 CFR Parts 308, 318, 320, 327, and 381 (USDA) (Chandrana, 1992).

2.1.1 Methods of thermal processing

There are several methods of thermal processing of foods with pasteurization and sterilization being the two most widely used. Pasteurization refers to heat treatment of a product to kill all pathogenic vegetative microorganisms in it. The time temperature combinations for the pasteurization of milk are 63 EC for 30 min which is referred to as a low temperature long time (LTLT) process and 71.5 EC for 15 s which is referred to as a high temperature short time (HTST) process. The heat treatment in pasteurization is not sufficient to inactivate all spoilage causing vegetative cells or heat resistant spores. Therefore, the shelf life of pasteurized low-acid products such as milks and dairy products is approximately 2 to 3 weeks under refrigerated conditions.

However, pasteurized high-acid products such as juices and beverages packed in hermetically sealed containers using the hot fill process may yield a commercially sterile shelf-stable product. Hot fill refers to filling unsterilized containers with a sterilized food product (usually acidic) that is still hot enough to render the container commercially sterile. A hermetically sealed container is a container that is designed and intended to be secure against the entry of microorganisms and thereby maintain the commercial sterility of its contents after processing. Commercial sterility refers to the absence of disease causing microorganisms, toxic substances, and spoilage causing microorganisms capable of multiplying under normal nonrefrigerated conditions of storage and distribution. Shelf-stable food products can be stored without refrigeration at ambient environmental conditions (David *et al.*, 1996).

Ultrapasteurization refers to pasteurization at temperatures of 138 EC or above for 2 s. This process further extends the shelf life of the product. Ultrapasteurization is sufficient to destroy a greater proportion of spoilage microorganisms, leading to extended shelf life of about 6 to 8 weeks. This process has been used for chocolate and flavored milks and non-dairy creamers in portion pack cups (David *et al.*, 1996).

Sterilization refers to killing of all living microorganisms including spores in the food product. Food products are never completely sterilized, instead they are rendered commercially sterile. Commercially sterilized food products are shelf-stable with a longer shelf life (1 to 2 years). Low-acid food products are commercially sterilized since they are capable of sustaining the growth of *Clostridium botulinum* spores. Traditionally, conventional canning has been used to process low-acid food products to ensure destruction of *Clostridium botulinum* spores. Conventional canning involves filling of the product in metal cans, glass jars, retortable semirigid plastic containers or pouches, double-seaming or heat sealing, followed by heating and cooling in a pressurized batch or continuous retort. Canning usually involves excessive thermal treatment of the product because heat transfer from the wall of the can to the center of the can is due to temperature gradient. Excessive thermal treatment of the product in conventional canning results in degradation of color, flavor, texture, and nutrients (David *et al.*, 1996).

2.1.2 Kinetics in thermal processing

When a homogeneous population of viable spores is subjected to a constant temperature, T, the rate of destruction of spores follows a first order reaction kinetics and is given by (David *et al.*, 1996):

$$\frac{-dN}{dt} = K_T N \quad \textbf{(1)}$$

where, N is the number of spores surviving after time t (s) and K_T is the reaction rate (s^{-1}). Integration of equation 1 from time 0 to time t yields:

$$\frac{N}{N_0} = e^{-k_T t} \quad \textbf{(2)}$$

where, N_0 is the number of spores at time t = 0.

Equation (2) can be re-written as:

$$\log_{10}\left(\frac{N}{N_0}\right) = \frac{-t}{D} \quad \textbf{(3)}$$

where, $D = 2.303/K_T$. D is the decimal reduction time which is the time required to reduce the number of surviving spores by 90%. The D value determined at a reference temperature (T_{ref}) is denoted by D_{ref}. D has been found to vary with temperature according to the equation below (Bigelow, 1921):

$$\log_{10}\left(\frac{D_T}{D_{ref}}\right) = \frac{(T_{ref} - T)}{z} \quad \textbf{(4)}$$

where, D_T is the D-value at temperature T and z is the change in temperature required to change the D value by 90%. The ratio of D_{ref} to D is refereed to as the lethal rate (L_r). F value for a process is defined as the processing time at any temperature to achieve a certain level of microbial kill. F value can be computed in terms of lethal rate as (Ball, 1923):

$$F = \int_0^t L\,dt = \int_0^t 10^{\frac{T - T_{ref}}{z}}\,dt = -D_{ref}\,\log\frac{N}{N_0} \quad \textbf{(5)}$$

The F value at a reference temperature of 121.1 °C (250 °F) and a z value of 10 °C (18 °F) is referred to as the F_0 value. The F_0 value required to achieve a 12 log reduction of *Clostridium botulinum* in a low-acid food product is 3 min. An F_0 value of 3 min indicates that the process is equivalent to a heat treatment of 3 min at 121.1 °C. Thus, many combinations of time and temperature can yield an F_0 value of 3 min. The ratio of F_0 value of the process to the F_0 value required for commercial sterility is known as lethality. Thus, process lethality must be at least 1.0 for commercial sterility. The product of F_0 value required for commercial sterility and $10^{(T_{ref} - T)/z}$ is known as the thermal death time (TDT). TDT is the time required for total destruction of a microbial population or the time required for destruction of microorganisms to an acceptable level.

The destruction of nutrients and inactivation of enzymes follow similar kinetics to that of the destruction of microorganisms. Destruction of nutrients in food products is quantified by cook value (C) which has been defined by Mansfield (1962) as:

$$C = \int_{0}^{t} 10^{\frac{T - T_{ref}}{z_c}} \, dt \quad \textbf{(6)}$$

The C value at a reference temperature of 100 °C (212 °F) and a z_c value of 33.1 °C (91.5 °F) is referred to as the C_0 value.

The objective of a food processor is to produce a commercially sterile product which retains nutritional and quality attributes at an acceptable level. Therefore, appropriate combination of time and temperature used for processing is based on factors such as nutrient retention and enzyme inactivation. D and z_c values for destruction of nutritional and quality attributes are larger than that of the microorganisms. This implies that the rate of destruction of microorganisms at higher temperature will be much higher than the rate of destruction of nutritional and quality attributes. This forms the basis for aseptic processing of food materials. The benefits of aseptic processing arises from ultra high temperature (UHT) sterilization of food materials. UHT sterilization involves exposing the food product to very high temperatures in the range of 130-145 °C for a very short time (2 to 45 s). Thus, aseptic processing of food materials can achieve commercial sterility with better retention of nutritional and quality attributes (David *et al.*, 1996).

2.2 Aseptic processing of food materials

With consumers becoming more health conscious and educated, the demand for convenient and high quality foods has increased over time. Aseptic processing offers a potential option from conventional canning to meet these demands. Aseptic processing is a thermal process in which the product and container are sterilized separately and brought together in a sterile environment. It involves pumping, deaeration, and sterilization of a food product, followed by holding it for a specified period of time in a holding tube, cooling it, and finally packaging it in a sterile container. The use of high temperature for a short period of time in aseptic processing yields a high quality product as compared to that by conventional canning. In addition, it is also possible to fill the finished product into flexible packages of different sizes and shapes. Some of the other advantages of aseptic processing include longer shelf life (1 to 2 years at ambient temperatures), less energy consumption, less space requirement, eliminating the need for refrigeration, easy adaptability to automation, and fewer operators. However, some of the disadvantages of aseptic processing include slower filler speeds, higher overall cost, need for better quality control of raw products, better trained personnel, better control of process variables and equipments, and stringent and extensive validation procedure (David *et al.*, 1996; Sastry and Cornelius, 2002).

2.2.1 History of aseptic processing

The work of Dr. C. Olin Ball and the American Can Research Department laid the foundation of aseptic processing in the U.S. as early as 1927 when the HCF (heat, cool, fill) process was developed. Although the process itself was not a big success, it was a milestone in the development of aseptic processing. In 1942, the Avoset process was developed by George Grindrod. In this process, steam injection was used to sterilize the product and the product was packaged in containers that were sterilized by hot air. The Dole-Martin aseptic process was developed in 1948. This process consisted of four steps -- product sterilization in a tubular heat exchanger, metal container sterilization using superheated steam at temperatures as high as 450 EF (since dry heat requires higher temperature than wet heat), aseptic filling of the product, and sealing of the cooled product in a superheated steam environment. The first commercial aseptic plant was built in 1951 in Washington State, USA by Roy Graves and Jack Stambaugh. The process was based on one of the aseptic canning machines used in the Dole process. The early 1960s was marked with the advent of a form-fill-seal tetrahedron package made of polyethylene in Switzerland for aseptic filling of milk. This development was the starting point for expanding to different package types and sizes. The late 1960s saw the advent of the Tetra Brick aseptic processing machine. The late 1970s saw the advent of the Combibloc aseptic system which used carton blanks instead of roll stocks. Soon, aseptic filling in drums and bag-in-box fillers were established. The use of hydrogen peroxide for the sterilization of packaging surfaces was approved by the FDA in 1981 (David *et al.*, 1996, Buchner, 1993).

Aseptic processing of liquid foods such as milk, fruit juices, salad dressings, and liquid eggs has been in place for several decades. Foods containing small particulates (smaller than 3.2 mm) such as baby foods, cottage cheese, soups, and rice desserts have also been aseptically processed. However, aseptic processing of low-acid multiphase foods containing large particulates (larger than 3.2 mm) such as soups containing meatballs or vegetables has not been a commercial reality in the U.S. even though it has been in place in the European market for several years (Morris-Lee, 2004). The main reason for this is that unlike in European countries, where approval of a process is based on spoilage tests, in the U.S., the FDA requires that the processor demonstrate by means of experiments and mathematical modeling that every part of the food product receives adequate heat treatment to ensure commercial sterility.

2.2.2 Aseptic processing of particulate foods

The development of aseptic processing of particulate foods has been hindered by the requirement to demonstrate an adequate thermal treatment for every part of the food product. In the mid-to-late 1980s, FDA received two filings for aseptically processed low-acid food products containing particles. FDA had several discussions with each firm about the establishment of a scheduled process. Both firms withdrew the filings from further consideration. In the early 1990s, the National Food Processors Association (NFPA) made an effort to develop protocols for the establishment of an aseptic multiphase food process. FDA was still unable to resolve several fundamental issues after lengthy discussions with NFPA (Larkin, 1997).

In an attempt to address the problem of validation of aseptic processing of multiphase foods, a workshop was organized by the Center for Advanced Processing and Packaging Studies (CAPPS) and the National Center for Food Safety and Technology (NCFST) in 1995 and 1996. The workshop participants included industry & university personnel and the FDA. The conclusions from the workshop were that determination of the residence time distribution (RTD) of at least 299 particulates in the products, determination of the heat transfer coefficient between the particulates and the fluid, mathematical modeling, and biological validation are vital in process validation (CAPPS and NCFST, 1995, 1996). Determination of RTD of 299 particles gives 95% confidence that at least one of these particles is in the fastest 1% of the particle's residence time population. This was determined using the following formula (Digeronimo *et al.*, 1997):

$$N = \frac{\log(1 - C_i)}{\log(1 - P)} \quad \textbf{(7)}$$

where, N is the population size, C_i is the confidence level (95%), and P is the fraction of fastest particle (1%).

Tetra Pak, Inc. (Vernon Hills, IL) made use of the results from the above mentioned workshop to successfully develop an aseptic process for a diced potato soup in a modified starch suspension and received a “no-objection letter” from the FDA for the process. Simulated potato particles made of epoxy with embedded small permanent magnets were used as the particulates in the suspension. The density of the simulated particles was adjusted so that the final density was less than the density of potato particles and close to the density of the carrier fluid. Since particles having a density similar to the density of the carrier fluid move faster along the holding tube, this yielded a conservative approach in the determination of residence times (less than the residence times of real potato particles). Real potato particles were also included in the RTD study to verify that the simulated particles yielded conservative residence time values. Magnetic coils were placed at the entrance and exit of the final heater, holding tube, and the pre-cooling unit. The electromotive force generated by the magnet-containing simulated particles was recorded by a data acquisition system, and the residence times in each section were obtained using the entrance and exit times. A finite difference program was used to calculate the temperatures of the fluid and the particle, and the accumulated lethality values were determined. The model was biologically validated using chicken-alginate cubes inoculated with *Clostridium sporogenes* PA 3679, which again had a density that would yield shorter residence times than those of real potato particles. The process proved to be safe based on the results of the mathematical model and the biological validation (Palaniappan and Sizer, 1997). Though this product was not commercially marketed, it demonstrated the feasibility of validating an aseptic process for multiphase foods.

2.2.2.1 Challenges in aseptic processing of particulate foods

Despite the success of Tetra Pak in developing a validated process for aseptic processing of multiphase foods, no such product has been commercially marketed by any company. There are several reasons for this. The first and most important factor is the cost and time involved in the extensive validation process (Larkin, 1997). This is partly due to the necessity to determine the RTD of 299 particles and the heat transfer coefficient between particles and the fluid. This necessity arises due to the fact that we are currently unable to measure the time-temperature history at the “critical point” within particulates as they flow through the processing system (Chandarana, 1992). The “critical point” is the slowest heating portion of the product. For a food product containing only one type of particle, this is the center of the fastest moving particle. However, for a food product containing several types of particles, the “critical point” is the center of the slowest heating particle which may not necessarily be the fastest particle in the system. Thus, if we are able to determine the “critical particle” in a system and use that as the carrier of the microorganism of concern for that particular food product, it would simplify the validation process and make the process relatively inexpensive.

FDA identified following elements that it expected a food processor to address when developing a filing of low-acid multiphase food product (Dignan *et al*., 1989).

1. Identification and selection of appropriate F_0 value
2. Development of a conservative model that predicts the F_0 value delivered to the critical point
3. Quantitative microbiological validation of the F_0 value delivered to the critical point
4. Listing of critical factors and procedures for controlling the F_0 value delivered to the critical point

2.2.3 Components of an aseptic processing system

An aseptic processing system has the following components -- product supply tank, pump, deaerator, heating section, holding tube, cooling section, flow diversion valve, aseptic surge tank, back pressure valve, and packaging system. A schematic representation of an aseptic processing system is shown in figure 1. The product is pumped from the product supply tank to the deaerator using a pump. The deaerator is a vessel maintained at a certain degree of vacuum by means of a vacuum pump. The product is fed into the deaerator at 55-70 EC. The product is deaerated to minimize oxidative reactions which may reduce the quality of the product during processing and storage. The deaerated product is discharged through the bottom and pumped to the heating section where the product is heated to the sterilization temperature.

Several heating systems are used for heating the product in an aseptic process. The choice of heating system is based on the characteristics of the product. Some of them have been described below (Skudder, 1993):

1. **Steam injection**: This is used for homogeneous and high viscosity products and is particularly suited for shear sensitive products such as creams, desserts, and viscous sauces. The liquid product is heated by injection of steam. The rapid heating by steam combined with rapid methods of cooling can yield a high quality product. However, this method is only suitable for liquids with no particles. Another disadvantage of this method is the reduced heat recovery of about 50%.
2. **Steam infusion**: This is used for homogeneous and high viscosity products

and is particularly suited for shear sensitive products such as creams and desserts. This method, similar to steam injection, involves infusing a thin film of liquid product into an atmosphere of steam which provides rapid heating.

3. **Plate heat exchangers**: These are used for homogeneous and low viscosity products (milk, juice, and thin sauce) containing particle sizes up to approximately 5 mm. These heat exchangers consist of closely spaced parallel stainless steel plates pressed together in a frame. Gaskets made of natural rubber or synthetic rubber seal the plate edges. These heat exchangers provide a rapid rate of heat transfer due to the large surface area for heat transfer and turbulent flow characteristics.

4. **Tubular heat exchangers**: These are used for homogeneous and high viscosity products (soups and fruit purees) containing particles of sizes up to approximately 10 mm. The simplest tubular heat exchanger is a double pipe heat exchanger consisting of a pipe located concentrically inside another pipe. These heat exchangers are the most widely used ones for UHT sterilization.

5. **Scraped surface heat exchangers**: These are used for viscous products (diced fruit preserves and soups) containing particles of sizes up to approximately 15 mm. Particle concentrations of up to 40% can be handled in these heat exchangers. These heat exchangers consist of a jacketed cylinder housing scraping blades on a rotating shaft. The rotating action of

the scraping blades prevents fouling on the heat exchanger surface and improves the rate of heat transfer. These exchangers are the best choice for viscous products containing particulates. A disadvantage of these exchangers is that particles are likely to be damaged because of excessive physical action of the blades (Carlson, 1991).

6. **Microwave heating unit**: Continuous flow microwave heating is an emerging technology in the food industry with a potential to replace the conventional heating process. In contrast to conventional heating, heating by microwave provide volumetric heating of the entire food product. Instant start-up and rapid heating of food products make microwave heating suitable for aseptic processing of low-acid multiphase food products. Continuous flow microwave sterilization is also associated with improved color, flavor, texture, and nutrient retention. Thus, aseptic processing of a low-acid multiphase food product using continuous flow microwave heating system can combine the advantages of an aseptic process along with those of microwave heating.

After the heating section, the product is held for a specified period of time in a holding tube, cooled in the cooling section, and finally packed in a sterile container. Commercial sterility of the product is achieved in the holding section. The hold tube must be inclined upwards at least 0.25" per foot in order to avoid air packets and ensure product draining (Lund and Singh, 1993). During aseptic processing, the FDA does not credit accumulation of F-value within a product in the heating and cooling sections. Credit is not given in the heating section because the temperature distribution within the product in the heating section is uncertain. Credit is not given in the cooling section because particulates could possibly break up in the cooling section and thus cool rapidly, thereby not accumulating significant amount of F-value. Therefore, an appropriate combination of time and temperature in the hold tube must be selected to ensure adequate accumulation of F-value for commercial sterility of the product.

Another important part of an aseptic processing system is the back pressure valve which provides sufficient pressure to prevent boiling of the product at processing temperatures which can be as high as 125-130 EC. An aseptic surge tank provides the means for the product to be continuously processed even if the packaging system is not operational due to any malfunction. It can also be used to package the sterilized product while the processing section is being resterilized.

Many methods are available for sterilizing packaging materials. These include use of heat, radiation, chemicals, and a combination of these methods for improved efficiency. Hydrogen peroxide (H_2O_2) is the only chemical allowed for sterilizing packages. It provides an effective method and even greater sterilization efficiency when combined with heat or ultraviolet radiation. It can be used at concentrations varying from 25 to 35%. Sterilization is performed either by spraying H_2O_2 into formed packages or dipping the packaging material into a H_2O_2 bath. The limit of residual H_2O_2 level has been set by the FDA at 0.1 ppm.

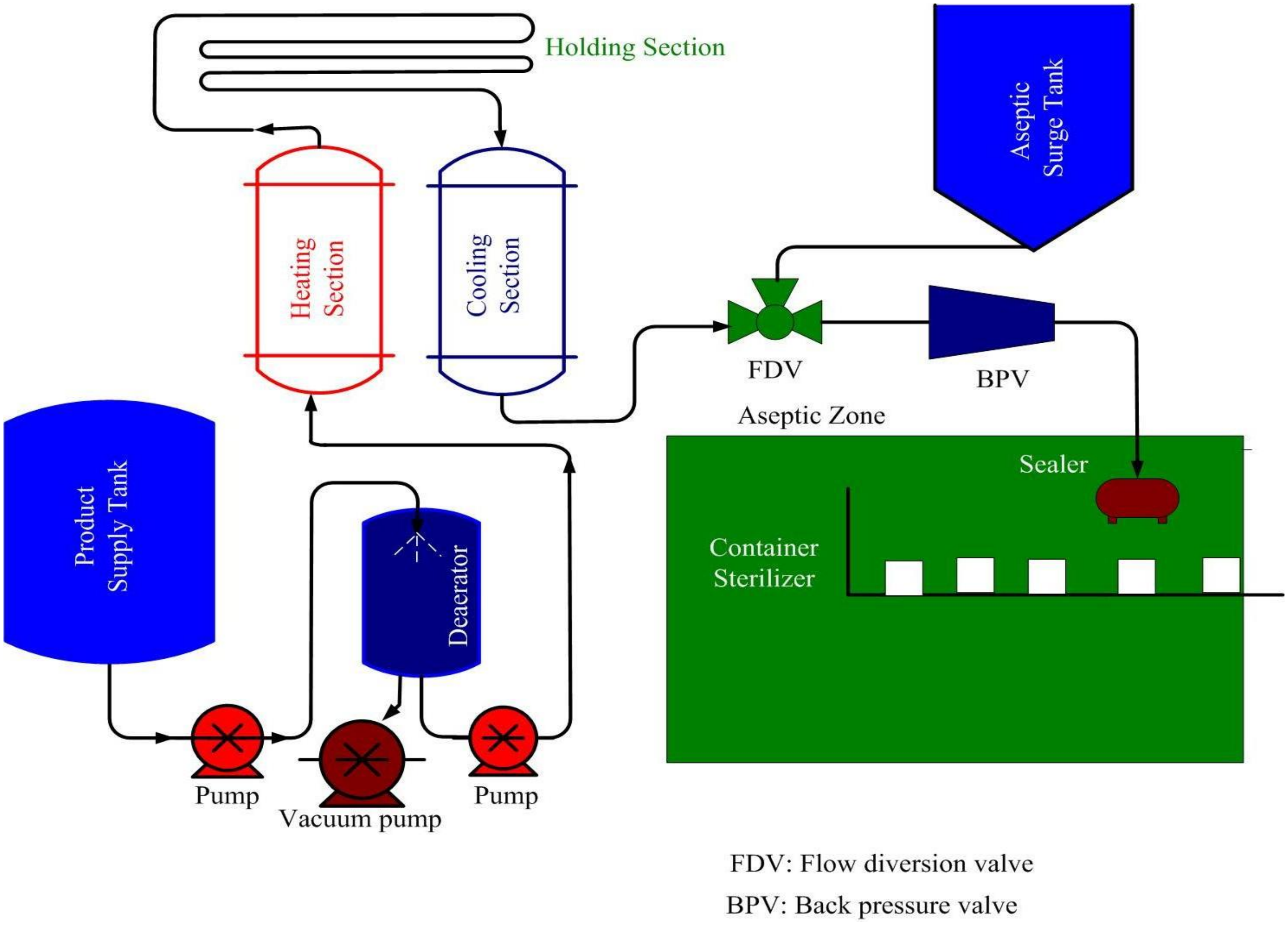


Figure 1. Schematic of an aseptic processing system

2.3 Microwave heating of food materials

Microwaves are a part of the electromagnetic spectrum and have a frequency between 300 MHz and 300 GHz. They lie between the radio (3 kHz - 300 MHz) and infrared (300 GHz - 400 THz) frequencies of the electromagnetic spectrum. Microwave radiation has the ability to heat materials by penetrating and dissipating heat in them. This behavior is used for obtaining controlled and precise heating for materials development and process engineering. Microwaves have got applications in the polymer and ceramic industries (joining, sintering, combustion synthesis, melting, epoxy curing, preheating rubbers, and thermosetting), medicine (thawing frozen tissues, warming blood, and tumor therapies), and textiles (drying) (Saltiel and Datta, 1999). Microwaves have been used for several food processing operations including thawing, blanching, pasteurization, sterilization, dehydration, baking, and roasting (Bengtsson and Ohlsson, 1973). Key components required for the design of a microwave processing system are: capability to produce radiation in the spectrum that will couple with the material being processed, efficient delivery of the radiation to the sample, and control of power distribution. In the U.S., only four microwave frequencies (915 ± 13, 2,450 ± 50, 5,800 ± 75, and 24,150 ± 125 MHz) are permitted by Federal Communications Commission (FCC) for industrial, scientific, and medical applications (47 CFR18.301, 2005).

The important advantages in microwave heating application as compared with conventional heating include instant start-up, faster and selective heating, and energy efficiency. The main disadvantage associated with microwave heating is non-uniform heating of the food materials.

2.3.1 History of microwaves

Percy Spencer (Raytheon Manufacturing Laboratories, Massachusetts, 1945) was the first to conceptualize the use of microwaves for heating food. The first patent (American Patent no. 2495-429) was filed by him in 1945 which described two magnetrons in parallel feeding a waveguide. The microwaves exiting the waveguide impinged upon food on a conveyor belt and cooked it. He filed another patent (American Patent no. 2480-679) which described the terms microwave oven and cavity for the first time. This patent was granted in 1949. Spencer got a patent (American Patent no. 2605-383) for conveying materials through a microwave oven in 1952. A prototype microwave oven operating at 915 MHz for thawing and precooking frozen meals was reported by General Electric in 1947. The Radarange®, the first commercial microwave oven, was demonstrated by Raytheon in 1947. This provided an output power of 1.6 kW from one water-cooled permanent magnet magnetron. K.J. Steiffel developed the first slotted waveguide (American Patent no. 2560-903) and first discussed the idea of using a metallic cover for reflecting microwaves surrounding foodstuffs. Raytheon established an industrial microwave division under the direction of W.C. Brown in the 1950s. The first commercial microwave oven for heating foods became available from Raytheon in the early 1950s. The first tunnel oven was manufactured in 1948 by Thomsan-Houston (French Patent no. 640783). The first multimode cavity incorporating wavetraps was introduced by Wolfgang Schmidt (German Patent no. 3048-686). The British company AEI developed the BM 25A magnetron that delivered 25 kW at 896 MHz. Microwave ovens were first introduced on a large scale in 1965 with expansive growth taking place in the 1970s and 1980s. Presently, microwaves are commonplace household applications (Osepchuk, 1984; Saltiel and Datta, 1999).

2.3.2 Components of a microwave heating system

A microwave heating system, either batch or continuous, have three major components -- a microwave generator, a wave guide, and an applicator. Apart from these three major components, circulator, directional coupler, and tuner form part of a continuous flow microwave heating system**.**

2.3.2.1 Microwave generator

Microwave radiation is generated by acceleration of charge. A number of electronic devices are capable of generating microwave radiation. Solid state microwave devices are not able to deliver the high power required for heating applications. Currently, vacuum tubes (linear beam tubes including klystron and traveling wave tubes and crossfield beam tubes which includes magnetron) are used for generating microwaves. The klystron modulates the movement of electrons using linear fashioned resonant cavities. The klystron was first invented in 1935 by Oscar Heil and A. Arsenjewa-Heil of the C. Lorenz AG company in Berlin.

Similar to klystrons, traveling wave tubes are linear-beam tubes that employ electron beams to amplify a microwave signal. Travelling wave tubes are used to generate variable frequency microwaves. A voltage controlled oscillator generates the microwave signal. The frequency of the microwave signal is controlled by the input voltage. Signal is then sent to the traveling wave tube for amplification. The traveling wave tube (figure 2) consists of a electron gun and a helical transmission line. The heated cathode emits a stream of electrons that is accelerated toward the anode. The electron stream is focused by an external magnetic field. The helical transmission line slows the velocity of the microwave in the axial direction (phase velocity) of the helix to make it equal to the velocity of the electron beam. The axial component of the microwave propagating along the helix interacts with the electron beam. This accelerates or decelerates the electrons within the electron beam. When the velocity of the electron beam becomes faster than phase velocity of the microwave, more electrons are decelerated than accelerated. Thus, microwave signal is amplified because energy is transferred from the electron beam to the microwave field (Thostenson and Chou, 1999).

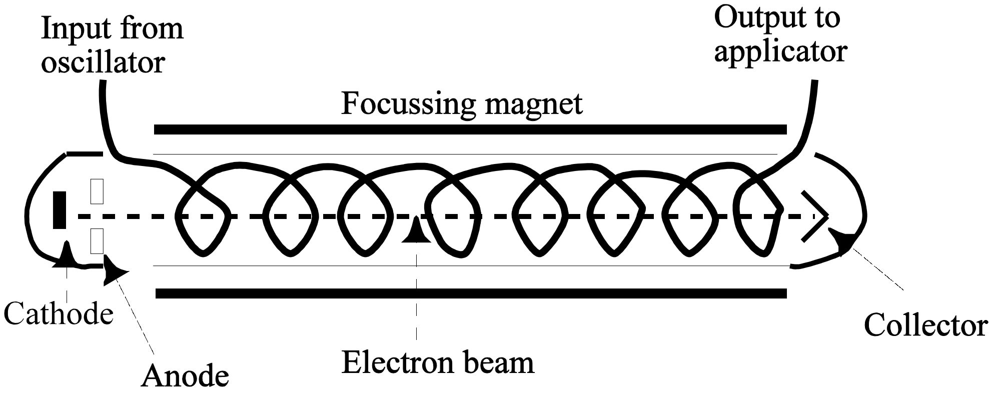


Figure 2. Schematic diagram of a traveling wave tube

The magnetron, a diode-type electronic tube that converts an electrical voltage to microwave radiation, was first conceived in 1921 by Albert Wallace Hull. Magnetron is a circular symmetric tube that consists of a metallic cathode as the central axis of the tube and a hollow cylindrical anode vanes around the circumference (figure 3). The cathode is usually a helix of tungsten that is heated to emit electron by passing electric current through it. The anode is usually made of copper and consists of a series of resonant cavities around the cathode. These resonant cavities act as tuned circuits to determine the output frequency of the microwave. Since anode is at a higher potential as compared to cathode, the potential difference between them produces a strong electric field. This strong electric field heats the cathode to remove loosely bound valence electrons. The magnetron operates by controlling the flow of electrons from the cathode toward the anode. A magnetic field is applied parallel to the axis of the anode and perpendicular to the electron path. This ensures that the electrons travel in a quasi-circular path around the cathode and form an electron cloud. The electron cloud moves continuously and induces positive charges in each cavity segment as it rotates around the axis. This leads to oscillations in the cavities and the frequency of oscillation depends on the size of the cavities. The excess microwave energy is extracted from one of the resonant cavities to the waveguide by a coupling probe. Antenna is an example of a coupling probe which transmits microwave energy from magnetron to waveguide in the form of a transverse electromagnetic (TEM) wave. The average power output of a magnetron can be controlled by adjusting the anode current and the magnetic field strength. Magnetrons are widely used because of their compactness and inexpensive cost (Saltiel and Datta, 1999; Thostenson and Chou, 1999).

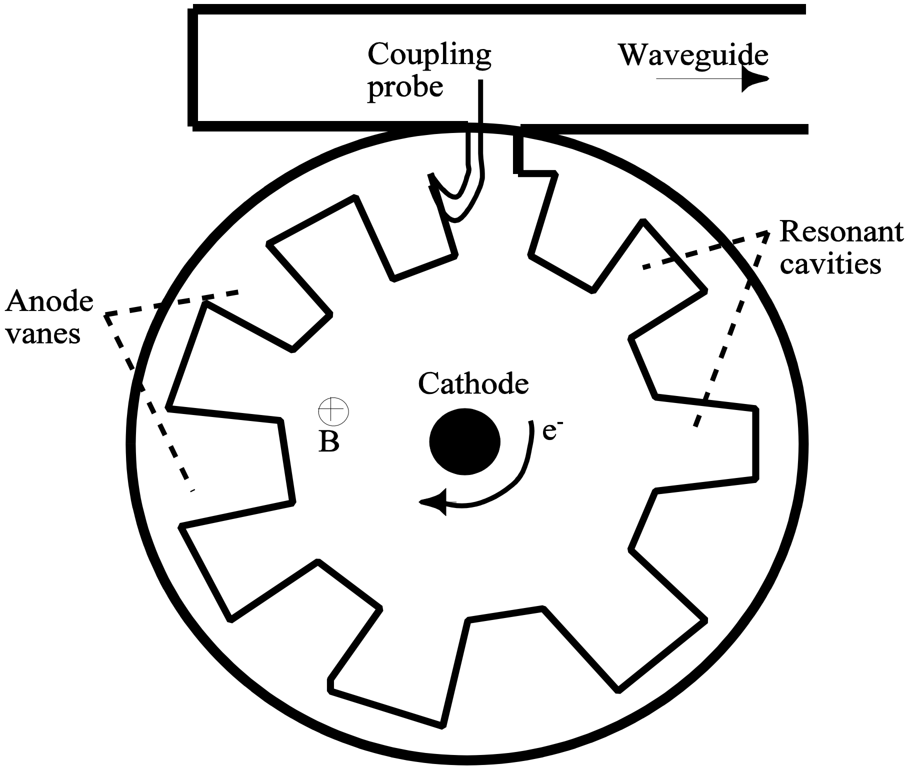


Figure 3. Schematic diagram of a magnetron

2.3.2.2 Waveguide

At low frequencies, wavelength of signals are much larger than the length of the circuit conductors. Therefore, a simple wire is used for carrying the signal. At higher frequencies, the wavelength of signals are comparable or much smaller than the length of the conductors. In this case, guided structure is needed for efficient power transmission. Two commonly used guided structures for propagation of microwaves are transmission lines and waveguides. Transmission line propagation (two-wire transmission lines and coaxial cable) is used extensively at lower frequencies because it becomes inefficient at higher frequencies (3-300 GHz) due to dielectric losses. Waveguides are hollow metal tubes used for propagation of microwaves at higher frequencies. The waveguide concept was first proposed by J.J. Thomson in 1893 and experimentally verified by O.J. Lodge in 1894 using electromagnetic waveguides. The mathematical analysis of the propagating modes of electromagnetic waves within a hollow metal cylinder was first performed by Lord Rayleigh in 1897. One major difference between a transmission line and waveguide is that a transmission line can only support transverse electromagnetic wave whereas a waveguide can support many possible field configurations (Sadiku, 2001).

Waveguides are hollow conductors which come in a variety of sizes and shapes such as rectangular and circular cross sections. Waveguides generally have a constant cross section with dimensions at least one-fourth the wavelength of the microwaves (Saltiel and Datta, 1999). Microwaves can spread out as modes within the waveguide. Mode defines the electromagnetic field distribution within the waveguide. Each mode is characterized by a set of integers l, m, and n. Integer l, m, and n equals the number of half cycle variations in the x, y, and z directions. These

modes of the electric and magnetic fields have been classified in four categories as follows (Sadiku, 2001).

1. Transverse electromagnetic modes (TEM) -- In this mode, both **E** and **H** fields are transverse to the propagation of the wave [$\mathbf{H}_z = \mathbf{E}_z = 0$]. TEM modes are not supported by rectangular waveguides.
2. Transverse electric modes (TE) -- The components of the electric field are transverse to the propagation of the wave, while the magnetic field is parallel to the propagation.
3. Transverse magnetic modes (TM) -- The components of the magnetic field are transverse to the propagation of the wave, while the electric field is parallel to the propagation of the wave.
4. Hybrid modes -- Neither the electric or magnetic field are transverse to the propagation of the wave

The TE and TM modes are the preferred modes of propagation because it is easier to control the electromagnetic fields into them. Both m and n can not be zero at the same time in TE mode. Thus, lowest order mode of all the TE_{mn} modes can be TE_{10} or TE_{01} depending on the dimension of the waveguide. Neither integer m nor n can be zero in TM mode. Thus, TM_{11} is the lowest order mode of all the TM_{mn} modes. For each mode, there is a cut-off frequency (f_c) which is the minimum frequency above which the wave propagate in the waveguide. The cut-off frequency is obtained by the solution of wave equations along with appropriate boundary conditions (Sadiku, 2001). Cut-off frequency is a function of of the waveguide. For a rectangular waveguide of width a and height b filled with a lossless dielectric material, cut-off frequency is given by:

$$f_c = \frac{u'}{2}\sqrt{\left(\frac{m}{a}\right)^2 + \left(\frac{n}{b}\right)^2} \quad \textbf{(8)}$$

where, u' is given by:

$$u' = \frac{1}{\sqrt{\mu\varepsilon}} \quad \textbf{(9)}$$

The intrinsic wave impedance (η_{TE}) of the mode is obtained from the following equation:

$$\eta_{TE} = \frac{\eta'}{\sqrt{1 - \left[\frac{f_c}{f}\right]^2}} \quad \textbf{(10)}$$

where, η' is the intrinsic impedance of the uniform plane wave in the dielectric medium and is given as:

$$\eta' = \sqrt{\frac{\mu}{\varepsilon}} \quad \textbf{(11)}$$

The dominant mode is the mode with the lowest cut-off frequency. For a rectangular waveguide with a > b, TE_{10} is the dominant mode because $f_{cTE10} = u'/2a < f_{cTE01} = u'/2b$

(Sadiku, 2001).

The transmission of microwaves in a waveguide generates a standing wave, but the application of a load changes the shape of the standing wave and increases reflection of the waves. The impedance of the load has to be matched to the impedance of the waveguide to maintain the standing wave ratio and minimize reflection of waves. Impedance matching in a waveguide by tuners.

2.3.2.3 Applicator

The applicator transfers electromagnetic energy from the waveguide to the material to be heated. The size and shape of the applicator depend on (1) the operating frequency, (2) the properties, size, and shape of the material, and (3) type of processing (batch vs. continuous). There are three types of applicator by the type of electromagnetic field configurations - near field applicators, single mode applicators, and multi mode applicators. Microwaves are applied directly to the material in near field applicator. A part of the microwaves, not absorbed by the material, should be transformed into heat by dielectric loads (usually water) behind the materials. These applicators can yield a homogeneous electric field distribution since a standing wave can not develop. They work best with materials which are good absorbers of microwaves.

The electric field is well defined in single mode applicators by the use of resonating structures. These resonating structures enhance the electric field at certain positions. The material to be heated are located at these positions. The design of single mode applicators is based on solution of Maxwell equations to support one resonant mode. However, they are ideal for heating small volumes only as the high intensity level is confined to small regions.

Many modes are established in multimode applicators as the cavity dimensions are large as compared to the wavelength of the microwaves. The design of multi mode applicators is based on trial and error and experience rather than the solution of Maxwell equations. Despite the high number of modes, a non-uniform electric field distribution is developed in mutli mode applicators as opposed to single mode applicators. Multi mode applicator is used in home microwave ovens. The presence of different modes results in multiple hot spots within a microwave cavity of multi mode applicator. These hot spots result in localized over heating. Several techniques are used to reduce the effects of hot spots and to obtain uniform heating. The size of the cavity can be increased to improve the uniformity of the microwave field. Turntables move the food material through areas of high and low power to achieve time averaged uniformity. Mode stirrers can also be used along with multi mode applicators to improve the uniformity of the microwave field. Mode stirrers are reflectors which redistribute the microwave field by rotating within the cavity near the waveguide input.(Saltiel and Datta, 1999; Thostenson and Chou, 1999; Regier and Schubert, 2001).

2.3.2.4 Circulator

A significant amount of power might be reflected back to the microwave generator if the material does not absorb microwave energy efficiently. Excessive reflected power can damage a microwave generator. The circulator protects the equipment of microwave heating system by allowing the microwaves to pass through it in only one direction. There are three ports in a circulator, one of which is connected to the microwave generator, another is connected to the applicator, and third is connected to a dummy load (usually water). The power that is reflected back to the microwave generator is absorbed by the dummy load (Thostenson and Chou, 1999).

2.3.2.5 Directional coupler

Directional coupler measures the magnitude of forward and reflected power in a microwave heating system. They are designed such that a small portion of forward and reflected microwaves are separated and measured by power meters (Thostenson and Chou, 1999).

2.3.2.6 Tuner

The impedance of the load has to be matched to the impedance of the waveguide to maximize power absorption and minimize reflection of waves. Tuners (irises, three stub tuners, and E-H plane tuners) are used for impedance matching which is done by placing small rods of a conductive material at a known distance from the load and changing the depth of their insertion (Thostenson and Chou, 1999).

2.3.3 Dielectric properties

Interaction of microwaves with materials depends on their dielectric properties. Metals are not heated significantly by microwaves as the metals are excellent reflectors. Dielectric materials are better absorbers and transmitters of microwaves. Dielectric materials are heated mainly through the absorption of microwave and the absorption depends on the composition, structure, and temperature of the materials and the frequency of the microwave. Dielectric properties determine the heating behavior of a material when subjected to a microwave field. Therefore, knowledge of dielectric properties is important for the design of a continuous flow microwave heating system.

Dielectric polarization and ionic conduction are primarily responsible for heat generation in dielectric materials under a microwave field. When an external electric field is applied, bound charges in polar molecules create electric dipoles by shifting relative to each other. The dipole is represented in vector form by the dipole moment. Polarization is the average dipole moment per unit volume. There are four main types of dielectric polarization, namely, electronic polarization, atomic polarization, orientation polarization and, space charge polarization. Electronic polarization, which occurs in atoms, is due to the displacement of electrons with respect to the nucleus. Atomic polarization is due to the separation of positive and negative ions in a molecule. Orientation polarization occurs when dipoles try to follow a rapidly changing electric field. Unlike electronic and atomic polarization, orientation polarization is dependent on temperature. Space charge polarization is due to the separation of charges when free electrons are restricted by obstacles. This is also referred to as interfacial or Maxwell-Wagner polarization. The other important mechanism is ionic conduction. When exposed to an external electric field, ions move in the direction of the electric field and energy is transferred by their movement. The relative importance of dielectric polarization and ionic conduction depends on the operating frequency and temperature. Ionic conduction predominates at frequencies below 1 GHz whereas polarization losses are important at frequencies above 1 GHz (Saltiel and Datta, 1999; Ryynanen, 1995).

2.3.3.1 Dielectric constant and loss factor

The electrical behavior of materials when they are subjected to electromagnetic fields is characterized by permittivity (ε), permeability (μ), and conductivity (σ_e). These three parameters relate field strength vectors to their respective flux densities by the following equations (Saltiel and Datta, 1999):

$$D = \varepsilon E \qquad \textbf{(12)}$$

$$B = \mu H \qquad \textbf{(13)}$$

$$J = \sigma_e E \qquad \textbf{(14)}$$

where, D, B, and J are the vector electric, magnetic, and current flux densities respectively, and E and H are the electric and magnetic field strengths. dielectric properties is important for the design of a continuous flow microwave heating system. Dielectric properties consist of dielectric constant (ε') and dielectric loss factor (ε"). Dielectric constant is a measure of the ability of a material to store electromagnetic energy whereas dielectric loss factor is a measure of the ability of a material to convert electromagnetic energy to heat (Metaxas and Meredith, 1983). Dielectric constant and dielectric loss factor can be defined in terms of complex permittivity (ε). The complex permittivity (ε) is composed of a real part (ε', relative dielectric constant) and an imaginary part (ε", effective relative dielectric loss factor) and is given by the equation (Saltiel and Datta, 1999):

$$\varepsilon = \varepsilon_0(\varepsilon' - j\varepsilon'') \qquad \textbf{(15)}$$

where, $j = (-1)^{0.5}$ and ε_0 is the permittivity of free space (8.86×10^{-12} F/m).

ε'' is the loss contribution from all microwave-material interaction mechanisms and is given by (Saltiel and Datta, 1999):

$$\varepsilon''_r(\omega) = \varepsilon''_d(\omega) + \varepsilon''_e(\omega) + \varepsilon''_a + \varepsilon''_s + \frac{\sigma_e}{\varepsilon_0 \omega} \quad \textbf{(16)}$$

where, ω is the angular frequency ($2\pi f$); the subscripts d, e, a, and s refer to dipolar, electronic, atomic, and space charge respectively; and the last term is the contribution due to ionic conduction losses. Loss tangent ($\tan \delta$), a parameter used to describe how well a product absorbs microwave energy, is the ratio of dielectric loss factor (ε'') to the dielectric constant (ε'). A product with a higher loss tangent will heat faster under microwave field as compared to a product with a lower loss tangent (Saltiel and Datta, 1999).

Similarly, losses in magnetic materials can be defined by a complex permeability (μ) as (Saltiel and Datta, 1999):

$$\mu = \mu_0(\mu' - j\mu'') \quad \textbf{(17)}$$

where, μ' is the relative permeability, μ'' is the effective relative magnetic loss factor, and μ_0 is the permeability of the free space ($4\pi \times 10^{-7}$ H/m). Magnetic loss is neglected while considering food materials as most of them are magnetically transparent (Saltiel and Datta, 1999).

Skin depth (δ_s) is the distance at which the amplitude of the electric field falls to e^{-1} of its value at the material surface. It can be expressed as a function of dielectric properties as (Saltiel and Datta, 1999):

$$\delta_s = \frac{\lambda}{\pi\sqrt{2\varepsilon'\left[\sqrt{1+\left(\frac{\varepsilon''}{\varepsilon'}\right)^2}-1\right]}} \tag{18}$$

where, λ is the wavelength of the microwave in free space in meters. The above equation is valid for a plane wave incident upon a semi-infinite slab.

In terms of the optimal parameters, it can be defined as:

$$\delta_s = \frac{n_r\lambda}{k_r 2\pi} \tag{19}$$

where, n_r and k_r are the real and imaginary indices of refraction respectively, and λ refers to the wavelength (Saltiel and Datta, 1999).

Power penetration depth (δ_p), often used in microwave heating applications, is the distance at which power drops to e^{-1} of its value at the surface of the material and is related to the skin depth as (Nelson and Datta, 2001):

$$\delta_p = \frac{\delta_s}{2} \tag{20}$$

2.3.3.2 Factors affecting dielectric properties

The extent of heating by microwaves is determined by dielectric properties, thermophysical properties, and the electromagnetic field of the microwave. The dielectric properties of most materials depend on frequency of the microwaves, temperature, composition, density, and physical structure of the material. Bulk density influences the dielectric properties of particulate materials.

2.3.3.2.1 Frequency

The dielectric properties of most materials vary considerably with the frequency of the applied electric field. Dielectric polarization contributes to the frequency dependence of the dielectric properties. The frequency dependence of dielectric properties for pure polar materials is described by the following equation (Debye, 1929):

$$\varepsilon = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + j\omega\tau} \quad \textbf{(21)}$$

where, ε_∞ is the dielectric constant at very high frequencies

ε_s is the dielectric constant at very low frequencies

j is $\sqrt{-1}$

τ is the relaxation time

ω is the frequency of radiation

Equation (21) can be separated into real and imaginary parts as:

$$\varepsilon' = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + \omega^2\tau^2} \quad \textbf{(22)}$$

$$\varepsilon'' = \frac{(\varepsilon_s - \varepsilon_\infty)\omega\tau}{1 + \omega^2\tau^2} \quad \textbf{(23)}$$

The dielectric constant has a maximum value of $\varepsilon_{s,}$ which is constant, at very low frequencies because the dipoles have enough time to follow the variations of the applied electric field. The dielectric constant has a minimum value of $\varepsilon_{4,}$ which is constant, at very high frequencies because the dipoles are not able to follow the rapidly changing electric field. The dielectric loss factor is small at both very high and very low frequencies. The polar molecules in a solution aligns themselves along the direction of any external electric field applied. The time taken by the molecules to achieve their random orientation from this aligned state on removing the electric field is known as the relaxation time (τ). The dielectric loss factor has a maximum value at the relaxation frequency ($\omega = 1/\tau$). The dielectric constant changes from a very high value before the relaxation frequency to a very low value after the relaxation frequency. The relaxation frequency for pure water at 20 EC is 17.004 GHz. Therefore, the most effective conversion of microwave energy into thermal energy should occur in this frequency region The maximum value of tan δ occurs at slightly higher frequency than the relaxation frequency (Gabriel *et al.*, 1998). Equation (21) can be represented in the complex plane as a 'Cole-Cole' diagram (Cole and Cole, 1941). The 'Cole-Cole' diagram is a semicircle with a locus of a point ranging from ($\varepsilon' = \varepsilon_{s,}\ \varepsilon'' = 0$) at the low frequency limit to ($\varepsilon' = \varepsilon_{4,}\ \varepsilon'' = 0$) at the high frequency limit.

The frequency dependency of dielectric properties of materials which are not pure polar can be given by the Cole-Cole equation (Cole and Cole, 1941):

$$\varepsilon \;'\; \frac{\varepsilon_s \;\&\; \varepsilon_4}{1 \;\%\; (j\omega\tau)^{1\&\alpha_s}} \qquad \textbf{(24)}$$

where, α_s is the spread of relaxation times and it ranges from 0 to 1.

2.3.3.2.2 Temperature

Temperature has a significant effect on the dielectric properties of materials. The temperature dependence of the dielectric properties of water is a function of the dielectric relaxation processes. The relaxation time decreases with increase in temperature which shifts the peak of the dielectric loss factor to higher frequencies. Since relaxation time is the time taken by polar molecules to align themselves along the direction of the field, a decrease in its value decreases the dipolar contribution to the dielectric constant. Thus, the dielectric constant in the region of relaxation frequency increases with an increase in temperature whereas the dielectric loss factor may either increase or decrease depending on whether the operating frequency is higher or lower than the relaxation frequency. (Nelson and Datta, 2001). The dielectric loss factor of a salt solution is due to the combined effect of dielectric polarization and ionic conduction. Contribution to dielectric loss factor from dielectric polarization decreases with temperature whereas contribution from ionic conduction increases with temperature. The increase in ionic conduction can be attributed to the decreasing viscosity, which in turn increases the mobility of ions (Datta *et al.*, 2005).

2.3.3.2.3 Composition

The composition of a food material influences its dielectric properties. Moisture, salt content, carbohydrate, protein, and fat and the important components of a food material. Water can exist in food materials in either free or bound form. Effect of bound water on the dielectric properties are smaller as compared to free water in the microwave frequency range. There is an increase in dielectric constant and dielectric loss factor with an increase in moisture content. Carbohydrates do not show dipolar polarization at microwave frequencies, but they can effect the dielectric properties by forming hydrogen bonds with water. For carbohydrate solutions, effect of free water on dielectric properties becomes significant. Gelatinization of starch increases dielectric properties by binding less water to its structure and thus increasing the amount of free water. Proteins do no significantly interact with microwaves. However, protein denaturation affects the dielectric properties of food materials. The dielectric properties of fats and lipids are very low and their only effect on dielectric properties is due to dilution of water (Datta *et al.*, 2005).

The dielectric constant of a fibrous material is higher than that of a granular material. The effect of pH is not significant at the pH levels typical in foods. There is a positive correlation between the dielectric constant and the water content whereas the correlation for the dielectric loss factor is uncertain. Fat influences the dielectric properties by diluting the water content of the material (Ryynanen, 1995).

2.3.3.2.4 Density

The permittivity of aqueous solutions or mixtures decreases by two mechanisms: replacement of water by a substance with lower permittivity, and binding of water molecules. When the size of particles in the mixture is much smaller than the wavelength of the waves, the effective permittivity depends only on the shape of the particles and is independent of their size (Ryynanen, 1995). Density will have a notable effect on the dielectric properties of particulate materials. For a two component mixture, dielectric properties can be calculated by the following equations (Nelson and You, 1990).

Complex refractive index mixture equation:

$$\varepsilon^{1/2} = \upsilon_1(\varepsilon_1)^{1/2} + \upsilon_2(\varepsilon_2)^{1/2} \tag{25}$$

Landau and Lifschitz, Looyenga equation (Landau and Lifschitz, 1967; Looyenga, 1965):

$$\varepsilon^{1/3} = \upsilon_1(\varepsilon_1)^{1/3} + \upsilon_2(\varepsilon_2)^{1/3} \tag{26}$$

Bottcher equation (Bottcher,1944):

$$\frac{\varepsilon - \varepsilon_1}{3\varepsilon} = \upsilon_2 \frac{\varepsilon_2 - \varepsilon_1}{\varepsilon_2 + 2\varepsilon} \tag{27}$$

Bruggeman-Hanai equation:

$$\frac{\varepsilon - \varepsilon_1}{\varepsilon_1 - \varepsilon_2}\left(\frac{\varepsilon_1}{\varepsilon}\right)^{1/3} = 1 - \upsilon_2 \tag{28}$$

Rayleigh equation:

$$\frac{\varepsilon - \varepsilon_1}{\varepsilon + 2\varepsilon_1} = \upsilon_2 \frac{\varepsilon_2 - \varepsilon_1}{2\varepsilon_1 + \varepsilon_2} \tag{29}$$

Lichtenecker equation (Lichtenecker and Rother, 1931):

$$\ln\varepsilon = \upsilon_1 \ln(\varepsilon_1) + \upsilon_2 \ln(\varepsilon_2) \tag{30}$$

where, ε represents the permittivity of the mixture

ε_1 is the permittivity of the medium in which particles of permittivity ε_2 are dispersed

υ_1 and υ_2 are the volume fractions of the respectivecomponents where $\upsilon_1 \ \% \ \upsilon_2 \ ' \ 1$

2.3.3.2.5 Change of phase

Change of phase is responsible for a very sharp change in the dielectric properties of food materials. Thus, accurate determination of dielectric properties of frozen and partially frozen products is important to determine the rate and uniformity of heating of those products during microwave thawing. The dielectric constant and the dielectric loss factor increase significantly when ice melts. Thus, portions of frozen products, that thaw first, can start boiling by absorbing more microwave energy and heating up at increasing rates while the other portions of the same product are still frozen. This undesirable phenomena is commnoly known as runaway heating. Runaway heating is the ability of a material to absorb increasing amount of microwave energy with an increase in its temperature (Datta *et al.*, 2005).

2.3.3.3 Measurement of dielectric properties

The dielectric properties of food products are important in determining the extent of heating of food materials in a microwave cavity. Dielectric properties govern the ability of food materials to heat when subjected to microwave fields. Therefore, there is a need for better understanding of the methods for measuring dielectric properties. The factors on which the measurement techniques for any particular application depend are: frequency of microwaves, physical and electrical properties material to be measured, and degree of accuracy required (Nelson, 1999).

Dielectric property measurement of a material-under-test (MUT) consist of the following steps -- generate a microwave signal at the frequencies of interest, direct the signal through the MUT, measure the changes in the signal caused by the MUT, and compute the permittivity (ε). Measurement of dielectric properties involves incident, reflected, and transmitted waves traveling along transmission lines. Specifically it involves accurate measurement of the ratio of the reflected signal to the incident signal and/or the transmitted signal to the incident signal. A network analyzer is an instrument used to analyze the properties associated with reflection and transmission of electrical signals**.** The basic principles of working of a network analyzer has been described below (Ball, 1998).

2.3.3.3.1. Network analyzer

There are 4 major signal processing sections in a network analyzer -- signal source, signal separation, receiver/detector, and processor/display (figure 4). The signal source supplies the sine wave for the test. Signal separation device measures a portion of the incident signal and separate the incident and reflected traveling waves at the surface of MUT. There are two basic ways of receiving signal in network analyzers -- diode detector and tuned receiver. Diode detectors convert the signal to a proportional DC level. The phase information of the signal is lost as it is a scalar detection device. The tuned receiver uses a local oscillator (LO) to change the signal to a lower intermediate frequency. This can be used in both scalar and vector network analyzers. The processor displays results from the reflection and transmission data obtained.

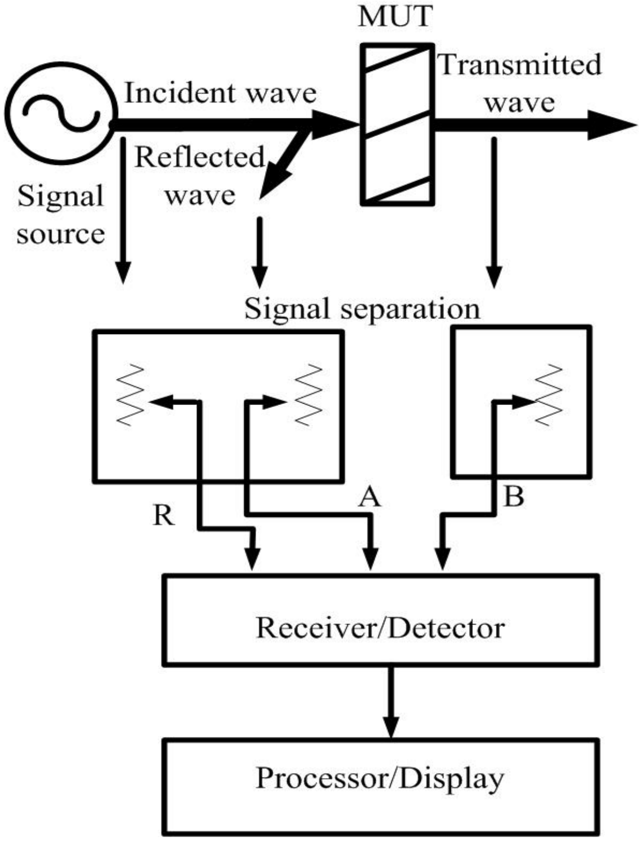


Figure 4. Block diagram of a network analyzer

Signal source section

A signal source produces sine waves. The sine wave in time domain is expressed as:

$$V(t) = V_o \sin(2\pi f_o t) \qquad \textbf{(31)}$$

There are three basic types of sources -- continuous wave (CW) source, swept source, and signal generator. A source that produces a single sine wave is known as a CW source.

The frequency and amplitude of the sine wave can be set to a desired value in most of the CW sources. A swept source can automatically vary the output frequency or amplitude of the sine wave over a range of frequencies or amplitudes. There are two types of frequency sweeps -- ramp sweep and step sweep. The frequency of the output sine wave is increased from a start frequency to a stop frequency in ramp sweep. The frequency of the output sine wave is abruptly changed from one frequency to another in step sweep. A signal generator can add modulation to the sine wave. Thus, signal generators output signals that can carry information. Basic signal generators have amplitude, frequency, and phase modulation capabilities. The ability to generate modulated signals is the main difference between a signal generator and a CW source.

There are three broad specifications for a CW source -- frequency, amplitude, and spectral purity. The main specifications for frequency are range, resolution, and accuracy. Range specifies the frequencies which the source can produce. Resolution is the smallest frequency increment. The stability of the reference oscillator and the amount of time since calibration affect accuracy of the source. The main specifications for amplitude are range, accuracy, resolution, switching speed, and reverse power protection. The range of a source is the maximum output power and the amount of internal attenuation built into the source. The resolution is the smallest amplitude increment. Switching speed measures how fast the source can change from one amplitude to another. The main specifications for spectral purity are phase noise and residual frequency modulation (FM). Phase noise is the random noise in the source which causes the power to spread from one frequency to a small range of frequencies. Residual FM is a measure of the small amount of FM inherent in a CW source output.

CW source can be divided into three major sections -- reference, synthesizer, and output. Reference oscillator is the main component of the reference section. A stable reference oscillator ensures that the frequency of the output signal remains accurate in between calibrations. The basic form of an oscillator is an electronic amplifier (transistor) with the output attached to a narrow-band electronic filter, and the output of the filter attached to the input of the amplifier. When the power supply to the amplifier is first switched on, the output of the amplifier consists only of noise. The noise travels around the circuit, gets filtered and re-amplified until it increasingly resembles the desired signal. The reference section sends a sine wave with a known frequency to the synthesizer section. This sine wave is used as a reference for the phase-locked loop (PLL) and the voltage controlled oscillator (VCO) in the synthesizer section produces a sine wave at the desired frequency. A simple VCO can be constructed from a varactor (voltage-variable capacitor). An example of common varactor is a reverse-biased pn junction diode. There is no flow of current in a reverse-biased pn junction diode. The capacitance across the diode decreases due to widening of the depletion region as the applied bias voltage to the diode increases. The varactor enables the output oscillation to be tuned. The output signal is unstable and is stabilized by a PLL. The synthesizer section supplies a sine wave to the output section where the overall amplitude and accuracy of the source is determined. Amplitude is determined by available amplification and attenuation. Accuracy of the source is maintained by monitoring the output power and is adjusted by the automatic level control (ALC).

Signal separation section

Signal separation section performs two functions. The first is to measure a portion of the incident signal to provide a reference for ratioing. This is done with splitters or directional couplers. Directional couplers are preferred as they have very low insertion loss and good isolation of the signal and directivity. Directivity is a measure of how well a coupler can separate signals moving in opposite directions. The second function of this section is to separate the incident wave and reflected wave at the surface of MUT. Directional couplers are ideal for this function, but due to difficulty in making broadband couplers, bridges are often used. Bridges operate similar to the simple wheatstone bridge and are very useful in measuring reflection because they can work over a very wide range of frequencies. If all four arms of the bridge are equal in resistance (usually 50 Ω), a zero voltage is measured and the bridge is balanced. If the test port load is not 50 Ω, the voltage across the bridge is proportional to the mismatch by MUT. The magnitude and phase angle of the voltage gives the complex impedance at the test port.

Receiver/detector section

There are two basic ways of receiving signal in network analyzers -- diode detector and tuned receiver. Diode detector converts the signal to a proportional DC level. The diode separates the signal from the modulation for amplitude modulated signal. Diode detection is scalar as the phase information of the signal carrier is lost. The main advantages of diode detector include broadband frequency coverage (< 10 MHz to > 26.5 GHz) and low cost. A tuned receiver uses a local oscillator (LO) to bring the signal to a lower intermediate frequency. Then, the signal is bandpass filtered to narrow the receiver bandwidth. The magnitude and phase information of the signal is extracted by an analog-to-digital converter (ADC) digital signal processing (DSP). Tuned receivers provide the best sensitivity and dynamic range. A tuned receiver can be used in both scalar and vector network analyzers.

Processor/display section

The processor displays the results from the reflection and transmission data obtained. This section has features such as linear and logarithmic sweeps, linear and log formats, polar plots, smith charts etc.

Overview of the HP 8753C network analyzer

The source produces a highly stable and accurate output signal by phase locking a YIG (Yttrium-iron-garnet) oscillator to a harmonic of the synthesized VCO (voltage controlled oscillator). The output is a CW or swept signal between 300 kHz and 3 GHz with a maximum leveled power of +20 dBm and minimum power of -5 dBm. The full frequency range of the source is produced in 11 subsweeps, 2 in low band (300 kHz to 16 MHz) and 9 in high band (16 MHz to 3 GHz). The high band frequencies are achieved by harmonic mixing with a different harmonic number for each subsweep. The low band frequencies are down-converted by fundamental mixing (Agilent Manual).

The three most popular methods for measuring dielectric properties are the open-ended coaxial probe, transmission line, and resonance cavity methods (Engelder and Buffler, 1991) and they are described in detail below.

2.3.3.3.2 Open-ended coaxial probe method

The open-ended coaxial dielectric probe (HP 85070B and HP 85070E) is a cut off section of a transmission line. The tip of the coaxial probe is in contact with the MUT. The source section of the network analyzer sends a microwave signal down the probe. The signal is reflected from the interface formed by the probe-end and the MUT. The receiver section of the network analyzer detects the magnitude and phase shift of the reflected signal. A computer program in the processor section calculates the dielectric constant (ε') and dielectric loss factor (ε'') from this data. The advantages of this method are -- measurement over a wide frequency range, easy to use as it needs no particular sample shape, and suitability to food products as the probe is water tight, easy to clean, and measures dielectric properties of both liquids and solids. Despite these advantages, the probe has limited accuracy when it is used to measure materials with low values of dielectric constant and dielectric loss factor (oils and fats). The sample should have a flat surface to have a direct contact between the probe and the sample.

Transmission line

A transmission line is the material, medium, or structure that forms all or part of a path from one place to another for directing the transmission of energy, such as electromagnetic waves or acoustic waves, as well as electric power transmission. A transmission line basically consists of two or more parallel conductors used to connect a source to a load. Components of transmission lines include wires, coaxial cables, dielectric slabs, optical fibers, electric power lines, and waveguides. An infinitely long lossless transmission line appears to be a resistive load. This resistive load is known as the characteristic impedance (Z_o). A characteristic impedance of 75 ohms is used for low loss transmission (cable TV) where as a characteristic impedance of 50 ohms is used for microwave applications (high power). For maximum transfer of energy from a source or transmission line to a load, impedance of source and load (Z_L) should match Z_o. When the load impedance is equal to Z_o, there is no reflected signal. When the transmission line is terminated in a short circuit, the magnitude of the reflected wave is equal to that of the incident wave and 180E out of phase with it. When the transmission line is terminated in open circuit, the reflected current wave is 180E out of phase with respect with the incident wave.

Transmission line parameters

The line parameters of a transmission line are resistance per unit length (R), inductance per unit length (L), conductance per unit length (G), and capacitance per unit length (C) (figure 5). The line parameters are distributed uniformly along the entire length of the transmission line as shown in figure 5. For each line, conductors are characterized by σ_c, μ_c, and $\varepsilon_c = \varepsilon_o$ and the homogeneous dielectric separating the conductors are characterized by σ, μ, and ε. Conductance per unit length (G) is not equal to the inverse of resistance per unit length (R). R is the AC resistance per unit length of the conductors comprising the line and G is the conductance per unit length due to the dielectric medium separating the conductors (Sadiku, 2001).

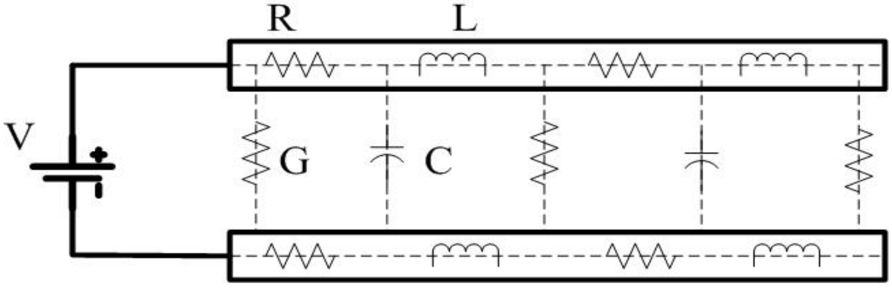


Figure 5. Distributed line parameters of a two conductor transmission line

For each line,

$$LC \; ' \; \mu\varepsilon \quad and \quad \frac{G}{C} \; ' \; \frac{\sigma}{\varepsilon} \qquad \textbf{(32)}$$

Propagation of an electromagnetic wave (EM) along transmission line

A coaxial transmission line probe connecting generator to the load has been shown in figure 6. When the inner conductor is made positive with respect to the outer conductor, Electric field (**E**) is radially outward from inner to outer conductor. Ampere's law states that the magnetic field (**H**) encircles the current carrying conductors. Thus, the Poynting vector (**E** × **H**) is long the transmission line. Thus, transmission line can transmit power by propagating a transverse electromagnetic (TEM) wave along its length (Sadiku, 2001).

Transmission line equations

Voltage and current in the circuit of figure 6 can be calculated by applying Kirchoff's law. Harmonic time dependence of voltage and current is considered. The equations for voltage (V_s) and current (I_s) in the circuit are (Sadiku, 2001):

$$\begin{aligned} \frac{d^2V_s}{dz^2} \; \& \; \gamma^2 V_s \; ' \; 0 \\ \frac{d^2I_s}{dz^2} \; \& \; \gamma^2 I_s \; ' \; 0 \end{aligned} \qquad \textbf{(33)}$$

where, γ is the propagation constant (m^{-1}) and is given by:

$$\gamma = \alpha_c + j\beta_c = \sqrt{(R + j\omega L)(G + j\omega C)} \quad \textbf{(34)}$$

where, α_c is the attenuation constant (in nepers per meter or decibels per meter) and β_c is the phase constant (radians per meter).

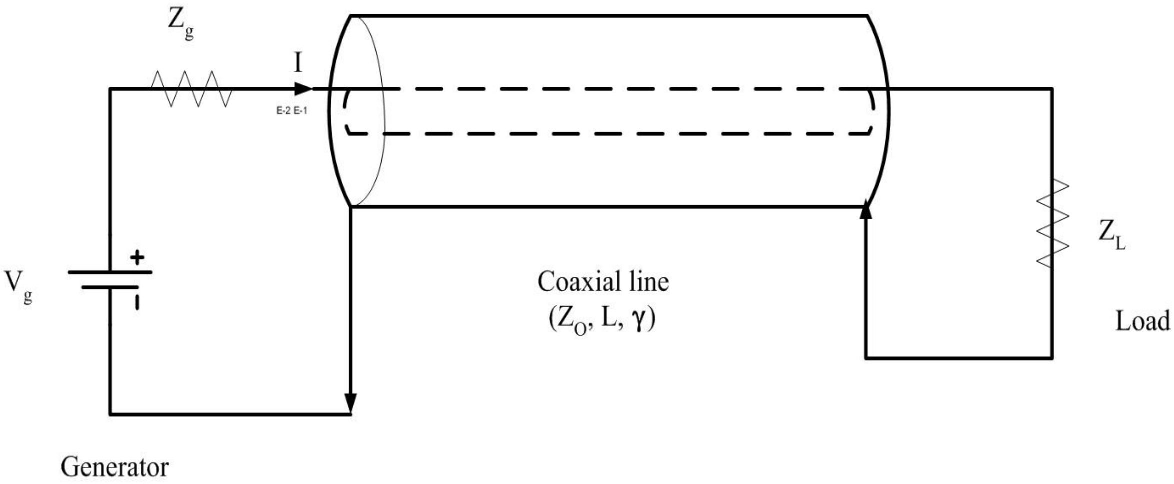


Figure 6. Coaxial line probe connecting the generator to the load

The wavelength (λ) is related to β_c by the following equation:

$$\lambda = \frac{2\pi}{\beta_c} \quad \textbf{(35)}$$

Characteristic impedance (Z_o) of the transmission line is the ratio of positively traveling voltage wave to current wave at any point in the line and is given by:

$$Z_o = \sqrt{\frac{(R + j\omega L)}{(G + j\omega C)}} = R_o + jX_o \quad \textbf{(36)}$$

where, R_o and X_o are the real and imaginary parts of Z_o.

The propagation constant (γ) and the characteristic impedance (Z_o) are important properties of transmission line because they depend on the line parameters R, L, G, and C and the frequency of operation. A transmission line is said to be lossless if the conductors ($\sigma_c = 4$) are perfect and the dielectric medium separating them is lossless ($\sigma = 0$). For such a line R = G = 0 and equation (36) becomes:

$$Z_o \;'\; \sqrt{\frac{L}{C}} \;'\; R_o \quad \textbf{(37)}$$

Voltage reflection coefficient (Γ) is the ratio of the reflected signal voltage to the incident signal voltage and is given as:

$$\Gamma \;'\; \frac{V_{reflected}}{V_{incident}} \;'\; \rho_r p \Phi \;'\; \frac{Z_L \& Z_O}{Z_L \% Z_O} \quad \textbf{(38)}$$

where, ρ_r is the magnitude portion of the reflection coefficient. The range for possible values of ρ_r is between 0 to 1.

Return loss is the number of dB that the reflected signal is below the incident signal and is given as:

$$\text{Return loss} \;'\; \&20 \log(\rho_r) \qquad \textbf{(39)}$$

The range for possible values of return loss is between 4 to 0 dB.

Voltage standing wave ratio (VSWR) is defined as the maximum value of voltage over the minimum value of voltage and is given as:

$$\mathrm{VSWR} \ ' \ \frac{V_{\max}}{V_{\min}} \ ' \ \frac{1 \% \rho}{1 \& \rho} \qquad \textbf{(40)}$$

The range for possible values of VSWR is between 1 to 4.

The Smith chart

The Smith chart is the most commonly used graphical techniques for calculating the characteristics of transmission lines. It is a graphical indication of the impedance of a transmission line as one moves along the line. The Smith chart is constructed within a circle of unit radius and is based on equation (38). All impedances are normalized with respect to Z_o to use the same Smith chart for transmission lines with different Z_o. For example, the normalized impedance (z_L) for the load impedance (Z_L) is given by (Sadiku, 2001):

$$z_L \ ' \ \frac{Z_L}{Z_O} \ ' \ r \ \% \ jx \qquad \textbf{(41)}$$

The normalized load impedance (z_L) can be written in terms of Γ as:

$$z_L \ ' \ r \ \% \ jx \ ' \ \frac{(1 \ \% \ \Gamma_r) \ \% \ j\Gamma_i}{(1 \ \& \ \Gamma_r) \ \& \ j\Gamma_i} \qquad \textbf{(42)}$$

Equating real and imaginary parts we obtain:

$$r = \frac{(1 - \Gamma_r^2) - \Gamma_i^2}{(1 - \Gamma_r)^2 + \Gamma_i^2} \qquad \textbf{(43)}$$

$$x = \frac{2\Gamma_i}{(1 - \Gamma_r)^2 + \Gamma_i^2} \qquad \textbf{(44)}$$

Rearranging terms in equations (43) and (44) leads to:

$$\left[\Gamma_r - \frac{r}{1 + r}\right]^2 + \Gamma_i^2 = \left[\frac{r}{1 + r}\right]^2 \qquad \textbf{(45)}$$

$$\left[\Gamma_r - 1\right]^2 + \left[\Gamma_i - \frac{1}{x}\right]^2 = \left[\frac{1}{x}\right]^2 \qquad \textbf{(46)}$$

Each of the equations (45) and (46) is similar to:

$$[x - h]^2 + [y - k]^2 = a^2 \qquad \textbf{(47)}$$

which is the general equation of circle of radius a and centered at (h, k).

Thus equation (45) is an r-circle (resistance circle) with

$$\begin{aligned} &\text{center at } (\Gamma_r\ ,\ \Gamma_i) = \left(\frac{r}{1 + r}\ ,\ 0 \right) \\ &\text{radius} = \frac{1}{1 + r} \end{aligned} \qquad \mathbf{(48)}$$

Similarly, equation (46) is an x-circle (reactance circle) with

$$\begin{aligned} &\text{center at } (\Gamma_r\ ,\ \Gamma_i) = \left(1\ ,\ \frac{1}{x} \right) \\ &\text{radius} = \frac{1}{x} \end{aligned} \qquad \mathbf{(49)}$$

Smith chart is the superimposition of r-circles and x-circles.

2.3.3.3.3 Transmission line method

This method involves placing a sample of material inside an enclosed transmission line. The transmission line may be either rectangular or coaxial. The sample must precisely fill the cross-section of the transmission line. A source in the network analyzer sends a microwave signal down the line. Once the signal hits the MUT in the transmission line, the analyzer measures both the energy reflection and transmission through the material. A computer program calculates the dielectric constant (ε') and dielectric loss factor (ε'') from these parameters. This method is more accurate and sensitive than the probe method. However, it provides results over a narrower range of frequencies. If the MUT do not fill in the cross section of the transmission line, then this method is difficult and time consuming.

2.3.3.3.4 Resonant cavity method

Cavities are tuned chambers which resonate like a bell at some ideal microwave frequency. Standing waves can exist in the cavities at the resonant frequencies. When a small sample of MUT is placed inside a cavity, the amount of microwave energy absorbed and the frequency of resonance change. The network analyzer can detect these changes. A computer program calculates the dielectric constant (ε') and dielectric loss factor (ε'') from these changes. This method is very accurate and sensitive to very low values of ε''. Another advantage of this method is its suitability to heterogeneous and larger sample. However, it provides the measurement at only one frequency.

2.3.3.4 Dielectric properties of food materials

Nelson *et al*. (1993) took measurements on fresh-cut tissue samples of 23 different fruits and vegetables to obtain dielectric properties data. The dielectric properties were measured by an open-ended, 3.6 mm diameter, semirigid, teflon-insulated coaxial line with copper conductors, connected to a microwave network analyzer (Model 8510B, Hewlett-Packard, Palo Alto, CA). The material in contact with the probe influences the reflection coefficient at the dielectric interface. This reflection coefficient is measured by the network analyzer. A Dielectric probe kit software (Model 85070A, Hewlett-Packard, Palo Alto, CA) was used with the network analyzer and a microcomputer (Model 98580A 310, Hewlett-Packard, Palo Alto, CA) to obtain the dielectric properties of the material. The dielectric measurements were taken by the network analyzer at 41 frequencies beginning at 200 MHz and ending at 20 GHz. Density, moisture content, and soluble solids content were also measured for the samples. Dielectric constant (ε') of fruits and vegetables of similar moisture content appears to be similar. The correlation between the dielectric constant and the moisture content is better at 20 GHz than at 200 MHz. This might be due to the disappearance of ionic conductivity influence above 10 GHz. Dielectric loss factor (ε'') decreases as the frequency increases from 200 MHz, reaches a minimum in the frequency range of 1 to 3 GHz, and then increases as the frequency approaches 20 GHz. The loss tangent ($\tan \delta = \varepsilon''/\varepsilon'$) decreases as the frequency increases from 200 MHz and then increases approaching unity around 20 GHz. There are no evident correlation between the dielectric properties and tissue density or soluble solid content for all of the fruits and vegetables.

Funebo and Ohlsson (1999) measured dielectric properties over a range of moisture and temperature. The dielectric properties of apple, chervil, mushroom, Parsley, and strawberry were measured. The measurements were performed with a cylindrical TM_{012} resonant cavity method designed for dielectric measurements at 2,800 Hz.. Pressure tubes of quartz glass (5 mm diameter, 80 mm long, and a volume of 2 ml) were filled with food samples. The tubes were inserted in the cylindrical cavity one at a time. This changes the electric field inside the cavity. The resonance frequency shift gives the dielectric constant (ε') and loss in strength of the transmitted signal gives the dielectric loss factor (ε''). The signal characteristics were recorded by a network analyzer (Model 5411A, Wiltron, Texas). A peak for dielectric loss factor was observed at intermediate moisture levels for all the five fruits and vegetables. There was a continuous decrease in dielectric constant with a decrease in moisture. A decrease in moisture content is expected to reduce the relaxation frequency because of a decrease in the availability of free water. The relaxation frequency (f_r) is defined as:

$$f_r \text{ ' } 2\pi\tau \text{ where, } \tau \text{ is the relaxation time} \tag{50}$$

The relaxation time (τ) is the time required by an agitated molecule to relax back to 37% of the normal state of the molecule. Peak value of dielectric loss factor is found at a point where the relaxation frequency and the operating frequency are close to each other. In the region where the dielectric loss factor is maximum, the dielectric constant increases with a increase in temperature whereas the dielectric loss factor may increase or decrease with temperature depending on whether the operating frequency is lower or higher than the relaxation frequency. The dielectric constant decreases with an increase in temperature at high moisture content whereas it increases with an increase in temperature at intermediate moisture content. Temperature does not have significant effect at low moisture contents (0.05-0.25 kg/kg on a dry basis). This could be explained by the binding of water and salts at low concentrations of water. Apple does not behave like strawberry though they have a similar low molecular sugar content. This could be explained by the stabilization of water molecules by hydrogen bonding with the pectic substances of apple.

Dielectric properties of materials influence interaction between microwave energy and the materials. The dielectric constant of hard red winter wheat decreased with increase in frequency whereas the dielectric loss factor showed very little dependence on frequency in the frequency range of 1 to 5.5 GHz. The dielectric constant of shelled, yellow-dent field corn varied linearly with natural bulk density over a narrow range of 0.55 to 0.75 g/cm^3. Non-linear behavior has been noted for bulk density greater than 0.75 g/cm^3 (Nelson, 1994).

Sharma and Prasad (2002) determined dielectric properties of garlic using cavity perturbation method (Model 5410B, Santa Clara, Palo Alto, CA) at 2,450 MHz as function of temperature and moisture content. The dielectric constant increased linearly from 2.2 to 54.2 at 35 °C as the moisture content increased from 6% (dry basis) to 185% (dry basis).The loss factor increased logarithmically from 0.82 to 8.5 at 35 °C as the moisture content increased from 6% (dry basis) to 185% (dry basis). The dielectric constant and the loss factor decreased linearly with an increase in temperature at moisture contents greater than 33% (dry basis) whereas it increased linearly at low moisture (6% dry basis).

Dielectric properties of various other food materials have been compiled in literature (Ohlsson and Bengtsson, 1975; Ryynanen, 1995; Nelson and Datta, 2001; Sipahioglu and Barringer, 2003; Guan *et al.*, 2004; Datta *et al.*, 2005; Nunes *et al.*, 2006).

2.3.4 Governing equations for microwave heating

Distribution and intensity of the electromagnetic field are the main factors that determine absorption of microwave energy in a food material. Heating of a material by microwaves is governed by the Maxwell's equations and the energy equation.

2.3.4.1 Maxwell's equation

Maxwell's equations are the basic laws governing the propagation of electromagnetic waves. The differential form of Maxwell's equations can be expressed in the form of the following four equations (Saltiel and Datta, 1999):

$$\nabla \times \mathbf{H} = \frac{\partial \mathbf{D}}{\partial t} + \mathbf{J} \qquad \mathbf{(51)}$$

$$\nabla \times \mathbf{E} = -\frac{\partial \mathbf{B}}{\partial t} \qquad \textbf{(52)}$$

$$\nabla \cdot \mathbf{D} = \rho_c \qquad \textbf{(53)}$$

$$\nabla \cdot \mathbf{B} = 0 \qquad \textbf{(54)}$$

where, ρ_c is the electric charge density.

Poynting vector (S) is defined as the cross product of electric field intensity and magnetic field intensity (E × H). It is associated with the direction of power flow and is equal to the sum of magnetic power density, electric power density, and the dissipated power density. Dissipated microwave energy (Q) is converted into thermal energy in a processed material and is given by (Saltiel and Datta, 1999):

$$Q = \mathbf{E} \cdot (\sigma_e \mathbf{E}) = \sigma_e |E|^2 \quad \textbf{(55)}$$

Boundary conditions must be applied at the interface between materials to account for the discontinuities in charge and current densities. The boundary conditions at the interface between two materials are:

$$\varepsilon_1 \mathbf{E}_1^n = \varepsilon_2 \mathbf{E}_2^n \quad \textbf{(56)}$$

$$\mathbf{E}_1^t = \mathbf{E}_2^t \quad \mathbf{(57)}$$

$$\mu_1 \mathbf{H}_1^n = \mu_1 \mathbf{H}_2^n \quad \mathbf{(58)}$$

$$\mathbf{H}_1^t = \mathbf{H}_2^t \quad \mathbf{(59)}$$

where, the subscripts 1 and 2 refer to the values in the materials 1 and 2 respectively and the superscripts t and n refer to the tangential and normal components of the field. It can be concluded from these boundary conditions that an electric field parallel to the surface of the load (material) will produce a much larger field than a field of equal strength but normal to the surface (Dibben, 2001).

2.3.4.2 Lambert's law

The microwave power absorption can be modeled using Lambert's law as:

$$Q(z) = Q_0 e^{-2\alpha z} = Q_0 e^{-z/\delta_p} \qquad \mathbf{(60)}$$

where, δ_p, power penetration depth (distance in which the value of the microwave power absorption falls to e^{-1} of its value at the material surface), is one half the value of the skin depth (δ_s). Lambert's law is valid for plane waves penetrating a semi-infinite slab. This assumption can be used if the load is a slab of thickness greater than a critical thickness $L_{crit,}$ given by the following equation (Ayappa *et al.*, 1991a):

$$L_{crit} = 5.4\,\delta_p - 0.08 \qquad \textbf{(61)}$$

where, both L_{crit}, δ_p and are in meters. If the thickness of the slab is below the critical thickness, Lambert's law is not applicable as the reflections from the far end are significant. The microwave radiation focuses near the center in cylindrical samples. Therefore, R_{crit} for cylindrical samples is more than that of L_{crit} and is given by the following equation (Oliveira and Franca, 2002):

$$R_{crit} = 7.03\,\delta_p - 0.0001 \qquad \textbf{(62)}$$

2.3.4.3 Energy equation

Materials are heated by microwaves due to coupling of the electromagnetic power distribution with the transport of heat due to conduction, convection, and radiation. This coupling is complicated by the temperature dependence of the electrical properties. The density of microwave power absorption (Q) is related to the electric and magnetic fields inside a material (Metaxas and Meredith, 1983):

$$Q = \omega\varepsilon_0\varepsilon_r|\mathbf{E}|^2 + \omega\mu_0\mu_r|\mathbf{H}|^2 \qquad \textbf{(63)}$$

Assuming no magnetic losses and using the root mean square value of the electric field intensity, equation (63) can be simplified to

$$Q = \omega \varepsilon_0 \varepsilon_r E_{rms}^2 = \sigma_e E_{rms}^2 \qquad \textbf{(64)}$$

If the load is a solid or an incompressible fluid, the energy transport equation may be written in terms of the local temperature, T, and Q (Saltiel and Datta, 1999)

$$\rho c_p \frac{\partial T}{\partial t} + \rho c_p v \cdot \nabla T = \nabla \cdot k \nabla T + Q \qquad \textbf{(65)}$$

where, ρ is density of the material in kg/m^3

c_p is specific heat capacity at constant pressure of the material in J/kg-K

v is velocity of the material in m/s

k is thermal conductivity of the material in W/m-K

The first term in the LHS of equation (65) is the rate of energy accumulation and the second term is convective energy flow. The first term in the RHS is the diffusive energy and the second term is the microwave power absorbed.

The generalized boundary condition can be written as (Saltiel and Datta, 1999)

$$-k \frac{\partial T}{\partial n} = h_c (T - T_\infty) + \sigma_{rad} \varepsilon_{rad} T^4 - \alpha_a q_{rad} + m_w h_{fg} \qquad \textbf{(66)}$$

where, T is the load surface temperature, n represents the normal to the surface, h_c is the convective heat transfer coefficient, σ_{rad} is the Stefan-Boltzmann constant, ε_{rad} is the radiative surface emissivity of the load, α_a is the surface absorptivity of the load, q_{rad} is the incident radiant heat flow rate per unit surface area to the load, and m_w and h_{fg} are the rate of evaporation and the latent heat of vaporization of the evaporated liquid, respectively.

2.3.5 Applications of microwave heating

Industrial applications of microwaves in food processing started with the development of continuous conveyor ovens in 1960s. The important food processes where microwave has been applied include tempering, drying, cooking, enzyme inactivation**,** baking, sterilization, and pasteurization (Bengtsson and Ohlsson, 1973; Giese, 1992). The primary objective in the present work is the applications of microwaves in sterilization and pasteurization of food products.

2.3.5.1 Batch heating

Copson (1954) studied the use of microwave energy to pasteurize orange juice. Pectin methylesterase (PME) was inactivated in orange juice concentrate at 66 EC with a 700 W microwave oven operating at 2,450 MHz.

Knutson *et al.* (1988) simulated high-temperature short-time (HTST, 71.7 EC for 15 s) and low-temperature long-time (LTLT, 62.8 EC for 30 s) pasteurization in a microwave oven (700 W, 2,450 MHz). Results showed that microwave heating caused a non-uniform distribution of heat in milk and failed to inactivate the cells of *Salmonella typhimurium, Escherichia coli, and Pseudomonas fluorescens.* Thompson and Thompson (1990) used a domestic microwave oven (2,450 MHz) to pasteurize goat's milk. The phosphatase test, which indicates pasteurization of milk, was not performed as the phosphatase of goat's milk is more heat sensitive than the phosphatase of cow's milk. They showed a reduction in the aerobic plate count in raw goat's milk by up to 6 log cycles without impairing the organoleptic quality. However, they did not undertake the test with *Coexiella burnetii* which is used as the basis for milk pasteurization.

Canumir *et al.* (2002) determined the effect of different microwave power levels (270-900 W) on the pasteurization of apple juice using a 2,450 MHz microwave oven. Pasteurization of apple juice resulted in a 2-4 log cycles reduction which showed that there were no significant differences between conventional and microwave pasteurization. The results also showed that there was no nonthermal effect of microwaves on microorganisms.

Gerald and Roberts (2004) evaluated the effect of microwave heat treatment of apple mash on juice yield, quality, and content of total phenolics and flavonoids in the juice. The microwave oven they used had variable power from 0 to 2.5 kW at 2,450 MHz. Results showed that microwave heating of apple mash before juice extraction resulted in high quality juice with increased phenolic and flavonoid content and juice yield.

Diaz-Cinco and martinelli (1991) established a method of sterilization using a microwave oven (700 W, 2,450 MHz) and compared this with conventional heating in a water bath and autoclave at 121 EC for 15 min. The results showed that microwaves produced only a thermal effect on the microorganisms.

2.3.5.2 Continuous heating

Jaynes (1974) demonstrated that microwave energy can be used for continuous pasteurization of milk in a system similar to a conventional HTST equipment. A variable power (0-1,500 W) microwave generator (Model H1-1200, Holaday Industries, MN) was used. The pasteurization system consisted of a teflon tube (0.635×10^{-2} m ID and 0.12 m long) placed across a waveguide. A regenerator section consisted of two glass condensers connected in series with the teflon tube. Incoming cold milk flowing through the core of the condenser was heated by the hot milk flowing through jackets in a counter-flow manner.

Milk was pasteurized at three flow rates (200, 300, and 400 ml/min). Phosphatase test was negative at all three flow rates. Standard plate count and coliform count reductions at all three flow rates showed adequate pasteurization of the milk.

Nikdel *et al.* (1993) studied pasteurization of orange juice with microwave energy. A 2,450 MHz and 600 W microwave oven was modified for continuous flow treatment of orange juice. Microwave heating of orange juice at 70 EC for 15 s resulted in 6 log cycles reduction in *Lactobacillus plantarum* which was better than the reduction by conventional pasteurization (90.5 EC with 15 s residence time) using a plate heat exchanger. PME inactivation in microwave pasteurization was between 98.5 and 99.5% at temperature greater than 75 EC with 10-15 s residence time which compared to 99% inactivation by conventional pasteurization. Tajchakavit and Ramaswamy (1997) compared kinetics of PME inactivation in orange juice under microwave heating and conventional heating. It was shown that there was enhanced inactivation of PME during microwave heating in a microwave oven (700 W, 2,450 MHz) as compared to conventional heating.

Tajchakavit *et al.* (1998) evaluated the destruction kinetics of *Saccharomyces cerevisiae* and *Lactobacillus plantarum* in apple juice under continuous flow microwave heating and conventional batch heating. Apple juice was inoculated with the cultures of the microorganisms and heated in a microwave oven (700 W, 2,450 MHz) under continuous flow conditions to the temperatures of 52.5 to 65 EC. Inoculated apple juice was subjected to batch treatment in a well-stirred water bath to temperatures of 50 to 80 EC. The range of temperatures were chosen to study the destruction kinetics during both the microwave and conventional heating. Microbial destruction occurred much faster under microwave heating than under thermal heating which suggested some enhanced destruction of microorganisms to be associated with microwave heating.

Coronel *et al.* (2003) studied heating of milk with different fat contents in a specially designed focused applicator operating at 915 MHz with 5 kW as the power output. There was uniform temperature distribution in the cross-sectional area of the tube at the exit of the applicator. The study also showed that higher temperature was achieved at the center of the tube as compared to the temperature achieved close to the walls of the tube.

Gentry and Roberts (2005) designed a laboratory scale continuous flow microwave system for pasteurization of apple cider. Water, apple cider from cold press extraction, and apple cider from hot press extraction, were used to study the heating characteristics. The microwave pasteurization system consisted of helical coils throughout a large cavity oven. This system was shown to produce uniform heating throughout the cavity and was evaluated based on the input power, volume load size, and inlet temperature. There was a 5.2 ± 0.10 log reduction in *E. coli 25922* Heat loss to the ambient air was calculated and it was up to 20% of the power available to the system.

Coronel *et al.* (2005) reported aseptic processing of sweetpotato puree using a 60 kW continuous flow microwave system operating at 915 MHz. A shelf stable product with no detectable microbial count during a 90 day storage period at room temperature was produced. The resulting product packed in flexible plastic containers had the color and apparent viscosity comparable to the untreated puree.

2.4 Biological validation of an aseptic process

Biological validation for traditionally processed canned food products is not required if the process is based on properly designed heat penetration tests. For aseptically processed multiphase food product, biological validation is necessary due to the unavailability of the time and temperature history of the particles (Chandrana, 1992). One of the elements that FDA identified for a food processor to address during filing of a low-acid multiphase food product is quantitative biological validation of the lethality delivered (Dignan *et al.*, 1989). Biological validation of an aseptic process for a low-acid multiphase food product requires use of a bioindicator that can be processed in an aseptic system, retrieved intact, and tested quantitatively for received lethality. An acceptable quantitative biological validation verifies that the target F_0 has been delivered to the critical point. A biological thermocouple system in which suspended spores are encapsulated in a carrier has been used successfully with canned products (Pflug and Smith, 1977; Pflug *et al.*, 1980; Marcy, 1997).

Simulated particles inoculated with bacterial spores have been used for biological validation. In contrast to real food particles, simulated particles normally have uniform size and spore concentration. Hunter (1972) embedded spores in polymethacrylate beads and applied thermal treatment to them. Apart from being time consuming, this process required acetone (deleterious to the heat-injured spores) for recovery of spores. Dallyn *et al.* (1977) suspended spores in alginate beads. This approach was easy for preparing the particles and recovering the spores. However, the particle size used in this study was small. This prevented its applicability to large particulate food products. Brown *et al.* (1984) achieved large particle size by embedding the spores in cubes formed from alginate mixed with potato, peas, or meat purees. The disadvantage of this approach was that the particles were not shelf-stable and they reduced in size during processing. The reduction in size of the particle resulted in significant differences between the calculated and predicted lethality of the particle. Sastry *et al.* (1988) developed a bioindicator with a large particle size by suspending spores in an alginate solution, infusing them into mushrooms, and immobilizing the spores by gelation of the alginate. The particles were made shelf-stabile by freeze drying and they were rehydrated before use. The disadvantage associated with this was the large inherent spore population in mushrooms which made quantification of infused spores difficult to determine. Bhamidipati and Singh (1996) used alginate spheres as model food particles and reported that the thermal response of the simulated particles were similar to those of the real food particles.

Workshops organized by CAPPS and NCFST in 1995 and 1996 assessed the status of biological validation of low-acid multiphase food products and produced a standard operating protocol for biological validation of an aseptic process. It was concluded that the test microorganism should have stable thermal characteristics with a D value which is not too low (to ensure that there are survivors) and not too high (to eliminate improbable results in count reduction). The D and z values should be determined in the media of incubation at temperatures associated with the aseptic process. Viability of the test microorganism in the incubation media should be established. It was further concluded that biological validation of a multiphase aseptic process should be done by demonstrating the effect of thermal processing on the critical particle. Two inoculation methods were discussed -- count reduction and inoculated pack methods. In the count reduction method, a known number of microorganisms are implanted into the center of a food particle. After the particle passes through the aseptic processing system, the lethality of the process is established by determining the number of surviving spores. In this method, additional labor to collect inoculated particles and enumerate the survivors is required. In the inoculated pack method, relatively larger number of particles are inoculated to ensure that an inoculated particle is in the containers to be incubated. The disadvantage of this method is the longer times required for incubation (Marcy, 1997).

The amount of heat received by a simulated particle is crucial for the biological validation of a multiphase aseptic process. A technique was developed by Jasrotia (2004) to design simulated particles with conservative behavior. Conservative behavior of simulated particles ensures that the particle heats up the slowest (due to its low thermal diffusivity) and receives least thermal treatment (because it is the fastest). These conservative particles are very important in understanding a multiphase aseptic process because they represent the real food particles in an aseptic system which receive least thermal treatment. Thus, if we ensure that the conservative particle receives the minimum required thermal treatment, we would ensure that all the particles in the processed food product receive a thermal treatment equal to or greater than the conservative particle. This will ensure the safety of the product. Systematic procedures for design, fabrication, and testing of conservative particles that would be used for biological validation of multiphase aseptic processing were developed by Jasrotia (2004).

2.4.1 Thermophysical properties of food materials

Thermophysical properties include density, viscosity, specific heat, thermal conductivity, and thermal diffusivity. They have a significant effect on the rate of heat transfer into the particles of a food product. Study of thermophysical properties of the food product and its particulate ingredients plays an important role in the design of conservative particles.

2.4.1.1 Density

Density (ρ) is defined as mass per unit volume with the units of kg/m^3. Materials with more compact molecular arrangement have higher densities. Specific gravity is defined as the ratio of density of a material to density of water at the same temperature. There are three types of densities for food materials -- solid density, particle density, and bulk density. Solid density does not take into account the internal pores in the food materials. Particle density is defined as the ratio of the actual mass of a particle to its actual volume. Particle density accounts for the internal pores in the food materials. Bulk density is defined as the mass of the food material per unit volume. Bulk density accounts for the void spaces between the particles. The void space in food materials is described by porosity which is the ratio of the void volume to the total volume of the material.

2.4.1.1.1 Measurement of density

a. Geometric dimension method -- The apparent density of a regular shape material can be determined by calculating the volume from characteristic dimensions and measuring the mass of the material. However, this method is not suitable for soft and irregularly shaped materials due to the difficulty in measuring the characteristic dimensions (Rahman, 1995).

b. Buoyant force method -- In this method, buoyant force (G_B, in kg) is determined by measuring the weight of the material in air and liquid. The apparent density (ρ_a) of the material is calculated as:

$$\rho_a = \frac{\rho_w m}{G_B} \qquad \textbf{(67)}$$

where, ρ_w is the is the density of the liquid and m is the mass of the material in air. Two errors are associated with this method. The first is due to mass transfer from the material to the liquid. This can be avoided by enclosing the material in cellophane or coating it with a thin layer of polyurathane varnish or wax. The second error may be due to partial floating of the sample. This error can be avoided by using a liquid with a density lower than that of the material (Rahman, 2005).

c. Hydrometer -- Densities of liquids can be measured by a hand hydrometer which gives the specific gravity. A weighted float is attached to a small diameter stem containing a scale of specific gravity values. The float sinks into the unknown liquid by an amount proportional to specific gravity and resulting liquid level is read on the stem scale (Singh and Heldman, 2001).

d. Liquid displacement method -- The volume of a sample can be measured directly by the volume of liquid displaced by the test material. The sample should be coated so that liquid does not penetrate in the pores. A non-wetting liquid such as mercury should be used if the sample is not coated.

e. Gas pycnometer -- Volume of a sample can be measured by a gas pycnometer which use gases such as air, nitrogen, and helium. The sample is placed in tank 2 with an empty volume of V_1 and air is supplied to tank 1 when valve 2 is closed. When suitable manometer displacement is achieved, valve 1 is closed and equilibrium pressure P_1 is read. Valve 3 is then closed and valve 2 is opened and pressure P_3 is read. Volume (V_s) of the sample in tank 2 is determined based on the ideal gas law as (Rahman, 2005):

$$V_s = V_1 + V_1 \times \frac{(P_3 - P_1)}{P_3} \qquad \textbf{(68)}$$

Commercial automatic helium gas pycnometers are also available to measure the volume of the samples (figure 7). A sample of volume V_s is placed in a sample cell with volume V_{sc} and pressure P_1 is applied to the sample cell. The gas is allowed to pass through an expansion cell with volume V_{ec} by opening valve 2 (V2). The pressure of the gas decreases to P_2 due to expansion of the gas. The volume of the sample is determined based on the ideal gas law as (Rahman, 2005):

$$V_s = V_{sc} + V_{ec} \times \frac{1}{1 - \frac{P_1}{P_2}} \qquad \textbf{(69)}$$

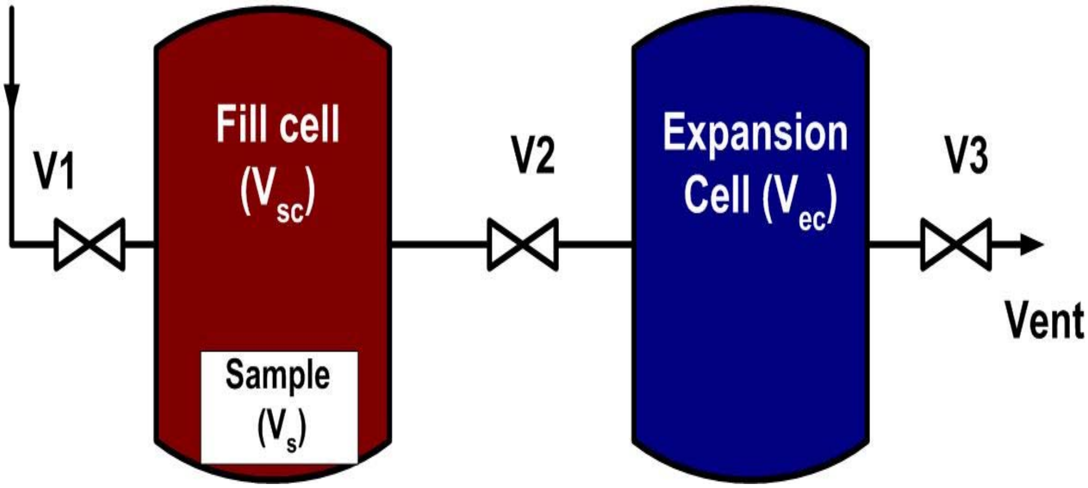


Figure 7. Schematic of a helium gas pycnometer

Apart from the above given experimental methods, density can also be determined by using predictive equations. Predictive equations to determine densities of milk and dairy products, fruits and vegetables, and meat and fish have been compiled by Rahman (2005).

2.4.1.2 Viscosity

Viscosity (μ) is the fluid property which denotes the resistance between the internal layers of a fluid to movement. It is also called absolute or dynamic viscosity. Its unit is Pa-s (N-s/m^2) or poise (dyne-s/cm^2) and is given by:

$$\mu = \frac{\sigma}{\dot{\gamma}} \qquad \textbf{(70)}$$

where, σ is the shear stress and $\dot{\gamma}$ is the shear rate. Force per unit are is defined as stress and when the force is applied parallel to the surface, the stress is called shear stress. Shear rate is the relative change in velocity divided by the distance. Liquids that follow the above equation exhibit a direct proportinality between shear rate and shear stress and are called Newtonian fluids. Examples of Newtonian fluids include water, fluid milk, honey, and fruit juices.

Liquids that do not have a direct proportionality between shear rate and shear stress are known as non-Newtonian fluids. The properties of non-Newtonian fluids can be classified as time-independent and time-dependent (figure 8).

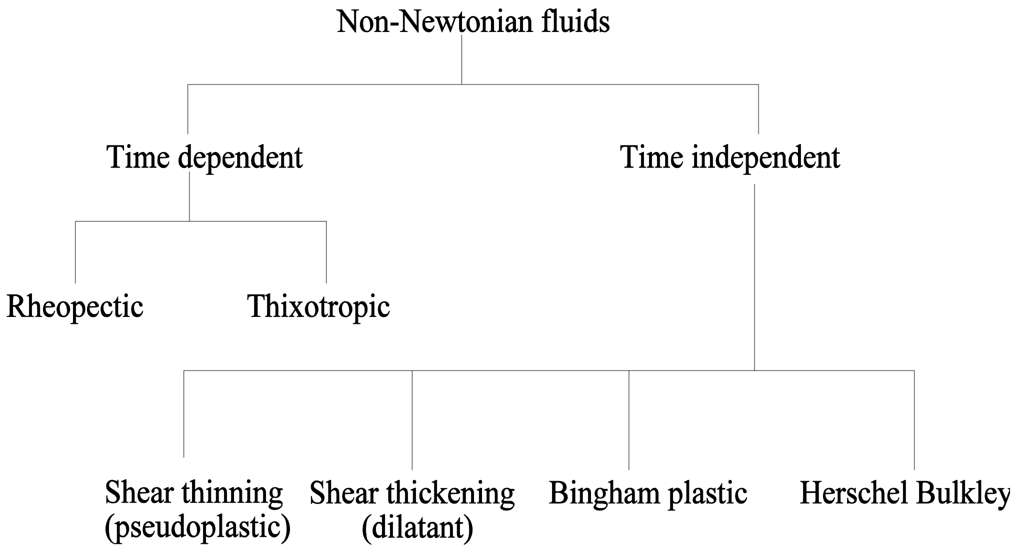


Figure 8. Classification of non-Newtonian fluids

Relationship between shear stress and shear rate for Newtonian and non-Newtonian fluids is shown in figure 9. Time-independent non-Newtonian fluids respond immediately with a flow as soon as a small amount of shear stress is applied. Two important types of time-independent non-Newtonian fluids are shear thinning (pseudoplastic) and shear thickening (dilatant). The difference between these two can be understood on the basis of apparent viscosity. An apparent viscosity is calculated by assuming that the non-Newtonian fluid obeys Newton's law of viscosity at a selected shear rate. The slope of the straight line from origin to a point on the curve at a given shear rate gives the value of apparent viscosity. For a shear thinning fluid, the apparent viscosity decreases as the shear rate increases. Some examples of shear thinning fluids are condensed milk, fruit purees, mayonnaise, mustard, and vegetable soups. For a shear thickening fluid, the apparent viscosity increases as the shear rate increases. Some examples of shear thickening fluids are 40% raw corn starch and some types of honey.

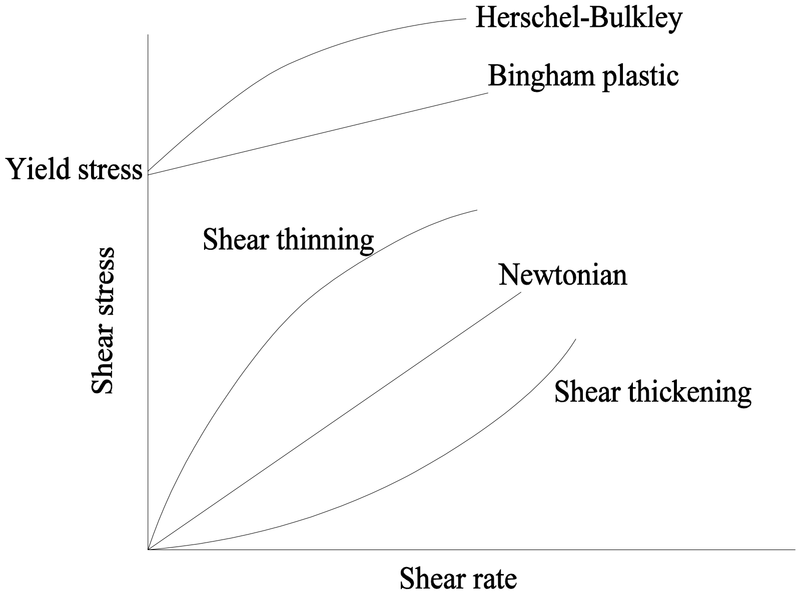


Figure 9. Relationship between shear stress and shear rate for Newtonian and non-Newtonian fluids

Another class of non-Newtonian fluids requires an application of a stress prior to any flow. This stress is known as the yield stress (σ_0). After the application of yield stress, those fluids which behave like a Newtonian fluid are known as Bingham plastic. If the fluids behave as shear thinning fluids, then they are known as Herschel-Bulkley fluids. The Herschel-Bulkley model, the most common mathematical model used to describe Newtonian and non-Newtonian fluids, is given as (Singh and Heldman, 2001):

$$\sigma = \sigma_0 + K\dot{\gamma}^n \qquad \textbf{(71)}$$

where, K is the consistency coefficient and n is the flow behavior index.

Time-dependent non-Newtonian fluids attain a constant value of apparent viscosity only after some time has elapsed after the application of shear stress. The fluid is known as thixotropic if its viscosity decreases over time and rheopectic if the viscosity increases over time.

Kinematic viscosity (ν) is an alternative term used to express viscosity**.** It is related to dynamic viscosity as:

$$\nu = \frac{\mu}{\rho} \qquad \textbf{(72)}$$

The unit of kinematic viscosity is m^2/s.

2.4.1.2.1 Measurement of viscosity

The viscosity of a fluid can be measured by many different methods, but the capillary tube and the rotational viscometers are the most common instruments used to measure viscosity.

a. Capillary tube viscometer -- In a capillary tube viscometer, fluid flows at a given volumetric flow rate (Q) through a tube of length L and radius R. Measurement of pressure drop across the tube allows the determination of viscosity as (Singh and Heldman, 2001):

$$\mu = \frac{\pi \Delta P R^4}{8 L Q} \qquad \mathbf{(73)}$$

This method is valid only for a Newtonian fluid.

b. Rotational viscometer -- A rotational viscometer consists of a coaxial-cylinder viscometer with fluid between the inner and outer cylinders. The principle involves measurement of torque (Ω) required to turn the inner cylinder at a given speed. Viscosity is calculated using the following expression (Singh and Heldman, 2001):

$$\mu = \frac{\Omega}{8\pi^2 N L}\left(\frac{1}{R_i^2 - R_o^2}\right) \qquad \mathbf{(74)}$$

where, N is the revolutions per second, R_i is the inner cylinder radius, R_o is the outer cylinder radius, and Ω is the torque in N-m.

2.4.1.3 Specific heat

Specific heat (c_p) is the amount of heat that is gained or lost by a unit mass of product for a unit change in temperature without changing state and is given by the following equation:

$$c_p = \frac{Q}{m(\Delta T)} \qquad \mathbf{(75)}$$

where, Q is the heat gained or lost (kJ), m is the mass (kg), ΔT is the temperature change (EC), and c_p is the specific heat (kJ/kg-K). For processes where a change of state takes place (freezing or thawing), an apparent specific heat is used. Specific heat plays an important role in the thermal analysis of food processing equipment used in heating or cooling of foods. Comprehensive databases (Singh, 1994), predictive equations, or experimental methods are generally used to obtain the values of specific heat.

Siebel (1892) proposed one of the earliest models to calculate specific heat as:

$$c_p = 0.837 + 3.349X_w \qquad \textbf{(76)}$$

where, X_w is the fraction of water content. This model does not take into consideration the effect of various food components and temperature. Charm (1978) proposed an empirical equation which considers various food components such as water, fat, and nonfat solids and is given as:

$$c_p = 2.093X_f + 1.256X_s + 4.187X_w \qquad \textbf{(77)}$$

where, X is the mass fraction and subscripts f, s, and w denote fat, non-fat solids, and water respectively. Heldman and Singh (1981) proposed the following expression for specific heat based on the components of a food product:

$${}_p = 1.424X_c + 1.549X_p + 1.675X_f + 0.837X_a + 4.187X \qquad \textbf{(78)}$$

where, X is the mass fraction and subscripts c, p, f, a, and w denote carbohydrate, protein, fat, ash, and water respectively.

None of the above predictive equations include a dependence on temperature even though specific heat is a function of temperature. Choi and Okos (1986) proposed the following model to predict specific heat based on composition and temperature:

$$c_p = \sum c_{pi} X_i \qquad \textbf{(79)}$$

where, X_i is the fraction of *i*th component and c_{pi} is the specific heat of the *i*th component as a function of temperature.

2.4.1.3.1 Measurement of specific heat

a. Mixing Calorimeter -- The basic principle involves mixing a known quantity of liquid at a known initial temperature with a known quantity of sample at a known temperature within an insulated container (calorimeter). Specific heat of the sample is calculated by an energy balance. However, this method does not allow measurement of specific heat as a function of temperature because the sample undergoes a temperature change from its initial value to that at equilibrium. Sources of error include energy loss or gain from the calorimeter and the thermal capacity of the calorimeter itself (Sastry and Cornelius, 2002).

b. Differential scanning calorimeter (DSC) -- DSC is the preferred method for the measurement of specific heat because it allows measurement as a function of temperature and it considers phase transition. The schematic representation of a DSC is shown in figure 10.

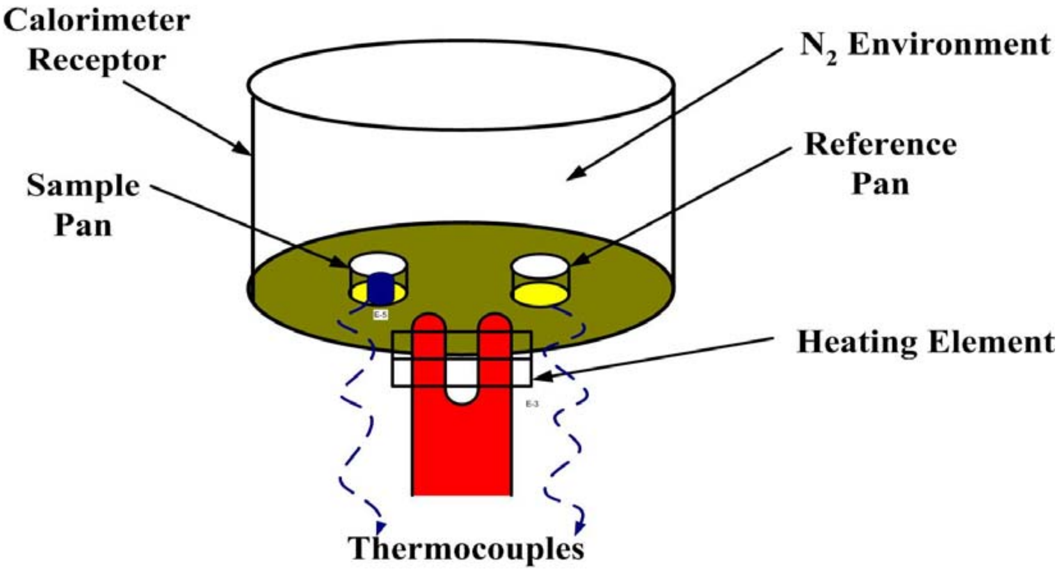


Figure 10. Schematic representation of a differential scanning calorimeter (DSC)

The first step in a DSC measurement is the establishment of a baseline which corresponds to an experimental run with an empty sample pan. Then, a small quantity of sample is placed in a container (sample pan) and an inert material of known heat capacity ($C = m\,c_p$) is placed in another similar container (reference pan). Both pans are placed inside a calorimeter receptor. A heating element is used to heat the sample pan at a constant rate of temperature rise, set to match the temperature of the reference pan. The result is a thermogram which gives the rate of heat input versus temperature. The deflection (d) of the thermogram from the baseline is proportional to the differential rate of energy input (dH/dt) required to maintain the sample at the temperature as of the reference pan (Mohsenin, 1970) and is given by:

$$d \;'\; \beta \frac{dH}{dt} \qquad \textbf{(80)}$$

where, β is a proportionality constant.

Temperature is linearly related to time because the rate of temperature rise is constant. Therefore, equation 80 can be written as:

$$d = \beta \frac{dH}{dT} \frac{dT}{dt} \quad \textbf{(81)}$$

The specific heat of the sample (c_p) is given by:

$$c_p = \frac{1}{m} \frac{dH}{dT} \quad \textbf{(82)}$$

From equations (81) and (82), deflection can be expressed as:

$$d = \beta m c_p \frac{dT}{dt} \quad \textbf{(83)}$$

A similar expression for a reference material of known specific heat can be written as:

$$d' = \beta' m' c_p' \frac{dT}{dt} \quad \textbf{(84)}$$

From equations (83) and (84), specific heat of the sample can be obtained as:

$$c_p = \left(\frac{d}{d'}\right)\left(\frac{m'}{m}\right) c_p' \quad \textbf{(85)}$$

One of the sources of error with this method is that the sample temperature is not actually measured. Another disadvantage is that due to very small sample size, the specific heat might not be representative for non-homogeneous materials (Sastry and Cornelius, 2002).

2.4.1.4 Thermal conductivity

Thermal conductivity (k) is the quantity of heat (Q) conducted per unit area (A) through a unit length (L) of material due to a unit temperature difference (ΔT) and is given by the equation:

$$k = \frac{QL}{A\Delta T} \qquad \textbf{(86)}$$

The units of thermal conductivity are W/m-K. Thermal conductivity of a food plays an important role in calculations involving rate of heat transfer by conduction. Thermal conductivities of water and air at 20 °C are 0.597 and 0.0251 W/m-K respectively (Singh and Heldman, 2001). Thermal conductivity of food materials is bounded by water and air on the upper and lower end respectively. Comprehensive databases (Singh, 1994, krokida *et al.*, 2001), predictive equations, or experimental methods are generally used to obtain the values of thermal conductivity.

Sweat (1974) proposed the following equation to predict thermal conductivity of fruits and vegetables with a water content of more than 60%:

$$k = 0.148 + 0.493 X_w \qquad \textbf{(87)}$$

where, X_w is the water content expressed as a fraction. Sweat (1975) proposed the following equation for meats and fish with a water content of 60-80% over a temperature range of 0 to 60 °C:

$$k = 0.08 + 0.52X_w \qquad \textbf{(88)}$$

Sweat (1986) developed another empirical equation for predicting the thermal conductivity of solid and liquid foods which is given below:

$$k = 0.25X_c + 0.155X_p + 0.16X_f + 0.135X_a + 0.58X_w \qquad \textbf{(89)}$$

where, X is the mass fraction, subscript c denotes carbohydrate, p denotes protein, f denotes fat, a denotes ash, and w denotes water.

None of the above predictive equations include a dependence on temperature even though thermal conductivity is a function of temperature. Choi and Okos (1986) developed the following expression that includes the influence of product composition and temperature:

$$k = \sum k_i X_i \qquad \textbf{(90)}$$

2.4.1.4.1 Measurement of thermal conductivity

a. Line heat source method -- In this method, a line heat source is inserted into a sample initially at a uniform temperature. The line source is then heated with a constant rate of heat input and temperature in close proximity to the heat source is measured and compared with the theoretical expression for temperature. A graph time (on a logarithmic scale) versus temperature is linear and the thermal conductivity of the sample is then calculated using the follwoing equation:

$$k = \frac{Q \ln\dfrac{(t_2 - t_0)}{(t_1 - t_0)}}{4\pi(T_2 - T_1)} \qquad \textbf{(91)}$$

where, Q is the power consumed by the probe heater in W/m, T_1 is the temperature of the probe thermocouple at time t_1 (s) in EC, T_2 is the temperature of the probe thermocouple at time t_2 (s) in EC, t_0 is a time correction factor in s.

The theory of this method has been discussed in detail by Hooper and Lepper (1950) and Nix *et al.* (1967). This method was developed as a thermal conductivity probe (figure 11) to measure thermal conductivity of food materials by Sweat and Haugh (1974). Only a small temperature rise is necessary to measure thermal conductivity. However, it can not be used with non-viscous liquids because convection currents develop around the heated probe (Sastry and Cornelius, 2002).

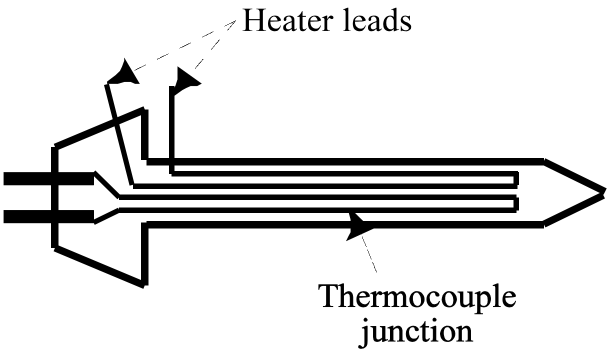


Figure 11. Cross-section of thermal conductivity probe

b. Modified Fitch method -- This method was originally developed for measuring thermal conductivity of materials with lower thermal conductivity by Fitch (1935). The device for this method consists of a heat source in the form of a vessel filled with a constant temperature liquid and a sink in the form of a copper plug insulated on all sides except one. The sample is sandwiched between the vessel and the uninsulated face of copper plug**.** The temperature of the copper plug changes depending on the rate of heat conduction through the material. The temperature change of the copper plug is recorded and used to calculate the thermal conductivity of the sample.

The Fitch apparatus was later modified by Bennet *et al.* (1962) and Zuritz *et al.* (1989). A one-dimensional analytical model was used to solve the heat transfer problem. The solution of the one-dimensional conduction equation through a slab with one face at constant temperature (in contact with vessel) and the other in contact with a perfect conductor (copper plug) is given as:

$$\ln\left(\frac{T_0 - T_\infty}{T - T_\infty}\right) = \frac{Akt}{m_c c_{pc}} \quad \textbf{(92)}$$

A graph of $\ln(T_0 - T_\infty)/(T - T_\infty)$ versus time (t) is a straight line and the thermal conductivity is calculated from the slope of the line. This method can be used to determine thermal conductivity of homogeneous particulate materials. However, This method in its present form can not be used to determine thermal conductivity under UHT conditions (Sastry and Cornelius, 2002).

Apart from the above mentioned methods, thermal conductivity of food materials has been measured by the differential scanning calorimetry method (Buhri and Singh, 1993), transient hot wire method (Shariaty-Nissar *et al.*, 2000), and point heat source method (Voudouris and Hayakawa (1994).

2.4.1.5 Thermal diffusivity

Thermal diffusivity (α) is the rate at which temperature at one point in a material is transmitted to another point. It is the ratio of thermal conductivity to volumetric heat capacity and is given as**:**

$$\alpha = \frac{k}{\rho c_p} \qquad \textbf{(93)}$$

The units of thermal diffusivity are m^2/s.

2.4.1.5.1 Measurement of thermal diffusivity

Thermal diffusivity can be calculated by determining the values of thermal conductivity, density, and specific heat. But, this approach requires considerable time and elaborate instrumentation.

Another approach is direct measurement of thermal diffusivity by experimental methods. The following methods have been most commonly used (Singh, 1982):

a. Least square estimation -- Temperature over a period of time is measured at the center of a well defined shape (infinite slab, infinite cylinder, or sphere). Temperature at various times at the same point is computed by solving the one-dimensional heat equation for some arbitrarily selected values of thermal diffusivity. The value of thermal diffusivity can be changed until the difference between the predicted temperature and the experimental temperature gives the minimum sum of squared deviations.

b. Use of time-temperature charts -- The analytical solutions of the governing partial differential equations for the one-dimensional heat equation have been used to obtain time-temperature charts (Schneider, 1963). The time-temperature charts contain dimensionless numbers such as Fourier number ($\alpha t/L^2$), Biot number (hd/k), and temperature ratio. The temperature ratio is experimentally determined for a particular location within an object at a known time. The time-temperature charts may be used to estimate the Fourier number. Thermal diffusivity can then be calculated from the Fourier number.

c. Use of analytical solutions -- The series solutions of the governing partial differential equations for the one-dimensional heat equation converges rapidly for a Fourier number great than 0.2. Experimentally determined temperature at a known location is used in the first term of the series solution to determine thermal diffusivity.

Several empirical models for predicting the thermal diffusivity of foods have also been proposed. Riedel (1967) proposed the following equation to predict thermal diffusivity as a function of water content:

$$\alpha = 0.088 \times 10^{-6} + (\alpha_w - 0.088 \times 10^{-6}) X_w \qquad \textbf{(94)}$$

where, α_w is the thermal diffusivity of water and X_w is the water content expressed as a percentage. Martens (1980) performed multiple regression analysis on 246 published values of thermal diffusivity of a variety of food products. The author found that temperature and water content are the major factors affecting thermal diffusivity whereas fat, protein, and carbohydrate content had a very small influence on thermal diffusivity. The following regression equation was obtained:

$$\alpha = 0.057363 X_w + 0.000288 (T + 273) \times 10^{-6} \qquad \textbf{(95)}$$

where, T is the temperature in °C.

2.4.2 Design of conservative particles

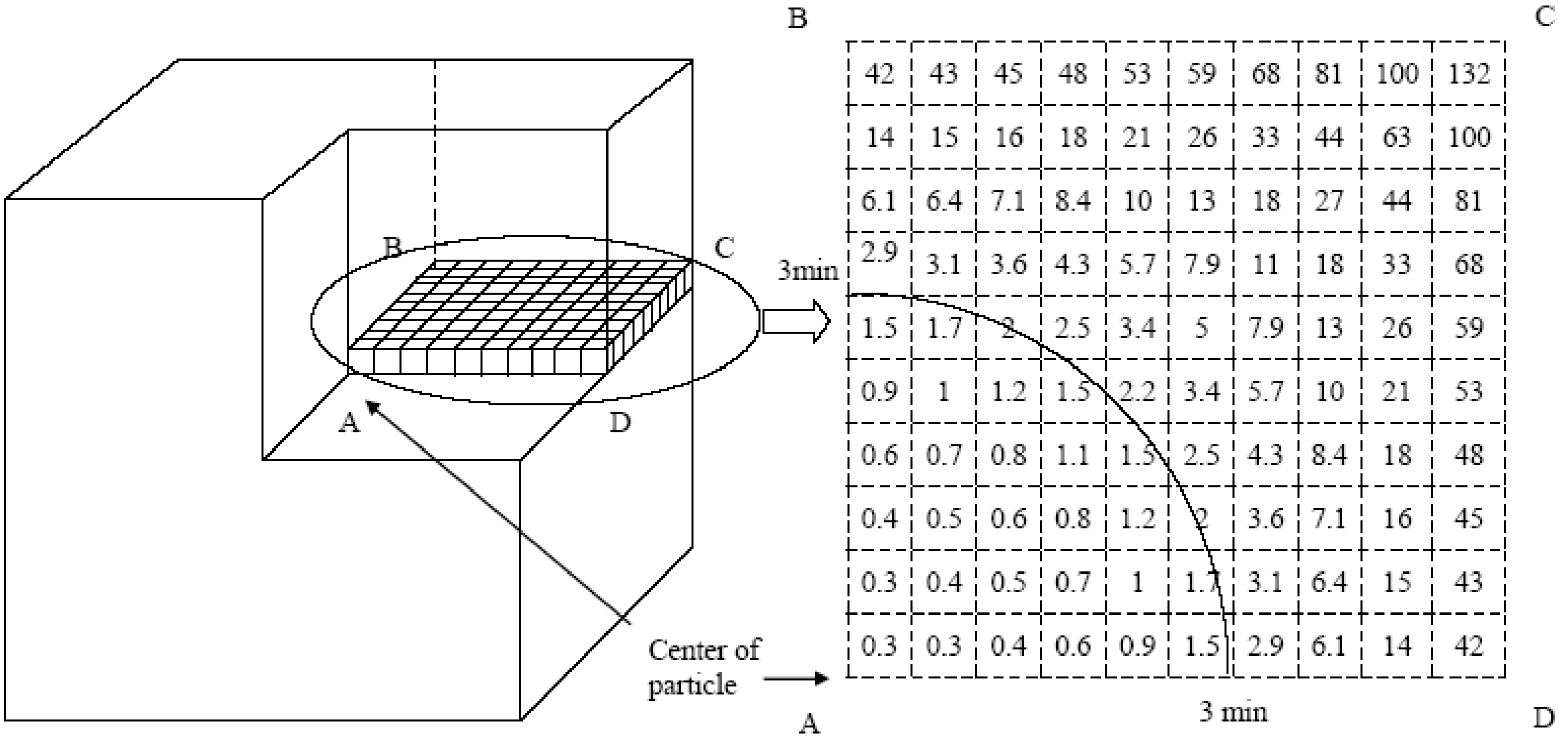
Simulated particles inoculated with bacterial spores have been used for biological validation of a multiphase aseptic process. In contrast to actual food particles, simulated particles normally have uniform size and inoculum distribution. The amount of heat received by a simulated particle is crucial for the biological validation of a multiphase aseptic process. A technique was developed by Jasrotia (2004) to design, fabricate, and test simulated particles with conservative behavior. Conservative behavior of a simulated particles ensures that the particle heats up the slowest (due to its low thermal diffusivity) and receives the least thermal treatment (because it is the fastest).

Design of simulated conservative particles was based on the Conservative Particle Design (CPD) software. The CPD software (Palazoglu *et al.*, 2004) was used to determine the design parameters (wall thickness and implant weight) for cubic, spherical, and cylindrical particles made of a polymer with thermal diffusivity lower than that of the food particles being studied. The CPD software simulates concurrent heating of a theoretical polymer particle and an identical theoretical food particle under identical sterilization-level conditions. For this, both particles are divided into grids. During these simulated sterilization treatment calculations, F_0 value accumulated in each grid is determined. Since the thermal diffusivity of polymer particles is lower than that of food particles, accumulation of F_0 values in the polymer particles will be slower.

Once the center of the food particle has reached the target F_0 (e.g. 3 minutes), F_0 of the concurrently heated polymer particle is examined to determine the points (depth) to which the penetration of target F_0 or lower has progressed under these identical heating conditions. All points deeper within the polymer particle beyond this level will therefore be thermally protected at least as well or better than the protection provided by the food particle to its geometric center. The depth of penetration of this pre-selected F_0 value into the particle establishes the most important design criterion -- particle wall thickness. A cavity is generated within each polymer particle that is supposed to be at least as equal or better thermally protected than the centers of the real food particles. Theoretically, under identical heating conditions, anything placed in the cavity will receive less thermal treatment than the center of an identical food particle.

In addition to being heated the slowest, conservative particle should also be the fastest flowing so that it receives the least thermal treatment. This is achieved by adjusting the effective density of the conservative particle to a value slightly lower than that of the carrier fluid density. This ensures that the conservative particle is the fastest flowing in vertical and slightly up-inclined configuration of the holding tube.

The CPD software requires the user to provide the thermophysical properties of the food particles, type of material that would be used for the construction of conservative particle (nylon, polypropylene (PP), teflon, polymethylpentene (PMP) or other plastic materials with known thermophysical properties), shape of the particle (spherical, cubical or cylindrical), and size of the food and simulated particle (i.e. half thickness for cube). The present study deals with the cubical particles. The results are generated as a lethality chart that shows the demarked region in a one-eighth portion of the particle (figure 12). The line shows the region inside which, the conservative particle will receive an accumulated F_0 value of less than three minutes (12D reduction process for *Clostridium botulinum*).



42	43	45	48	53	59	68	81	100	132
14	15	16	18	21	26	33	44	63	100
6.1	6.4	7.1	8.4	10	13	18	27	44	81
2.9	3.1	3.6	4.3	5.7	7.9	11	18	33	68
1.5	1.7	2	2.5	3.4	5	7.9	13	26	59
0.9	1	1.2	1.5	2.2	3.4	5.7	10	21	53
0.6	0.7	0.8	1.1	1.5	2.5	4.3	8.4	18	48
0.4	0.5	0.6	0.8	1.2	2	3.6	7.1	16	45
0.3	0.4	0.5	0.7	1	1.7	3.1	6.4	15	43
0.3	0.3	0.4	0.6	0.9	1.5	2.9	6.1	14	42

Figure 12. F_0 map showing the quarter cube of a particle and the F_0 values at different points inside the cube

2.4.3 Calculation of hold tube length

The hold tube is one of the most important components of the aseptic processing system because it provides the heat treatment for commercial sterility of a product. For a homogeneous fluid food product, length of the hold tube is calculated on the basis of two important considerations -- the temperature profile of the food product within the hold tube and the residence time of any given fluid element in the hold tube. The temperature of the product leaving the hold tube indicates the minimum bulk average product temperature throughout the entire hold tube. Residence time of the fluid is affected by the product flow characteristics (laminar or turbulent flow). The fastest fluid element flows no more than twice as fast as the bulk average velocity for fully developed laminar flow and 1.2 times as fast for fully developed turbulent flow. The length of the hold tube is calculated such that the fastest fluid element receives the required thermal treatment for commercial sterility (Gavin, 1985).

For a multiphase food product, considerably more heat needs to be applied to the particle surface in the hold tube to bring the center temperature of the particle to the required temperature because the particles heat slowly by conduction. This makes the calculation of hold tube length for a multiphase food product more complex as compared to that for a homogeneous food product. The temperature of the carrier fluid leaving the hold tube indicates the minimum bulk average temperature of the carrier fluid. Different particles are heated at different rates by the carrier fluid. Therefore, the calculation of hold tube is done on the basis of the critical particle because the center of the critical particle receives the least heat treatment. Since the inside of the critical particle heats by conduction, long hold tubes are required to bring the center temperature of the particle to the required sterilization temperature. Residence time of the critical particle should be known to calculate the length of the hold tube. Residence time of the critical particle depends on the flow characteristics of the carrier fluid. Once residence time and center temperature of the critical particle is determined, the length of the hold tube which gives the required thermal treatment to the center of the critical particle is calculated.

AseptiCAL™ (FMC FoodTech, Chicago, IL) is a software based on an advanced finite difference based mathematical modeling for aseptic process development of low-acid food products with or without particulates. This software is divided into four modules to accommodate 3-D particulates, 2-D particulates, 1-D particulates, and homogeneous products. The software simulates the center temperature of the fastest moving particle and its accumulated lethality to calculate the length of hold tube.

2.4.4 Biological validation

Biological validation of an aseptic process for a low-acid multiphase food product requires use of a bioindicator that can be processed in an aseptic system, retrieved intact, and tested quantitatively for received lethality (Marcy, 1997). Design particles could be used as thermo-sensitive implant carriers for bacterial spores in biological validation of an aseptic process. The validation tests should be conducted at different temperatures to achieve a positive/negative result at the end of the process. This will aid in determining the minimum allowable process temperature that will result in a safe process. Based on all of these tests, a process is designed for final validation of the established process (Sandeep and Puri, 2001).

The final step in establishing an aseptic process for *salsa con queso* would require processing of *salsa con queso* along with more than 299 design particles having spore suspension in their cavity using a continuous flow microwave system. To ensure a safe process, microbial tests should be conducted to show that the required log reduction of the spores is achieved.

The main objective of this research was to develop a systematic approach for biological validation of aseptic processing of *salsa con queso* products using a continuous flow microwave system operating at 915 MHz. The objective was separated into smaller goals as follows: Measurement of dielectric (dielectric constant and dielectric loss factor) and thermophysical(density, specific heat, and thermal conductivity) properties of different *salsa con queso* products, their ingredients, and spore suspension of *Clostridium sporogenes*, fabrication of conservative design particles, and processing of *salsa con queso* in a 5 kW microwave unit.

The rest of this thesis is organized in the form of three manuscripts. Manuscript I presents a new approach (under continuous flow conditions) to measure dielectric properties of pumpable food materials. Thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients (tomatoes, bell peppers, jalapeno peppers, and onions) at a temperature range of 20 to 130 EC have been determined and compiled in manuscript II. Manuscript III investigates the feasibility of aseptic processing of *salsa con queso* using a continuous flow microwave system.

SYMBOLS

B	Electric flux density
c_p	Specific heat capacity at constant pressure of the material (J/kg-K)
C_i	Confidence interval (%)
C	Capacitance per unit length
C value	Cook value (s)
C_0 value	F-value at 100 EC and a z_c-value of 33.1 EC (s)
D	Magnetic flux density
D value	Decimal reduction time (s)
E	Electric field intensity
F value	Processing time at any temperature to achieve a certain level of microbial kill (s)
F_0 value	F-value at 121.1 EC and a z-value of 10 EC (s)
f	Frequency (s^{-1})
f_c	Cut-off frequency (s^{-1})
f_r	Relaxation frequency (s^{-1})
G	Conductance per unit length
G_B	Buoyant force (kg)
h_c	Convective heat transfer coefficient (W/m^2-K)
h_{fg}	Latent heat of vaporization of the evaporated liquid
H	Magnetic field intensity
I_s	Current (Ampere)

J	Current flux density
k	Thermal conductivity of the material (W/m-K)
k_r	Imaginary index of refraction
K	Consistency coefficient
L_r	Lethal rate
L	Inductance per unit length
m_w	Rate of evaporation
n	Flow behavior index
N	Number of spores surviving after time t
N_0	Number of spores surviving at time t = 0
n_r	Real indices of refraction
K_T	Reaction rate (s^{-1})
P	Fraction of the fastest particle (%)
q_{rad}	Incident radiant heat flow rate per unit surface area to the load
Q	microwave power absorbed
R	Resistance per unit length
S	Poynting vector
tan δ	Loss tangent
u**r**	Velocity of the wave in a lossless dielectric medium (ms^{-1})
v	Velocity of the material (ms^{-1})
V_s	Voltage (V)
X_w	Fraction of water content

z value	Temperature change required to change D-value by a factor of ten (EC)
z_c value	z value for nutrient destruction (EC)
Z_o	Characteristic impedance
Z_L	Impedance of source and load

Greek letters

α	Thermal diffusivity (m^2/s)
α_a	Surface absorptivity of the load
α_s	Spread of relaxation times
α_c	Attenuation constant (in nepers per meter or decibels per meter)
β_c	Phase constant (radians per meter)
γ	Propagation constant (m^{-1})
ε	Complex permittivity (F/m)
ε'	Relative dielectric constant
ε''	Effective relative dielectric loss factor
ε_0	Permittivity of free space (8.86×10^{-12} F/m)
ε_4	Dielectric constant at very high frequencies
ε_s	Dielectric constant at very low frequencies
ε_{rad}	Radiative surface emissivity of the load
μ	Complex permeability (H/m)
μ	Viscosity (Pa-s)
μ'	Relative permeability
μ''	Effective relative magnetic loss factor

μ_0	Permeability of the free space ($4\pi \times 10^{-7}$ H/m)
σ_e	Conductivity
σ_{rad}	Stefan-Boltzmann constant
σ	Shear stress (Pa)
$\dot{\gamma}$	Shear rate (s^{-1})
δ_s	Skin depth (m)
δ_p	Power penetration depth (m)
λ	Wavelength of the microwave in free space (m)
ω	Angular frequency (rad/s)
η'	Intrinsic impedance of the uniform plane wave in the dielectric medium
η_{TE}	Intrinsic wave impedance of the mode
τ	Relaxation time (s)
ν	Volume fraction
ν	Kinematic viscosity (m^2/s)
ρ	Density of the material in kg/m^3
ρ_a	Apparent density of the material in kg/m^3
ρ_r	Magnitude portion of the reflection coefficient
ρ_c	Electric charge density
Γ	Voltage reflection coefficient

Subscripts

c	Carbohydrate

p	Proteins
f	Fat
w	Water
ref	Reference

Abbreviations

ADC	Analog-to-digital converter
ALC	Automatic level control
CAPPS	Center for Advanced Processing and Packaging Studies
CPD	Conservative Particle Design
CW	Continuous wave
DSP	Digital signal processing
FCC	Federal Communications Commission
FDA	Food and Drug Administration
FM	Frequency modulation
HCF	Heat, cool, and fill
HTST	High temperature short time
LO	Local oscillator
LTLT	Low temperature long time
MUT	Material under test
NCFST	National Center for Food Safety and Technology
NFPA	National Food Processors Association
PLL	Phase-locked loop

PMP	Polymethylpentene
PP	Polypropylene
RTD	Residence time distribution
TDT	Thermal death time
TE	Transverse electric
TM	Transverse magnetic
TEM	Transverse electromagnetic
UHT	Ultra high temperature
USDA	United States Department of Agriculture
VCO	Voltage controlled oscillator
VSWR	Voltage standing wave ratio

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Chapter 3

MANUSCRIPT I

Measurement of dielectric properties of pumpable food materials under static and continuous flow conditions

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Abstract

Continuous flow microwave sterilization is an emerging technology which has the potential to replace the conventional heating processes for viscous and pumpable food products. Dielectric properties of pumpable food products were measured by a new approach (under continuous flow conditions) at a temperature range of 20 to 130 EC and compared with the dielectric properties measured by the conventional approach (under static conditions). The food products chosen for this study were skim milk, green pea puree, carrot puree, and *salsa con queso*. Second order polynomial correlations for the dependence of dielectric properties at 915 MHz of the food products on temperature were developed. Dielectric properties measured under static and continuous flow conditions were similar for homogeneous food products such as skim milk and vegetable puree, but they were substantially different for *salsa con queso* which is a multiphase food product. The results from this study suggest that, for a multiphase product, dielectric properties measured under continuous flow conditions should be used for designing a continuous flow microwave heating system.

Keywords: microwave heating, continuous flow, multiphase product, sterilization

Introduction

Microwaves are a part of the electromagnetic spectrum and have a frequency between 300 MHz and 300 GHz. They lie between the radio (3 kHz - 300 MHz) and infrared (300 GHz - 400 THz) frequencies of the electromagnetic spectrum. Microwave radiation has the ability to heat materials by penetrating and dissipating heat in them. Due to this ability, microwaves have been used in the polymer and ceramic industries (joining, sintering, combustion synthesis, melting, epoxy curing, preheating rubbers, and thermosetting), medicine (thawing frozen tissues, warming blood, and tumor therapies), and textiles (drying) (Saltiel and Datta, 1999). Microwaves have been used for several food processing operations including thawing, blanching, pasteurization, sterilization, dehydration, baking, and roasting (Bengtsson and Ohlsson, 1973). Continuous flow microwave sterilization is an emerging technology which has the potential to replace conventional heating processes for viscous and pumpable food products. Some of the advantages associated with microwave heating are instant start-up, faster and selective heating, improved color, flavor, texture, and nutrient retention.

Interaction of microwaves with materials depends on their dielectric properties. Dielectric properties determine the extent of heating of a material when subjected to electromagnetic fields. Therefore, knowledge of dielectric properties is important for the design of a continuous flow microwave heating system. Dielectric properties consist of dielectric constant (ε') and dielectric loss factor (ε''). Dielectric constant is a measure of the ability of a material to store electromagnetic energy whereas dielectric loss factor is a measure of the ability of a material to convert electromagnetic energy to heat (Metaxas and

Meredith, 1983). Dielectric constant and dielectric loss factor can be defined in terms of complex permittivity (ε). The complex permittivity (ε) is composed of a real part (ε', relative dielectric constant) and an imaginary part (ε''_r, relative dielectric loss factor) and is given by the equation (Saltiel and Datta, 1999):

$$\varepsilon = \varepsilon_0(\varepsilon' - j\varepsilon'') \qquad \textbf{(1)}$$

where, $j = (-1)^{0.5}$ and ε_0 is the permittivity of free space (8.86×10^{-12} F/m). Loss tangent (tan δ), a parameter used to describe how well a product absorbs microwave energy, is the ratio of dielectric loss factor (ε") to the dielectric constant (ε'). A product with a higher loss tangent will heat faster under microwave field as compared to a product with a lower loss tangent (Nelson and Datta, 2001). Dielectric properties of food products depend on the frequency of the microwaves, temperature, composition, and density of the materials (Datta *et al.*, 2005).

Power penetration depth (δ_p), often used in microwave heating applications, is the distance at which power drops to e^{-1} of its value at the surface of the material and is given by the following equation (Nelson and Datta, 2001):

$$\delta_p = \frac{\lambda}{2\pi\sqrt{2\varepsilon'\left[\sqrt{1 + \left(\frac{\varepsilon''}{\varepsilon'}\right)^2} - 1\right]}} \qquad \textbf{(2)}$$

where, λ is the wavelength of the microwave in free space in meters. The above equation is valid for a plane wave incident upon a semi-infinite slab. Power penetration depth is used to calculate the tube diameter for a continuous flow microwave heating system.

The three most popular methods for measuring dielectric properties are the open-ended coaxial probe, transmission line, and resonance cavity methods. Of the three methods, the open-ended coaxial probe method is the preferred one for measuring dielectric properties of food products because it can measure dielectric properties over wide frequency range, it is easy to use, and it can be used for liquids and solids equally well (Engelder and Buffler, 1991). Many studies have been undertaken to determine the dielectric properties of food products using the open-ended coaxial probe method (Tran *et al.*, 1984; Nelson *et al.*, 1993; Nelson and Bartley, 2002; Sipahioglu and Barringer, 2003; Guan *et al.*, 2004; Nunes *et al.*, 2006). In all of these studies, dielectric properties were measured by heating the food product in a water or oil bath. There are inherent problems associated with this approach of measuring dielectric properties under static conditions. The dielectric properties of only a portion of the material which is in contact with the dielectric probe is measured. Thus, the measurement by this approach may not be representative of the dielectric properties of the bulk for a multiphase food product. It is well known that physical structure of the food products have an effect on dielectric properties (Ryynanen, 1995). The rate of heating under static conventional heating conditions is slower as compared to the rate of heating under continuous flow microwave heating conditions. At the slower rate of heating under static conditions, there is higher degree of protein denaturation and starch gelatinization (Li *et al.*, 2006; Sakonidou *et al.*, 2003). Protein denaturation and starch gelatinization change the physical structure of food products. Thus, for the food products which have protein or starch as one of the major constituents, dielectric properties measured under static conditions will be different from those measured under continuous flow microwave heating.

The present study was undertaken to develop a new approach to measure dielectric properties of pumpable food products under continuous flow microwave heating conditions at a temperature range of 20 to 130 EC and compare it with the dielectric properties measured under static conditions. In this new measurement method, the food product is continuously pumped across the dielectric probe. Therefore, different parts of the food product are in contact with the probe at different times. Thus, this approach gives a better representation of the dielectric properties of the bulk of the food product.

Materials and methods

Sample selection

The food products chosen for this study were skim milk, green pea puree, carrot puree, and *salsa con queso.* Skim milk and three brands of *salsa con queso* (Brands A, B, and C, representative of the category of *salsa con queso*) were purchased from a local supermarket (Food Lion, Raleigh, NC). These samples were stored in a refrigerator prior to use in the experiments. Frozen green pea and carrot puree were purchased from Stahlbush Islands Farm Inc. (Corvallis, OR). These purees were thawed at room temperature for several hours prior to processing.

Measurement under static conditions

Dielectric properties of the samples under static conditions were measured using an open-ended coaxial probe (Model HP 85070B, Agilent Technologies, Palo Alto, CA) connected to a network analyzer (Model HP 8753C, Agilent Technologies, Palo Alto, CA) (figure 1). HP 85070B is a large diameter probe with an outer diameter (OD) of 19 mm. The network analyzer was calibrated by leaving the probe in contact with air, metal, and 25 ºC de-ionized water and measuring the dielectric properties. The dielectric properties were measured at 20, 75, 90, 100, 110, 120, 125, and 130 ºC and at frequencies from 300 to 3,000 MHz with an increment of 5 MHz. The variable step size for temperature increment was chosen because the aim in this study was to determine the dielectric properties at sterilization temperatures. The samples were placed in a pressurized cell and heated in an oil bath (Model RTE111, Neslab Instruments Inc., Newington, NH) to attain the testing temperature.

Measurement under continuous flow conditions

Dielectric properties of the samples under continuous flow conditions were measured inline using an open-ended coaxial probe (Model HP 85070E, Agilent Technologies, Palo Alto, CA) connected to a network analyzer (Model HP 8753C, Agilent Technologies, Palo Alto, CA) (figure 1). HP 85070E is a small diameter probe with an outer diameter (OD) of 2.2 mm which can be inserted in one of the three ports of a smart gasket (Rubber-Fab, Newton, NJ). The dielectric properties were measured at temperatures from 20 to 130 ºC and frequencies from 300 to 3,000 MHz. A schematic of the experimental system to measure dielectric properties during continuous flow microwave heating is shown in figure 2. The product was heated using a 5 kW microwave unit which has been described below.

5 kW microwave unit

The microwave unit, shown in figure 2, consists of a 5 kW microwave generator (Industrial Microwave Systems, Morrisville, NC) operating at 915 MHz, a waveguide of rectangular cross-section, and a specially designed focused applicator (Drozd and Joines, 1999). A tube of 1.5" nominal diameter (0.038 m ID) made of Polytetrafluoroethylene (PTFE or Teflon®) was placed at the center of the applicator through which the product was pumped using a positive displacement pump (Model MD012, Seepex GmbH+ Co, Bottrop, Germany) with a variable speed motor (Tri- Clover Rotary Pump, Model PRE3-1M, Ladish Co., Kenosha, WI). The temperatures at the inlet and outlet of the applicator were recorded using a thermocouple arrangement described by Coronel *et al.* (2003) and a datalogging system (Model DAS-16, Keithley Metrabyte Inc., Taunton, MA). The dielectric probe (HP 85070E) was inserted at the outlet of the applicator in one of the three ports of the smart gasket (Coronel *et al.*, 2003) as shown in figure 2.

Results and Discussion

Skim milk

The dielectric properties of skim milk at 915 MHz were measured under continuous flow conditions and were compared with those measured under static conditions and published by Coronel *et al.* (2003), as shown in figure 3. Dielectric properties of skim milk measured under static and continuous flow conditions from 20 to 120 EC were similar. This is expected because skim milk is a homogeneous product and it does not have protein or starch as a major constituent. However, dielectric properties measured under continuous flow conditions at 130 EC were substantially different from those under static conditions which might be a result of a change of phase at that high temperature under static conditions. The effect of temperature on dielectric properties under static and continuous flow conditions was similar with ε' decreasing with an increase in temperature and ε'' increasing with an increase in temperature which is in accordance with the observations of Datta *et al.* (1997) for food products with moisture content greater than 60%. Second order polynomial correlations for the dependence of dielectric properties of skim milk on temperature at 915 MHz were developed and are shown in table 1.

Vegetable purees

The dielectric properties of green pea and carrot puree at 915 MHz measured under static and continuous flow conditions are shown in figures 4 and 5 respectively. Dielectric properties of both the purees measured under static and continuous flow conditions were similar. This is expected because these purees are homogeneous. The values of dielectric constant (ε') and dielectric loss factor (ε'') for green pea puree were similar to those reported by Tong *et al.* (1994). The values of ε' and ε'' at 20 °C and 915 MHz (figure 4) are 66.74 and 15.31 respectively and the correlation by Tong *et al.* (1994) predicts these values to be 64.49 and 13.24 respectively. The effect of temperature on dielectric properties under static and continuous flow conditions was similar with ε' decreasing with an increase in temperature and ε'' increasing with an increase in temperature which is similar to the results obtained for skim milk. Second order polynomial correlations for the dependence of dielectric properties of both vegetable purees on temperature at 915 MHz were developed and are shown in table 1.

Salsa con queso

The dielectric properties of three different brands (Brands A, B, and C) of *salsa con queso* at 915 MHz measured under static and continuous flow conditions are shown in figures 5, 6, and 7 respectively. Dielectric properties of *salsa con queso* at 915 MHz measured under static and continuous flow conditions are substantially different for all the three brands. This result is expected for a multiphase product such as *salsa con queso*. Under static conditions, dielectric properties of only a small portion of the product is measured. Therefore, measurement by this approach might under-predict or over-predict dielectric properties based on the components of the product in contact with the probe surface. Thus, measurement under static conditions is not representative of the dielectric properties of the bulk of the product. Second order polynomial correlations for the dependence of dielectric properties of the three brands of *salsa con queso* on temperature at 915 MHz were developed and are shown in table 1.

It was observed that the measurement under static conditions under-predicted ε'' and over-predicted ε' for all the brands. This resulted in a lower value of loss tangent for the measurements under static conditions. Thus, dielectric properties under static conditions for *salsa con queso* will predict a slower rate of heating under a microwave field. Equation (2) was used to calculate the power penetration depth for the three brands of *salsa con queso*. The deviations in power penetration depths for the measurement under static and continuous flow conditions were 0.6 to 18%, 3.9 to 32% and 4.6 to 22.8% for brands A, B, and C respectively.

Apart from measuring the true values of dielectric properties for a multiphase product, this new approach to measure dielectric properties can be used to monitor physico-chemical changes such as protein denaturation and starch gelatinization in a food product during thermal processing. Monitoring such changes can become a tool for research and development in the food industry. Thus, it is recommended that, for a multiphase product, dielectric properties measured under continuous flow conditions should be used for designing a continuous flow microwave heating system.

Conclusions

Dielectric properties of pumpable food products were measured by a new approach (under continuous flow conditions) at a temperature range of 20 to 130 EC and compared with the dielectric properties measured by the conventional approach (under static conditions). Second order polynomial correlations for the dependence of dielectric properties of the food products on temperature at 915 MHz were developed. The results showed that the dielectric properties measured under static and continuous flow conditions were similar for homogeneous food products such as skim milk and vegetable puree, but they were substantially different for *salsa con queso* which is a multiphase food product. The results suggest that, for a multiphase product, dielectric properties measured under continuous flow conditions should be used for designing a continuous flow microwave heating system. This new approach to measure dielectric properties can be used to monitor physico-chemical changes in a food product during thermal processing which can be used a tool for research and development in the food industry.

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The use of trade names in this publication does not imply endorsement by the North Carolina Agricultural Research Service of the products named nor criticism of similar ones not mentioned.

Symbols

δ_p	Power penetration depth (m)
tan δ	Loss tangent

Greek letters

ε	Complex permittivity (F/m)
ε_0	Permittivity of free space (8.86×10^{-12} F/m)
ε'	Relative dielectric constant
ε''	Relative dielectric loss factor
λ	Wavelength of microwave in free space (m)

Abbreviations

ID	Inside diameter
OD	Outside diameter
PTFE	Polytetrafluoroethylene

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Table 1. Dielectric properties of different food materials as a function of temperature at 915 MHz

Sample	Correlations (T in EC)	r^2
Skim milk (Static conditions)	$\varepsilon N = 67.3 + 0.0878\ T - 0.0021\ T^2$	0.960
	$\varepsilon 0 = 16.0 - 0.0590\ T + 0.0020\ T^2$	0.903
Skim milk (Continuous flow conditions)	$\varepsilon N = 73.4 - 0.1572\ T - 0002\ T^2$	0.999
	$\varepsilon 0 = 13.5 + 0.0577\ T + 0.0009\ T^2$	0.989
Green pea puree (Static conditions)	$\varepsilon N = 70.4 - 0.1792\ T + 0.0001\ T^2$	0.988
	$\varepsilon 0 = 13.1 + 0.0864\ T + 0.0005\ T^2$	0.977
Green pea puree (Continuous flow conditions)	$\varepsilon N = 74.8 - 0.2983\ T + 0.0008\ T^2$	0.994
	$\varepsilon 0 = 13.1 + 0.0910\ T + 0.0004\ T^2$	0.986
Carrot puree (Static conditions)	$\varepsilon N = 77.7 - 0.1584\ T - 0.0004\ T^2$	0.994
	$\varepsilon 0 = 18.8 - 0.0207\ T + 0.0022\ T^2$	0.999
Carrot puree (Continuous flow conditions)	$\varepsilon N = 77.5 - 0.2064\ T + 3E\text{-}05\ T^2$	0.969
	$\varepsilon 0 = 18.6 + 0.0267\ T + 0.0020\ T^2$	0.995
Salsa con queso (Brand A) (Static conditions)	$\varepsilon N = 58.3 + 0.0157\ T - 0.0008\ T^2$	0.998
	$\varepsilon 0 = 34.33 - 0.0401\ T + 0.0039\ T^2$	0.998
Salsa con queso (Brand A) (Continuous flow conditions)	$\varepsilon N = 58.2 + 0.0333\ T - 0012\ T^2$	0.832
	$\varepsilon 0 = 19.9 + 0.5961\ T + 0.0003\ T^2$	0.925
Salsa con queso (Brand B) (Static conditions)	$\varepsilon N = 63.2 + 0.0167\ T - 0.0006\ T^2$	0.998
	$\varepsilon 0 = 56.6 - 0.2449\ T + 0.0065\ T^2$	0.999
Salsa con queso (Brand B) (Continuous flow conditions)	$\varepsilon N = 70.9 - 0.1961\ T - 2E\text{-}05\ T^2$	0.997
	$\varepsilon 0 = 25.1 + 0.7591\ T + 0.0007\ T^2$	0.996
Salsa con queso (Brand C) (Static conditions)	$\varepsilon N = 53.9 + 0.0398\ T - 0.0005\ T^2$	0.998
	$\varepsilon 0 = 36.0 + 0.0389\ T + 0.0046\ T^2$	0.999
Salsa con queso (Brand C) (Continuous flow conditions)	$\varepsilon N = 62.2 - 0.1609\ T - 4E05\ T^2$	0.944
	$\varepsilon 0 = 25.3 + 0.8009\ T + 0.0009\ T^2$	0.998

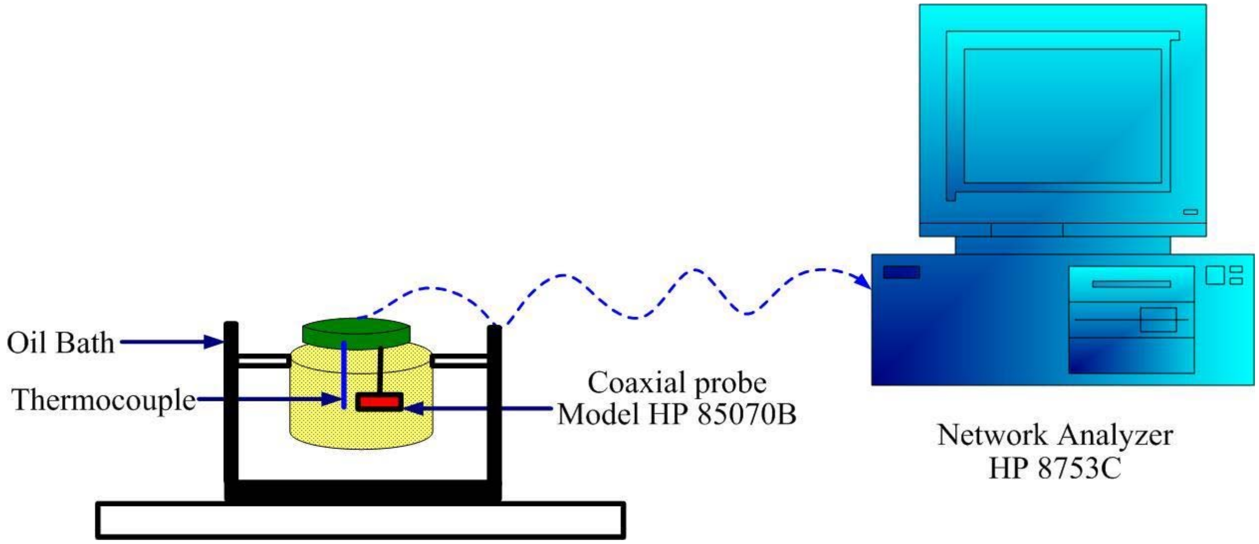

Figure 1. Schematic of the experimental system to measure dielectric properties under static conditions

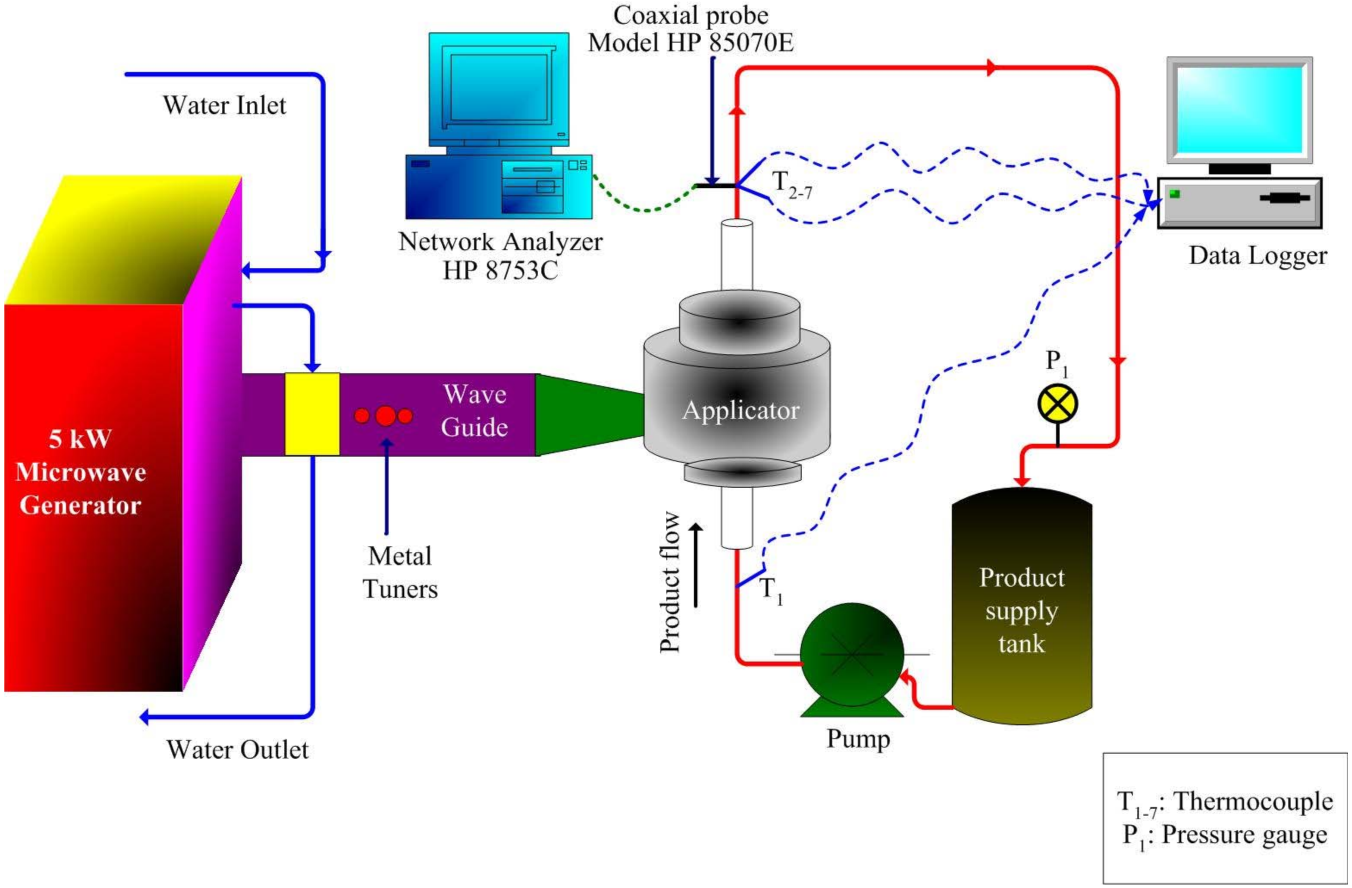

Figure 2. Schematic of the experimental system to measure dielectric properties under continuous flow conditions

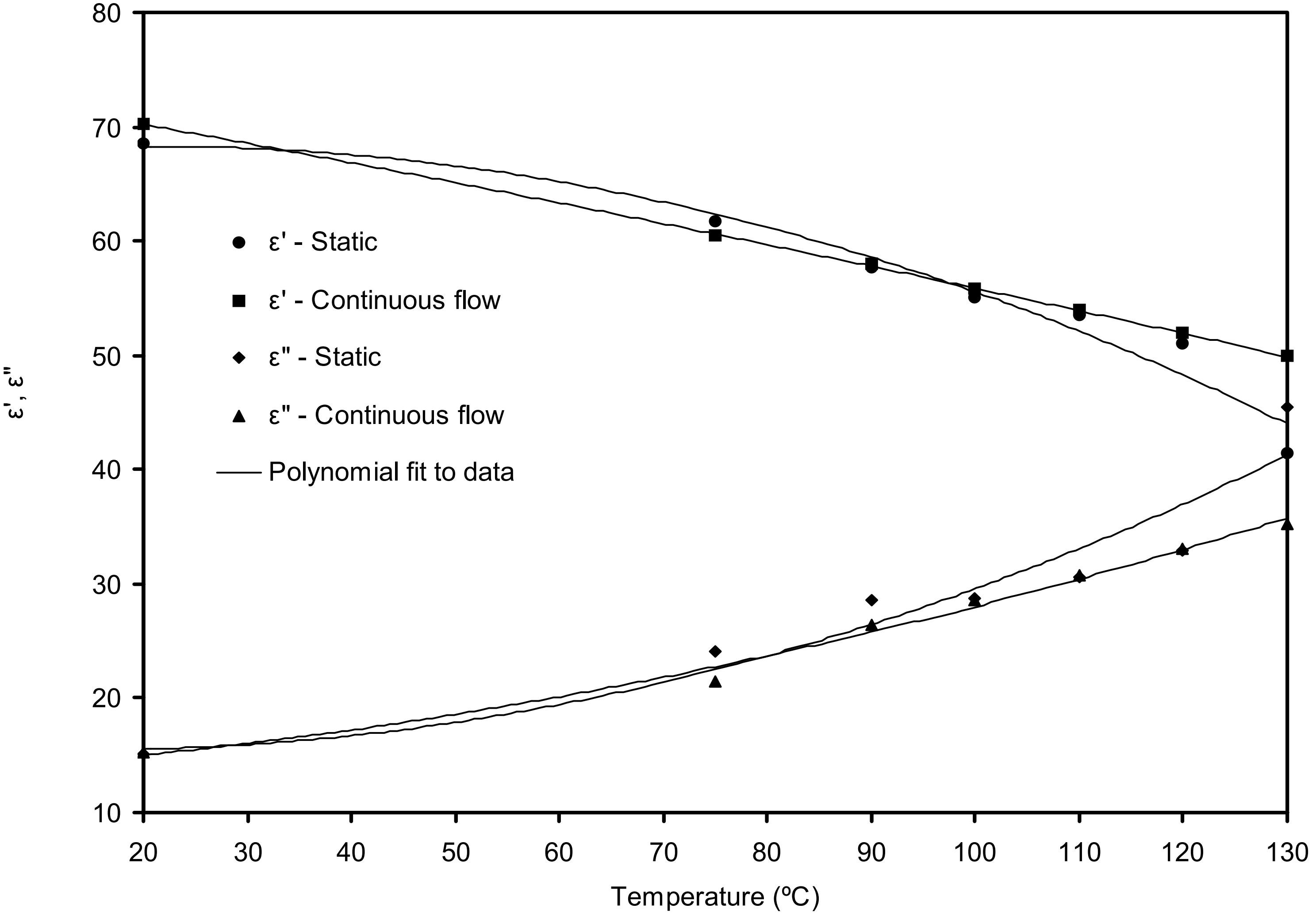

Figure 3. Dielectric constant (εℕ) and dielectric loss factor (ε0) of skim milk at 915 MHz

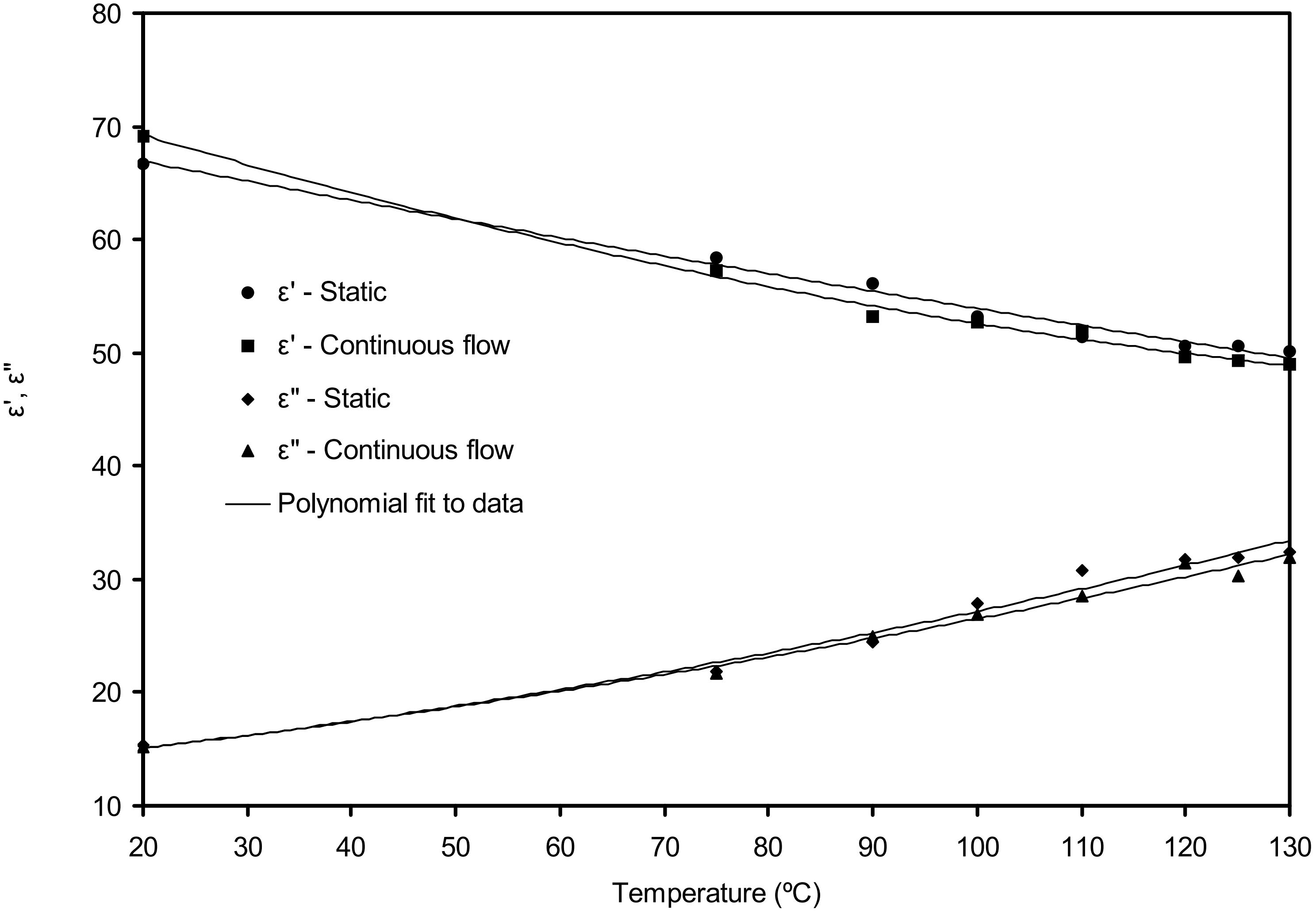

Figure 4. Dielectric constant (εℕ) and dielectric loss factor (ε0) of green pea puree at 915 MHz

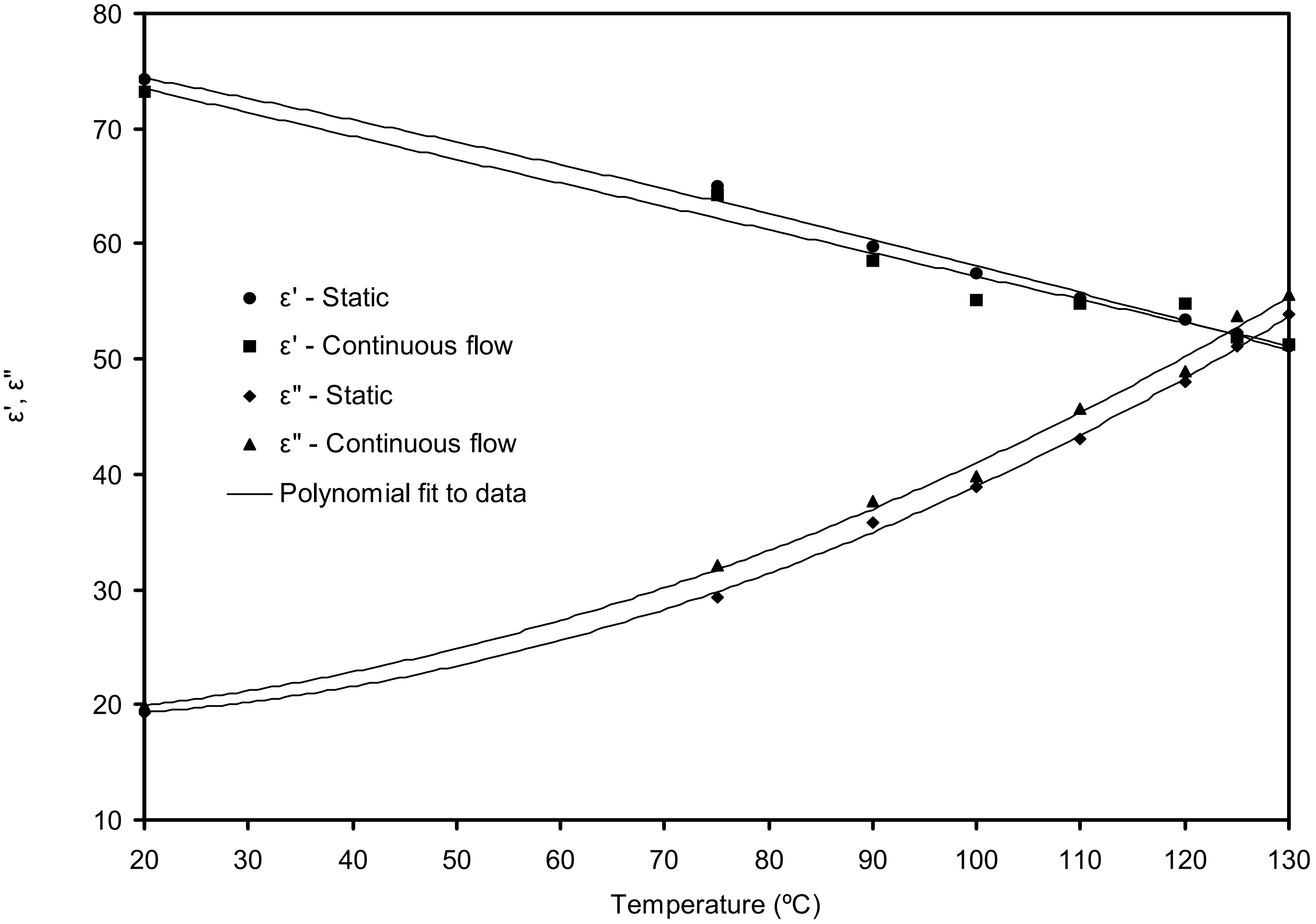

Figure 5. Dielectric constant (εℕ) and dielectric loss factor (ε0) of carrot puree at 915 MHz

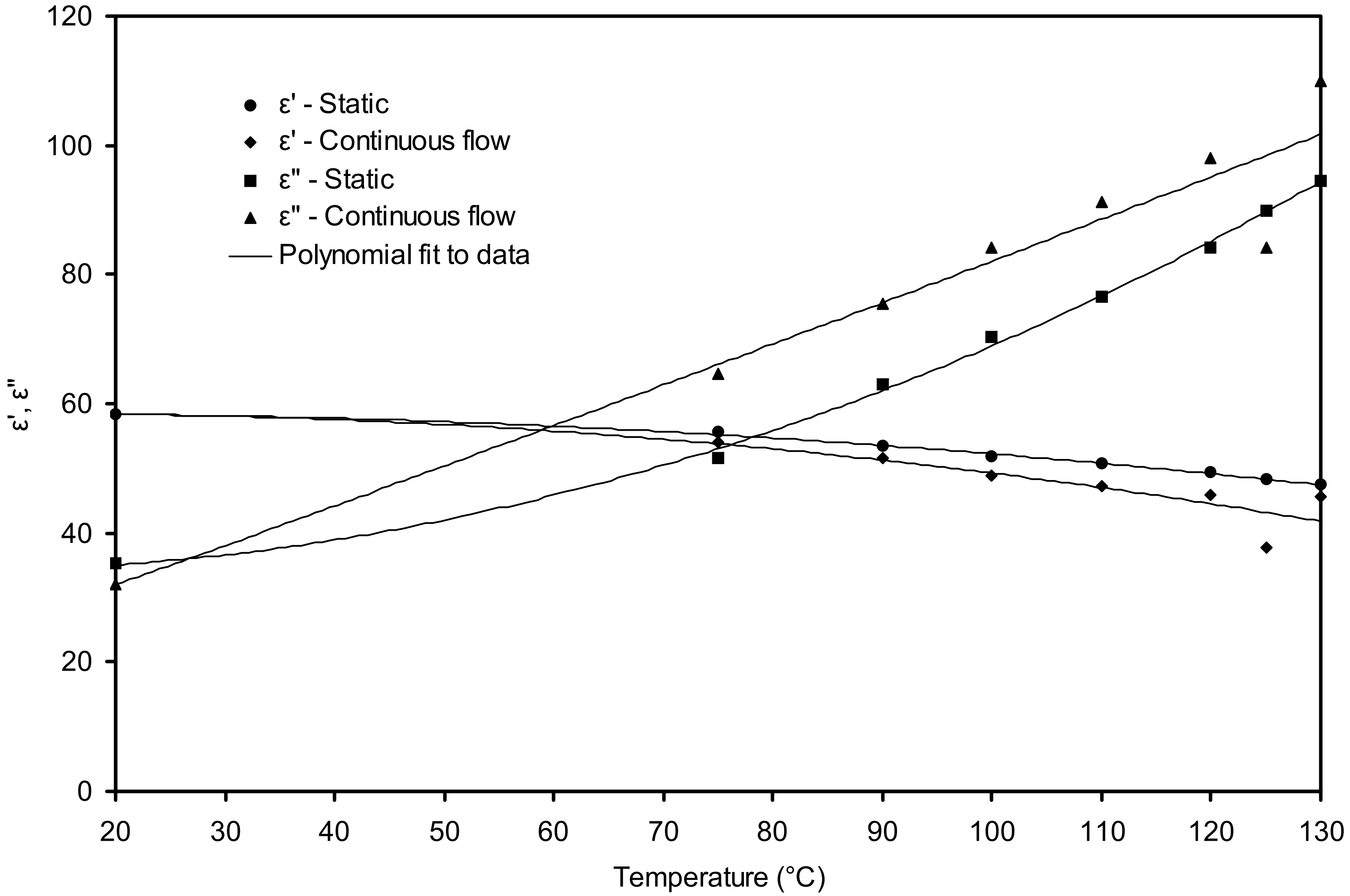

Figure 6. Dielectric constant (εN) and dielectric loss factor (ε0) of *salsa con queso* (Brand A) at 915 MHz

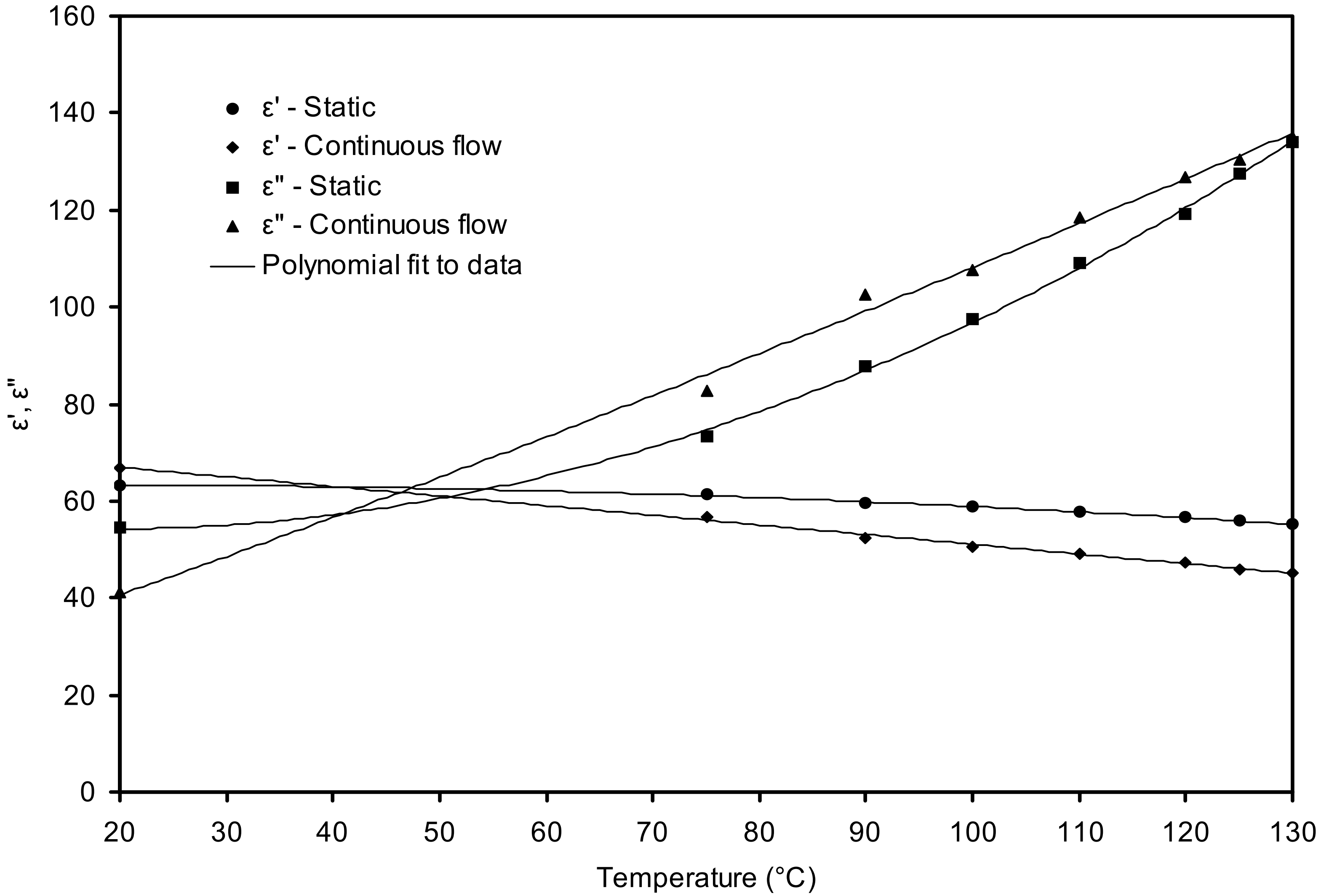

Figure 7. Dielectric constant (εℕ) and dielectric loss factor (ε0) of *salsa con queso* (Brand B) at 915 MHz

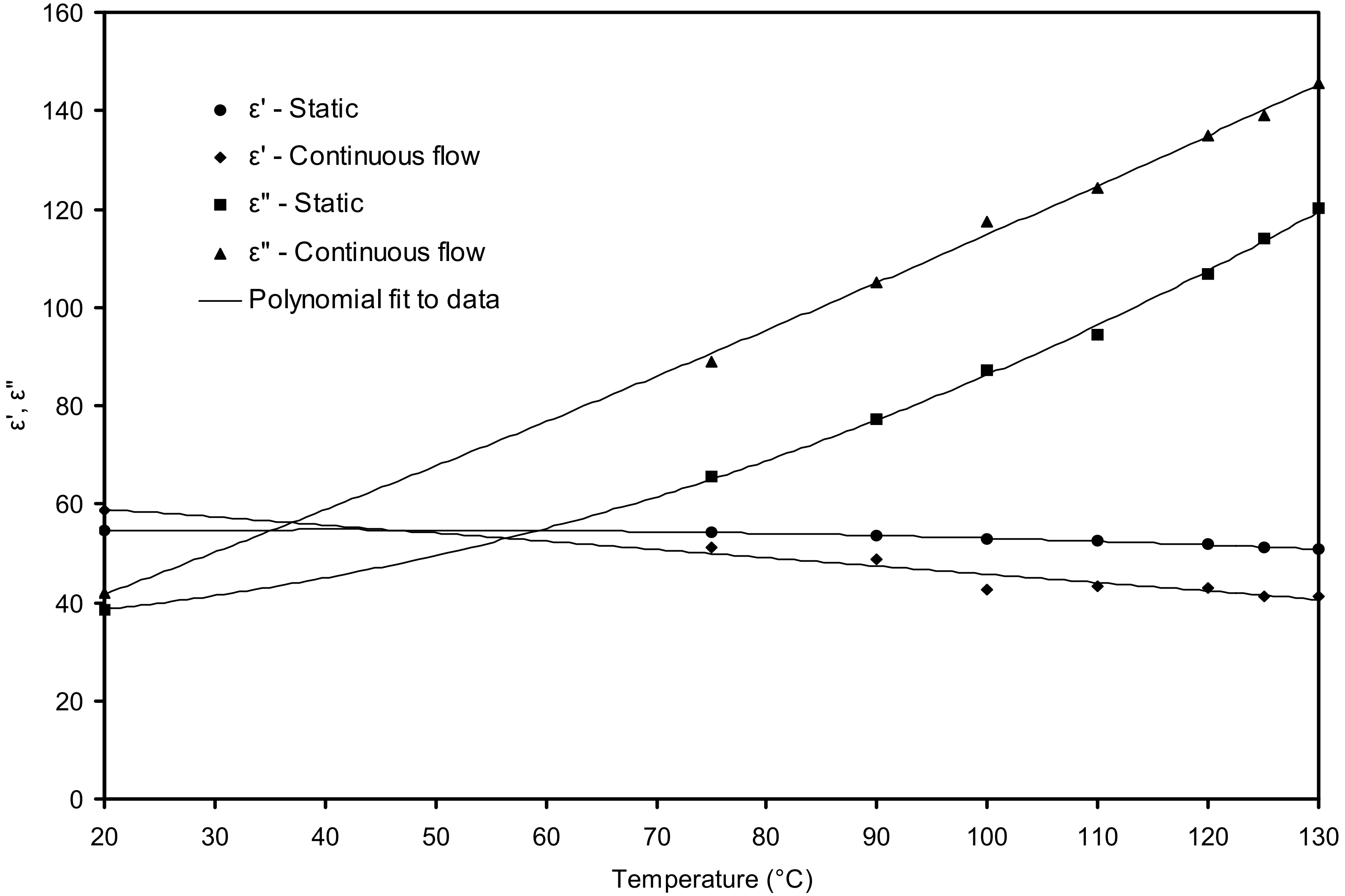

Figure 8. Dielectric constant (εℕ) and dielectric loss factor (ε0) of *salsa con queso* (Brand C) at 915 MHz

Chapter 4

MANUSCRIPT II

Thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients at sterilization temperatures

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Abstract

Aseptic processing of a low-acid multiphase food product using continuous flow microwave heating system can combine the advantages of an aseptic process along with those of microwave heating. The objective of this study was to determine the thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients (tomatoes, bell peppers, jalapeno peppers, and onions) at a temperature range of 20 to 130 EC. The influence of temperature on apparent viscosity of *salsa con queso* was described by an Arrhenius-type relationship. Second order polynomial correlations for the dependence of thermophysical and dielectric properties (at 915 MHz) of *salsa con queso* and its vegetable ingredients on temperature were developed. The results showed that the dielectric constant decreased with an increase in temperature and the dielectric loss factor increased with an increase in temperature. The results from this study can be used to design a safe process for aseptic processing of *salsa con queso* using a continuous flow microwave system.

Keywords: Aseptic processing, microwave heating, thermophysical and dielectric properties

Introduction

With consumers becoming more health conscious and educated, the demand for convenient and high quality foods has increased over time. Aseptic processing offers a potential option from conventional canning to meet these demands. As opposed to conventional canning, aseptic processing is a thermal process in which the product and container are sterilized separately and brought together in a sterile environment. The use of high temperature for a short period of time in aseptic processing yields a high quality product with the same level of microbiological safety as that in a conventional canning system. Aseptic processing of liquid foods such as milk, fruit juices, salad dressings, and liquid eggs has been in place for several decades. Foods containing small particles (smaller than 3.2 mm) such as baby foods, cottage cheese, soups, and rice desserts have also been aseptically processed. However, aseptic processing of low-acid multiphase foods containing large particulates (larger than 3.2 mm) such as soups containing meatballs or vegetables has not been a commercial reality in the U.S. even though it has been in place in the European market for several years (Morris-Lee, 2004). In recent years, there has been an increasing interest in aseptic processing of low-acid multiphase food products. One such product is *salsa con queso* (salsa with cheese) which is a commercially successful product with continued expansion. The main vegetable ingredients in *salsa con queso* are tomatoes, bell peppers, jalapeno peppers, and onions. Currently, *salsa con queso* products are sterilized by the conventional retort process which is associated with degradation of color, flavor, texture, and nutrients (David *et al.*, 1996; Sastry and Cornelius, 2002).

Continuous flow microwave heating is an emerging technology in the food industry with a potential to replace the conventional retort process. Instant start-up and rapid heating of food products makes microwave heating suitable for aseptic processing. Continuous flow microwave sterilization is also associated with improved color, flavor, texture, and nutrient retention. Thus, aseptic processing of a low-acid multiphase food product using a continuous flow microwave heating system can combine the advantages of an aseptic process along with those of microwave heating (Coronel *et al.*, 2005).

The extent of heating by microwaves is determined by the thermophysical and dielectric properties of the food materials. Dielectric properties consist of dielectric constant (ε') and dielectric loss factor (ε''). Dielectric constant is a measure of the ability of a material to store electromagnetic energy whereas dielectric loss factor is a measure of the ability of a material to convert electromagnetic energy to heat (Metaxas and Meredith, 1983). Loss tangent ($\tan \delta$), a parameter used to describe how well a product absorbs microwave energy, is the ratio of dielectric loss factor (ε'') to the dielectric constant (ε'). A product with a higher loss tangent will heat faster under microwave field as compared to a product with a lower loss tangent (Nelson and Datta, 2001). Thermophysical properties include density (ρ), viscosity (μ), specific heat (c_p), thermal conductivity (k), and thermal diffusivity (α). They have a significant effect on the rate of heat transfer into the particulates within the food product (Singh and Heldman, 2001). Therefore, knowledge of dielectric and thermophysical properties is important for the design of a safe aseptic process using a continuous flow microwave system.

Thermophysical and dielectric properties of various food materials have been compiled in literature (Polley *et al.*, 1980; Rahman, 1995; Rahman, 2005; Singh 1994;

Krokida *et al.*, 2001; Singh and Heldman, 2001; Nelson *et al.*, 1993; Ryynanen, 1995; Sipahioglu and Barringer, 2003; Datta *et al.*, 2005). However, there is currently no data available on the thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients at sterilization temperatures. Therefore, the objective of this study was to determine the thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients (tomatoes, bell peppers, jalapeno peppers, and onions) at a temperature range of 20 to 130 EC.

Materials and methods

Sample selection

Salsa con queso and four of its vegetable ingredients (tomatoes, bell peppers, jalapeno peppers, and onions) were purchased from a local supermarket (Food Lion, Raleigh, NC). The samples were stored in a refrigerator prior to use in the experiments.

Measurement of thermophysical properties

Particle densities of the four vegetables were measured at room temperature (22 EC) using a helium gas pycnometer (Micrometrics Accupyc 1330, Micrometrics, Norcross, GA). Particle density of *salsa con queso* was determined by measuring the weight of a fixed volume of *salsa* and dividing the weight by the fixed volume. All the measurements were performed in duplicate.

Rheological characterization of *salsa con queso* without the particulates was conducted from 20 to 130 EC at an interval of 10 EC using a Reologica StressTech Rheometer (ATS Rheosystems, Bordentown, NJ). Duplicate shear rate sweeps between 10 and 100 s^{-1} were performed at each of the temperatures.

Specific heat of *salsa con queso* and its vegetable ingredients was measured from 20 to 130 EC at an interval of 10 EC using a differential scanning calorimeter (DSC7, Perkin Elmer Instruments, Norwalk, CT). The DSC was calibrated using de-ionized water (temperature and enthalpy) and indium (temperature). Water was used as a standard and its specific heat was within 2.5% of the known values given by Osborne *et al.* (1930). Samples were subjected to a heating rate of 3 EC/min and the purge gas was nitrogen at a flow rate of 30 cc/min. Specific heat was calculated using the Pyris software version 5.0 (DSC7, Perkin Elmer Instruments, Norwalk, CT). All the measurements were performed in duplicate.

Thermal conductivity and thermal diffusivity of *salsa con queso* and its vegetable ingredients were measured from 20 to 130 EC at an interval of 10 EC using a thermal properties analyzer probe (KD2 Pro, Decagon devices Inc., Pullman, WA). The samples were placed in a pressurized cell and heated in an oil bath (Model RTE111, Neslab Instruments Inc., Newington, NH) to attain the testing temperature. All the measurements were performed in duplicate.

Measurement of dielectric properties

Dielectric properties of *salsa con queso* and its vegetable ingredients were measured from 20 to 130 EC using an open-ended coaxial probe (Model HP 85070B for static conditions and 85070E for continuous flow conditions, Agilent Technologies, Palo Alto, CA) connected to a network analyzer (Model HP 8753C, Agilent Technologies, Palo Alto, CA). Dielectric properties of *salsa con queso* were measured under static as well continuous flow conditions because the study done by Kumar *et al.* (2006) shows that there is a substantial difference between the dielectric properties measured under static and continuous flow conditions for a multiphase product such as *salsa con queso*. Dielectric properties of the vegetable ingredients were measured in particulate as well as homogenized form because density has a notable effect on the dielectric properties of particulate materials (Nelson and You, 1990). Samples were homogenized using a blender. The network analyzer was calibrated by leaving the tip of the probe in contact with air, metal, and 25 ºC de-ionized water and measuring the dielectric properties. The dielectric properties were measured at 20, 75, 90, 100, 110, 120, 125, and 130 ºC and at frequencies from 300 to 3000 MHz with an increment of 5 MHz. The variable step size for temperature increment was chosen because the aim in this study was to determine the dielectric properties at sterilization temperatures. All the measurements were performed in duplicate. For measurement during static conditions, the samples were placed in a pressurized cell and heated in an oil bath (Model RTE111, Neslab Instruments Inc., Newington, NH) to attain the testing temperature. For measurement during continuous flow conditions, the dielectric probe was inserted in one of the three ports of a smart gasket (Rubber-Fab, Newton, NJ) at the exit of the applicator and the product was heated using a 5 kW microwave generator operating at 915 MHz. The 5 kW unit consisted of a 5 kW microwave generator (Industrial Microwave Systems, Morrisville, NC) operating at 915 MHz, a waveguide of rectangular cross-section, and a specially designed focused applicator (Drozd and Joines, 1999). The setup to measure dielectric properties under static as well continuous flow conditions have been described in detail by Kumar *et al.* (2006).

Results and Discussion

Particle Density

The particle density of *salsa con queso* and its vegetable ingredients at 22 EC is shown in table 1. *Salsa con queso* was found to be least dense with a density of 1012 kg/m^3 and onions were found to be the most dense with a density of 1068 kg/m^3. The density of onions is in close agreement with the value of 1066 kg/m^3 as determined by Rapusas and Driscoll (1995). No data was available to compare the densities of other materials.

Magnus lift force, Saffman lift force, drag force, and buoyancy force are the forces experienced by particles suspended in a viscous fluid (Sandeep and Puri, 2001). As the density of particulates (vegetable ingredients) are higher than that of the carrier fluid (*salsa con queso*), particulates will sink due to the negative buoyant force. This will result in longer residence time of the particulates in a continuous flow system. Drag force on the particulates is a function of both fluid viscosity and relative velocity between carrier fluid and particulates. A high fluid viscosity and/or a sufficient relative velocity are required to overcome the negative buoyant force in order to transport the particulates through a continuos flow thermal processing system.

Rheological properties of salsa con queso

The apparent viscosity of *salsa con queso* (without the particulates) over a temperature range of 20 to 130 EC at three different shear rates (10, 50, and 100 s^{-1}) is shown in figure 1. The value of apparent viscosity (3.09 Pa-s at 30 EC) of *salsa con queso* was much higher than that of water (792.37 ×10^{-6} Pa-s at 30 EC), but is comparable to that of apple sauce (2.53 Pa-s at 30 °C) (Steffe and Daubert, 2006). Shear stress and shear rate data were fit into power law model and is given belows:

$$\text{At } 20\ ^\circ\text{C},\quad \sigma = 15.545\,\dot{\gamma}^{0.329} \qquad r^2 = 0.998 \quad \textbf{(1)}$$

$$\text{At } 100\ ^\circ\text{C},\quad \sigma = 5.836\,\dot{\gamma}^{0.399} \qquad r^2 = 0.994 \quad \textbf{(2)}$$

$$\text{At } 110\ ^\circ\text{C},\quad \sigma = 4.690\,\dot{\gamma}^{0.409} \qquad r^2 = 0.994 \quad \textbf{(3)}$$

$$\text{At } 120\ ^\circ\text{C},\quad \sigma = 5.333\,\dot{\gamma}^{0.358} \qquad r^2 = 0.991 \quad \textbf{(4)}$$

$$\text{At } 130\ ^\circ\text{C},\quad \sigma = 5.239\,\dot{\gamma}^{0.349} \qquad r^2 = 0.986 \quad \textbf{(5)}$$

where, σ is the shear stress in Pa and $\dot{\gamma}$ is the shear rate in s^{-1}.

In equations (1-6), the exponent of $\dot{\gamma}$, popularly denoted by n, is known as the dimensionless flow behavior index. *Salsa con queso* is a non-Newtonian shear thinning (pseudoplastic) fluid because the value of the flow behavior index is less than 1. The critical value (2100 + 875(1-n)) of the power law Reynolds number, which determine wether a flow is laminar or turbulent, depends on the flow behavior index. The maximum velocity for a power law fluid in laminar flow also depends on the flow behavior index and is less than the maximum velocity for a Newtonian fluid in laminar flow with the same average velocity (Steffe and Daubert, 2006). *Salsa con queso* will have a flatter velocity profile in a continuous flow thermal processing system because of the low value of flow behavior index. Therefore, the residence time distribution of particulates will be narrower when the carrier fluid is *salsa con queso*. This will result in a product of high quality in an aseptic processing system.

The influence of temperature on viscosity for a food product may be described by an Arrhenius-type relationship (Singh and Heldman, 2001):

$$\ln(\mu) = \ln(B_A) + \frac{E_a}{R_g T_A} \quad \textbf{(6)}$$

where, μ is the apparent viscosity in Pa-s, B_A is the Arrhenius constant (Pa-s), E_a is the activation energy constant (J/mol), and R_g is the gas constant (8.314 J/mol-K), and T_A is the temperature in Kelvin (K). The plot of ln (μ) vs $1/T_A$ was used to determine the Arrhenius constant and activation energy constant. The values of the Arrhenius constant and activation energy constant are shown in table 2.

Specific heat

Specific heat of *salsa con queso* and its vegetable ingredients over a temperature range of 20 to 130 °C is shown in figure 2. Among the four vegetable ingredients, bell peppers had the highest specific heat values and tomatoes had the lowest specific heat values at sterilization temperatures. Second order polynomial correlations for the dependence of specific heat of *salsa con queso* and its vegetable ingredients on temperature were developed and are shown in table 3.

Thermal conductivity

Thermal conductivity of *salsa con queso* and its vegetable ingredients over a temperature range of 20 to 130 EC is shown in figure 3. Among the four vegetable ingredients, jalapeno peppers had the highest thermal conductivity values and tomatoes had the lowest thermal conductivity values at sterilization temperatures. Second order polynomial correlations for the dependence of thermal conductivity of *salsa con queso* and its vegetable ingredients on temperature were developed and are shown in table 4.

Thermal diffusivity

Thermal diffusivity of *salsa con queso* and its vegetable ingredients over a temperature range of 20 to 130 EC is shown in figure 4. Among the four vegetable ingredients, jalapeno peppers had the highest thermal diffusivity values and tomatoes had the lowest thermal diffusivity values at sterilization temperatures. Second order polynomial correlations for the dependence of specific heat of *salsa con queso* and its vegetable ingredients on temperature were developed and are shown in table 5.

Dielectric properties

The dielectric properties of *salsa con queso* (high salt content product) at 915 MHz under static and continuous flow conditions are shown in figure 5. There is similar effect of temperature on dielectric properties under static and continuous flow conditions with ε' decreasing with an increase in temperature and ε" increasing with an increase in temperature. Decrease in ε' with an increase in temperature is in accordance with the observations of Datta *et al.*, (1997) for solutions containing salt. This might be explained based on the decrease in relaxation time with an increase in temperature. Since relaxation time is the time taken by polar molecules to align themselves along the direction of the field, a decrease in its value decreases the dipolar contribution to ε'. Increase in ε" with an increase in temperature is in accordance with the observations of Datta *et al.,* (1997) for solutions containing salts. This might be explained based on the relative importance of ionic conduction over dipolar contribution to ε". Thus, even though the dipolar contribution to ε" decreases with an increase in temperature, ε" increases with temperature due to increased ionic conduction. The increase in ionic conduction can be attributed to the decreasing viscosity, which in turn increases the mobility of ions (Datta *et al.*, 2005).

From figure 5, it can be seen that the dielectric properties of *salsa con queso* at 915 MHz under static and continuous flow conditions are substantially different. This result is expected for a multiphase product such as *salsa con queso*. Under static conditions, dielectric properties of only a small portion of the product (in close proximity to the probe) is measured. Therefore, measurement by this approach might under-predict or over-predict dielectric properties based on the components of the product in contact with the probe surface. It was observed that the measurement under static conditions under-predicted ε" and over-predicted ε'. This resulted in a lower value of loss tangent for the measurements under static conditions. Therefore, it is recommended that, for a multiphase product, dielectric properties be measured under continuous flow conditions for designing a continuous flow microwave heating system.

The dielectric properties of the vegetable ingredients of *salsa con queso* at 915 MHz in particulate as well as homogenized form are shown in figures 6 through 9. Density has a notable effect on dielectric properties (Nelson and You, 1990). A continuous flow microwave heating system should be designed based on the dielectric properties of the vegetable ingredients in either particulate or homogenized form depending on which one of the two has a lower value of loss tangent. This will ensure that every portion of the product receives required thermal treatment in the heating system. There is similar effect of temperature on dielectric properties with ε' decreasing with an increase in temperature and ε" increasing with an increase in temperature. This observation is in accordance with the results of Sipahioglu and Barringer (2003) for vegetables and fruits and is due to similar reasons as that for *salsa con queso*.

Salsa con queso was found to have a higher loss tangent than its vegetable ingredients and thus it will heat faster than its vegetable ingredients under a microwave field. Among the four vegetables, tomatoes were the fastest heating and onions were slowest heating under a microwave field. Second order polynomial correlations for the dependence of dielectric properties of *salsa con queso* and its vegetable ingredients on temperature at 915 MHz were developed and are shown in table 6.

Thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients presented in this study will aid in the development of a comprehensive database of these properties. The results from this study can be used to design a safe process for aseptic processing of *salsa con queso* using a continuous flow microwave system.

Conclusions

The influence of temperature on apparent viscosity of *salsa con queso* was described by an Arrhenius-type relationship. Second order polynomial correlations for the dependence of thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients on temperature were developed. The results showed that the dielectric constant decreased with an increase in temperature and the dielectric loss factor increased with an increase in temperature. The dielectric properties of *salsa con queso* at 915 MHz under static and continuous flow conditions are substantially different. The results from this study suggest that, for a multiphase product, dielectric properties measured under continuous flow conditions should be used for designing a continuous flow microwave heating system. The results from this study can be used to design a safe process for aseptic processing of *salsa con queso* using a continuous flow microwave system.

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The use of trade names in this publication does not imply endorsement by the North Carolina Agricultural Research Service of the products named nor criticism of similar ones not mentioned.

Symbols

c_p	Specific heat (J/kg-K)
B_A	Arrhenius constant (Pa-s)
E_a	Activation energy constant (J/mol)
k	Thermal conductivity (W/m-K)
R_g	Gas constant (J/mol-K)
T_A	Temperature (K)
$\tan \delta$	Loss tangent

Greek letters

ε'	Relative dielectric constant
ε''	Relative dielectric loss factor
ρ	Density (kg/m^3)
μ	Viscosity (Pa-s)
α	Thermal diffusivity (m^2/s)
σ	Shear stress (Pa)
$\dot{\gamma}$	Shear rate (s^{-1})

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Table 1. Density of *salsa con queso* and its vegetable ingredients at 22 EC

Sample	**Density (ρ in kg/m^3)**
Salsa con queso	1012
Tomatoes	1032
Bell peppers	1040
Jalapeno peppers	1060
Onions	1068

Table 2. Arrhenius constant (B_A) and activation energy constant (E_a) for *salsa con queso* at different shear rates

Shear rate (s^{-1})	**B_A (Pa-s)**	**E_a (J/mol)**	**r^2**
10	0.0168	13251.29	0.982
50	0.0115	11809.59	0.991
100	0.0104	10894.19	0.994

Table 3. Specific heat (c_p) of *salsa con queso* and its vegetable ingredients as a function of temperature

Sample	**Correlations (c_p in J/kg-K, T in EC)**	**r^2**
Salsa con queso	$c_p = 3529.2 + 0.391\ T + 0.0071\ T^2$	0.980
Tomatoes	$c_p = 3913.5 + 1.016\ T - 0.0017\ T^2$	0.938
Bell peppers	$c_p = 3922.0 + 1.017\ T + 0.0026\ T^2$	0.965
Jalapeno peppers	$c_p = 3888.6 + 0.644\ T + 0.0052\ T^2$	0.850
Onions	$c_p = 3802.7 + 2.059\ T + 0.0009\ T^2$	0.985

Table 4. Thermal conductivity (k) of *salsa con queso* and its vegetable ingredients as a function of temperature

Sample	**Correlations (k in W/m-K, T in EC)**	**r^2**
Salsa con queso	k = 0.4067 - 0.0003 T + 1E-05 T^2	0.991
Tomatoes	k = 0.4655 - 0.0015 T + 1E-05 T^2	0.995
Bell peppers	k = 0.3955 - 0.0012 T + 5E-05 T^2	0.986
Jalapeno peppers	k = 0.0374 - 0.0092 T + 1E-06 T^2	0.987
Onions	k = 0.5450 - 0.0004 T + 5E-05 T^2	0.974

Table 5. Thermal diffusivity (α) of *salsa con queso* and its vegetable ingredients as a function of temperature

Sample	**Correlations ($\alpha \times 10^6$ in m^2/s, T in EC)**	**r^2**
Salsa con queso	$\alpha = 0.1248 + 0.0005\ T - 2E\text{-}07\ T^2$	0.995
Tomatoes	$\alpha = 0.1273 + 0.0003\ T + 4E\text{-}06\ T^2$	0.995
Bell peppers	$\alpha = 0.0328 + 0.0021\ T - 3E\text{-}06\ T^2$	0.995
Jalapeno peppers	$\alpha = 0.1200 + 0.0006\ T + 7E\text{-}06\ T^2$	0.999
Onions	$\alpha = 0.1520 + 0.0006\ T + 2E\text{-}06\ T^2$	0.998

Table 6. Dielectric properties of *salsa con queso* and its vegetable ingredients as a function of temperature at 915 MHz

Sample	Correlations (T in EC)	r^2
Salsa con queso (Static conditions)	$\varepsilon N = 53.9 + 0.0398\ T - 0.0005\ T^2$	0.998
	$\varepsilon 0 = 36.0 + 0.0389\ T + 0.0046\ T^2$	0.999
Salsa con queso (Continuous flow conditions)	$\varepsilon N = 62.2 - 0.1609\ T - 4E\text{-}05\ T^2$	0.994
	$\varepsilon 0 = 25.3 + 0.8009\ T + 0.0009\ T^2$	0.998
Tomato (Particulates)	$\varepsilon N = 84.5 - 0.0980\ T - 0.0005\ T^2$	0.999
	$\varepsilon 0 = 26.3 - 0.0615\ T + 0.0022\ T^2$	0.994
Tomato (Puree)	$\varepsilon N = 70.7 + 0.0280\ T - 0.0010\ T^2$	0.997
	$\varepsilon 0 = 28.5 + 0.1055\ T + 0.0031\ T^2$	0.998
Bell peppers (Particulates)	$\varepsilon N = 63.8 - 0.0882\ T - 0.0001\ T^2$	0.995
	$\varepsilon 0 = 10.0 + 0.0377\ T + 0.0009\ T^2$	0.997
Bell peppers (Puree)	$\varepsilon N = 56.5 + 0.0696\ T - 0.0010\ T^2$	0.997
	$\varepsilon 0 = 10.2 - 0.0483\ T + 0.0009\ T^2$	0.997
Jalapeno peppers (Particulates)	$\varepsilon N = 58.8 + 0.0621\ T - 0.0009\ T^2$	0.946
	$\varepsilon 0 = 14.0 + 0.0683\ T + 0.0015\ T^2$	0.992
Jalapeno peppers (Puree)	$\varepsilon N = 64.5 + 0.0803\ T - 0.0011\ T^2$	0.999
	$\varepsilon 0 = 17.6 - 0.0800\ T + 0.0017\ T^2$	0.999
Onion (Particulates)	$\varepsilon N = 65.3 - 0.1289\ T - 7E\text{-}05\ T^2$	0.894
	$\varepsilon 0 = 11.6 - 0.0317\ T + 0.0007\ T^2$	0.959
Onion (Puree)	$\varepsilon N = 67.6 + 0.0465\ T - 0.0011\ T^2$	0.999
	$\varepsilon 0 = 13.9 + 0.0854\ T + 0.0011\ T^2$	0.998

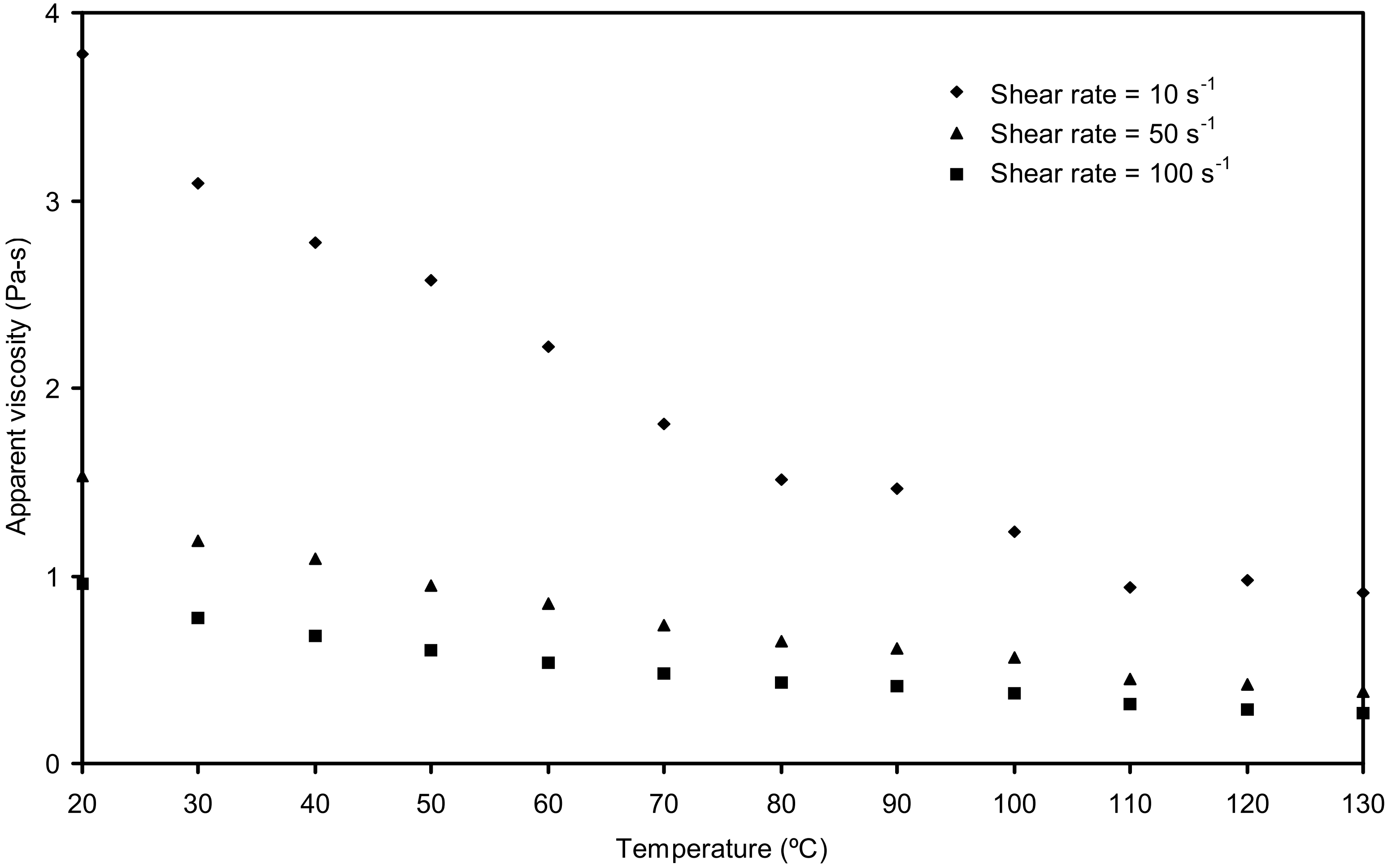

Figure 1. Apparent viscosity of *salsa con queso* at different shear rates

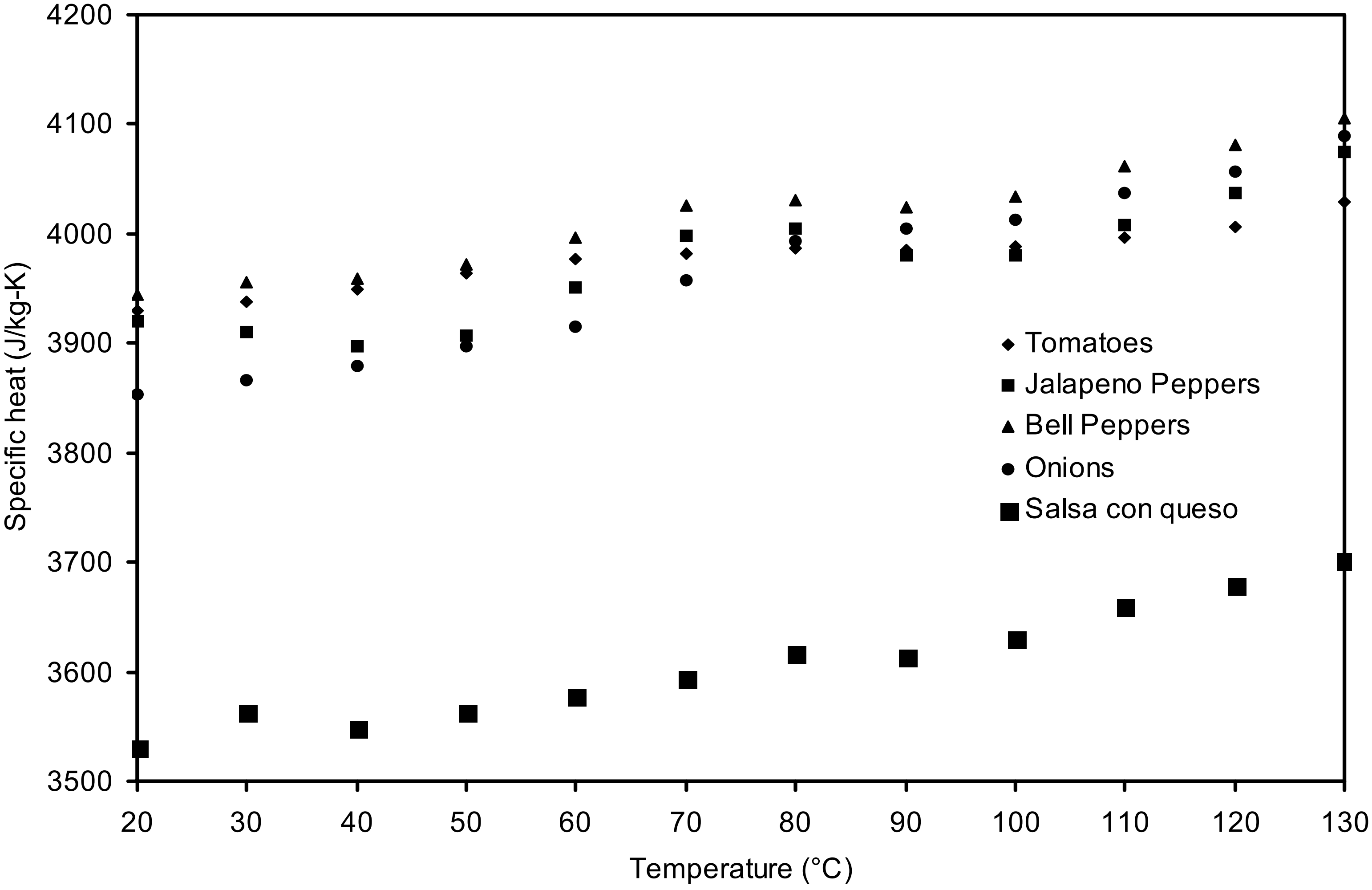

Figure 2. Specific heat of *salsa con queso* and its vegetable ingredients

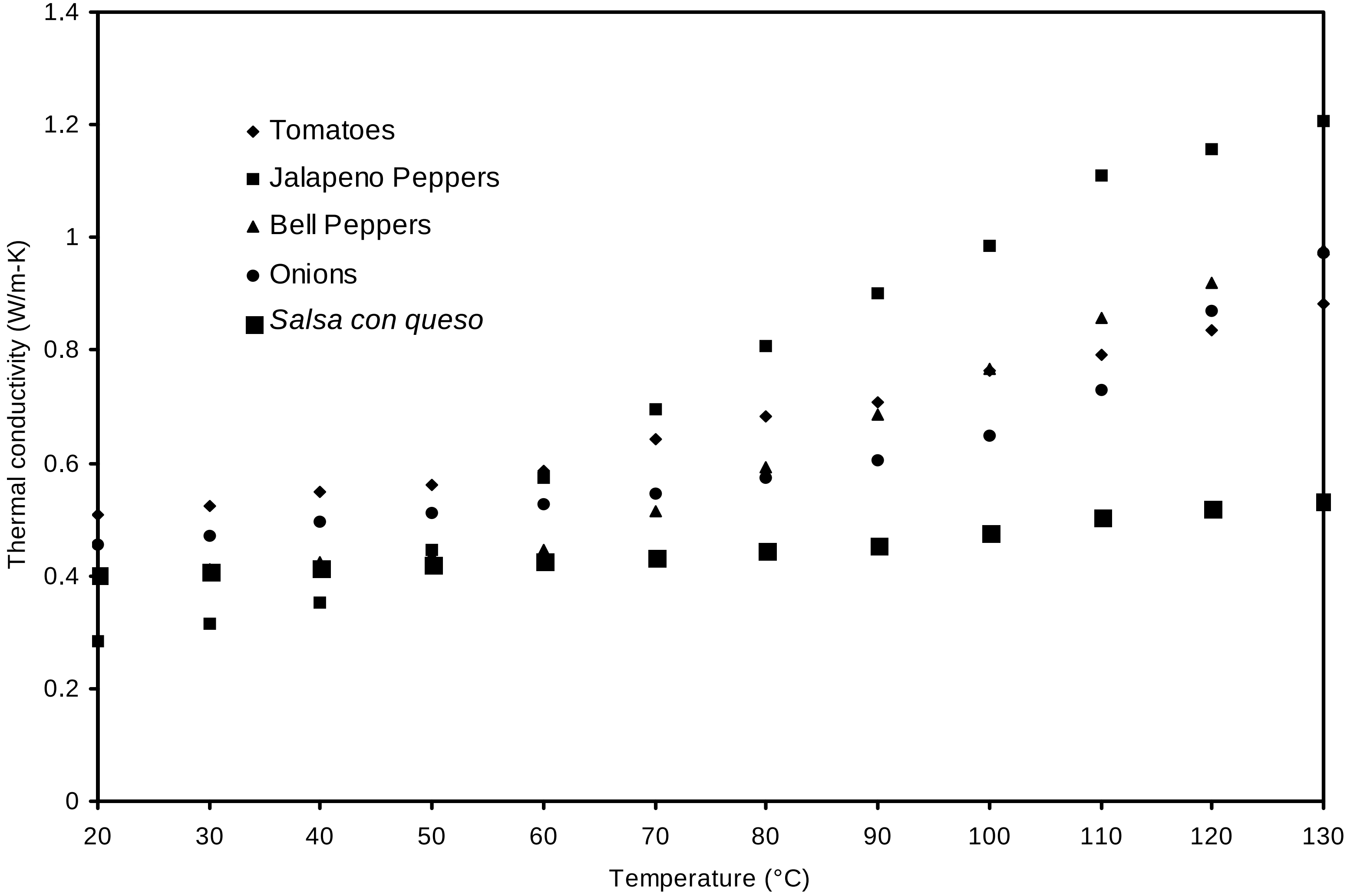

Figure 3. Thermal conductivity of *salsa con queso* and its vegetable ingredients

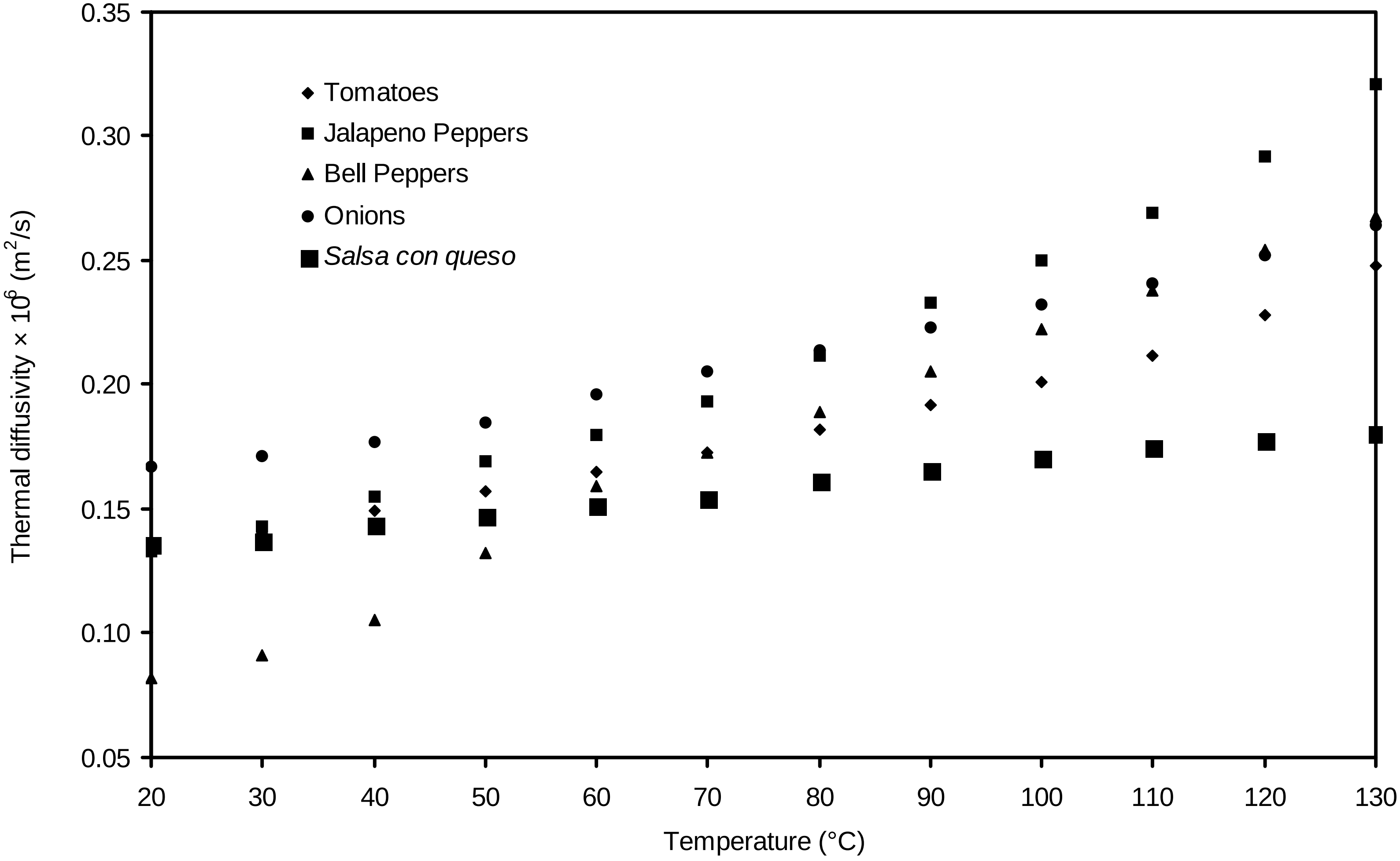

Figure 4. Thermal diffusivity of *salsa con queso* and its vegetable ingredients

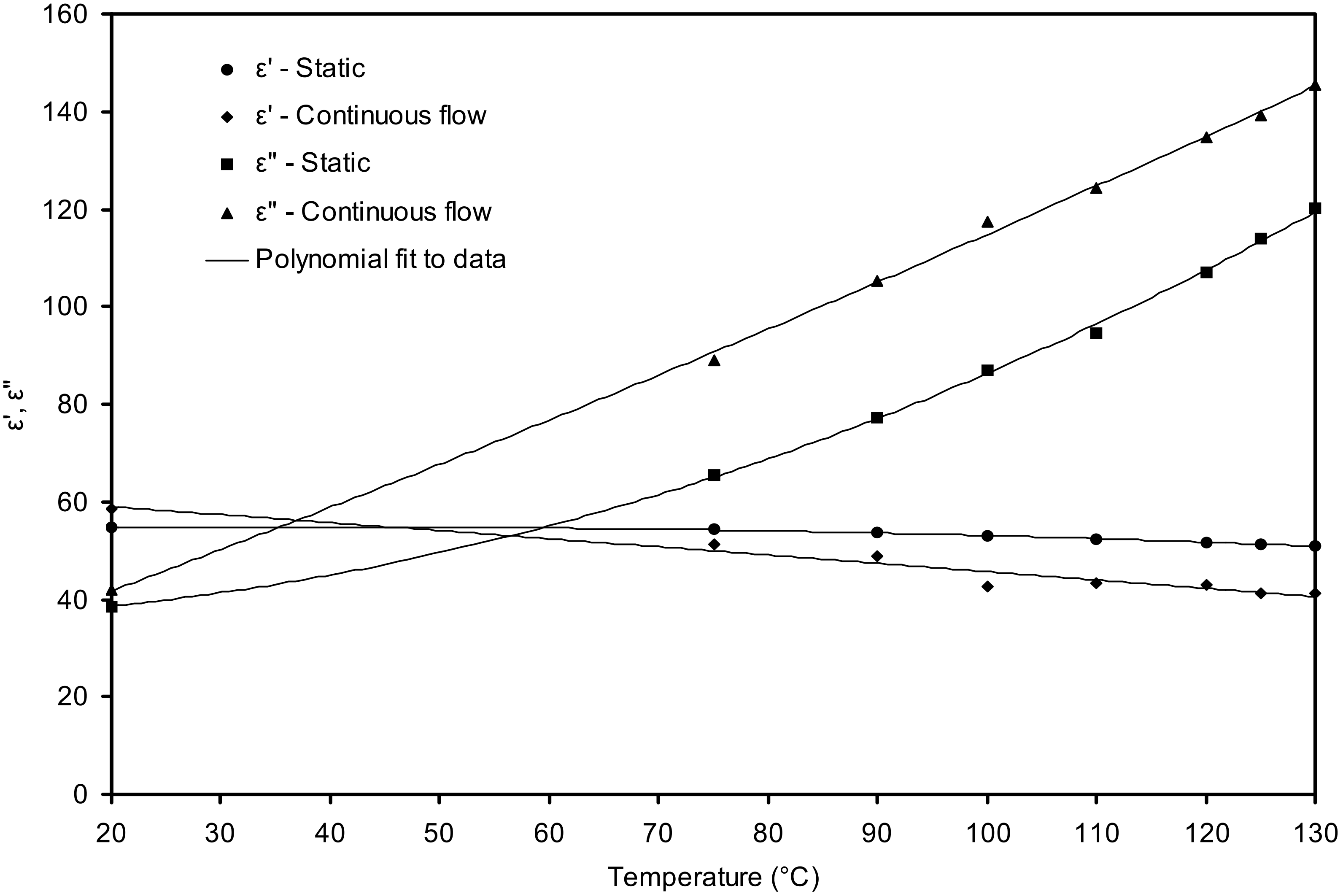

Figure 5. Dielectric constant (εℕ) and dielectric loss factor (ε0) of *salsa con queso* at 915 MHz

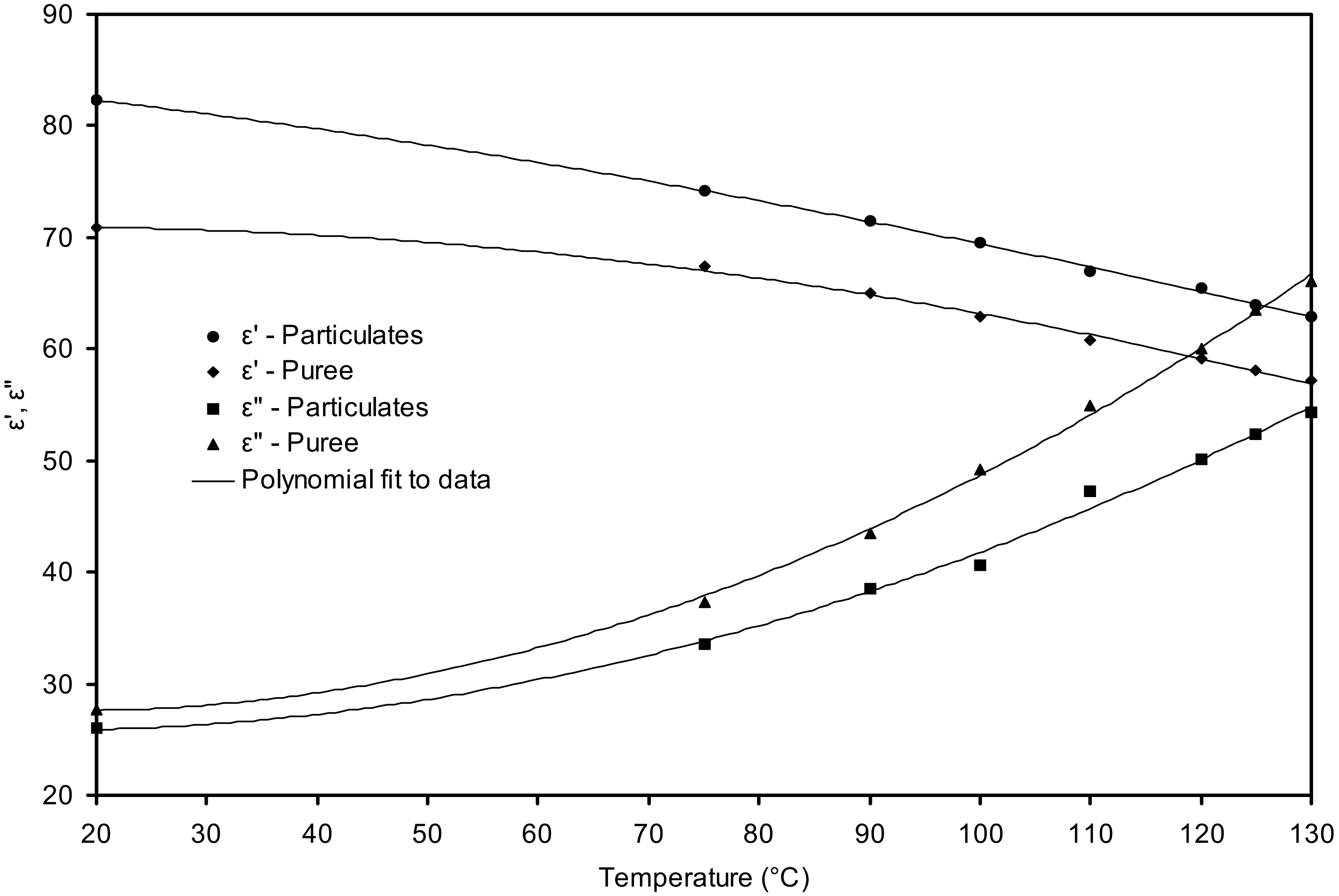

Figure 6. Dielectric constant (εℕ) and dielectric loss factor (ε0) of tomatoes at 915 MHz

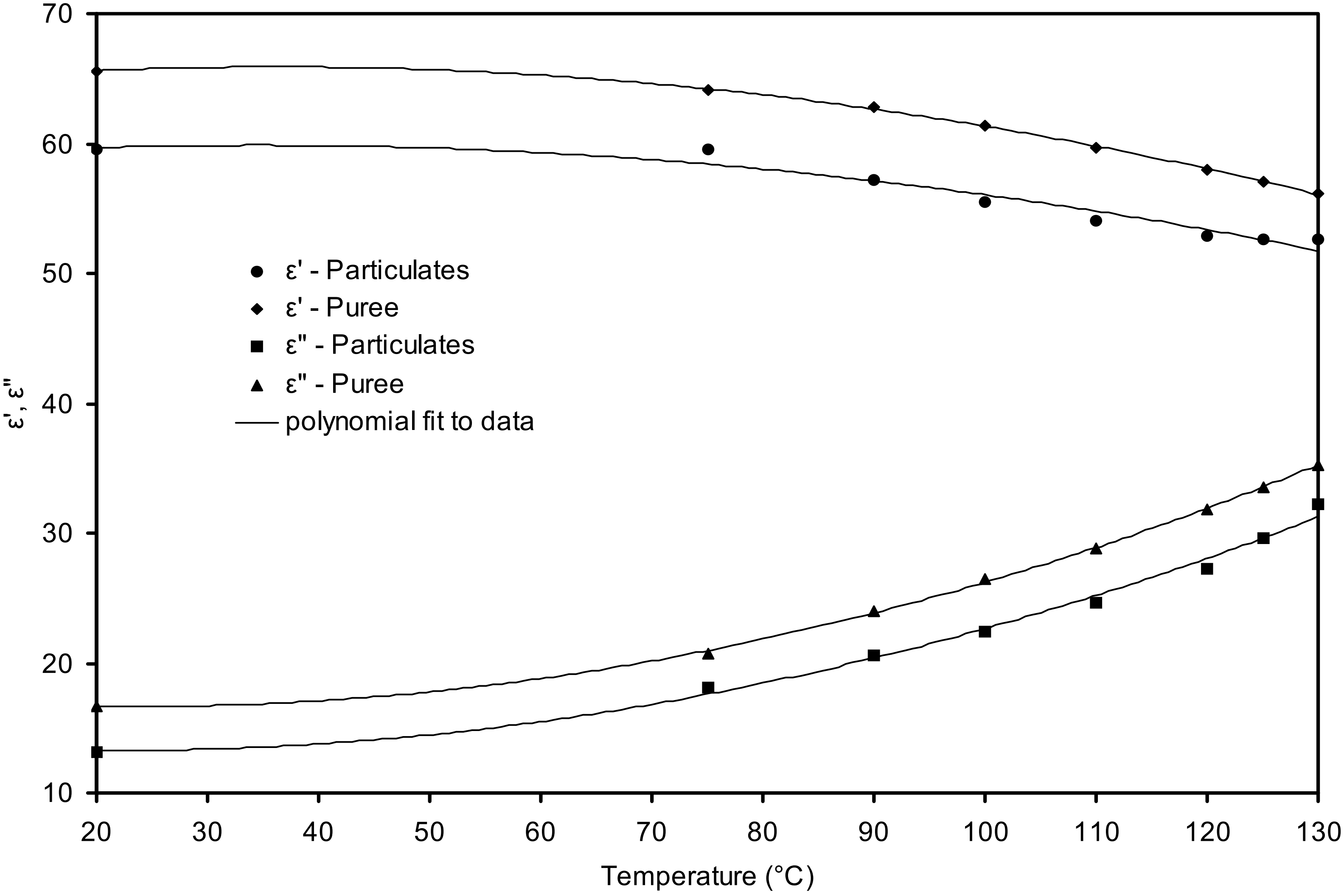

Figure 7. Dielectric constant (εN) and dielectric loss factor (ε0) of jalapeno peppers at 915 MHz

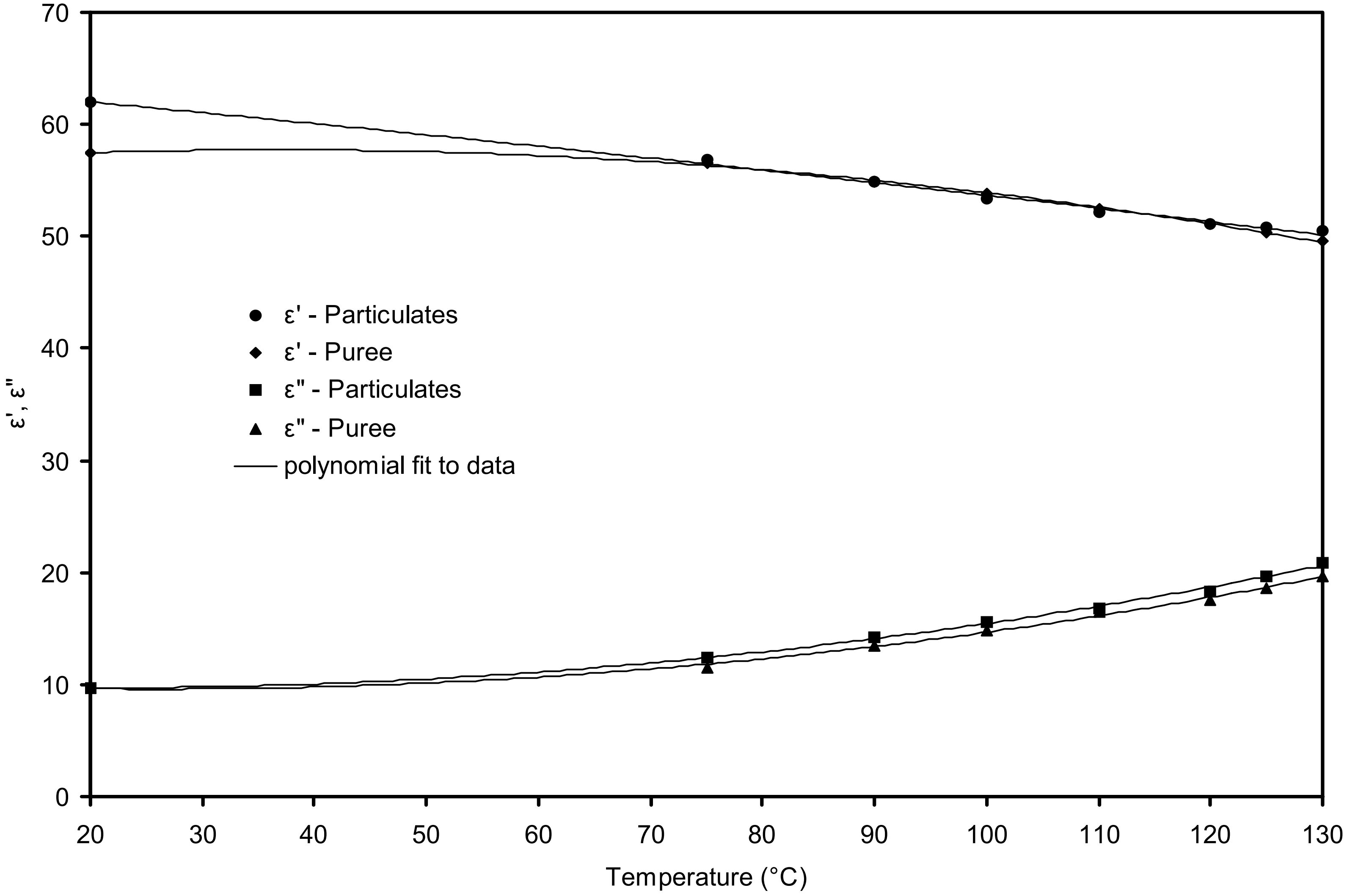

Figure 8. Dielectric constant (εN) and dielectric loss factor (ε0) of bell peppers at 915 MHz

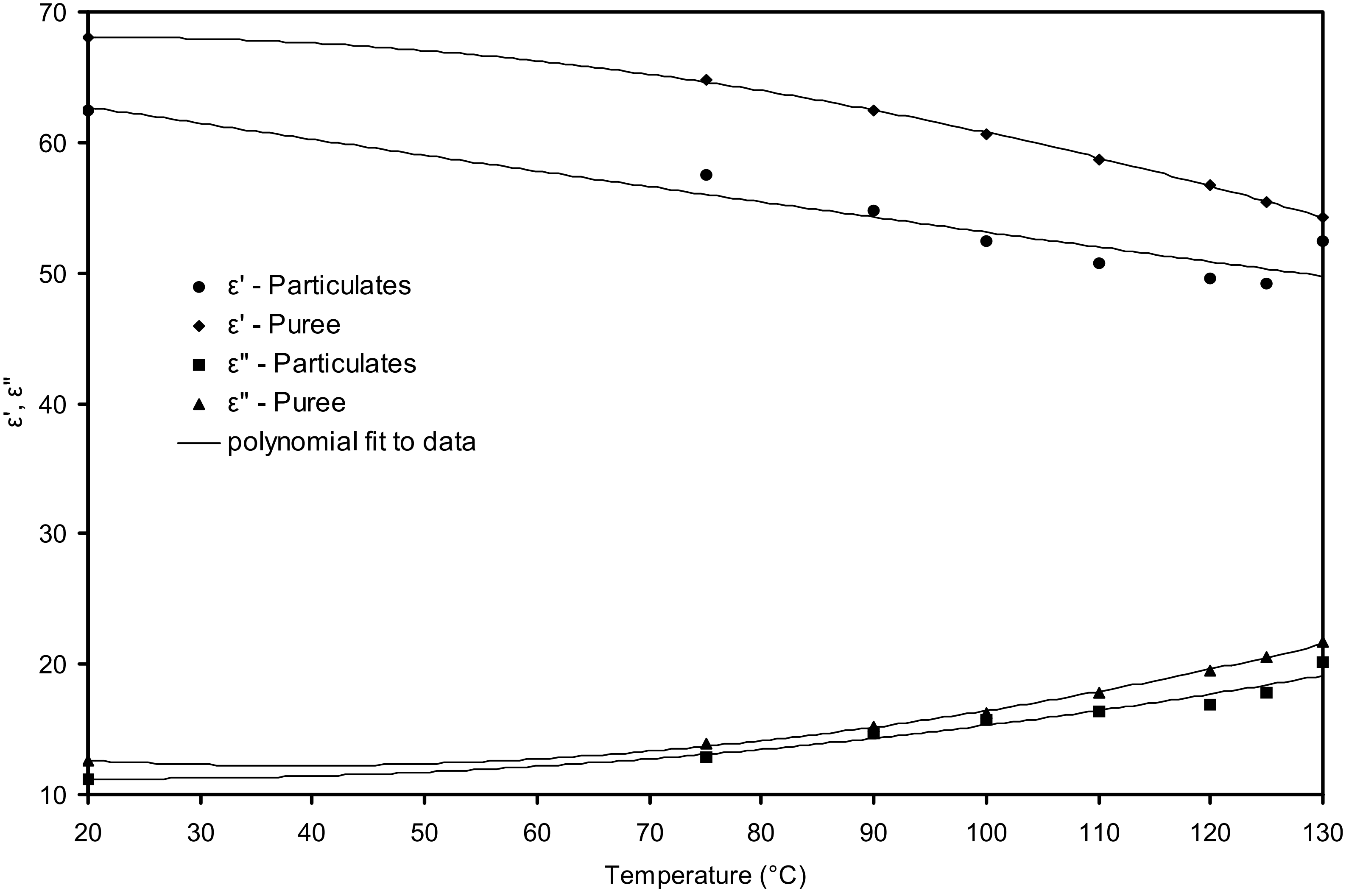

Figure 9. Dielectric constant (εN) and dielectric loss factor (εO) of onions at 915 MHz

Chapter 5

MANUSCRIPT III

Feasibility of aseptic processing of a low-acid multiphase food product using a continuous flow microwave system

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Abstract

Aseptic processing of a low-acid multiphase food product using continuous flow microwave heating system can combine the advantages of an aseptic process along with those of microwave heating. Dielectric properties of two different brands of one such product (*salsa con queso*) were measured under continuous flow conditions at a temperature range of 20 to 130 EC. At 915 MHz, dielectric constant ranged from 58.7 at 20 EC to 41.3 at 130 EC with dielectric loss factor ranging from 41.0 at 20 EC to 145.5 at 130 EC. The loss tangent at 915 MHz ranged from 0.61 at 20 EC to 3.52 at 130 EC. The temperature profiles at the outlet during processing of *salsa con queso* in a 5 kW microwave unit showed a narrow temperature distribution between the center and the wall of the tube. The study showed the feasibility of aseptic processing of *salsa con queso* using a continuous flow microwave system.

Keywords: aseptic processing, microwave heating, dielectric properties, *salsa con queso*

Introduction

Thermal processing of food products is the most widely used method of food preservation. The extent of thermal treatment given to a food product depends on whether the food product is a high-acid product or a low-acid product. Traditionally, conventional canning has been used to process low-acid food products to ensure the destruction of *Clostridium botulinum* spores (U.S. Food and Drug Administration, 1992). Excessive thermal treatment of the product in conventional canning results in degradation of color, flavor, texture, and nutrients (David *et al.*, 1996).

Aseptic processing offers a potential option from conventional canning to meet the demand for convenient and high quality foods. As opposed to conventional canning, the use of high temperature for a short period of time in aseptic processing yields a high quality product with the same level of microbiological safety as that in a conventional canning system. Aseptic processing of liquid foods and foods containing small particles (smaller than 3.2 mm) have been in place for several decades. However, aseptic processing of low-acid multiphase foods containing large particulates (larger than 3.2 mm) such as soups containing meatballs or vegetables has not been a commercial reality in the U.S. even though it has been in place in the European market for several years (Morris-Lee, 2004).

Continuous flow microwave heating is an emerging technology in the food industry with a potential to replace the conventional retort process. Microwave heating offers instant start-up and rapid heating of food products. Continuous flow microwave heating is also associated with improved color, flavor, texture, and nutrient retention. Heating of food products using continuous flow microwave systems have been reported by various researchers (Nikdel *et al.*, 1993; Tajchakavit *et al.*, 1998; Coronel *et al.*, 2003; Gentry and Roberts, 2005). Recently, aseptic processing of sweetpotato puree using a continuous flow microwave system has also been reported (Coronel *et al.*, 2005).

Dielectric properties determine the extent of heating of a material when subjected to a microwave field. Therefore, knowledge of dielectric properties is important for the design of a continuous flow microwave heating system. Dielectric properties consist of dielectric constant (ε') and dielectric loss factor (ε"). Dielectric constant is a measure of the ability of a material to store electromagnetic energy whereas dielectric loss factor is a measure of the ability of a material to convert electromagnetic energy to heat (Metaxas and Meredith, 1983). Loss tangent (tan δ), a parameter used to describe how well a product absorbs microwave energy, is the ratio of dielectric loss factor (ε") to the dielectric constant (ε'). A product with a higher loss tangent will heat faster under microwave field as compared to a product with a lower loss tangent (Nelson and Datta, 2001).

Aseptic processing of a low-acid multiphase food product using continuous flow microwave heating system can combine the advantages of an aseptic process along with those of microwave heating. One such product is *salsa con queso* (salsa with cheese) which is a commercially successful product. Aseptic processing of this category of products could expand its market by enhancing quality and enabling the use of packages of different sizes and shapes. Therefore, the objective of this study was to evaluate the feasibility of aseptic processing of a low-acid multiphase food product (*salsa con queso*) using a continuous flow microwave system.

Materials and methods

Sample selection

Two brands of *salsa con queso* (Brands B and C, representative of the category of *salsa con queso*) were purchased from a local supermarket (Food Lion, Raleigh, NC). The samples were stored in a refrigerator prior to use in the experiments.

Measurement of dielectric properties

Dielectric properties of *salsa con queso* were measured from 20 to 130 EC using an open-ended coaxial probe (Model HP 85070E, Agilent Technologies, Palo Alto, CA) connected to a network analyzer (Model HP 8753C, Agilent Technologies, Palo Alto, CA). Dielectric properties of *salsa con queso* were measured under continuous flow conditions because a study done by Kumar *et al.* (2006) recommends that, for a multiphase product such as *salsa con queso*, dielectric properties measured under continuous flow conditions should be used for designing a continuous flow microwave heating system. The network analyzer was calibrated by leaving the tip of the probe in contact with air, metal, and 25 ºC de-ionized water and measuring the dielectric properties. The dielectric properties were measured at 20, 75, 90, 100, 110, 120, 125, and 130 ºC and at frequencies from 300 to 3,000 MHz with an increment of 5 MHz. The variable step size for temperature increment was chosen because the aim in this study was to determine the dielectric properties at sterilization temperatures. For measurement of dielectric properties during continuous flow conditions, the dielectric probe was inserted in one of the three ports of a smart gasket (Rubber-Fab, Newton, NJ) at the exit of the applicator and the product was heated using a

5 kW microwave generator operating at 915 MHz. The 5 kW unit consisted of a 5 kW microwave generator (Industrial Microwave Systems, Morrisville, NC) operating at 915 MHz, a waveguide of rectangular cross-section, and a specially designed focused applicator (Drozd and Joines, 1999).

5 kW microwave unit

The microwave unit, shown in figure 1, consists of a 5 kW microwave generator (Industrial Microwave Systems, Morrisville, NC) operating at 915 MHz, a waveguide of rectangular cross-section, and a specially designed focused applicator (Drozd and Joines, 1999). A tube of 1.5" nominal diameter (0.038 m ID) made of Polytetrafluoroethylene (PTFE or Teflon®) was placed at the center of the applicator through which the product was pumped at a rate of 0.9 liters per minute using a positive displacement pump (Model MD012, Seepex GmbH+ Co, Bottrop, Germany) with a variable speed motor (Tri- Clover Rotary Pump, Model PRE3-1M, Ladish Co., Kenosha, WI). The output power of the microwave was maintained at 3 kW. The temperatures at the inlet and outlet of the applicator were recorded using a thermocouple arrangement described by Coronel *et al.* (2003) and a datalogging system (Model DAS-16, Keithley Metrabyte Inc., Taunton, MA). The dielectric probe (HP 85070E) was inserted at the outlet of the applicator in one of the three ports of the smart gasket (Coronel *et al.*, 2003) as shown in figure 1.

Results and discussion

Dielectric properties

The dielectric properties of two different brands (Brands B and C) of *salsa con queso* (high salt content product) at 915 MHz are shown in figure 2. There is similar effect of temperature on dielectric properties with ε' decreasing with an increase in temperature and ε" increasing with an increase in temperature which is in accordance with the observations of Datta *et al.*, (1997) for solutions containing salt. At 915 MHz, ε' ranged from 58.7 at 20 EC to 41.3 at 130 EC and ε" ranged from 41.0 at 20 EC to 145.5 at 130 EC. The loss tangent at 915 MHz ranged from 0.61 at 20 EC to 3.52 at 130 EC. The loss tangent of *salsa con queso* is very high in comparison to that of other food materials (loss tangent for water is 0.02 at 20 EC). Thus, *salsa con queso* has the potential to be aseptically processed using a continuous flow microwave system. Second order polynomial correlations for the dependence of dielectric properties of both brands of *salsa con queso* on temperature at 915 MHz were developed and are shown in table 1.

Processing in the 5 kW microwave unit

Salsa con queso was processed using the 5 kW microwave unit with a microwave power output of 3 kW and at a flow rate of 0.9 liters per minute. Temperatures at the inlet and outlet of the applicator for brands B and C are shown in figures 3 and 4 respectively. For brand B, the average temperature difference between the inlet and outlet of the applicator varied from 10.6 to 20.0 EC with 16.6 EC being the average for a product flow rate of 0.9 liters per minute. For brand C, the average temperature difference between the inlet and outlet of the applicator varied from 6.4 to 20.0 EC with 11.9 EC being the average for a product flow rate of 0.9 liters per minute.

From figures 3 and 4, it can be seen that the differences in the temperatures between the center and the wall of the tube at the outlet became smaller as the output temperature increased. This might be due to a more uniform absorption of microwave energy by *salsa con queso* at higher temperature as its loss tangent increased with an increase in temperature. The wide temperature distribution between the center and the wall in a conventional heating system results in excessive thermal treatment of the product at the wall. Thus, aseptic processing of low-acid multiphase product such as *salsa con queso* using a continuous flow microwave system could overcome the problems (degradation of color, flavor, texture, and nutrients) associated with the conventional retort process.

This study shows the feasibility of aseptic processing of *salsa con queso* using a continuous flow microwave system. However, further research is required to biologically validate such a process as the final step in establishing an aseptic process for *salsa con queso* using a continuous flow microwave system.

Conclusions

The results showed that the dielectric constant of *salsa con queso* decreased with an increase in temperature and the dielectric loss factor increased with an increase in temperature. Second order polynomial correlations for the dependence of dielectric properties of the products on temperature at 915 MHz were developed. The temperature profiles at the outlet during processing of *salsa con queso* in a 5 kW microwave unit showed a narrow temperature distribution between the center and the wall of the tube. Thus, continuous flow microwave heating could overcome the problems (degradation of color, flavor, texture, and nutrients) associated with the wider temperature distribution between the center and the wall in a conventional heating system. The study showed the feasibility of aseptic processing of *salsa con queso* using a continuous flow microwave system. However, further research is required to biologically validate such a process as the final step in establishing an aseptic process for *salsa con queso* using a continuous flow microwave system.

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The use of trade names in this publication does not imply endorsement by the North Carolina Agricultural Research Service of the products named nor criticism of similar ones not mentioned.

Symbols

$\tan \delta$	Loss tangent

Greek letters

ε'	Relative dielectric constant
ε''	Relative dielectric loss factor

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Table 1. Dielectric properties of two different brands of *salsa con queso* as a function of temperature at 915 MHz

Sample	**Correlations (T in EC)**	**r^2**
Salsa con queso (Brand B)	εN = 70.9 - 0.1961 T - 2E-05 T^2	0.997
	ε0 = 25.1 + 0.7591 T + 0.0007 T^2	0.996
Salsa con queso (Brand C)	εN = 62.2 - 0.1609 T - 4E05 T^2	0.994
	ε0 = 25.3 + 0.8009 T + 0.0009 T^2	0.998

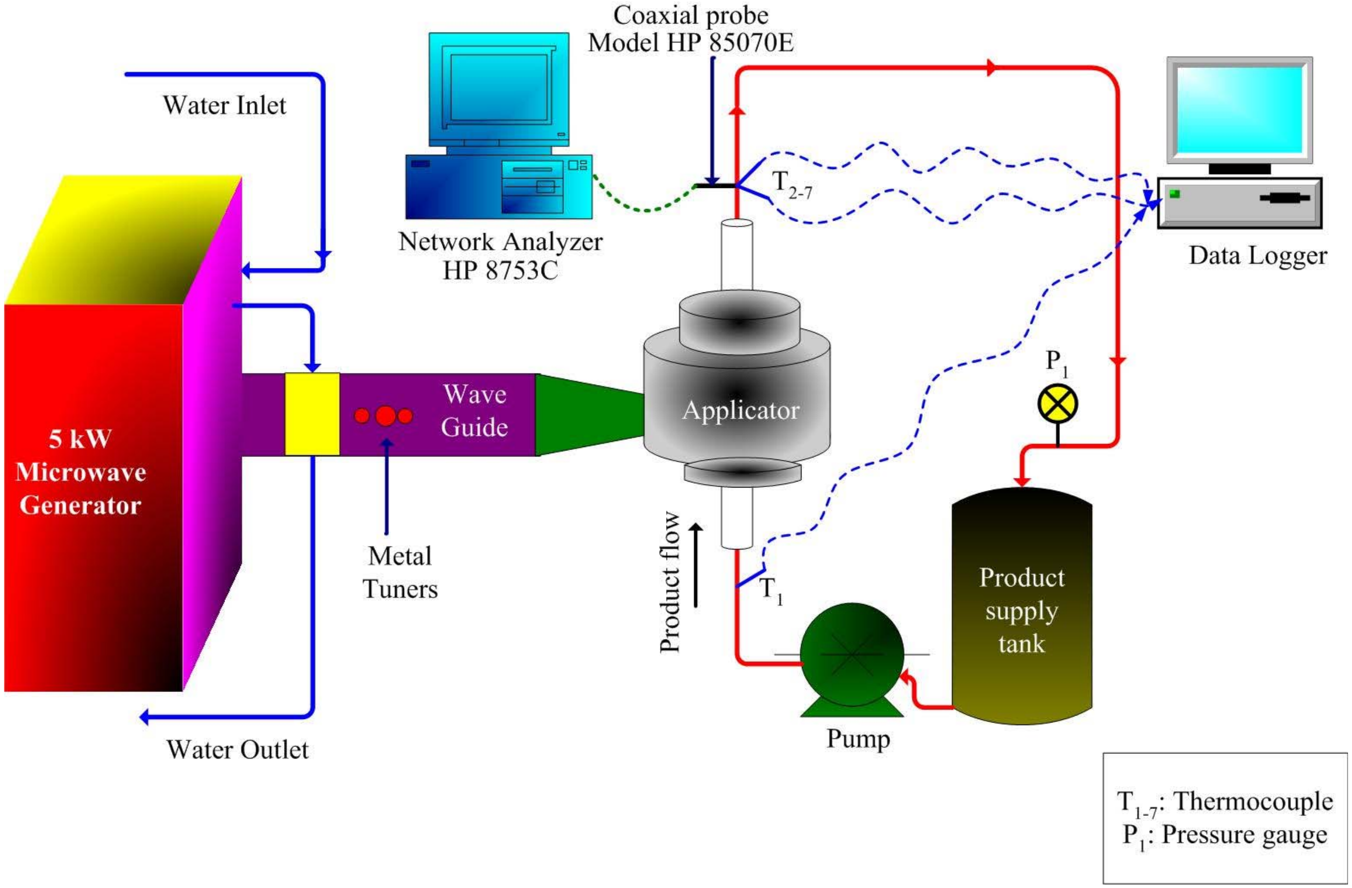

Figure 1. Schematic of the 5 kW microwave unit

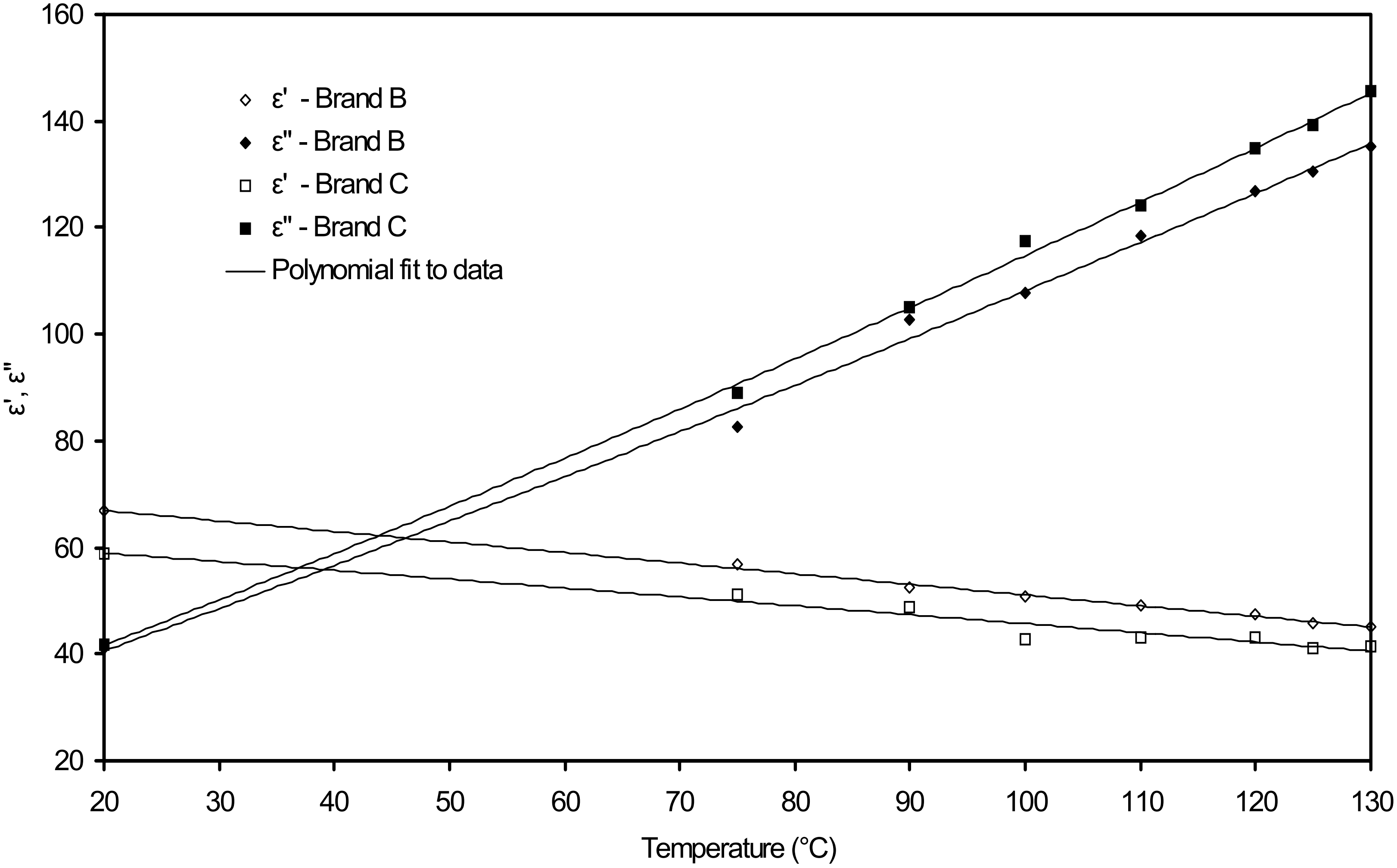

Figure 2. Dielectric constant (εℕ) and dielectric loss factor (ε0) of two different brands of *salsa con queso* at 915 MHz

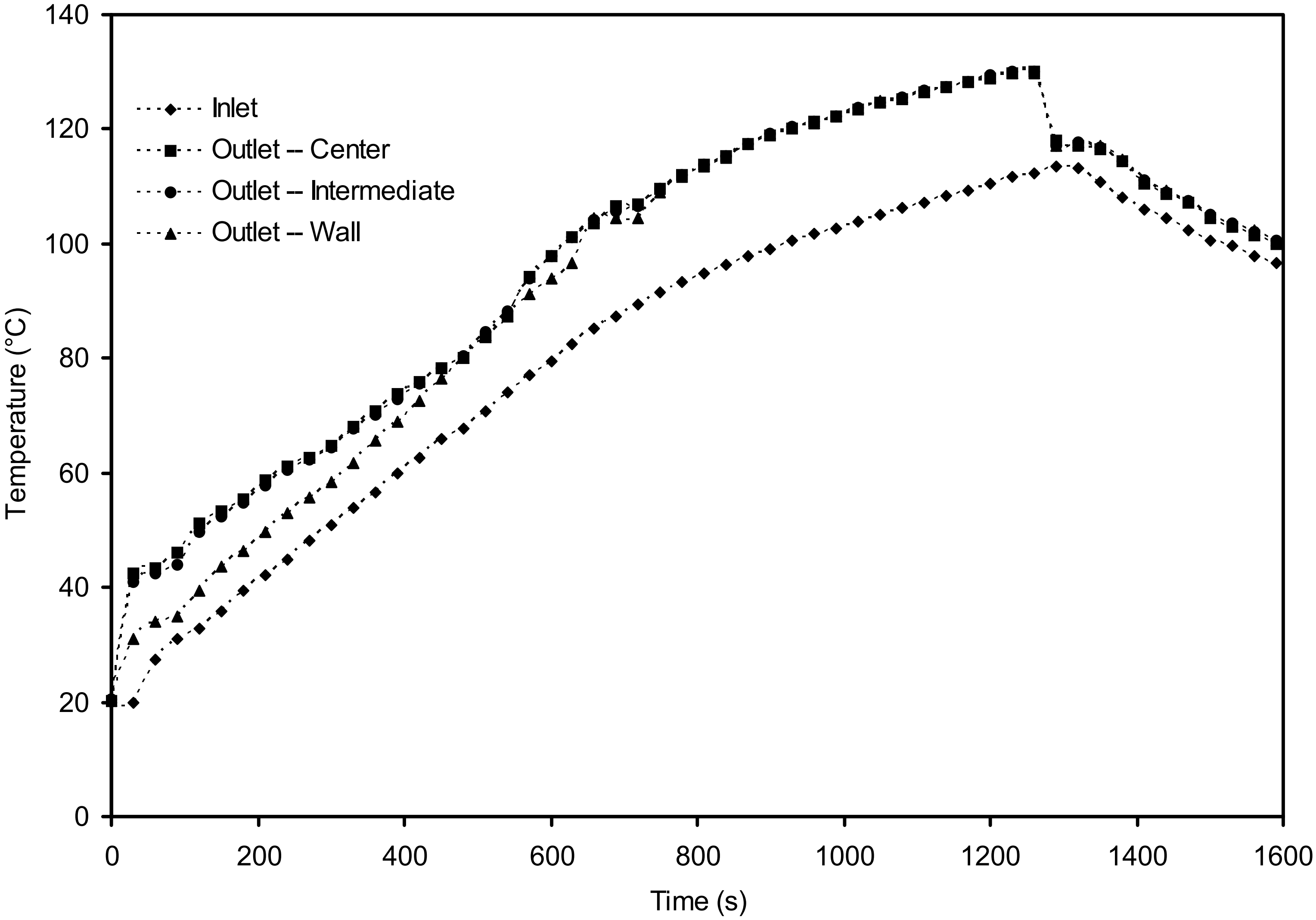

Figure 3. Temperature profile during processing of *salsa con queso* (Brand B) in the 5 kW microwave unit

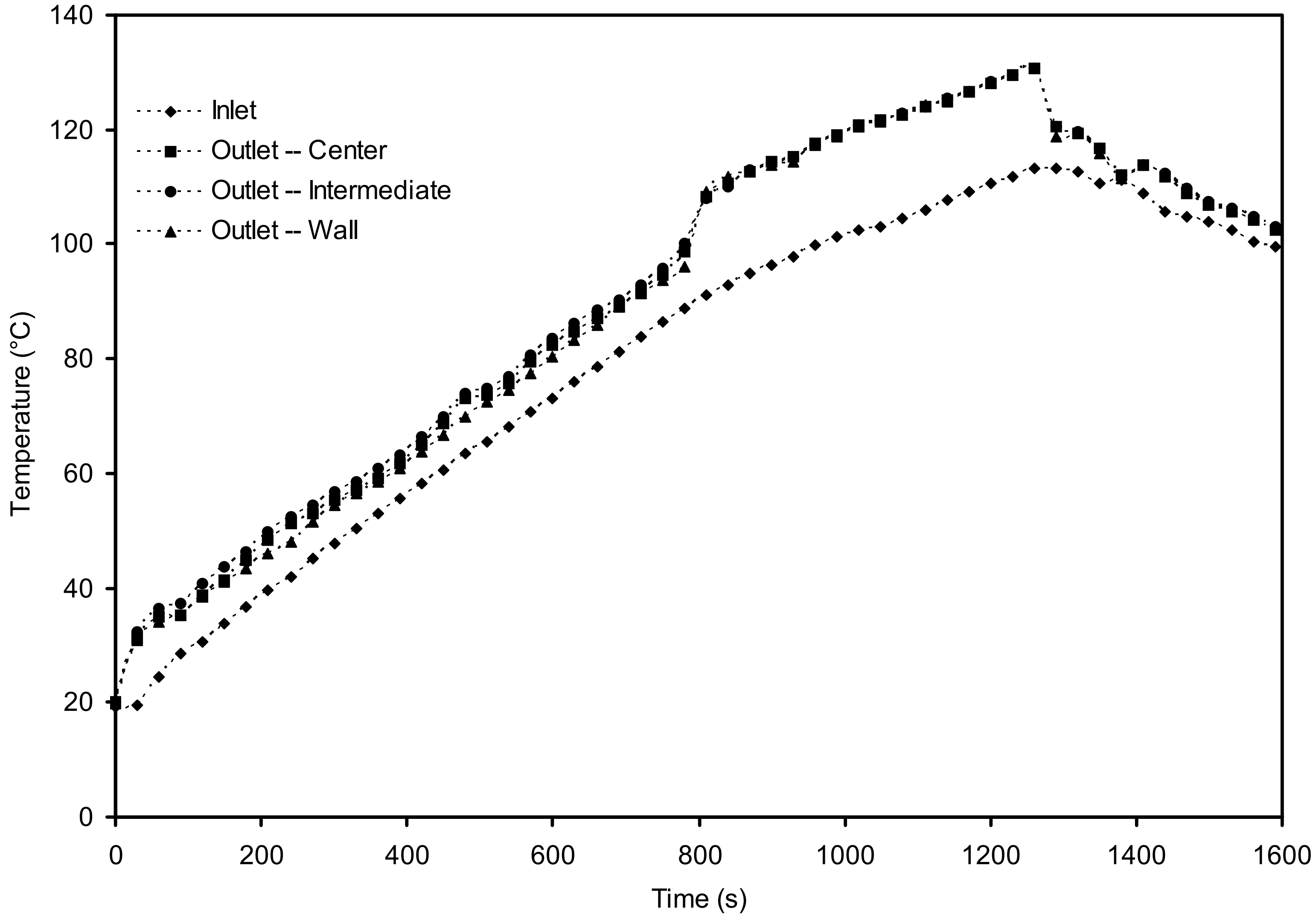

Figure 4. Temperature profile during processing of *salsa con queso* (Brand C) in the 5 kW microwave unit

Chapter 6

CONCLUSIONS

The results from this study will assist processors in designing a safe process for aseptic processing of low-acid multiphase food products such as *salsa con queso* using a continuous flow microwave system and in biologically validating such a process as the final step in establishing an aseptic process for *salsa con queso*.

The results showed that the dielectric properties measured under static and continuous flow conditions were similar for homogeneous food products such as skim milk and vegetable puree, but they were substantially different for *salsa con queso* which is a multiphase food product. Therefore, it is suggested that, for a multiphase product, dielectric properties measured under continuous flow conditions should be used for designing a continuous flow microwave heating system. This new approach to measure dielectric properties can also be used to monitor physico-chemical changes in a food product during thermal processing which can be used a tool for research and development in the food industry.

Second order polynomial correlations for the dependence of thermophysical and dielectric properties of *salsa con queso* and its vegetable ingredients on temperature were developed. The results were used to fabricate design particles from PP (polypropylene) and PMP (polymethylpentene) using a custom developed CPD (Conservative Particle Design). These design particles could be used as thermo-sensitive implant carriers for bacterial spores in biological validation of a multiphase aseptic process.

The temperature profiles at the outlet during processing of *salsa con queso* in the 5 kW microwave unit showed a narrow temperature distribution between the center and the wall of the tube. The results showed feasibility of aseptic processing of *salsa con queso* using a continuous flow microwave system. Thus, continuous flow microwave heating could overcome the problems (degradation of color, flavor, texture, and nutrients) associated with the wider temperature distribution between the center and the wall in a conventional heating system.

Chapter 7

RECOMMENDATIONS FOR FUTURE WORK

Results from this study showed the feasibility of aseptic processing of *salsa con queso* using a continuous flow microwave system. Some recommendations for future work are presented below:

There is a need to better understand the interactions between multiphase food products and microwaves. This study was limited to the processing of only one multiphase product (*salsa con queso*) using microwaves. Therefore, further experiments should be conducted to process a wide variety of multiphase food products using a continuous flow microwave system and comparing the results with a mathematical model.

The design particles could be used as carriers for bacterial spores in biological validation of a multiphase aseptic process using a continuous flow microwave heating system. The conservative nature of the design particles should be experimentally validated by acquiring time-temperature data for the design and the real food particles under sterilization conditions in an autoclave (>120 EC and 24 psi).

The final step in establishing an aseptic process for *salsa con queso* would require biological validation of the process. The validation tests should be conducted at different temperatures to achieve a positive/negative result at the end of the process. This will aid in determining the minimum allowable process temperature that will result in a safe process. Based on these tests, a process should be designed for final validation by processing *salsa con queso* along with more than 299 design particles having a spore suspension in their cavity using a continuous flow microwave system. To ensure a safe process, microbial tests should be conducted to show that the required reduction of the spores is achieved. Once validated, the process can be used for commercial production of the food product.