# INVESTIGATION OF METHODS TO IMPROVE HEATING UNIFORMITY FOR RADIO FREQUENCY PASTEURIZATION OF FOOD POWDERS

by

### DAMLA DAG

(Under the Direction of Fanbin Kong and Rakesh K. Singh)

### ABSTRACT

Hot air and steam heating are the most commonly used techniques for food powder pasteurization in the food industry. In conventional heating, heat is transferred from the hot air to the food surface and then it is transferred to the inside of the food matrix. Conventional heating is time and energy-consuming and the airflow may not be uniformly distributed in the powder. In this regard, radio frequency (RF) heating is a promising alternative technology for food powder pasteurization. The working principle of RF heating is similar to microwave heating but RF heating has a relatively deeper penetration depth in foods and lower equipment cost. Heat is generated by the interaction between RF energy and the molecules inside the food; thus, pasteurization is evolved from inside the products. Despite the effectiveness of this method, heating uniformity is still a major concern of RF heating which is caused by the heterogeneity of food compositions and the non-uniform distribution of the electric field. In this study, to overcome the non-uniform heating limitation, the food powder container was surrounded by a liquid medium. Firstly, the effect of container dimension and geometry on RF heating was evaluated to address the problem to be solved. Then, the effect of the surrounding medium on RF heating performance was investigated. As a final step, the proposed oil-immersion system was validated via microbiological inoculation and subsequent RF assisted thermal processing. RF heating of whole milk powder surrounded by soybean oil increased the heating rate by 25% compared to the one surrounded by air. *E. faecium* NRRL B-2354 was considered as a surrogate of *Salmonella* spp. for whole milk powder to validate RF pasteurization due to its higher thermal resistances than *Salmonella* spp. More than 5 log CFU/g reduction of *Salmonella* spp. was achieved by RF heating the whole milk powder to 75 °C followed by holding it in the oven at 75 °C for 2 h. The study indicated that soybean oil as a surrounding material improves RF heating uniformity and increases the heating rate of food powders. This information is useful for developing RF pasteurization technology for commercialized applications in food powders.

INDEX WORDS: Radio frequency heating, Heating uniformity, Dielectric properties, Food powder, Milk powder, Corn flour

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A Dissertation Submitted to the Graduate Faculty of The University of Georgia in Partial Fulfillment of the Requirements for the Degree

DOCTOR OF PHILOSOPHY

ATHENS, GEORGIA

2021

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To my beloved family,

#### ACKNOWLEDGEMENTS

I would first like to express my sincere gratitude to my Ph.D. advisor Prof. Fanbin Kong and co-advisor Prof. Rakesh K. Singh for the continuous support of my dissertation study, related research, academic career, and their patience, motivation, and immense knowledge. They were and remain my best role models as scientists, mentors, and teachers. It has been an honor and a privilege to be involved in their project under their guidance.

I am also highly thankful to my other committee members: Drs. Jinru Chen, Abhinav Mishra and Samir Trabelsi. I would also like to extend my gratitude to my M.Sc. advisor Assoc. Prof. Mecit Halil Oztop for his support, guidance, and encouragement.

Every result described in this dissertation was accomplished with the support of current and past lab-mates, friends, and all the faculty, staff, and technicians in the Department of Food Science and Technology who were always willing to help and give their best suggestions.

Finally, it is a pleasure to express my deepest thanks and gratitude to Tayfun Efe Ertop for his continuous support and encouragement throughout my years of study. I would not be able to start this journey without your endless support and love. I owe a deep sense of gratitude to my mother, Huriye Dag, father, Saban Dag, and brother, Ugur Dag, for providing me with unfailing support and continuous encouragement throughout my life with their endless love. I am indebted to my parents for giving me this opportunity and experience that have made me who I am.

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### CHAPTER 1

#### INTRODUCTION

Low moisture foods include a large category of food products such as nuts, dried fruits, and vegetables, herbs and species, flours, powders, legumes, grains, etc. In general, food with less than 0.7 water activity is categorized as low moisture food (Jiao et al., 2015). Due to the low water activity of this food category, they are generally assumed microbiologically safe.

Nowadays, there is a growing demand from health consciousness consumers for certain low moisture foods in the market. The powder and/or dried form of the food materials have been widely preferred by consumers and food manufacturers due to easy handling, transportation, storage, and longer shelf life compared to the liquid or granular form of the products.

In most of the food facilities, there is no pasteurization unit for low moisture foods since pasteurization was seen as unnecessary for the food products with low water activity. Only in a few food plants, the food powders are pasteurized either by steam or hot room heating. Conventional pasteurization technologies (steam and hot air heating) with hightemperature treatment might negatively impact protein solubility, soluble whey protein nitrogen, and functional and sensory properties of the dairy powders. Moreover, the conventional heating of low moisture food is very slow and inefficient due to the low thermal diffusivity of food powders. Among the pasteurization techniques, RF pasteurization is considered a promising technique that can be used to ensure safety and increase the shelf life of food powders. In RF treatment, heat is generated within the product resulting in volumetric and more uniform heating compared to the traditional conventional heating in which heat is transferred from a hot medium to a product (Piyasena et al., 2003). Because RF heating can provide faster treatment and more uniform temperature distribution within the food sample, it can be considered as a solution for slow and inefficient heating of the conventional method (Jiao et al., 2018). Furthermore, a faster heating rate achieved in RF heating allows maintaining the product quality (Jiao et al., 2014). RF heating has also the advantage of having a high penetration depth with a long wavelength, which can make this technology suitable for bulk materials. Besides all of its benefits, non-uniformity in temperature distribution is still a major limitation for RF heating to be used as a pasteurization step.

The researchers/scientists are currently developing different RF designs in which heating uniformity can be further improved. The effect of hot air circulation, water immersion, preconditioning the sample, mixing, intermittent stirring/shaking, rotating the sample its central axis, conveyor belt movement, adjusting the electrode gap during RF heating, electrode modification, surrounding the container with Polyetherimide (PEI) are some of the methods which were conducted for enhancing the RF heating uniformity (Boreddy et al., 2016; Chen et al., 2017; Choi et al., 2017; Huang et al., 2016; Jiao et al., 2014; Lau et al., 2017; Palazoğlu and Miran, 2018; Wang et al., 2006). Although the beforementioned techniques and designs have improved the RF heating uniformity, there is still a need to work on the different techniques/designs to make RF heating uniformity acceptable for food industrial applications.

In recent years, the potential use of RF pasteurization/sterilization has been studied for various low moisture foods including in-shell almond (Gao et al., 2011; Li et al., 2017), almond kernel (Li et al., 2018; Villa-Rojas et al., 2013), black peppercorn (Wei et al., 2018), and confectionery formulation, chicken meat powder, pet food and savory seasoning (Rachon et al., 2016). Moreover, several studies indicated that RF heating is a promising technique to inactivate the pathogens in food powders including ground black pepper (Wei et al., 2019), corn flour (Ozturk et al., 2019), wheat flour (Liu et al., 2017; Villa-Rojas et al., 2017; Xu et al., 2020), broccoli powder (Zhao et al., 2017), cumin, paprika and white pepper (Ozturk et al., 2020), red and black pepper (Jeong and Kang, 2014) and nonfat dry milk (Michael et al., 2014).

Until now, only a few studies were conducted on the application of RF heating in either whole or nonfat milk powders. Previously, the effect of RF heating on 1) the functionality of low-heat nonfat dry milk (Sanchez Alan et al., 2019), 2) the composition, microstructure, flowability, and rehydration characteristics of infant milk powder (Zhong et al., 2017), and 3) the solubility and whey protein nitrogen index of nonfat dry milk was studied (Chen et al., 2013). Moreover, in the study by Michael et. al. (2014), RF heating was validated for the decontamination of low-heat and high-heat nonfat dry milk. All of the studies done for milk powder RF heating were performed under batch system conditions in which the container containing milk powder was heated in the center of the RF electrodes without conveying along the electrodes. Moreover, no study was found in the literature investigating the effect of the surrounding media (air, deionized (DI) water, and soybean oil) on the RF heating uniformity and heating rate of food powders yet. In this regard, in this study, the effect of a continuous system and surrounding media were performed for whole and nonfat milk powders and corn flour. The study provided useful information that may lead to the novel design of RF pasteurization system for powdered foods.

The central hypothesis was that the RF heating uniformity and heating rate of food powders can be improved by proper design of the dimension and geometry of the food container, conveying the sample through RF heating cavity, and surrounding the powder container with selected mediums such as water and oil.

The hypothesis was formulated, in large part, based on the literature and our preliminary findings which demonstrated that (a) the dielectric properties of powders and the geometry of the container used in RF heating are important parameters affecting heating uniformity, (b) the continuous movement of the food powder during heating affects the heating uniformity, (c) the types of the surrounding media has an impact on the temperature distribution and heating rate. By completing this study, we will be able to understand the effect of the container shape, movement, and surrounding media effect on RF pasteurization uniformity.

The overall goal of the proposed work was to improve RF heating uniformity and heating rate of the selected food powders by investigating several approaches that can be easily applied in the food industry for low moisture food pasteurization.

### To test our hypothesis, three specific aims were proposed:

1) To determine the dielectric properties of milk powder and whether the geometry of the container affects RF pasteurization uniformity by studying temperature profile, thermal

images, uniformity index, penetration depth, the electric field strength in a batch system.

- 2) To determine whether the type of the surrounding medium and the continuous movement of corn flour during heating have an influence on heating uniformity and heating rate by investigating temperature profile, thermal images, and uniformity index in both batch and continuous systems.
- 3) To validate the proposed RF heating system by testing the microbial load of the inoculated whole milk powder after RF treatment by plate counting method.

In brief, the effect of the geometry and dimension of the container, continuous movement of the sample, and the types of the surrounding medium in RF heating uniformity and heating rate were evaluated in this study. This work allowed us to develop innovative methods for RF pasteurization of low moisture foods for the best results. Overall, the present study laid a foundation for system design and scale-up for the commercialized application of RF pasteurization in low moisture foods in the food industry.

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## CHAPTER 2

### LITERATURE REVIEW

## DEVELOPMENTS IN RADIO FREQUENCY PASTEURIZATION OF FOOD

### POWDERS<sup>1</sup>

<sup>&</sup>lt;sup>1</sup>Dag D., Singh R.K., Kong F. 2020. Food Reviews International. Published Online. Reprinted here with permission of the publisher.

### Abstract

Radio frequency (RF) pasteurization has been used in several industries and has a great potential for further scale up to commercial applications in the food industry. Efforts have been undertaken to develop uniform heating throughout the food to ensure product safety and extend its shelf life. Among the food products, food powder is a large category of low moisture foods and generally considered microbiologically safe due to its low water activity. However, recent outbreaks indicate that certain microbial species can survive for a significant time period in contaminated low moisture foods. For this reason, the development of effective pasteurization techniques for low moisture foods has become more of an issue. This chapter focuses on the literature on RF heating for the pasteurization of food powders. In addition to a brief introduction to food powders and RF heating applications, the factors affecting heating uniformity, dielectric properties of various food powders found in the literature, and microbial validation studies were covered.

### Introduction

Food safety is an extremely important priority for the food industry. Every year, about 1 out of 6 people suffers from foodborne illnesses in North America causing approximately 3,000 deaths, 128,000 hospitalizations and enormous economic loss due to product recalls (CDC, 2011). Thermal processing techniques hold great potential to ensure food safety by eliminating foodborne pathogens in foods. Among the thermal processes, electromagnetic radiation has the advantage of shorter heating time preserving the nutritional value and sensory quality of the final product.

Low moisture foods are generally considered as "low-risk foods" due to their low water activity (lower than 0.7) limiting bacterial growth and multiplication (Blessington, Theofel, & Harris, 2013; Jiang, Gu, Gou, Xia, & Wang, 2020; Wei, Lau, Stratton, Irmak, & Subbiah, 2019). However, several food pathogens can still survive for several months in low moisture foods resulting in outbreaks related to the consumption of raw almond (Isaacs et al., 2005), ground black pepper (Keller, VanDoren, Grasso, & Halik, 2013), peanut (Kirk et al., 2004), peanut butter (Park, Oh, & Kang, 2008), confectionery products (Komitopoulou & Peñaloza, 2009), spices and herbs (Zweifel & Stephan, 2012), paprika and paprika powdered potato chips (Lehmacher, Bockemühl, & Aleksic, 1995) and powdered infant formula (Drudy, Mullane, Quinn, Wall, & Fanning, 2006), etc. Thus, alternative technologies to traditional pasteurization methods are either developed or improved by the researchers to ensure food safety of low moisture foods with desired microbial reduction.

Current pasteurization techniques such as fumigation, steam, ozone treatment, and conventional heating have limitations including quality deterioration, minimal customer acceptance, long treatment time, and high energy consumption. Nowadays, RF pasteurization of low moisture foods is being studied to overcome the limitations of current pasteurization technologies. In this regard, RF pasteurization is a promising technology to be used in the food industry since heating is rapid, volumetric, and can penetrate much deeper into most packaged foods.

### **Food Powders**

The powder form of the food materials has been widely preferred by consumers and food manufacturers due to easy handling, transportation, storage, and longer shelf life compared to the liquid or granular form of the products. As a result of the great demand for food powders, powder technology has been progressively developing over the past few decades.

Food powders are particulate solid materials produced from either granular solids or liquids. Particulate solid materials can be categorized as granules (200-4000  $\mu$ m), flour (100-5000  $\mu$ m), powder (50-200  $\mu$ m), and dust (5-100  $\mu$ m) based on the size of the material, although all terms are commonly considered as a powder (Bhandari, 2013).

Spray drying, freeze-drying, drum drying, belt drying, and crystallization are the techniques widely used in powder production from liquid or paste. The particle size of the material can be further decreased by crushing, grinding, milling and pulverization. In addition to the size reduction process, granulation and mixing are the processes utilized in powder production from the solid state of the material. Particle density, shape, size, and size distribution have a large impact on the functional properties of the food powder and are highly dependent on raw material and processing conditions during manufacturing (Bhandari, 2013).

### **Pasteurization and Sterilization**

Pasteurization and sterilization are common terms that are used to extend the shelf life of a food product. Although both processes perform the same function which is the elimination of pathogenic and spoilage bacteria from the food material their distinct differences should be noted. Pasteurization is defined as the process used to destroy/inactivate the vegetative cells of pathogenic microorganisms but not their spores. On the other hand, sterilization is the process in which all forms of microbial life, including fungi, spores, viruses, and bacteria were eliminated from food.

### **Radio Frequency Heating**

Among the pasteurization techniques, RF heating is a promising pasteurization technique to ensure safety and increase the shelf life of food powders by providing fast and volumetric heating. In RF heating, heat is generated within the product providing more uniform heating than the traditional conventional heating in which heat is transferred from a hot medium to a food sample (Piyasena, Dussault, Koutchma, Ramaswamy, & Awuah, 2003). Moreover, the conventional heating of low moisture food is very slow and inefficient due to their low thermal diffusivities. RF heating can be a solution for the limitation of conventional heating by providing volumetric heating with deep penetration into the food (Jiao, Tang, Wang, & Koral, 2018). The faster heating rate in RF heating allows maintaining the product quality with reduced heat treatment time (Jiao, Tang, Wang, 2014).

### Working Principle of Radio Frequency Heating

RF heating is a non-ionizing radiation thermal process since insufficient energy (less than 10 eV) is produced to ionize biologically important atoms. Since RF waves (1-

300 MHz) lie in the radar range, only three selected frequencies 13.56, 27.12, and 40.68 MHz are permitted by the US Federal Communications Commission (FCC) for industrial, scientific, and medical applications (ISM) to avoid interference with the communication systems (Piyasena et al., 2003).

The water, salts, and other polar and charged molecules in the food can absorb electromagnetic radiation, which is then converted to heat. In RF heating, the alternating electric field is generated between two electrodes in the RF chamber (shown in Fig. 2.1) where the product is placed. The food is heated with the friction generated by (1) spinning polar dielectric molecules and (2) moving ions as a result of an alternating electric field (Marra, Zhang, & Lyng, 2009).

### Dipolar Polarization:

The dipolar rotation occurs due to the tendency of dipolar molecules, such as water, to align themselves appropriately with the change in polarity of the alternating field (Zhou & Wang, 2019). Heat is generated by internal molecular friction as a result of the polarization of dipolar molecules with the changing electric field.

### Ionic Polarization:

Ionic depolarization occurs due to the movement of positive ions in the food sample towards negative regions of the electric field and the movement of negative ions in the food towards positive regions of the electric field with the alternating electric field (Marra, Bedane, Uyar, Erdogdu, & Lyng, 2014). Heat is generated due to the friction between the charged ions in food.

### Advantages and Disadvantages of Radio Frequency Heating

RF heating has the advantage of high penetration depth due to the long wavelength, which makes it suitable to heat bulk material with better heating uniformity. The other advantages of RF heating over conventional heating are high energy densities, reduced operation time and production floor-space requirements, compatibility with automated production batch and/or continuous flow processing, and being environmentally friendly (Zhao, Flugstad, Kolbe, Park, & Wells, 2000). However, besides its advantages, nonuniformity in temperature distribution is a major challenge for RF heating. The different RF systems were developed to improve heating uniformity with the help of hot air circulation, water immersion, preconditioning the sample, mixing, intermittent stirring/shaking, rotating the sample its central axis, conveyor belt movement, adjusting the electrode gap during heating, electrode modification, and surrounding the container with Polyetherimide (PEI) (Boreddy, Thippareddi, Froning, & Subbiah, 2016; Chen, Lau, Chen, Wang, & Subbiah, 2017; Choi, Park, Yang, Kim, & Chun, 2017; Huang, Zhang, Marra, & Wang, 2016; Jiao et al., 2014; Lau, Thippareddi, & Subbiah, 2017; Palazoğlu & Miran, 2018; Wang et al., 2006).

#### **Radio Frequency Applications in Industry**

RF heating has been used in the wood, textile, and paper industries, and also in the food industry with limited applications. Post-baking of biscuits and thawing/tempering of the meat are two RF applications that have been successfully commercialized in the food industry so far.

RF drying and disinfestation are the processes that are partially commercialized in the food industry. The combination of hot air circulation with RF treatment has been found effective for drying and is commonly used to minimize the operation cost. Moreover, RF heating has been found as an alternative method to fumigation which is the chemical treatment to inactivate pests in food products. Recently, RF postharvest disinfestation of agricultural samples has been commercialized in the food industry (Jiao et al., 2018).

RF pasteurization/sterilization, roasting, and enzyme inactivation (also known as blanching) are the applications still under development. In recent years, the potential RF pasteurization/sterilization of various foods including in-shell almond (Gao, Tang, Villa-Rojas, Wang, & Wang, 2011; Li, Kou, Cheng, Zheng, & Wang, 2017), almond kernel (Li, Kou, Hou, Ling, & Wang, 2018; Villa-Rojas et al., 2013), shell eggs (Geveke, Bigley, & Brunkhorst, 2017; Lau et al., 2017), black peppercorn (Wei et al., 2018), and confectionery formulation, chicken meat powder, pet food and savory seasoning (Rachon, Peñaloza, & Gibbs, 2016) have been studied. Moreover, several studies indicated that RF heating is a promising technique to inactivate the pathogens in food powders including ground black pepper (Wei et al., 2019), corn flour (Ozturk et al., 2019), wheat flour (Liu et al., 2018; Villa-Rojas, Zhu, Marks, & Tang, 2017; Xu, Yang, Jin, Barnett, & Tang, 2020), broccoli powder (Zhao, Zhao, Yang, Singh Sidhu, & Kong, 2017), cumin, paprika and white pepper (Ozturk, Kong, & Singh, 2020), red and black pepper (Jeong & Kang, 2014) and nonfat dry milk (Michael et al., 2014). The schematic of free-running oscillator 6 kW, 27.12 MHz RF oven used in most of the RF studies and its components were illustrated in Fig. 2.2. In some studies, improvements in RF ovens such as conveyor belt installation, hot air circulation, and vacuum systems were incorporated.

### Heat Transfer of Food Powders in Radio Frequency Heating

Maxwell's equations are a set of four differential equations describing the change in magnetic and electric fields with time. Because the wavelength of an electromagnetic wave in RF range is much longer than the RF cavity the electric field can be calculated by the Laplace equation which is the simplified form of Maxwell's equations with the quasisteady-state approximation (Choi & Konrad, 1991; Huang, Marra, Subbiah, & Wang, 2018):

$$-\nabla \cdot \left( \left( \sigma + j 2\pi f \varepsilon_o \varepsilon' \right) \nabla V \right) = 0 \tag{2.1}$$

where  $\sigma$  is the electrical conductivity (S/m) of food, f is the frequency (Hz),  $j = \sqrt{-1}$ ,  $\varepsilon_0$ is the free space permittivity (8.854×10<sup>-12</sup> F/m),  $\varepsilon'$  is the dielectric constant of food, V is the voltage (V) between the electrodes.

The amount of the power (P, W/m<sup>3</sup>) converted to the thermal energy from the electromagnetic energy in food can be calculated by the following equation (Choi & Konrad, 1991; Erdogdu, Altin, Marra, & Bedane, 2017):

$$P = 2\pi f \varepsilon_0 \varepsilon'' \left| \vec{E} \right|^2 \tag{2.2}$$

where  $\varepsilon''$  is the dielectric loss factor of food material and  $\vec{E}$  is the electric field intensity (V/m) in the food with the expression  $\vec{E} = -\nabla V$ .

The unsteady heat transfer in food is calculated by Fourier's equation (Huang, Marra, et al., 2018; Tiwari, Wang, Tang, & Birla, 2011b):

$$\frac{\partial T}{\partial t} = \nabla \alpha \nabla T + \frac{P}{\rho c_p}$$
(2.3)

where  $\partial T/\partial t$  is the heating rate (°C/s), *T* is the temperature of food sample (°C), *P* is the power absorbed by the food (W/m<sup>3</sup>),  $\alpha$  is the thermal diffusivity (m<sup>2</sup>/s),  $\rho$  is the density (kg/m<sup>3</sup>), and  $C_p$  is specific heat (J/kg.K) of the food.

### **Factors Affecting Radio Frequency Heating of Food Powders**

### **Dielectric Properties**

Dielectric properties (dielectric constant and dielectric loss factor) play a key role in the understanding of the interaction between food materials and electromagnetic energy in RF heating. The determination of the dielectric properties of foods has gained enormous attention in the last years since knowledge of the dielectric properties of a food product at a specific frequency and temperature is necessary for the development of RF heating.

The dielectric constant is defined as the ability of food material to store electrical energy while the dielectric loss factor is defined as the ability of food to dissipate the electrical energy in the form of heat (Sosa-Morales, Valerio-Junco, López-Malo, & García, 2010). Dielectric properties of food powders depend on the frequency of the applied electric field, temperature, characteristics of food such as bulk density of the air-particle mixture, moisture content, chemical composition (fat, protein, salt, mineral, *etc.*), and structure of the powder (Icier & Baysal, 2004).

According to Kraszewski & Nelson (1990), the frequency and temperature dependence of the dielectric properties of the materials is complex and the measurement of the dielectric properties at the desired frequency and temperature is recommended as the best approach. In general, the food sample having a higher density has higher dielectric properties as a result of a lower volume fraction by air in the food. Because the dielectric properties of materials are highly influenced by mobile ions and the permanent dipole moments associated with water, the food materials with higher moisture content have higher dielectric properties (Marra et al., 2009; Nelson & Datta, 2001). Overall, food powders have a low dielectric loss factor resulting in less energy absorption due to their low moisture content. However, it should be noted that dielectric properties are only one of the parameters affecting dielectric heating.

Many studies on the dielectric properties of food powders have been investigated at different frequencies, temperatures, and moisture contents as well as bulk and compact densities. Summarization of measurement technique, frequency, temperature, and moisture content in some recent studies in dielectric properties measurement of food powders was given in Table 2.1. The information found in Table 2.1 includes the current studies available in the literature related to the dielectric properties of food powders which might be a guideline for researchers for their further investigations. Furthermore, Table 2.2 demonstrates the dielectric constant and loss factor of the selected food powders at a particular temperature (mostly room and target temperature), moisture content (similar to the moisture content of a specific powder found in the food market), frequency (13.56 and/or 27.12 MHz). The data found in Table 2.2 can be taken as a reference to develop a novel RF pasteurization model specific to food powders.

### Shape, Size, Position of Food Powder and Container

The shape, size, and position of food samples have a major impact on RF heating uniformity by affecting the electric field distribution. In the study carried by Tiwari, Wang, Tang, & Birla (2011a), the effect of size, shape, vertical position of the sample between the electrodes and top electrode configuration on heating uniformity was shown by using a developed finite element computer model. Wheat flour was heated in cuboid, ellipsoid,
and cylinder containers at three different vertical positions (on the ground electrode, in the middle of the electrodes, and contact with the top electrode). The simulated results indicated that the larger sample size and the smaller dielectric properties provide better RF heating uniformity. Edge heating was predominant in cube and cylinder containers while central heating was seen in the ellipsoid container. The effect of the top electrode configuration on RF heating was also examined by bending the top electrode upward on both ends at different lengths and angles. It was revealed that the RF power uniformity index (PUI) was lower when the top electrode was bent at the middle point of the electrode (called as 200 mm position) compared to bending at 0, 100, 300, 400, 500 mm positions. Moreover, the lowest PUI was achieved at 20 °C bending angles when the bending position was fixed at 200 mm.

In another study conducted by Wang, Zhang, Gao, Tang, & Wang (2014), the influence of polyurethane foam on RF heating uniformity was investigated by comparing the heating patterns of Macadamia nuts with and without foam. It was suggested that polyurethane foam was a proper material to improve the RF heating uniformity of Macadamia nuts. In a different study, it was concluded that RF heating uniformity of corn flour increased by surrounding the glass and polystyrene cylindrical container with polyurethane foam (Ozturk, Kong, Singh, Kuzy, & Li, 2017).

In summary, the shape, dimension, and position of the food between the RF electrodes have a significant effect on the RF heating uniformity. The particular importance should be taken into account in the development of the pasteurization system specific to food powders.

# Container Selection

To develop effective RF heating, the proper selection of container/package material in accordance with a food sample is necessary. The dielectric constant is the predominant factor in the electric field distribution when it is much greater than the dielectric loss factor of the sample at a desired frequency and temperature (Jiao et al., 2014; Metaxas, 1996). The minimum difference between the dielectric constant of the food material and the container could improve the RF heating uniformity by providing a more uniform electric field distribution.

In the study conducted by Huang et al. (2016), the effect of the container material (polystyrene and polypropylene), shape (sharp and rounded corners and edges), and thickness (2 to 12 cm with 2 cm increment) on RF heating uniformity was investigated for dried soybeans by computer simulation. The significant change in uniformity index (from 0.122 to 0.095) indicated that an increase in container thickness from 2 to 8 cm improved the temperature distribution volumetrically. Moreover, reducing the sharp areas on edges and corners led to a decrease in uniformity index (from 0.095 to 0.085) demonstrating an improved heating uniformity. The results demonstrated that using the polystyrene container with 8 cm thickness and a corner radius of 8 cm significantly improved RF heating uniformity. In another study, the positive impact of Polyetherimide (PEI) on RF heating uniformity due to having a similar dielectric constant with low moisture foods was reported for peanut butter (Jiao, Shi, Tang, Li, & Wang, 2015; Jiao et al., 2014) and corn flour (Ozturk et al., 2017). These findings suggest that a similar dielectric constant of the sample and container material can provide uniformity in the temperature distribution.

# <u>Electric Field</u>

Another important parameter affecting the RF heating uniformity is the distribution of the electric field during the process (Wang, Monzon, Johnson, Mitcham, & Tang, 2007). Uniform electric field distribution is directly associated with uniform temperature distribution throughout the sample (Guo, Mujumdar, & Zhang, 2019). For this reason, the solutions to create uniform electric field distribution within the food samples should be focused on achieving uniform temperature distribution.

The shape and geometry of the electrodes are the main parameters affecting the electric field distribution. In the study conducted by Choi et al. (2017), the newly designed curved-electrode RF system was introduced for the tempering of cylindrical frozen pork loin. According to frozen pork tempering time-temperature profiles, quality changes, internal temperature measurements at the top, middle and bottom, histological and microbiological analysis, it was shown that curved-electrode RF tempering of cylindrical-shaped frozen pork loin allowed a uniform temperature distribution with a reduced treatment time compared to parallel-electrode RF system. Moreover, some studies have been carried out with the help of computer simulation to predict the electric field distribution for low moisture foods such as wheat flour (Tiwari et al., 2011b), peanut butter (Jiao et al., 2015, 2014), and potato starch (Zhu et al., 2018). The studies involving computer simulations provided valuable knowledge to predict electric field and temperature distribution in the products that can be obtained without the necessity of experiments (Huang, Marra, et al., 2018).

Movement and rotation of the sample in the RF chamber were found effective to improve the electric field distribution within the product. In the study carried by Palazoğlu

& Miran (2018), the effect of heating wheat flour on an inclined conveyor belt with the combined translational and rotational movement on RF heating uniformity was investigated. The results from the study indicated that a temperature distribution was improved by rotating a wheat flour horizontally about its central axis on an inclined conveyor. The wheat flour heated on an inclined conveyor with rotation had a lower uniformity index (0.076±0.001) compared to the sample heated without rotation  $(0.125\pm0.003)$  at the top surface which is an indication of better heating uniformity. Moreover, it was suggested that heating uniformity can be further improved by increasing the speed of rotation. In another study conducted by Chen et al. (2017), it was reported that the heating uniformity of egg white powder was not improved by moving on the conveyor belt when compared to egg white powder heated at the stationary condition. In their study, a 3D finite element-based multiphysics model with a moving mesh approach was used to simulate the RF heating of egg white powder continuously moving on a conveyor belt. The difference between the studies could be due to the rotation of the sample.

# **Radio Frequency Heating Uniformity Evaluation**

#### Penetration Depth

When an electromagnetic wave propagates through the food sample, the portion of the wave is reflected while the other portion penetrates the food (Boreddy & Subbiah, 2016). Penetration depth ( $d_p$ ) is defined as the depth where the power is reduced to 1/e (where e=2.718) or approximately 37% of its value at the surface of the material (Marra et al., 2009).  $D_p$  is useful in the determination of the material thickness at the selected frequency and can be calculated using the following equation (Von Hippel, 1954):

$$d_p = \frac{c}{2\pi f \sqrt{2\varepsilon' \left[\sqrt{1 + \left(\frac{\varepsilon'}{\varepsilon'}\right)^2 - 1}\right]}}$$
(2.4)

where *c* is the speed of light in free space ( $3x10^8$  m/s), *f* is the frequency in Hz,  $\varepsilon'$ , and  $\varepsilon''$  are dielectric constant and loss factor of the sample, respectively.

As can be seen in Equation 1, the penetration depth is dependent on frequency, dielectric constant, and loss factor. It was assessed by Bengtsson & Risman (1971) the largest penetration depth was recorded when both the dielectric constant and loss factor were low. Moreover, the composition of the sample influences penetration depth due to the dependency of the sample's dielectric properties on composition.

It is important to note that if the thickness of the food sample is several times more than the penetration depth, the interior parts of the food will be cooler while the exterior parts will be warmer. On the other hand, if the thickness of the sample is too small compared to penetration depth the center of the product will have a warmer interior and cooler exterior (Schiffman, 1995). Thus, it is recommended to determine the container dimensions by considering the penetration depth of the food to be heated.

# Heating Uniformity Index

Uniformity Index ( $\lambda$ ) is a parameter associated with heating uniformity in RF treatment and can be calculated using the following equation (Wang, Yue, Tang, & Chen, 2005):

$$\lambda = \frac{\Delta\sigma}{\Delta\mu} \tag{2.5}$$

where  $\Delta \sigma$  is the change in the standard deviation of product temperature and  $\Delta \mu$  is the change in mean product temperature.

The uniformity index is affected by RF unit design and the interaction between the RF unit and the food sample. It indicates the increase in standard deviation when mean product temperature increases by one degree. Small uniformity index means that the rise in mean product temperature leads to a small difference in standard deviation which is an indication of better heating uniformity (Wang et al., 2005).

In the study conducted by Huang, Zhang, Marra, & Wang (2016), the effect of container thickness on the heating uniformity index was evaluated. A significant difference in uniformity index (from 0.122 to 0.095) was observed when container thickness has been increased from 2 to 8 cm. In another study, it was reported that hot air assistance and continuous movement of legumes on conveyor belts improved heating uniformity providing a smaller uniformity index (Wang, Tiwari, Jiao, Johnson, & Tang, 2010).

## <u>Heating Rate from Temperature Profile</u>

Heating rate (°C/min) is another parameter affecting heating uniformity and can be calculated by dividing temperature increase in food with treatment time. Heating rates of hot air and RF treatment for low moisture foods are considerably different due to the low thermal conductivity of low moisture foods. In the study carried by Ozturk et al. (2017), the efficiency of RF and hot air heating systems for corn flour was evaluated by comparing the treatment time of both heating techniques. It was stated that it took approximately 210 min with a heating rate of 0.28 °C/min to heat corn flour to 80 °C in a hot air oven. On the other hand, the sample could reach to same target temperature in 9 min with a 6.53 °C/min heating rate in an RF oven with a 13 cm electrode gap. In general, the high heating rate is desirable by the food manufacturers because it is associated with quality retention as well

as the lower operational cost; however, it is more likely to result in overheating and thermal runaway due to fast heating (Ling, Lyng, & Wang, 2018).

Heating rates of sample to be heated in RF oven is dependent on dielectric properties, electrical conductivity, bulk density, moisture content, composition and size and shape of the food material, the configuration of the food container, the distance between two electrodes, and RF oven power. In the study carried by Marra, Lyng, Romano, & McKenna (2007), the cylindrical meat batters were heated in 50  $\Omega$  RF system at 27.12 MHz with output powers of 100, 200, 300, and 400 W. Heating rates at five different locations at 5, 10 and 15 min were calculated using temperature profiles. It was concluded an increase in RF output power caused an increase in heating rate. Moreover, the uneven temperature distribution within the sample was observed as a result of different heating rates throughout the sample. The higher heating rate was recorded at the location close to the bottom of the container. The results from the study showed that higher RF output power resulted in uneven temperature distribution.

The effect of moisture, bulk density, and electrode gap on heating uniformity was investigated in the study conducted by Ozturk et al. (2017). The corn flour in PEI container (with inner dimension: 7 (H) × 24 (W) × 30 (L) cm<sup>3</sup>) was heated in 6 kW, 27.12 MHz RF oven at the different electrode gaps (11, 13, and 15 cm). Heating rate, heating uniformity index, and thermal images were used to evaluate the RF heating uniformity. The heating rate in corn flour during the RF heating decreased from  $12.71\pm1.84$  to  $5.52\pm0.32$  °C/min when the electrode gap was increased from 11 to 15 cm. The heating rate slightly increased from  $5.52\pm0.32$  to  $5.91\pm0.54$  °C/min when the PEI container was covered with a 5 mm foam sheet at a 15 cm electrode gap. The uniformity index was decreased from

 $0.044\pm0.006$  to  $0.033\pm0.013$  at the middle surface as the electrode gap increased from 11 to 15 cm due to reduced over-heating and run-away energy. Covering the PEI container with a foam sheet caused a decrease in uniformity index from  $0.033\pm0.013$  to  $0.029\pm0.021$  at the middle surface. To observe the effect of moisture and bulk density on heating uniformity, corn flour with various moisture contents (10.4 and 16.7 % wet basis) and bulk density levels (0.42, 0.53, and 0.61 g/cm<sup>3</sup>) were heated in a smaller container made of polystyrene with a 15 cm electrode gap. It was concluded that the heating uniformity was improved with an increase in bulk density, but decreased with an increase in moisture content.

In the study conducted by Lee (2018), the effect of the package dimension on the heating rate and uniformity was evaluated by heating corn flour in the cardboard paper boxes with five different side lengths of a square base at 10, 15, and 20 cm electrode gap. Each box was filled with the corresponding amount of corn flour to keep the bulk density constant. Heating uniformity was assessed with the help of heating rate, thermal images, heating profile, uniformity index, and electric field strength. The equations based on the linear relationship between heating rate and side length and the exponential relationship between heating rate and height were developed with  $R^2>0.93$  for each electrode gap. Overall, it was observed that the heating rate decreased with an increase in the box side length. On the contrary, an exponential increase in heating rate was reported with increasing box height.

In general, food powders having lower dielectric properties due to their low moisture content have a lower heating rate which may lead to a better heating uniformity compare to high moisture foods. Therefore, the heating rates should be optimized to a reasonable value for specific food products by changing parameters affecting heating.

# Temperature Distribution

Thermal imaging captured by an infrared camera provides quick and valuable information about the surface temperature distribution of the food. Thermal images can be further analyzed to obtain a temperature map demonstrating hot and cold spots at the sample surface. Moreover, the average surface temperature and heating uniformity index in different layers can be calculated via thermal images taken in different cross-sections of the food. Temperature measurement with thermocouples and/or fiber optic sensors is an alternative way for temperature measurement but it only measures the temperature at a single point at a time. Temperature vs time graph can be obtained by using a fiber optic sensor at the different locations in the sample to observe the heating trend of a sample during RF treatment. Infrared thermometers are easy and quick temperature readers; but similar to thermocouples and fiber optic sensors, it measures the temperature at a certain location. The examples of successful applications of thermal images in heating uniformity evaluation can be found in several RF studies (Huang, Datta, & Wang, 2018; Huang, Zhu, Yan, & Wang, 2015; Tiwari et al., 2011b; Wang, Olsen, Tang, & Tang, 2007; Zhu et al., 2018).

## **Microbial Validation**

It is crucial to validate the RF technology with microbial decontamination studies since RF pasteurization/sterilization is still under development for food industry applications. Non-pathogenic surrogate microorganisms with similar or slightly higher thermal resistance than the target microorganisms have gained great attention in microbial validation of RF pasteurization systems since pathogen microorganism is not allowed to study in food processing plants to avoid possible cross-contamination (Liu et al., 2018; Wei et al., 2019).

Microbial inactivation by RF heating occurs predominantly through thermal effects, such as denaturation of enzymes, proteins, nucleic acids, or other vital components, as well as the disruption of membranes resulting in the alterations in the microbial cell structure and functionality (Dwivedi, 2019; Kim, Sagong, Choi, Ryu, & Kang, 2012). Bacterial population reduction of pathogens in food powders by RF heating depends on the strain, growing conditions, cell wall structure, age, and the number of the bacterial cells as well as heating treatment conditions including setpoint temperature, heating rate, and heating uniformity (Kou, Li, Hou, Zhang, & Wang, 2018; Syamaladevi et al., 2016).

Because non-uniform heating is a major problem in RF heating, the researchers investigated the microbial reduction at the least heated location (cold spot) in the sample. However, the studies indicated that the least heated zone is not the same for different food samples. For instance, the coldest part after RF heating was reported as the geometric center for corn flour (Ozturk et al., 2019) while it was at the center on the top surface for ground black pepper (Wei et al., 2019). The difference in cold spots may result from the ratio of penetration depth to the sample thickness, the electrode gap, the air gap between container and electrode, and any parameters affecting electric distribution. Moreover, the lowest lethality zones location change due to heat conduction and ambient heat loss if the RF heated sample is naturally cooled down to room temperature (Xu et al., 2020). For this reason, it is suggested to conduct microbial validation studies at multiple locations in food. The temperature, composition, pH, water activity (a<sub>w</sub>), and the presence of the organic acids in the sample have a great influence on microbial thermo-tolerance in heat treatments (Doyle, Mazzotta, Wang, Wiseman, & Scott, 2001; Doyle & Mazzotta, 2000; Jagannath, Tsuchido, & Membré, 2005). Among these parameters, a<sub>w</sub> has a dominant effect on the heat resistance of the microorganisms (Villa-Rojas et al., 2013). In general, the thermal resistance of pathogens is higher in a low moisture environment compared to a moist environment (Li et al., 2017). Thus, the thermal resistance of pathogens can be decreased by increasing the a<sub>w</sub> of the food sample before RF heating. In several RF pasteurization studies, it was proposed to increase a<sub>w</sub> of the sample before RF heating to control pathogens in low moisture food including wheat flour (Villa-Rojas et al., 2017), peanut kernels (Zhang et al., 2018) and almond kernels (Villa-Rojas et al., 2013).

In addition to treatment temperature and sample a<sub>w</sub>, the heating rate has a significant effect on microbial inactivation. In the study carried by Xu et al. (2020), microbial reduction of *Enterococcus faecium* NRRL B-2354 which is a valid surrogate for *Salmonella* Enteritidis PT30 was evaluated at 15 different locations (evenly distributed in the top, middle and bottom layers) in wheat flour with 0.45 a<sub>w</sub> at room temperature after RF heating to 80 °C with three different RF heating rates (36.0, 11.3, 5.5 °C/min), followed by a 20 min nature cooling. It was concluded a high heating rate led to a non-uniformity in temperature and microbial inactivation. It was suggested a relatively low RF heating rate assisted with hot-air circulation is helpful to improve the temperature uniformity to obtain even microbial inactivation.

The pathogen reduction level in food facilities is determined by the regulatory requirements such as a 6.5-log *Salmonella* reduction in meat, a 7-log *Salmonella* reduction

in poultry (USDA FSIS, 1999), a 5-log pathogen reduction in juice (FDA, 2001), and 4-log CFU/g reduction in almond (USDA AMS, 2007). In RF pasteurization studies, a 5 log CFU/g reduction in low moisture foods was aimed to meet the product safety criteria. The potential of RF pasteurization to achieve the desired pathogen and/or its surrogate reduction was reported in several RF studies (Ozturk et al., 2019; Wei et al., 2019; Xu et al., 2018). In one of those studies, a 5 log CFU/g reduction of freeze-dried Enterococcus faecium NRRL B-2354 was reported by Xu et al. (2018) as a result of heating 3 kg wheat flour for 27 min in an RF oven. However, from most of the RF studies, it was observed that it is necessary to combine RF heating system with an additional process such as preheating, subsequent freezing, holding at a specific temperature, and forced air cooling to meet the 5-log pathogen reduction requirement. For instance, the effect of post-freezing treatment on microbial survival of Salmonella Enteritidis PT30 and Enterococcus faecium in RF heated with and without holding corn flour was reported by Ozturk et al. (2019). In their study, RF heated inoculated corn flour was stored at -20 °C for 96 h and the samples were taken and analyzed in every 24 h to determine the effect of post-freezing treatment on the survival of microorganisms. Holding the inoculated sample in the RF cavity for 10 min after RF heating led to an additional  $1.92 \pm 0.17$  and  $1.24 \pm 0.09 \log \text{CFU/g}$  reduction for Salmonella Enteritidis PT30 and Enterococcus faecium, respectively. It was concluded that RF treatment followed by holding and subsequent freezing storage enhanced microbial inactivation of Salmonella Enteritidis PT30 and Enterococcus faecium in corn flour compared to RF heating only. Microbial validation studies were conducted for RF pasteurization of food powders including RF oven specifications, treatment parameters, target microorganisms, the major results from the studies were summarized in Table 2.3.

#### **Future Aspects of Radio Frequency Heating**

Studies in the literature indicate that RF heating has various applications in food processing including thawing (Erdogdu et al., 2017; Uyar et al., 2015), cooking (Schlisselberg et al., 2013; Zhang, Lyng, & Brunton, 2004), drying (Zhou & Wang, 2019; Zhou et al., 2018), post-baking (Palazoğlu, Coşkun, Kocadağli, & Gökmen, 2012), roasting (Liao, Zhao, Gong, Zhang, & Jiao, 2018), blanching (Gong et al., 2019), disinfestation (Gao, Tang, Wang, Powers, & Wang, 2010; Wang et al., 2010), sterilization (Wang, Wig, Tang, & Hallberg, 2003), pasteurization (Gao et al., 2011; Geveke et al., 2017). The RF pasteurization was not commercialized yet due to the main limitation of RF heating (i.e. non-uniformity in temperature distribution). However, the computer simulations have a great ability to provide an understanding of the heating mechanism by solving the governing equations demonstrating the 3D distribution of the desired parameters including temperature and electric field distribution with minimum time, labor, and experimental work (Li et al., 2018). Moreover, the RF microbial validation studies highlight the potential of RF heating in the pasteurization of food powders. Further improvements in RF heating uniformity with the combination of experimental, computer simulation, and microbiological validation studies will help RF heating to be scaled up for the commercialized application of RF pasteurization in the food industry. Furthermore, thanks to the advantages of RF oven compared to microwave ovens such as relatively better heating uniformity, deeper penetration, and higher preservation of sensory and nutritional qualities of food it is expected to see RF ovens at our home/offices for daily use as a replacement of microwave ovens (Alternimi et al., 2019).

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# Tables

Table 2.1 The summary of studies on dielectric properties of food powders as a function of moisture, temperature, and frequency where

Sample	Measurement Technique	Moisture Content	Temperature (°C)	Frequency (MHz)	Reference	
Red Pepper Powder	Coaxial Probe	10.4, 14.7, 17.6, 22.8, 27.5, 30.8 (%wb)	25-85	27.12	Guo and Zhu (2014)	
Barley Grass Powder	Coaxial Probe	0.09 (%db)	25	27.12	Cao, Zhang, Chitrakar et al. (2019)	
Powdered Infant Formu	laCoaxial Probe	2.5, 3.5, 4.5, 5.5 (%wb)	20-80	13.56, 27.12, 40.68	Lin, Gao, Wang et al. (2016)	
Broccoli Powder		3.9, 6.9, 9.1, 12.2, 14.9(%wb)	20-80			
Onion Powder		1.4 (%wb)				
Tapioca Flour	Parallel Plate	5.7 (%wb)				
Chili Powder		7.8 (%wb)		20, 23, 27.12, 30	(2010)	
Potato Starch		12.6 (%wb)	_			
Corn Flour	Parallel Plate	10.4, 13.6, 16.7 (%wb)	20, 80	13.56, 27.12	Ozturk, Kong, Singh et al., (2017)	
Egg White Powder	Coaxial Probe	5.5, 6.6, 8.0, 9.8 (%db)	20-100	10-3000 MHz	Boreddy and Subbiah (2016)	
Chickpea Flour		7.9, 11.4, 15.8, 20.9 (%wb)		10-1800		
Green Pea Flour		10.8, 14.6, 17.5, 21.6 (%wb)		10-1800	- Com Ward Time int 1 (2010)	
Lentil Flour	-Coaxial Probe	8.4, 12.9, 16.9, 21.5 (%wb)		10-1800	-Guo, Wang, Hwari et al., (2010)	
Soybean Flour		8.9, 11.7, 16.0, 19.9 (%wb)		10-1800	-	
Whole Milk Powder	D	1.77 (%wb)	20.00	1, 5, 10, 13.56, 15	, 15, Dec. Sinch Kana (2010)	
Nonfat Milk Powder	-Parallel Plate	4.71 (%wb)	-20-90	20, 25, 27.12, 30	Dag, Singh, Kong (2019)	
White Pepper		10.2, 13.7, 17.1, 21.7 (%wb)	20-90			
Red Pepper		10.5 (%wb)				
Black Pepper		11.02 (%wb)	23±2			
Paprika		12.3 (%wb)		1, 5, 10, 13.56, 15	20 - t = 1 - K = 0	
Curry	-Parallel Plate	8.3 (%wb)		20, 25, 27.12, 30	Ozturk, Kong, Singh et al., (2018)	
Turmeric		9.5 (%wb)				
Cumin		9.2 (%wb)				
Garlic		3.1 (%wb)	_			
Chestnut Flour	Coaxial Probe	11.6, 20.6, 30.2, 39.3, 48.0 (%wb)	20-60	10-4500	Zhu, Guo, Wu et al., (2012)	
Wheat Flour	Coaxial Probe	8.8 (%wb)	20-70	27.12	Tiwari, Wang, Tang et al., (2011)	

wb is wet basis and db is dry basis.

Sample	<b>Moisture Content</b>	Temperature (°C)	Frequency (MHz)	<b>Dielectric Constant</b>	<b>Dielectric Loss Factor</b>	Reference
Red Pepper Powder	10.40 (%wb)	25 85	27.12	3.75 8.55	0.28 3.82	Guo and Zhu (2014)
Barley Grass Powder	0.09 (%db)	25	27.12	7.45	1.2	Cao, Zhang, Chitrakar et al., (2019)
Broccoli Powder	3.9 (%wb)		13.56 27.12	4.37 4.31	-	
Onion Powder	1.4 (%wb)	22±3	13.56 27.12	2.96 2.93	-	Ozturk, Kong, Trabelsi et al., (2016)
Tapioca Flour	5.7 (%wb)		13.56 27.12	4.71 4.60	0.07 0.02	
Chili Powder	7.8 (%wb)		13.56 27.12	5.69 5.46	0.31 0.17	
Potato Starch	12.6 (%wb)		13.56 27.12	4.32 4.29	0.01 0.06	
Corn Flour	10.3 (%wb)	20 80	13.56 27.12 13.56	3.86 3.68 9.11	0.16 0.13 1.11	Ozturk, Kong, Singh et al., (2017)
Egg White Powder	8.0 (%db)	20 100	27.12 27.12	8.43 2.38 3.59	0.74 0.180 0.762	Boreddy and Subbiah (2016)
White Pepper	17.1 (%wb)	20 80 20 70	27.12	5.35 19.72	0.59 6.89	Ozturk, Kong, Singh et al., (2019)
Chestnut Flour	11.6 (%wb)	20 60	27	2.1 10.7		Zhu, Guo, Wu et al., (2012)
Wheat Flour	8.8 (%wb)	20 70	27.12	3.27 4.77	0.23 0.42	Tiwari, Wang, Tang et al., (2011)

Table 2.2 Dielectric constant and loss factor of selected food powders at certain frequencies, temperatures, and moisture content.

**Table 2.3** The summary of RF microbial validation studies (in 6 kW, 27.12 MHz free-running oscillator system) for food powder

 pasteurization application including the major results from the studies.

Sample	Heating Procedure	Target Microorganism	Major Results	Reference
Wheat Flour	to 85 °C, followed by 18 and 25 min holding time	Salmonella Enteritidis Enterococcus faecium NRRL B-2354	<ul> <li><i>Enterococcus faecium</i> NRRL B-2354 is more heat resistant than <i>Salmonella</i> Enteritidis</li> <li><i>Enterococcus faecium</i> NRRL B-2354 is a valid surrogate for <i>Salmonella</i> Enteritidis in wheat flour</li> </ul>	Liu, Ozturk, Xu et al., (2017)
	to 80 °C followed by a 20 min nature cooling	<i>Enterococcus faecium</i> NRRL B-2354	• An average microbial reduction of 1.21–4.64 log	Xu, Yang, Jin et al., (2020)
Corn Flour	to 85 °C followed by 10 min holding time in an RF oven and stored at $-20$ °C for 48 h.	Salmonella enterica Enteritidis PT30 Enterococcus faecium NRRL B-2354	<ul> <li>6.59 ± 0.21 log CFU/g reduction for Salmonella enterica Enteritidis PT30</li> <li>4.79 ± 0.17 5 log CFU/g reduction for Enterococcus faecium NRRL B-2354</li> <li>Freezing storage treatment has a significant effect on microbial reduction of both microorganisms in corn flour.</li> </ul>	Ozturk, Liu, Xu et al., (2019)
Barley Grass Powder	to 80 °C followed by ice cooling treatment	Total bacteria	• Up to 3.8 log CFU/g reduction	Cao, Zhang, Chitrakar et al., (2019)
Broccoli Powder	1-6 min RF heating followed by cold- shock treatment	Total bacteria	<ul> <li>4.2 log CFU/g reduction after 5 min RF heating without cold-shock treatment</li> <li>Less than 30 CFU/g after 5 min RF heating followed by cold-shock treatment</li> </ul>	Zhao, Zhao, Yang et al., (2017)

Ground Black Pepper	120 and 130 s RF heating followed by ice-water bath cooling treatment for 3 min	Salmonella spp. Salmonella Agona 447967 Salmonella Reading Salmonella Tennessee K4643 Salmonella Montevideo 488275 Salmonella Mbandaka 698538 Enterococcus faecium NRRL B-2354	<ul> <li>More than 5.93 log CFU/g reduction for <i>Salmonella</i> spp.</li> <li><i>Enterococcus faecium</i> is a suitable surrogate for <i>Salmonella</i> spp.</li> <li>No significant quality changes after 130 s RF heating.</li> </ul>	Wei, Lau, Stratton et al., (2019)
Cumin Paprika White Pepper	to 80 °C followed by ice-water bath treatment for 90 s	Salmonella cocktail (S. Typhimurium, S. Agona, S. Montevideo, and S. Tennessee) Enterococcus faecium NRRL B-2354	<ul> <li>Enterococcus <i>faecium</i> is a suitable surrogate of <i>Salmonella</i> in packaged cumin, paprika, and white pepper.</li> <li>No significant color change was observed after RF pasteurization of cumin, paprika, and white pepper.</li> </ul>	Ozturk, Kong, Singh (2020)

# Figures



Figure 2.1 Simplified diagram of a radio frequency heating system.



**Figure 2.2** Schematic of free-running oscillator 6 kW, 27.12 MHz radio frequency oven and its components.

# CHAPTER 3

# DIELECTRIC PROPERTIES OF WHOLE, NONFAT MILK POWDER AND THEIR MIXTURES ASSOCIATED WITH RADIO FREQUENCY HEATING, AND THE EFFECT OF GEOMETRY<sup>1</sup>

<sup>&</sup>lt;sup>1</sup>Dag D., Singh R.K., Kong F. 2019. Journal of Food Engineering. 261: 40-50. Reprinted here with permission of the publisher.

# Abstract

Radio frequency (RF) heating is a promising pasteurization technique providing a fast and more uniform heat generation throughout a sample. In this chapter, the effect of composition, frequency (1-30 MHz), and temperature (20-90 °C) on the dielectric properties of the whole, nonfat milk powder and their mixtures were studied to develop an effective RF pasteurization. Moreover, the effect of the shape and dimension of the container on RF heating was evaluated. Milk powders were loaded into rectangular and cylindrical boxes with the same volume and different heights (3, 6, and 9 cm), and heated in a 6 kW, 27.12 MHz pilot-scale free-running oscillator RF oven. The heating rate, temperature profile, thermal image penetration depth, and electric field strength were determined. The quality of the RF heated milk powders was evaluated by measuring moisture content, water activity, solubility, and color. Overall, heating milk powder in the 6 cm height cylindrical box showed a relatively uniform temperature distribution with a moderate heating rate as well as improved quality in the finished product with fewer changes in moisture content, water activity, solubility, and color compared to 6 cm height rectangular box. Electric field strength increased with the increase in the height of the container.
## Introduction

Milk powder is considered an ideal nutritional source providing minerals, vitamins, and proteins. In the food industry, milk is pasteurized, and then made into powder by spray drying in which milk is dried rapidly to protect the nutritional components in the milk. Contamination of milk powder could occur when the dried product becomes contaminated from environmental sources (Michael et al., 2014), or the pasteurized milk to be fed to the spray dryer was contaminated (*Cronobacter sakazakii* and *Salmonella* spp. can survive the spray-drying conditions). According to the Centers for Disease Control and Prevention (CDC), the outbreak of Staphylococcus aureus linked to the consumption of powdered milk was reported in 2006 in Michigan, USA. That outbreak resulted in 36 powdered-milk related infections. Moreover, from 1985 to 2005 at least 6 outbreaks of Salmonella infections linked to the consumption of the powdered infant formula have been reported. Additionally, CDC has warned of Cronobacter contamination in powdered infant formulas, and to a lesser degree herbal teas, starches, and powdered milk in 2016. The main reason for the outbreaks was due to contamination that occurred in the spray driers (Angulo, Cahill, Wachsmuth, Costarrica, & Embarek, 2008). Therefore, a further pasteurization step for milk powder after spray drying will be useful to ensure its safety.

Conventional pasteurization technology including steam and hot air heating involves high-temperature processing which influences protein denaturation, soluble whey protein nitrogen, and functional and sensory properties of the final product (Karagül-Yüceer, Drake, & Cadwallader, 2001). As a rapid heating technology, radio frequency (RF) heating could be a promising pasteurization method for milk powder providing a fast and deep heat generation throughout a sample due to friction generated by (1) oscillating polar dielectric molecules and (2) moving ions. Heat is generated within the product in RF heating compared to conventional heating in which heat is transferred from a hot medium to a cooler product (Piyasena, Dussault, Koutchma, Ramaswamy, & Awuah, 2003). The other advantages of RF heating over conventional heating are high energy efficiency, high energy densities, heating independent of the product's thermal conductivity, reduced production floor-space requirements, and compatibility with automated production batch and/or continuous flow processing (Zhao, Flugstad, Kolbe, Park, & Wells, 2000).

Although RF waves occupy the range between 1 and 300 MHz in the electromagnetic spectrum, only 13.56, 27.12, and 40.68 MHz are permitted for use in industrial, scientific, and medical applications (Marra, Bedane, Uyar, Erdogdu, & Lyng, 2014). Previously, several promising food applications of RF heating have been reported such as the thawing of pork sirloin (Kim et al., 2016), tempering of frozen beef (Li et al., 2018), roasting of cashew nut kernels (Liao, Zhao, Gong, Zhang, & Jiao, 2018), disinfestation of dried fruits (Alfaifi et al., 2014), postharvest disinfestation of almonds (Gao, Tang, Wang, Powers, & Wang, 2010), pasteurization of in-shell almonds (Gao, Tang, Villa-Rojas, Wang, 2011), raw milk (Zhu, Guo, & Jia, 2014), honey (Guo, Liu, Zhu, & Wang, 2011), heating of infant milk powder (Zhong et al., 2017), *etc*.

The dielectric properties of the food sample are significant to achieve effective RF heating since the amount of power conversion from electromagnetic energy to thermal energy within the sample depends on the dielectric properties of the product (Jiao, Tang, & Wang, 2014). According to previous studies, it is well known that the dielectric properties of the food are influenced by temperature, moisture content, water activity, bulk density, and the chemical composition of the food as well as frequency applied alternating

electric field (Venkatesh & Raghavan, 2004; Zhu, Guo, Jia, & Kang, 2015). Moreover, the recent studies have emphasized the significant effect of the sample geometry, shape, position, and size on the effectiveness of RF heating system (Huang, Datta, & Wang, 2018; Li et al., 2018; Marra, Zhang, & Lyng, 2009; Romano & Marra, 2008).

Previously, the dielectric properties of the raw, whole, and skim milk as a function of protein and lactose content, frequency and temperature were extensively studied (Liu & Guo, 2018; Zhu et al., 2015). Moreover, Lin et al. (2016) have described the dielectric properties of the powdered infant formula milk as influenced by frequency, temperature, and main components. In the study conducted by Zhong et al. (2017), the effect of RF heating on the composition, microstructure, flow characteristic, and rehydration property of infant milk powder was evaluated. The effect of RF heating on the solubility and whey protein nitrogen index of nonfat dry milk (Chen et al., 2013), and the destruction of Cronobacter sakazakii and Salmonella species in nonfat dry milk by RF heating (Michael et al., 2014) were investigated previously. To the best of our knowledge, there is a lack of scientific information on the comparison of the dielectric properties of whole and nonfat milk powder and their mixture as influenced by temperature, frequency, and composition (specifically fat content). Furthermore, the effect of the configuration and dimension of the containers on RF heating uniformity has not been investigated yet. In this regard, the present study should provide useful information to design an effective RF pasteurization system for whole and nonfat milk powder as an alternative technique to conventional heating commonly used in the food powder pasteurization in the industry, such as steam treatment.

The objective of this study was to investigate the effect of the composition, frequency, and temperature on dielectric properties of milk powders, the effect of the geometry (cylindrical and rectangular), and dimension (3, 6, 9 cm) of the boxes on RF heating and the quality of the milk powders after RF heating. The milk powder mixtures were prepared by mixing whole and nonfat milk powder in different mass ratios to examine the effect of composition on dielectric properties. The temperature profile, heating rate, temperature distribution via thermal images, electric field strength, and penetration depth of milk powders after RF heating were determined. The quality of the whole and nonfat milk powders heated in an RF oven was evaluated by conducting moisture content, water activity, solubility, and color measurements.

#### **Materials & Methods**

#### Materials and Sample Preparation

The whole and nonfat milk powder (Hoosier Hill Farm, Fort Wayne, Indiana, USA) were purchased from a local supermarket. Milk powder mixtures were prepared by mixing whole and nonfat milk powder in different mass ratios, including 1:1, 1:2, 2:1, 1:3, 3:1 (whole:nonfat w:w) to obtain mixtures with various compositions. According to the manufacturer's database, the fat content of the whole milk powder was 28.57 % while nonfat milk powder had no fat.

#### Physical Characterization of Milk Powders

Previous studies indicated that the dielectric properties are mainly influenced by frequency, temperature, composition, moisture content, water activity, and density of the food products (Liu & Guo, 2018; Nelson & Trabelsi, 2012; Sacilik, Tarimci, & Colak, 2006; Zhang, Lyng, & Brunton, 2007). Therefore, moisture content, water activity, and

bulk density of the milk powders were determined to explain the effect of the physical properties on the dielectric heating in the following section.

The moisture content of the milk powders was determined in triplicate by drying approximately 2 g milk powder samples in aluminum dishes in a NAPCO vacuum oven (Model 5831, Precision Scientific, Chicago, IL) at 105 °C for 24 h at 57.6 kPa vacuum gauge pressure (AOAC, 2005). The samples were left to be cooled in a desiccator before weighing. Moisture content was calculated from the initial and final weights of the milk powders. The initial moisture content of whole, nonfat milk powder, and their 1:1 mixture were measured as 1.77, 4.71, and 3.04 % (wet basis) respectively.

Since the moisture content of the powders is related to the water activity, the water activity of milk powders was also measured using a water activity meter (Aqualab serious 3TE, Decagon 221 Devices Inc., Pullman, WA, USA) at room temperature. The initial water activity of whole, nonfat milk powder, and their 1:1 mixture were measured as 0.183, 0.284, and 0.236, respectively.

The bulk density  $(\rho)$  of each mixture was determined gravimetrically as:

$$\rho = \frac{m_t}{v} \tag{3.1}$$

where  $m_t$  is the total mass of the sample (g) and v is its volume (cm<sup>3</sup>) (Trabelsi, Kraszewski, & Nelson, 2001).

Bulk density of whole, nonfat milk powder, and their 1:1 mixture was measured as  $0.56, 0.44, \text{ and } 0.51 \text{ g/cm}^3$  respectively.

Volumetric heating capacity (VHC) was measured using a KD2 Pro thermal analyzer (Decagon Devices, Inc., USA) by inserting an SH1 probe into the 20 g milk powder placed in a beaker. Then, specific heat capacity ( $C_p$ ) was calculated by dividing

VHC by the density of the sample. The specific heat capacity of whole and nonfat milk powder was 2.13 and 1.79 kJ/kg°C respectively and the values were used to calculate heat generated within a food sample.

## **Determination of Dielectric Properties**

There are several measurement techniques used to determine the dielectric properties of the materials namely parallel plate, lumped circuit, coaxial probe, transmission line, cavity resonator, free space, and time-domain spectroscopy. The determination of the technique depends on the material type, frequency range, accuracy, availability, and cost of the equipment (Nelson & Kraszewski, 1990). In the current study, the parallel plate technique was used due to its being an inexpensive method, working at the desired frequency, ability of the powder to create a flat and smooth sheet, and a relatively simple computation of the dielectric properties (Içier & Baysal, 2004).

The dielectric constant ( $\varepsilon'$ ) and loss factor ( $\varepsilon''$ ) of the milk powders were determined by measuring the parallel capacitance ( $C_p$ ) and resistance ( $R_p$ ) with an Inductance Capacitance and Resistance (LCR) meter (4285A, Agilent Technologies, Palo Alto, CA) and a liquid test fixture (16452, Agilent Technologies, Palo Alto, CA). Before the measurement, the analyzer was turned on for 30 min for a warm-up and then the electrical performance of the test fixture was confirmed by checking the short residual. Air capacitance ( $C_0$ ) was measured after assembling the test fixture with the spacer to be used to measure the milk powders.

For each test, 2 g milk powder was equally spread onto the bottom half of the test fixture. The fixture was tightly closed after a 1.5 mm spacer was assembled to the test fixture. The test fixture was placed into a temperature chamber (625G, Thermo Fisher

Scientific Inc., Waltham, MA, USA) and connected to the LCR meter by its BNC connector. The sample was heated in the chamber for 60 min to reach the target temperatures ranging from 20 to 90 °C with 10 °C intervals, which were based on the inactivation temperature for bacteria present in low moisture foods. The change in temperature of the sample during heating was monitored using a fiber optic temperature sensor (Model FOT-L-NS-967B, Fiso Technologies, Quebec, Canada) connected to a four-channel fiber-optic thermometer (Fiso UMI 4, Fiso Technologies, Quebec, Canada). The C<sub>p</sub> and R<sub>p</sub> of the milk powders were measured in a range from 1 to 30 MHz at each temperature. The measured values of C<sub>p</sub> and R<sub>p</sub> by the LCR meter were used to calculate the dielectric constant and loss factor using the following equations (Agilent Technologies, 2000).

$$\varepsilon' = \frac{DC_p}{A\varepsilon_0} \tag{3.2}$$

$$\varepsilon'' = \frac{D}{2\pi f R_p \varepsilon_0 A} \tag{3.3}$$

Vacuum permittivity ( $\varepsilon_0$ ) was calculated from the capacitance of the vacuum (approximately equal to air capacitance, C<sub>0</sub>) using the following equation:

$$\varepsilon_0 = \frac{D C_0}{A} \tag{3.4}$$

where D is the gap between electrodes of the test fixture (m),  $C_p$  is parallel capacitance (F),  $R_p$  is the resistance ( $\Omega$ ), f is the frequency (Hz), and A is the electrode area (m<sup>2</sup>).

## Penetration Depth

The power penetration depth which is an important parameter to evaluate the heating uniformity was calculated as (Marra et al., 2014);

$$d_p = \frac{c}{2\pi f \sqrt{2\varepsilon' \left[\sqrt{1 + \left(\frac{\varepsilon''}{\varepsilon'}\right)^2 - 1}\right]}}$$
(3.5)

where c is the speed of light in free space ( $3x10^8$  m/s), f is the frequency in Hz,  $\varepsilon'$  and  $\varepsilon''$  are calculated dielectric constant and loss factor of the sample respectively.

## Radio Frequency Heating Rate and Uniformity Tests

A 6 kW, 27.12 MHz pilot-scale free-running oscillator RF machine (COMBI 6-S, Strayfield International, Wokingham, UK) with parallel-plate electrodes (L: 83 cm and W: 40 cm) was used to heat the milk powders (Fig. 3.1). Table 3.1 indicates the specification of the boxes made from poster board paper occupying the same volume ( $V=175 \text{ cm}^3$ ) with different dimensions. The same amount of milk powders (100 g) was transferred to the boxes for each trial. After closing the lid of the box, it was placed at the center of the bottom electrode. To obtain a better heating rate and uniformity, the gap between the two parallel electrodes was fixed at 10 cm based on our preliminary experimental results. The samples in 3, 6, and 9 cm boxes in both rectangular and cylindrical shapes were heated in the RF oven for 10, 4.5, and 3 min, respectively. Heating uniformity was assessed by heating rates, heating profiles, and thermal images. Temperature profile during RF heating at the geometric center point of the container was measured using a fiber optic temperature sensor (Model FOT-L-NS-967B, Fiso Technologies, Quebec, Canada) connected to a fiber-optic thermometer (Fiso UMI 4, Fiso Technologies, Quebec, Canada). The heating rate (°C/min) was calculated from the obtained temperature profile by dividing temperature increase with heating time. Thermal images of the top surface were captured by an infrared thermal imaging camera having an accuracy of ±2 °C (FLIR T440, FLIR Systems, Inc., North Billerica, MA, USA).

## Electric Field Strength

When the milk powder was heated in an RF oven, the electromagnetic energy was absorbed by the powder. The absorbed energy was converted to the thermal power (P), which can be calculated by the following equation (Ling, Liu, Zhang, & Wang, 2018):

$$P = 2\pi f \varepsilon_0 \varepsilon'' |E|^2 = \rho C_{pp} \frac{\Delta T}{\Delta t}$$
(3.6)

where *P* is the power generated per unit volume (W/m<sup>3</sup>), f is the frequency (Hz),  $\varepsilon_0$  is vacuum permittivity (8.854 × 10<sup>-12</sup> F/m),  $\varepsilon''$  is dielectric loss factor of food sample, and |*E*| is the electric field strength inside the load (V/m),  $\rho$  is the density of food sample (kg/m<sup>3</sup>), C<sub>pp</sub> is the specific heat capacity of milk powder (J/g°C), and  $\Delta T/\Delta t$  is the heating rate (°C/s).

It should be noted that the rate of temperature increase at the geometric center was used to estimate the heat generated within the sample, and that heat loss to air was neglected.

#### Milk Powder Quality Evaluation

#### Moisture Content and Water Activity

The moisture content and water activity of the milk powders before and after RF heating in boxes 3 and 4 were measured by following the method described in the physical characterization of the milk powders section.

## <u>Solubility</u>

The method described by Zhong et al. (2017) was used with slight modifications. Milk powder solution (5% w/w) was prepared by dissolving the calculated amount of milk powder in distilled water. The solution was stirred in a magnetic stirrer at 400 rpm at room temperature for 30 min and centrifuged at 2250 rpm for 15 min. The total solid of the supernatant was measured by drying it in a vacuum oven at 105 °C for 24 h. The solution before centrifugation was used as the control. The solubility was calculated using the following equation:

Solubility (%) = 
$$\frac{Total \ solid \ in \ the \ supernatant}{Total \ solid \ content \ in \ the \ control} * 100$$
 (3.7)

#### Color Measurement

The color of the milk powders was measured using a Konica Minolta chroma meter (Model CR-410 HS, Minolta, Tokyo, Japan) with illuminant C, 50 mm aperture size, widearea illumination/0° viewing angle, 2° standard observer. The color values were expressed as L\* (lightness: 100, white; 0, black), a\* (+, red; –, green), and b\* (+, yellow; –, blue) respectively. After initial calibration against a calibration plate (Y=93.6, x=0.3137, y=0.3199) provided by the company, three replicate measurements on the external surface of the samples placed in the plastic Petri dishes were performed. The total color difference ( $\Delta$ E) was calculated by equation 8 where L<sub>0</sub>, a<sub>0</sub>, and b<sub>0</sub> represent reference values. Chroma (Eqn. 9) and hue angle (Eqn. 10) were also calculated (Sant'Anna, Gurak, Ferreira Marczak, & Tessaro, 2013).

$$\Delta E = \sqrt{(a^* - a_0)^2 + (b^* - b_0)^2 + (L^* - L_0)^2}$$
(3.8)

Chroma = 
$$\sqrt{(a^*)^2 + (b^*)^2}$$
 (3.9)

Hue angle =  $\arctan(b^*/a^*)$  for quadrant I (+a\*,+b\*)

Hue angle =  $180 + \arctan(b^*/a^*)$  for quadrant II (-a\*,+b\*) and quadrant III (-a\*,-b\*)

(3.10)

Hue angle =  $360 + \arctan(b^*/a^*)$  for quadrant IV (+a\*,-b\*)

## Data Processing and Analysis

All measurements were done in triplicate and all results were reported as the mean value  $\pm$  standard error. Excel (Microsoft Office, Redmond, WA, USA) was used in data processing and analysis. Analysis of variance, Tukey's pairwise comparison test, and Student's t-test with a significant level of 0.05 were performed with the JMP Pro 13 software package (SAS Institute, Cary, NC).

## **Results & Discussion**

#### Effect of Frequency and Temperature on Dielectric Properties of the Milk Powders

The effect of frequency and temperature on  $\varepsilon'$  and  $\varepsilon''$  of milk powders over the frequency range of 1-30 MHz and the temperature range of 20-90 °C are shown in Fig. 3.2 and 3.3. Overall,  $\varepsilon'$  and  $\varepsilon''$  decreased with increasing frequency and increased with increasing temperature (Liu & Guo, 2018; Zhu et al., 2015). Previous studies showed that dielectric properties are affected by ionic conduction and dipole orientation. The increase in  $\varepsilon'$  with an increased temperature might be explained by the increased water dipole activity (Boreddy & Subbiah, 2016) and an increase in  $\varepsilon''$  with temperature could be attributed to the increased mobility of bound water at higher temperatures (Calay, Newborough, Probert, & Calay, 1994).

As seen in Fig. 3.2, whole milk powder had higher values of  $\varepsilon'$  and  $\varepsilon''$  than nonfat milk powder. It is known that fat globules in the whole milk occupy the volume of the conducting medium and limit the movement of conducting ions that would lead to lower  $\varepsilon'$  and  $\varepsilon''$  in whole milk at a low-frequency range (Liu & Guo, 2018). Meanwhile, it is well known that the materials with higher bulk density have the higher dielectric constant and loss factor (Nelson & Trabelsi, 2012). In the previous section, it was reported that the bulk density of the whole milk powder  $(0.56 \text{ g/cm}^3)$  was higher than that of nonfat milk powder  $(0.44 \text{ g/cm}^3)$ . In this regard, the effect of the bulk density on the dielectric properties might predominate the effect of fat content.

Both  $\varepsilon'$  and  $\varepsilon''$  decreased sharply with increasing frequency at higher temperatures. For example, when the frequency increased from 1 to 5 MHz,  $\varepsilon'$  of whole milk powder decreased from 1.55 to 1.47 at 20 °C, and from 10.13 to 6.23 at 90 °C. The possible reason for this behavior can be related to the stability changes of different components such as whey protein, fat, and lactose in milk powder with increased temperature, indicating an interaction between frequency, temperature, and composition (Lin et al., 2016).

A sharp increase in both  $\varepsilon'$  and  $\varepsilon''$  at the same frequency was observed over 60-70 °C compared to the lower temperature. For instance, the  $\varepsilon'$  of milk powder increased from 1.55 to 1.81 when the temperature increased from 20 to 30 °C while from 4.69 to 8.95 with the temperature increased from 70 to 80 °C. This effect can be explained by the fact that the dielectric loss factor of the milk powder increased rapidly above the glass transition due to increased mobility of the polar groups of molecules (Silalai & Roos, 2010).

## Penetration Depth

To gain a better understanding of the effect of the dielectric properties of samples in the heating uniformity, the penetration depth was calculated. When an electromagnetic wave propagates through the sample, the portion of the wave is reflected while another portion penetrates the material (Boreddy & Subbiah, 2016). Penetration depth is useful in the determination of the material thickness at the selected frequency. The thickness of the food sample should not be more than 2 or 3 times the penetration depth to achieve an effective RF pasteurization with uniform heating (Schiffmann, 1995). Penetration depth in

dielectric materials varies with the dielectric properties and composition of the sample and frequency (Bengtsson and Risman, 1971). The penetration depths calculated from the measured dielectric properties of whole and nonfat milk powder at 27.12 and 13.56 MHz at different temperatures are shown in Fig. 3.4. The decrease in the penetration depth with increasing frequency was observed for both whole and nonfat milk powder after 30 °C. The penetration depth of the milk powders at 13.56 MHz increased between 20 and 30 °C and then decreased with the temperature. In the study conducted by Guo et al. (2011), it was reported that the penetration depth of the honey sample indicated a fluctuation at RF frequencies with the temperature. The decrease in the penetration depth with the temperature was reported for various food powders including egg white powder (Boreddy & Subbiah, 2016), chestnut flour (Zhu, Guo, Wu, & Wang, 2012), chickpea flour (Guo, Tiwari, Tang, & Wang, 2008) and red pepper powder (Guo & Zhu, 2014). The penetration depth in the nonfat milk powder was greater than that in the whole milk powder. This can be explained by the fat content dependence of the penetration depth. Bengtsson and Risman (1971) reported that the penetration depth decreases with an increase in the salt and fat content.

## Heating Rate and Uniformity Tests

The temperature profiles of the whole, nonfat milk powder and their 1:1 mixture at the geometric center of the box placed in the middle of two parallel electrodes of the RF oven are shown in Fig. 3.5. The electrode gap was fixed at 10 cm, and the heating time required to reach the temperature from 22 to 80 °C ranged from 4 and 12 min depending on the geometry and height of the box as well as the sample type. A linear increase in the temperature of the milk powders with time was observed for all types of the samples heated

in each box. A similar trend was previously shown for different kinds of low moisture foods including broccoli, onion, chili, tapioca powders and potato starch (Ozturk et al., 2018), egg white powder (Boreddy, Thippareddi, Froning, & Subbiah, 2016), wheat flour (Palazoğlu and Miran, 2018), lentil (Jiao, Johnson, Tang, & Wang, 2012), wheat germ (Ling, Lyng, & Wang, 2018).

The heating rates from the temperature profile were calculated as 4.85, 4.30, 8.74, 9.04, 12.65, 12.23 °C/min for whole milk powder, 7.63, 7.38, 12.76, 12.97, 16.51, 16.83 °C/min for non-fat milk powder and 6.04, 6.02, 10.39, 10.52, 14.20, 14.61 °C/min for 1:1 mixture when heated in box 1, 2, 3, 4, 5 and 6, respectively. The reason for the different heating rates observed for the boxes with different heights is that the milk powder heated in the taller box has a smaller air gap between the sample and the top electrode. Thus, the electric field intensity and energy intensity were higher when the gap between the top surface of the sample and the electrode was smaller based on electric field distribution theory (Li et al., 2018). Even though the high heating rate is associated with high throughput, it causes non-uniformity in heating due to very rapid and runaway heating (Jiao et al. 2012). Therefore, heating the milk powders in the boxes with the 6 cm height providing a relatively short heating time (4.5 min) with a moderate heating rate could be a good selection compared to the boxes with 3 and 9 cm height. For this reason, the quality parameters were evaluated for the whole, non-fat milk powder and their mixture heated in a 6 cm rectangular and cylindrical box in the following sections. It was observed that the increase in the fat content in the milk powder led to a decrease in the heating rate. This might be explained by the water activity of the milk powders. As mentioned before, the initial water activity of whole, nonfat milk powder, and their 1:1 mixture were determined

as 0.183, 0.284, and 0.236, respectively. Calay et al., (1994) reported that the amount of free water in a given sample contributes to dielectric polarization much more than bound water when the sample is placed in an alternating electric field. Another reason could be the effect of the fat content on the electrical conductivity of the milk powders. Mabrook and Petty, (2003) concluded that the increase in the fat content of the milk resulted in a decrease in the milk electrical conductivity. Electrical conductivity is defined as the ability of a material to conduct an electric current and it is associated with the ionic depolarization in dielectric heating (Marra et al., 2009). The sample with high electrical conductivity will have high heating rates since it will be heated faster compared to the sample with low electrical conductivity (Awuah, Ramaswamy, & Tang, 2014). Lastly, the difference in the heating rates might be associated with the dielectric properties of the sample. In general, it is believed that the food material with the higher loss factor will have a higher heating rate since the loss factor is related to the dissipation of electric energy into heat (Piyasena et al., 2003). Moreover, the research conducted by Jiao et al., (2014b) revealed that the peanut samples with the comparable dielectric constant and loss factor have higher heating rates. On the other hand, Wang et al., (2007) investigated the negative relationship between dielectric loss factor and heating rate of mashed potato sample having different salt content. Similarly, in this study, the whole milk powder having a higher dielectric loss factor compared to nonfat milk powder had a lower heating rate. It should be pointed out more systematic research should be done to examine the effect of the dielectric properties on the heating rate in free-running oscillator RF systems.

Thermal images of the top surface of the whole milk powder heated in the boxes with different geometry and height are in shown Fig. 3.6. The low temperature was observed at the outer edge of the sample due to heat loss that occurred during the time spent transferring the sample from the RF oven to the camera. Thermograms indicate that the coldest location is at the center of the top layer whereas hotspots are located around the edges for all samples due to the edge heating effect of the RF. The main reason for the edge heating effect in RF oven is that electromagnetic energy is focused on the edges of the sample causing an even more severe thermal-runaway of the heating due to the increase in the dielectric loss with an increase in temperature. Similar observations have been reported for various low moisture foods subjected to RF heating including wheat flour (Palazoğlu & Miran, 2018), wheat germ (Ling, Lyng, et al., 2018), black peppercorn (Wei et al., 2018), lentil (Jiao et al., 2012). The shape and height of the boxes did not cause any significant change in the temperature distribution at the upper surface of the sample. This can be due to the same amount of input power penetrates a certain amount of the sample heated in the boxes with different geometry and dimension (Li et al., 2018).

#### Electric Field Strength

The power density indicating the energy absorbed by the sample in the AC electric field is proportional to the frequency of the applied electric field, the dielectric loss factor of the sample, and the square of the local electric field (Marra et al., 2009). The electric field inside the food is dependent on the dielectric properties, and the geometry and configuration of the sample in the oven (Buffler, 1993). To examine the effect of the geometry and the dimension of the box on electric field strength, the milk powders were heated in the boxes having a different geometry (rectangular and cylindrical) with different heights. Since the dielectric loss factor of the sample changes with the temperature the

electric field strength during the heating was estimated in the different temperature intervals by using the corresponding dielectric loss factor at that temperature.

The electric field strength of the whole and nonfat milk powder at the different temperature intervals were illustrated in Fig. 3.7. The electric field strength during the heating has been decreased indicating the energy absorbed by the food during heating decreased with temperature. The decrease in the electric field strength with the temperature can be explained by the increase in the dielectric loss factor of the milk powders with the temperature since the electric field strength is inversely proportional to the dielectric loss factor of the sample. Although the dielectric loss factor of the whole and nonfat milk powder was different as discussed in the previous section, their electric field strength was very similar. This can be explained by the difference in the heating rate of whole and nonfat milk powder which was used to estimate power density. The electric field strength increased with the height of the boxes since more power was absorbed by the sample when heated in the taller boxes as a result of the high heating rate. Generally, the electric field strength was lower when the samples were heated in the cylindrical boxes. It should be pointed out the determination of the electric field distribution is very complex since it is influenced by many factors such as the geometry and configuration of the sample in the oven. Thus, the equation used to estimate the electric field strength is generally impractical but provides an understanding of the energy absorbed by the food during the heating.

## Quality Parameters

#### Moisture Content and Water Activity

The moisture contents of the whole and nonfat milk powders before and after RF heating are illustrated in Fig. 3.8. The decrease in the moisture content of the powders after

RF treatment can be explained by the moisture migration to the bottom and edges during heating. Similar findings were reported for several low moisture foods including black peppercorn (Wei et al., 2018), in-shell almonds (Gao et al., 2011), and wheat flour (Tiwari, Wang, Tang, & Birla, 2011). It should be pointed out the milk powder with the lower final moisture contents after RF heating is expected to exhibit better storability (Boreddy & Subbiah, 2016).

The water activity of whole and nonfat milk powder before and after RF heating was shown in Fig. 3.9. The water activity of nonfat milk powder and the 1:1 mixture decreased while the water activity of the whole milk powder increased after RF heating. In general, the reduction in the water activity of RF heated food samples was observed in the previous studies including cashew nut kernels (Liao et al., 2018), corn flour (Ozturk, Kong, Singh, Kuzy, & Li, 2017), and black peppercorn (Wei et al., 2018). On the other hand, the increase in the water activity of whole milk powder at 90 °C for 70 min heat treatment was reported by the study conducted by Baechler et al., (2005). The reason for an increase in the water activity of whole milk powder was associated with the release of water from the amorphous lactose during crystallization (Vuataz, 2002). The milk powders become sticky before crystallization temperature. The observation of clumping of milk powder in RF heating in this study supports that crystallization could be a possible explanation for an increase in water activity. Alternatively, the increase in water activity of whole milk powder might be explained by its moisture sorption isotherms indicating the increase in the water activity with increasing temperature at the same moisture level (Langová & Stencl, 2014). It should be noted that there was no significant difference in the moisture content of whole milk powder before and after RF heating (Fig. 3.8).

**Solubility** 

Solubility is a demanding property for dairy powders since higher solubility accounts for better functional property resulting in higher customer satisfaction (Zhong et al., 2017). It should be noted that the solubility of milk powders depends on many factors mainly chemical nature of the protein, physical state, processing conditions, moisture, and also properties of dissolving solution such as pH, temperature, and ionic concentration (Atuonwu, Ray, & Stapley, 2017; Jaskulski, Atuonwu, Tran, Stapley, & Tsotsas, 2017; Semagoto et al., 2014). As shown in Fig. 3.10, the solubility of the unheated whole, nonfat milk powder, and their 1:1 mixture were 81, 98, and 95 % respectively. An increase in the solubility of whole milk powder was observed after heating the sample in box 4 (6 cm height box in cylindrical shape) while the solubility of whole milk powder did not show a significant difference (p>0.05) after heating in box 3 (6 cm height box in a rectangular shape). The solubility increase after heat treatment has been investigated by Pelegrine and Gasparetto, (2005), and it was interpreted as an indication that neither coagulation nor aggregation between the protein molecules has occurred. Moreover, in the study of Boreddy et al., (2016) the solubility of RF heated egg white powder at 7.0 and 9.5 pH increased while the solubility of the egg white powder at 10.4 pH heated at the same conditions decreased. This behavior can be explained by the temperature and pH dependency of the solubility. The solubility of nonfat milk powder decreased after RF treatment, probably due to the conformational changes in the proteins that occurred in milk powders during heating (Chen et al., 2013). The decrease in solubility was not significant (p>0.05) when the milk powders were heated in box 4. Overall, heating the milk powders in box 4 having cylindrical shape did not cause a significant solubility loss in nonfat milk

powder and the 1:1 mixture but caused a significant increase in the solubility of whole milk powder.

#### Color Measurement

The color of the milk powders was measured to check whether the reactions resulting in the color change such as the Maillard reaction after RF heat treatment have occurred or not (Morales and van Boekl, 1998). As shown in Table 3.2, the color parameters of heated milk powders which are L\* (lightness), a\* (redness), and b\* values (yellowness) decreased for whole milk powder heated in box 3 when compared to the unheated milk powders (p>0.05). For the 1:1 mixture, L\* and a\* and b\* values increased when the sample was heated in both boxes 3 and 4. The change in a\* and b\* values were closely associated with a brown coloration which might have been initiated by the Maillard browning during RF heating (Zhong et al., 2017).  $\Delta E^*$ , the attribute of total color change from the control sample was the highest for whole milk powder heated in box 3. Chroma that indicates the strength and degree of saturation of the color decreased when the milk powders were heated except the mixture of whole and nonfat milk powder. The hue angle value was proportional to the received color and in general, an angle of 0 or  $360^{\circ}$  represents a red hue, while angles of 90, 180, and 270° represent yellow, green, and blue hues, respectively (Khazaei, Jafari, Ghorbani, & Hemmati Kakhki, 2014). Unheated and heated milk powders showed greenness with no significant change between the samples (p>0.05).

## Conclusion

To develop an effective pasteurization process for milk powders the dielectric properties of the whole, nonfat milk powder and their mixtures were measured at different temperatures (20-90 °C) and frequencies (1-30 MHz). The dielectric properties of milk

powders increased with increasing temperature and decreasing frequency. The dielectric properties of the whole milk powder were higher than nonfat milk powder due to its bulk density. Heating uniformity in RF pasteurization was evaluated by heating rate, heating profile, thermal images, electric field strength, and penetration depth. The hot spots around the edges were observed in RF heating for all boxes. An increase in the fat content in the milk powder led to a decrease in heating rate associated with the water activity, electrical conductivity, and dielectric properties of the milk powders. A decrease in the electric field strength during heating was observed for milk powders. Whole and nonfat milk powders had comparable electric field strength. Moreover, for the boxes with the same volume, the electric field strength increased with increased height. The penetration depth of the nonfat milk powder was greater than the whole milk powder. A small decrease in the moisture content of RF heated milk powder was observed due to the moisture migration during heating. The solubility of whole milk powder increased while the solubility of the nonfat milk powder decreased the solubility of the 1:1 mixture remained constant after RF heating. Overall, the findings from the study show the importance of the geometry and dimension of the container used in the RF heating on the heating uniformity as well as the quality of the end product.

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## Tables

<b>Box Number</b>	Geometry	Dimension (cm)		
1	Rectangular	h=3; l and w=7.62		
2	Cylindrical	h=3; r=4.30		
3	Rectangular	h=6; 1 and w=5.39		
4	Cylindrical	h=6; r=3.04		
5	Rectangular	h=9; l and w=4.44		
6	Cylindrical	h=9; r=2.48		

**Table 3.1** Specification of the boxes used in RF heating (h: height; l: length; w: width; r:radius).

		L*	a*	b*	$\Delta E^*$	Chroma	Hue (°)
Whole	Control	78.72 <sup>a</sup>	-4.63 <sup>b</sup>	9.45ª		10.52 <sup>a</sup>	178.88ª
	Box 3	75.20 <sup>b</sup>	-4.25 <sup>a</sup>	8.36 <sup>b</sup>	3.71ª	9.38 <sup>b</sup>	178.89 <sup>a</sup>
	Box 4	78.22ª	-4.31ª	8.72 <sup>ab</sup>	2.82ª	9.73 <sup>ab</sup>	$178.88^{a}$
Non-Fat	Control	78.98ª	-4.16 <sup>b</sup>	8.95 <sup>a</sup>		9.87ª	178.86ª
	Box 3	77.71 <sup>b</sup>	-3.83 <sup>ab</sup>	$8.48^{ab}$	1.43 <sup>a</sup>	9.31 <sup>ab</sup>	178.85 <sup>a</sup>
	Box 4	78.51ª	-3.51ª	7.34 <sup>b</sup>	1.95ª	8.14 <sup>b</sup>	$178.87^{a}$
1:1 W:N	Control	$78.47^{a}$	-4.01 <sup>a</sup>	8.86 <sup>a</sup>		9.72ª	178.85 <sup>a</sup>
	Box 3	79.09 <sup>a</sup>	-4.26 <sup>a</sup>	9.58ª	1.19 <sup>a</sup>	10.49 <sup>a</sup>	178.85 <sup>a</sup>
	Box 4	79.74ª	-4.14 <sup>a</sup>	9.23ª	1.46 <sup>a</sup>	10.11 <sup>a</sup>	$178.85^{a}$

**Table 3.2** Color parameters (L\*, a\*, b\*,  $\Delta E^*$ , Chroma, and Hue Angle) of unheated (control) and RF heated (in box 3 and 4) whole, nonfat, and 1:1 W:N milk powder.

# Figures



Figure 3.1 Simplified schematic diagram of the RF pasteurization system.



Figure 3.2 Dielectric constant and loss factor in log scale (mean  $\pm$  SE of three replicates) of (a) whole milk powder; (b) nonfat milk powder; (c) their 1:1 mixture.



**Figure 3.3** Dielectric constant and loss factor in log scale (mean  $\pm$  SE of three replicates) of (d) 1:2; (e) 2:1; (f) 1:3; (g) 3:1 whole:nonfat milk powder mixture.



△ Whole Milk Powder at 13.56 MHz ○ Nonfat Milk Powder at 13.56 MHz ◇ Whole Milk Powder at 27.12 MHz □ Nonfat Milk Powder at 27.12 MHz

**Figure 3.4** Penetration depths (m) in log scale calculated from the measured dielectric properties of whole and nonfat milk powder at 13.56 and 27.12 MHz at different temperatures.


**Figure 3.5** Temperature profiles of (a) whole milk powder; (b) nonfat milk powder; (c) their 1:1 mixture during RF heating at 27.12 MHz in the boxes with different geometry and dimension.



Figure 3.6 Top surface temperature distribution (°C) of whole milk powder heated in the

RF system in (a) 3 cm height box; (b) 6 cm height box and (c) 9 cm height box.



**Figure 3.7** Electric field strength in (a) whole milk powder; (b) nonfat milk powder during RF heating at 27.12 MHz heated in the boxes with different geometry and dimension where interval # (1) 20-30 °C; (2) 30-40 °C; (3) 40-50 °C; (4) 50-60 °C; (5) 60-70 °C.



**Figure 3.8** Moisture content (mean  $\pm$  SE of three replicates) of unheated (control) and RF heated (in boxes 3 and 4) whole, nonfat milk powder and their 1:1 mixture.



**Figure 3.9** Water activity (mean  $\pm$  SE of three replicates) of unheated (control) and RF heated (in boxes 3 and 4) whole, nonfat milk powder and their 1:1 mixture.



**Figure 3.10** Solubility (mean ± SE of three replicates) of unheated (control) and RF heated (in boxes 3 and 4) whole, nonfat milk powder and their 1:1 mixture.

## CHAPTER 4

# EFFECT OF SURROUNDING MEDIUM ON RADIO FREQUENCY (RF) HEATING UNIFORMITY OF CORN FLOUR<sup>1</sup>

<sup>&</sup>lt;sup>1</sup>Dag D., Singh R.K., Kong F. 2021. Journal of Food Engineering. 307: 110645. Reprinted here with permission of the publisher.

#### Abstract

Radio frequency (RF) heat treatment holds great potential for packaged food powder pasteurization by providing rapid and volumetric heating with a high penetration depth. Edge and corner overheating create high-temperature differences throughout the product, which is a major drawback in its industrial applications. In this study, the effect of the surrounding media (air, DI water, and soybean oil), medium level, heating mode, electrode gap, and heating time on the heating uniformity was investigated by the thermal distributions, heating uniformity index, and the heating rate at the top and middle layers of RF heated corn flour. The thermal images indicated that the edge and corner overheating were improved by immersing the corn flour container in soybean oil. A higher heating rate (7.77 and 9.86 °C/min at the top and middle layers, respectively) with a better heating uniformity (as indicated by lower heating uniformity indices: 0.047 and 0.037 at the top and middle layers, respectively) was obtained for the corn flour heated in soybean oil with an 11 cm electrode gap compared to those heated in air. The study proposed a method for improving the heating uniformity of RF heated food powders which can be easily implemented in industrial applications of RF treatment.

#### Introduction

Low moisture foods (LMFs) including but not limited to flour, cereals, nuts, spices, chocolate, butter, and dried milk and egg products, etc., are considered shelf-stable due to their long term storability associated with their low water activity (typically <0.7) (Huang, Marra, & Wang, 2016; Jiao, Shi, Tang, Li, & Wang, 2015). Although low water activity has a clear advantage for controlling the growth of foodborne pathogens, there are still major concerns such as a longer period of survival and higher thermal resistance of these foodborne pathogens in low water activity environments (Beuchat et al., 2013; Syamaladevi et al., 2016). Recently, the studies focusing on microbial inactivation in LMFs have drawn increased attention from both industry and research communities due to an increase in foodborne infections and outbreaks linked to LMFs (Beuchat et al., 2013; Podolak, Enache, Stone, Black, & Elliott, 2010).

Corn flour is widely used as an ingredient in a variety of food products such as baked goods, infant foods, biscuits, wafers, cereals, and breading, and also as a filler, binder, and carrier in meat products (Rosentrater & Evers, 2018). Corn flour is also used as a substitute for rice and wheat flour in the production of gluten-free products or can be mixed with other flour types to reduce gluten content in food products. Although corn flour is usually consumed after heat treatments such as baking and cooking, it is also used in confectionery products without heating treatment. Therefore, it is important to conduct a study giving insightful information on heating systems in which corn flour is used.

Fumigation, wet and dry steam, ozone treatment, and conventional heating are common pasteurization techniques used in pasteurization, sterilization, and disinfestation of the LMFs. These methods have limitations due to minimal customer acceptance,

significant-quality deterioration, long treatment time resulting in high energy consumption (Ozturk, Kong, Singh, Kuzy, & Li, 2017). RF heating holds great potential to tackle the aforementioned issues by providing rapid and volumetric heating with a long penetration depth (Dag, Singh, & Kong, 2020). RF heating is advantageous over conventional heating in terms of having better quality retention associated with the shorter treatment time and large-scale processing of the products due to deeper penetration and uniform heating (Boreddy, Thippareddi, Froning, & Subbiah, 2016). However, non-uniformity in temperature distribution is the main limitation of RF heating that hinders its industrial applications. Although higher temperatures at some locations would be beneficial for pasteurization and sterilization, it might cause severe quality deteriorations in the sample (Huang, Zhang, Marra, & Wang, 2016; Ling, Cheng, & Wang, 2019). Several theoretical, modeling, and design-based works such as hot air circulation (Wang, Monzon, Johnson, Mitcham, & Tang, 2007), preconditioning the sample (Zhu et al., 2018), intermittent mixing (Wang et al., 2006), rotating the sample at its central axis and conveyor belt movement (Palazoğlu & Miran, 2018), electrode shape modification (Choi, Park, Yang, Kim, & Chun, 2017), and surrounding the container with polyetherimide (Jiao, Tang, & Wang, 2014) were conducted to improve the uniformity in temperature distribution for RF processes.

The immersion of food into the surrounding media having similar dielectric properties with the food was found to improve heating uniformity, as shown in previous studies involving immersing the shell eggs in deionized water (Lau, Thippareddi, & Subbiah, 2017), fresh fruits in water (Birla, Wang, & Tang, 2008; Tiwari, Wang, Birla, & Tang, 2008) and cuboid and step-shaped minced frozen beef in glycerol solution (Li et al.,

2020); however, the method has not been extended to RF heating of food powder yet. In the previous RF studies, food powders in the sealed plastic containers were directly placed in the RF oven without surrounding by another medium. Thanks to the very low dielectric properties of air, the food powder was targeted for heating rather than the surrounding air. However, the use of air as a surrounding medium could not provide a solution to reduce the edge and corner heating that occurred due to the fringe effect at the interface between the side of the food and the air (Wang, Wig, Tang, & Hallberg, 2003). In this regard, the surrounding medium chosen specifically for the sample might be a solution to reduce the temperature differences in the RF-treated food powders by providing a combination of surface cooling effect and electric field modification (Lau et al., 2017). In the light of this idea, the objectives of this study were to investigate the effect of the (1) surrounding medium type (air, deionized water, and soybean oil), (2) surrounding medium level, (3) heating mode (stationary and continuous), (4) electrode gap and (5) heating time on the temperature distribution of RF heated food powder.

#### **Materials & Methods**

## Materials and Physical Characterization

Corn flour was obtained from the Georgia Spice Company (Atlanta, GA). The initial moisture content of corn flour was determined in triplicate by drying samples in aluminum dishes in a NAPCO vacuum oven (Model 5831, Precision Scientific, Chicago, IL) at 105 °C for 24 h (AOAC, 2000). Deionized water (18.2 M $\Omega$  cm) was obtained with a Purelab Ultra water purification system (ELGA, High Wycombe, Buckinghamshire, United Kingdom) and 100% soybean oil (Great Value, Walmart) was purchased from the local market. The deionized water and soybean oil were selected as the surrounding

medium since both media do not convert a significant proportion of RF energy into heat compared to corn flour and both media are food-grade and can be reused via a recycle unit in a larger-scale RF oven for industrial applications.

The bulk density  $(\rho)$  of corn flour was determined gravimetrically as:

$$\rho = \frac{m}{V} \tag{4.1}$$

where *m* is the total mass of the sample (g) and *V* is its volume (cm<sup>3</sup>)

The dielectric constant ( $\varepsilon'$ ) and loss factor ( $\varepsilon''$ ) of corn flour were determined by measuring the parallel capacitance ( $C_P$ ) and resistance ( $R_P$ ) with an Inductance Capacitance and Resistance (LCR) meter (4285A, Agilent Technologies, Palo Alto, CA) and a liquid test fixture (16452, Agilent Technologies, Palo Alto, CA). The analyzer was turned on for 30 min for a warm-up and then the electrical performance of the test fixture was confirmed by checking the short residual. Air capacitance ( $C_0$ ) was measured after assembling the test fixture with the spacer to be used for the measurements.

For each test, 6.12 g corn flour (giving a density of 9.0 g/ml) was equally spread onto the bottom half of the test fixture. The fixture was tightly closed after a 3.0 mm spacer was assembled to the test fixture. The test fixture was connected to the LCR meter by its BNC connector. The measured values of  $C_P$  and  $R_P$  at 27.12 MHz were used to calculate the dielectric constant and loss factor using the following equations (Agilent Technologies, 2000).

$$\varepsilon' = \frac{DC_P}{A\varepsilon_0} \tag{4.2}$$

$$\varepsilon'' = \frac{D}{2\pi f R_p \varepsilon_0 A} \tag{4.3}$$

Vacuum permittivity ( $\varepsilon_0$ ) was calculated from the capacitance of the vacuum (approximately equal to air capacitance,  $C_0$ ) using the following equation:

$$\varepsilon_0 = \frac{DC_0}{A} \tag{4.4}$$

where *D* is the gap between electrodes of the test fixture (m),  $C_P$  is parallel capacitance (F),  $R_P$  is the resistance ( $\Omega$ ), *f* is the frequency (Hz), and *A* is the electrode area (m<sup>2</sup>).

## Radio Frequency Heating

The corn flour (288 g) at  $21.2\pm0.4$  °C was placed in a Rubbermaid plastic food storage container with a 7.32 cm (H) x 12.7 cm (W) x 12.7 cm (L) dimension given in the product specification provided by the company. The inner dimension of the food storage container was measured as 5.0 cm (H) x 10.2 cm (W) x 10.2 cm (L). The food container and its lid were made of polypropylene and low-density polyethylene, respectively. The plastic container filled with corn flour was horizontally separated into two equal layers from the middle by the cheese cloth. The cheese cloth was used to remove the top half layer as quickly as possible to capture the thermal image of the middle layer after heating. The plastic container having corn flour was then placed into a pyrex cylindrical glass container with 17 cm diameter, 9 cm height, and 0.24 cm wall thickness. The space between the plastic and glass container was filled with either deionized water or soybean oil which are at room temperature (approximately 22 °C). Both media were added to the levels at 6.5, 7.1, and 8.8 cm to fill the space up to the lid of the corn flour container, just covered the lid (1 cm over the lid) and filled glass container, respectively. Wooden chopsticks with 0.3 cm diameter were used to fit the plastic container into the glass container and to allow the deionized water and soybean oil to have contact with the bottom of the plastic container.

The electrode gap is defined as the distance between the top and bottom electrodes while the air gap is the distance between the top electrode and the sample. When the electrode gap is moved vertically by keeping the sample thickness constant, both electrode and air gap are changed in the system. In this study, 3 different electrode gaps were set (11, 11.5, and 12 cm) to change the heating rate for each system. The experimental setup can be seen in Fig 4.1.

For stationary heating mode, the glass container having corn flour container was positioned at the center of the bottom plate and heated for 5 min in a pilot-scale 6 kW, 27.12 MHz free-running oscillator RF machine (COMBI 6-S, Strayfield International, Wokingham, UK) with parallel-plate electrodes (L:83 cm and W:40 cm). For continuous heating mode, the glass container was connected to the twin syringe pump (Model 33, Harvard Apparatus, Holliston, MA) via nylon thread and pulled from one end to the other to convey the sample in the RF cavity. The speed of the pump was set to achieve 4, 5, and 6 min heating time. The experimental setup was shown in Fig. 4.2 to visualize the system.

Twenty-eight experimental setups (shown in Table 4.1) were conducted to investigate the effect of the surrounding medium (air, deionized water, and soybean oil), the medium level (6.5, 7.1, and 8.8 cm), the heating mode (stationary and continuous), electrode gap (11, 11.5, and 12 cm), and heating time (4, 5, and 6 min) on the temperature distribution, heating uniformity index, and heating rate of RF heated corn flour.

## Heating Uniformity Evaluation

Thermal images (with 320x240 IR resolution) of the top and middle layers were captured immediately by an infrared thermal imaging camera having an accuracy of  $\pm 2$  °C (FLIR T440, FLIR Systems, Inc., North Billerica, MA, USA). The image analysis system

(FLIR Tools Software, FLIR Systems, Inc., North Billerica, MA, USA) was used to collect and analyze the surface temperature data points for each layer. 135x135 pixels within each sample layer were used to calculate the average product temperature and standard deviation. The heating rate (°C/min) was calculated from the average temperatures obtained from the image analysis system by dividing temperature increase with heating time. The average surface temperature at the top and middle layers before and after heating was used in the heating rate calculation.

The uniformity index ( $\lambda$ ) is a unique parameter to a specific RF unit and the sample at the fixed conditions such as electrode gap, mode of heating, and the surrounding medium, etc. It is derived experimentally from product temperature measurements before and after heating, using the following equation (Wang et al., 2007):

$$\lambda = \frac{\Delta\sigma}{\Delta\mu} \tag{4.5}$$

where  $\Delta \sigma$  is the rise in the standard deviation of product temperature and  $\Delta \mu$  is the rise in mean product temperature over the treatment time. A lower value of  $\lambda$  indicates higher heating uniformity.

#### Data processing and analysis

All experiments were done in triplicate. Minimum, maximum, and average temperatures, heating uniformity index, heating rate, and temperature distributions at the top and middle layers of each heating system were compared to investigate the effect of the surrounding medium, medium level, heating mode, electrode gap, and heating time on RF heating uniformity. The mean value and standard error were reported in the tables. Analyses of variance were performed with the JMP Pro 15 software package (SAS

Institute, Cary, NC). Differences between the mean values were considered significant at P<0.05. The mean values were separated with Tukey's method at a significance level of 0.05. The effect of the single variables and their all interactions were included in each analysis. The effect of the single variable was given in the discussion section by providing a p-value while the combined effect of the variables was shown in the tables with the different letters indicating the significant difference.

#### **Results & Discussion**

#### The effect of the surrounding medium

The relationship between the rise in the standard deviation and mean temperature of the sample was investigated before comparing the heating uniformity index of the samples heated in different media to various temperatures for temperature distribution evaluation. Fig. 4.3 indicates the high correlation between the rise in the standard deviation and the average temperature of corn flour heated in air, DI water, and soybean oil. Overall, corn flour heated in DI water had the lowest slope indicating the highest heating uniformity associated with the low-temperature increases. Corn flour heated in soybean oil had a slightly lower slope compared to those heated in the air although a higher temperature increase was achieved. After validating the use of a heating uniformity index for the samples heated in different media to the different temperatures, it was further used to evaluate the heating uniformity within the sample.

The thermal images captured by an infrared camera after RF heating is a quick tool to examine the heating uniformity on the sample surface. In this study, the temperature scales in all thermal images were set using an automatic setting to observe as many details as possible in each layer. Thus, the temperature scale should be taken into account while interpreting the temperature distributions from the images. Fig. 4.4 indicates the surface temperature distribution at the top and middle layers of RF heated corn flour surrounded with air, DI water, and soybean oil under stationary and continuous heating mode for 5 min with an 11 cm electrode gap. It was seen that the temperatures near the edges and at the corners are higher than those at the center for corn flour surrounded by air and soybean oil due to the edge and corner heating. The edge/corner heating is a common drawback of RF heating resulting in the higher temperature difference in the treated sample. This heating trend could be explained by the electric field behavior in the sample during RF heating. The computer simulation results obtained by Tiwari et al., (2011a) showed that the electric field was normal on the central parts of wheat flour in a cuboid container while it was deflected at the corners and edges resulting in an increased net electric field at the corners, edges, and lower sections of the sample due to the fringe effect. RF power density has also increased with an increase in the electric field since it is proportional to the square of the electric field. Thus, the temperatures at the edges and corners were higher compared to the central area due to the increased power density at these parts. The edge heating patterns in RF heated food samples in a rectangular container were also observed in the several RF studies for corn flour (Ozturk et al., 2017), wheat flour (Tiwari et al., 2011a), and whole and nonfat milk powder (Dag, Singh, & Kong, 2019). However, it should be noted that placing the corn flour container in soybean oil has decreased the edge and corner heating according to the thermal images (Fig. 4.4) and heating uniformity index (Table 4.2). For example, the heating uniformity index has declined significantly (P < 0.05) from 0.088 to 0.064 at the top layer and 0.069 and 0.051 at the middle layer by immersing the corn flour container in soybean oil and heat for 5 min in stationary RF heating mode with an 11 cm

electrode gap. The movement of the sample has further improved heating uniformity which can be supported by a decrease in the heating uniformity index from 0.064 to 0.047 at the top layer and 0.051 to 0.037 at the middle layer when corn flour was heated in soybean oil for 5 min with an 11 cm electrode gap.

The improvement in heating uniformity with soybean oil might be explained by the dielectric and thermal properties of each material. The density and moisture content of corn flour were determined as 0.90 g/ml and 7.80% (wet basis) respectively, and the dielectric constant and loss factor were measured as 4.31 and 0.27 at 20 °C and 27.12 MHz. The dielectric constant and loss factor of air and deionized water were taken from the literature as 1 and 0 for air, 80 and 0.11 for water at 20 °C and 27.12 MHz, respectively (Lau, Dag, Ozturk, Kong, & Subbiah, 2020). The dielectric properties of soybean oil at 27.12 MHz could not be measured experimentally due to the very low dielectric loss factor; thus, the dielectric constant and loss factor of soybean oil was taken as 3 and 0.15 for soybean oil at 20 °C and 27.12 MHz, respectively according to the results obtained by Inoue et al., (2002). The dielectric constant is a measure of the ability of the sample to store the electromagnetic energy whereas the dielectric loss factor is a measure of the ability of the sample to convert the electromagnetic energy into heat (Alternimi et al., 2019). Corn flour heated in deionized water showed a lower temperature increase compared to the sample heated in the air and soybean oil due to the high dielectric constant of deionized water. Deionized water absorbed more electromagnetic energy than corn flour resulting in low average temperature in the sample. The low heating uniformity indices for corn flour heated in deionized water might be associated with a lower electric field strength causing a less temperature increase in corn flour. The corn flour was heated with a higher heating rate with a lower uniformity

index when surrounded with soybean oil instead of air since the dielectric properties of soybean oil match better with corn flour compared to those of air.

The effect of the surrounding medium on RF heating of corn flour might be also explained by the temperatures of both surrounding media after heating. The final temperatures of deionized water and soybean oil were found as  $26.45\pm0.45$  °C and  $47.05\pm0.15$  °C, respectively, after 5 min RF heating with an 11 electrode gap and 8.8 cm medium level. Although deionized water absorbed more electromagnetic energy due to its high dielectric constant, this absorbed electromagnetic energy would not be converted into heat due to its low dielectric loss factor. On the other hand, soybean oil allowed corn flour heated due to the comparable dielectric constant values with corn flour. Soybean oil was heated to lower temperatures than corn flour due to the lower dielectric loss factor compared to the sample. Thus, soybean oil might act as a cooling medium by allowing the corn flour heated simultaneously.

## The effect of surrounding medium level

The effect of surrounding medium level on RF heating uniformity was shown in Fig. 4.5 and Table 4.3 by temperature distributions and uniformity indices. Heating uniformity indices decreased for corn flour surrounded with DI water (0.074, 0.061, and 0.045 at the top layer for DI water level at 6.5, 7.1, and 8.8 cm, respectively) while the uniformity indices increased for corn flour surrounded with soybean oil (0.048, 0.053 and 0.064 at the top layer for soybean oil level at 6.5, 7.1 and 8.8 cm, respectively). On the other hand, the heating uniformity index and heating rate were reported as 0.088 and 5.53 °C/min for corn flour surrounded with the level of DI water and soybean

oil can be explained by the effect of the surrounding medium type. It was observed that the higher heating rates were achieved by surrounding the corn flour with soybean oil as opposed to DI water when compared to corn flour heated in air. Increasing the level of DI water and soybean oil has further enhanced this trend resulting in lower and higher heating rates, respectively. The effect of the surrounding medium might be also associated with the top electrode voltage which depends on the air gap, material thickness, dielectric properties, density and specific heat of the sample, and heating rate (Metaxas, 1996; Zhu, Huang, & Wang, 2014). The material thickness is also linked with the amount of sample in the system which affects the heating rate. The analytical method used in the calculation of top voltage has become even more complicated when the level of the surrounding medium was investigated since all of the variables mentioned previously were changed either due to the temperature change or the level of the surrounding medium. The current study has pointed out the importance of the level of the surrounding medium, however; an individual study is in need to understand the effect of the surrounding medium on RF heating in a more controlled manner.

Although corn flour heated in DI water and soybean oil with 6.5 cm level had similar heating rates (5.10 and 5.26 °C/min in DI water and soybean oil at the top layer, respectively), the heating uniformity indices were significantly different for each system (P<0.05) (0.074 and 0.048 in DI water and soybean oil at the top layer, respectively). This result showed the impact of the surrounding medium at the bottom and sides of the corn flour container even though there was no liquid at the top of its lid. The corn flour heated in soybean oil with the lowest uniformity with the highest uniformity index (0.064) and heating rate (7.44 °C/min) at the 8.8 cm liquid level still showed better temperature distribution than corn flour surrounded by air.

#### The effect of the heating mode

The effect of the heating mode on RF heating uniformity is shown in Fig. 4.4 (heated in the different media), Fig. 4.6 (heated at the different electrode gaps), and Table 4.2 with temperature distributions at the top and middle layers, maximum, minimum, and average temperature values, heating uniformity indices and heating rates. From Fig. 4.4 and 4.6, the improvement in the temperature distribution with the sample movement can be observed from the thermal images captured from the corn flour surface after heating for 5 min with an 11 cm electrode gap. This improvement in heating uniformity can be supported by the uniformity indices shown in Table 4.2 with the lower uniformity index for corn flour conveyed along the RF cavity. For instance, the uniformity index has decreased from 0.088 to 0.063 and from 0.064 to 0.047 at the top layer for corn flour heated at an 11 cm electrode gap when surrounded with air and soybean oil, respectively. The improvement in the heating uniformity with the sample movement might be explained by the exposure of the sample to the different electromagnetic patterns and strengths while conveying in the RF cavity (Chen, Lau, Chen, Wang, & Subbiah, 2017). On the other hand, no significant difference (P>0.05) in the heating uniformity index was observed (changed from 0.045 to 0.046) at the layer for the corn flour sample surrounded by DI water. The higher heating uniformity index for the stationary heating mode might be explained by the thermal runaway effect of the RF process where the electromagnetic energy generation is higher in the locations with the higher temperatures as a result of the higher dielectric loss factor. This thermal runaway effect was lower in the continuous heating mode. From the

thermal images, it can be also seen that the edge or corner over-heating was noticeably reduced at both top and middle layers especially for the corn flour surrounded by soybean oil.

Although there is no clear trend in the heating mode and heating rate, a slight increase in the heating rate was observed with the sample movement for corn flour surrounded by air and soybean oil heated for 5 min with an 11 cm electrode gap. In general, the heating rates were comparable when corn flour was heated either in stationary and continuous heating mode. This is an advantage for the application of continuous RF heating in the food industry. The higher throughput could be achieved with a continuous heating system with similar heating rates compared to the batch system.

## The effect of the electrode gap

Fig. 4.6 shows the temperature distributions of RF heated corn flour in soybean oil for 5 min with 11, 11.5, and 12 cm electrode gaps. The thermal images demonstrated the improvement in the temperature distributions with an increasing electrode gap which might be associated with the lower heating rates. According to the results shown in Table 4.2, the heating rates decreased by increasing the electrode gap which corresponds to the decreased power in the RF systems (Gao, Tang, Wang, Powers, & Wang, 2010). For instance, heating rates for corn flour heated in soybean oil were 7.77, 6.72, and 6.14 °C/min at the top layer and 9.86, 8.39, and 7.64 °C/min at the middle layer when heated in the continuous RF heating mode with 11, 11.5, and 12 cm electrode gaps, respectively. In general, a lower heating rate is selected in RF systems since a higher heating rate may result in thermal runaway resulting in poor heating uniformity. On the other hand, the low heating rates reduce the throughput of the system which is a limitation for industrial applications. Thus, the highest heating rate possible with a reasonable heating uniformity was aimed at the industrial applications of RF heating. In the previous RF studies, the heating rates around 6-8 °C/min (which were also achieved in this study for corn flour) have been selected for an effective and even heating of low moisture foods (Gao et al., 2010; Zhou & Wang, 2016). The heating uniformity indices for corn flour heated in soybean oil with the continuous heating mode for 5 min were 0.047, 0.041, and 0.045 at the top and 0.037, 0.032, and 0.033 at the middle layer for 11, 11.5, and 12 cm electrode gaps, respectively. Therefore, the electrode gap of 11 cm corresponding to a higher heating rate was suggested for RF heating of corn flour since it has a comparable heating uniformity index than those of longer electrode gaps.

## The effect of heating time

The adjustment in the movement speed varies the product residence times and corresponding throughputs in a continuous RF industrial application (Zhou & Wang, 2016). In the microbial validation RF studies, the electrode gap and movement speed were optimized in a manner allowing the sample cold spots to reach the target temperature. The individual effect of heating time/movement speed which will be discussed in this section might provide information in the development of the RF system ensuring even temperature distribution.

Corn flour heated in soybean oil with an 11 cm electrode gap was selected as a model to explain the effect of heating time on RF heating uniformity. Thermal images in Fig. 4.7 indicated that the increase in heating time (or decreasing the movement speed) led to uneven heating which is more predominant at the top layer. However, the change in the heating uniformity indices was not significant (P>0.05) according to the results shown in

Table 4.4 showing that the heating uniformity indices as 0.049, 0.047, and 0.050 for 4, 5, and 6 min RF heating at the top and 0.041, 0.037, and 0.031 at the middle layer. The improvement in temperature distribution at the middle layer with treatment time was observed for corn flour in soybean oil. Longer heating time might allow more heat conduction resulting in more even temperature distribution within the sample (Ling, Lyng, & Wang, 2018; Villa-Rojas, Zhu, Marks, & Tang, 2017).

Table 4.4 also indicates that the increase in the heating time resulted in a decline in the heating rates. For example, heating rates were 9.40, 7.77, and 7.68 °C/min at the top and 11.24, 9.86, and 8.92 °C/min at the middle layer when heated for 4, 5, and 6 min, respectively. The decline in the heating rates with the treatment time can be explained by the temperature-time profile of RF heated low moisture foods reported previously. Low moisture foods have demonstrated the temperature-time profiles having a decreasing slope with time (meaning a decrease in heating rate with treatment time) for corn flour (Ozturk et al., 2017), red pepper (Zhang, Zhao, Gong, & Jiao, 2020), powdered infant formula milk (Lin, Subbiah, Chen, Verma, & Liu, 2020), wheat flour (Palazoğlu & Miran, 2018) and wheat germ (Ling et al., 2018). The longer heating time increased the final product temperature which may cause more moisture loss or migration during the RF heating process and lead to relatively lower heating rates. At the early stage of heating, the sample temperature has increased rapidly and then the moisture vapor pressure in the sample has exceeded that of the environment resulting in the moisture expulsion process (Zhou et al., 2018).

## Conclusions

The effect of immersing corn flour in the air, DI water, and soybean oil on RF heating uniformity was investigated experimentally in this study. Our results showed corn flour heated in soybean oil has a lower uniformity index when compared with air medium at similar temperature levels. Overall, the corn flour container immersed in soybean oil was heated at a higher rate and with better heating uniformity which is an advantage for the industrial application of RF treatment. Thermal images have demonstrated that a thermal runaway at the edges and corners was reduced and more uniform temperature distributions were obtained at the middle and top layers of the RF heated corn flour immersed in soybean oil. The heating uniformity of corn flour surrounded with soybean oil was further improved by conveying the sample in the RF cavity during heating. Additionally, the effect of medium level, electrode gap, and heating time on RF heating uniformity was investigated since the target temperature specific to the food samples can be achieved easily by optimizing the electrode gap and heating time/movement speed in a more controlled manner.

It should be noted that although heating uniformity indices suggested the improvement in the heating uniformity, the difference between the maximum and minimum temperature in each layer and temperature difference between the layers are not at the desired level yet. For this purpose, the combination of other techniques such as mixing and rotating with immersing the sample in liquids is recommended for future studies.

In addition to the parameters investigated in the study, the effect of the secondary container radius which was filled with the medium, and the ratio of the containers filled with the sample and surrounding medium on RF heating performance are recommended for future studies. Although the proposed method is still at an early stage, it is strongly recommended to extend the current study to higher pasteurization temperatures and conduct the microbial inactivation studies in which the microbial inactivation level of each system are compared. The computer simulation studies might also provide valuable information to understand the system in detail and to explore the effect of the other parameters such as container materials or sample properties when the sample is heated in a different medium than soybean oil and DI water.

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## Tables

Surrounding Medium	Heating Mode	Surrounding medium leve (cm)	l Electrode Gap (cm)	Heating Time (min)
Air	Stationary	N/A	11, 11.5, and 12	5
	Continuous	N/A	11 11.5, and 12	4, 5, and 6 5
Deionized Water	Stationary	6.5 and 7.1 8.8	11 11, 11.5, and 12	5
	Continuous	8.8	11 11.5, and 12	4, 5, and 6 5
Soybean Oil	Stationary	6.5 and 7.1 8.8	11 11, 11.5, and 12	5
	Continuous	8.8	11 11.5, and 12	4, 5, and 6 5

 Table 4.1 Experimental design.

**Table 4.2** Minimum, maximum, and average temperature, heating uniformity index, and the heating rate at the top and middle layers of corn flour surrounded with air, DI water (at 8.8 cm level), and soybean oil (at 8.8 cm level) under stationary and continuous heating mode for 5 min with 11, 11.5, and 12 cm electrode gaps.

Layer	Heating Mode	Electrode Gap (cm)	Surrounding Medium	Min Temperature (°C)	Max Temperature (°C)	Average Temperature (°C)	Heating Uniformity Index	Heating Rate (°C/min)
Тор	Stationary	11	Air	$43.20{\pm}1.46^{\scriptscriptstyle ijk}$	$59.03{\pm}0.46^{\scriptscriptstyle ghij}$	48.83±0.30 <sup>ij</sup>	$0.088{\pm}0.004^{a}$	$5.53{\pm}0.06^{ij}$
			DI Water	30.77±0.151	35.00±0.17 <sup>k</sup>	$33.37{\pm}0.30^{\rm klm}$	$0.045{\pm}0.004^{\rm defghij}$	$2.43{\pm}0.06^{\rm klm}$
			Soybean Oil	$51.10{\pm}1.30^{\rm defg}$	$65.87{\pm}1.28^{\text{bcde}}$	$58.40{\pm}0.80^{\rm cdef}$	$0.064{\pm}0.003^{\text{bcde}}$	$7.44{\pm}0.16^{\text{cdef}}$
		11.5	Air	$43.30{\pm}0.40^{ijk}$	$57.10{\pm}0.38^{\scriptscriptstyle ghij}$	$47.90{\pm}0.45^{\rm j}$	$0.076{\pm}0.004^{\text{ab}}$	$5.34{\pm}0.09^{\rm j}$
			DI Water	30.17±1.181	35.90±2.11 <sup>k</sup>	$33.77{\pm}1.19^{\rm klm}$	$0.043{\pm}0.001^{\rm efghij}$	$2.51{\pm}0.24^{\rm klm}$
			Soybean Oil	$49.47{\pm}0.27^{\text{fgh}}$	$61.50{\pm}1.30^{\scriptscriptstyle defgh}$	$56.07{\pm}1.02^{\rm efg}$	$0.040{\pm}0.003^{\rm fghij}$	$6.97{\pm}0.20^{\rm efg}$
		12	Air	42.27±1.33 <sup>k</sup>	$55.40{\pm}0.46^{\rm ij}$	$47.73{\pm}0.72^{j}$	$0.059{\pm}0.001^{\rm bcdefg}$	5.31±0.14 <sup>i</sup>
			DI Water	30.60±0.251	32.67±0.13 <sup>k</sup>	$31.97{\pm}0.03^{\rm klm}$	$0.023{\pm}0.005^{j}$	$2.15{\pm}0.01^{\rm klm}$
			Soybean Oil	$47.60{\pm}1.46^{\rm fghi}$	$58.10{\pm}1.44^{\scriptscriptstyle ghij}$	$53.00{\pm}0.47^{\text{gh}}$	$0.040{\pm}0.001^{\rm fghij}$	$6.36{\pm}0.09^{\text{gh}}$
	Continuous	11	Air	$47.50{\pm}0.50^{\text{ghij}}$	$61.97{\pm}1.07^{\rm defg}$	$52.03{\pm}0.20^{\rm hi}$	$0.063{\pm}0.003^{\rm bcdef}$	$6.17{\pm}0.04^{\rm hi}$
			DI Water	29.23±0.571	$33.77{\pm}0.09^{k}$	$32.37{\pm}0.15^{\rm klm}$	$0.046{\pm}0.002^{{\scriptscriptstyle cdefghi}}$	$2.23{\pm}0.03^{\rm klm}$
			Soybean Oil	$50.27 \pm 1.17^{efg}$	$66.67 \pm 0.54^{\text{bcd}}$	$60.07 \pm 0.47^{\text{bcd}}$	$0.047{\pm}0.005^{\text{cdefghi}}$	$7.77{\pm}0.09^{\text{bcd}}$
		11.5	Air	$43.00{\pm}0.60^{jk}$	$54.40{\pm}1.01^{j}$	$47.87{\pm}0.98^{\rm j}$	$0.065{\pm}0.002^{\text{bcde}}$	5.33±0.20 <sup>j</sup>
			DI Water	31.07±0.971	$32.23{\pm}1.39^{k}$	$32.13{\pm}0.09^{\rm klm}$	$0.038 {\pm} 0.007^{\rm ghij}$	$2.19{\pm}0.02^{\rm klm}$
			Soybean Oil	$48.47{\pm}0.39^{\rm fgh}$	$58.77 {\pm} 1.21^{\text{ghij}}$	$54.80{\pm}1.00^{\rm fgh}$	$0.041{\pm}0.003^{\rm fghij}$	$6.72{\pm}0.20^{\rm fgh}$
		12	Air	41.63±0.52 <sup>k</sup>	$55.47{\pm}0.88^{ij}$	47.10±0.30 <sup>j</sup>	$0.069{\pm}0.005^{\text{abc}}$	$5.18{\pm}0.06^{i}$
			DI Water	28.77±0.121	32.03±0.17 <sup>k</sup>	31.00±0.06 <sup>m</sup>	$0.034{\pm}0.006^{\rm hij}$	1.96±0.01 <sup>m</sup>
			Soybean Oil	$45.37{\pm}0.81^{\scriptscriptstyle hijk}$	$56.03{\pm}0.62^{\rm hij}$	$51.90{\pm}0.35^{\text{hi}}$	$0.045{\pm}0.005^{\rm efghij}$	$6.14{\pm}0.07^{\rm hi}$

			Air	55.00±0.36 <sup>bcd</sup>	68.20±0.61 <sup>b</sup>	61.77±0.47 <sup>bc</sup>	$0.069 \pm 0.001^{abc}$	$8.11 \pm 0.09^{bc}$
		11	DI Water	32.23±0.351	36.30±0.67 <sup>k</sup>	$35.00{\pm}0.49^{\rm kl}$	$0.043{\pm}0.007^{\rm efghij}$	$2.76{\pm}0.10^{kl}$
Stati Middle —— Cont			Soybean Oil	63.27±0.24ª	$75.83{\pm}1.23^{a}$	70.53±0.55ª	$0.051{\pm}0.002^{\rm cdefghi}$	9.87±0.11ª
		11.5	Air	$51.33{\pm}0.88^{\rm defg}$	$60.23{\pm}1.08^{\text{fghi}}$	$56.33{\pm}0.78^{\rm defg}$	$0.055{\pm}0.002^{{}_{bcdefghi}}$	$7.03{\pm}0.16^{\rm defg}$
			DI Water	31.97±1.021	36.93±1.21 <sup>k</sup>	35.47±1.22 <sup>k</sup>	$0.055{\pm}0.003^{{}_{bcdefghi}}$	2.85±0.24 <sup>k</sup>
	Stationary		Soybean Oil	57.63±1.59 <sup>b</sup>	$66.77 \pm 1.94^{\text{bcd}}$	63.27±1.38 <sup>b</sup>	$0.037 {\pm} 0.005^{\rm ghij}$	$8.41{\pm}0.28^{\text{b}}$
	Stational y		Air	$50.27{\pm}1.11^{\text{efg}}$	$59.53{\pm}0.97^{\rm fghij}$	$55.37{\pm}0.85^{\rm fgh}$	$0.059{\pm}0.002^{\rm bcdefg}$	$6.83{\pm}0.17^{\rm fgh}$
		12	DI Water	30.07±0.321	$32.83{\pm}0.23^{k}$	$31.90{\pm}0.32^{\rm klm}$	$0.033{\pm}0.002^{\rm hij}$	$2.14{\pm}0.06^{\rm kim}$
			Soybean Oil	$55.03{\pm}0.95^{\scriptscriptstyle bcd}$	$64.67{\pm}1.41^{\rm bcdef}$	$61.23{\pm}0.86^{\rm bc}$	$0.042{\pm}0.003^{{\scriptscriptstyle \text{efghij}}}$	$8.01 \pm 0.17^{\text{bc}}$
	Continuous	11	Air	$56.37 {\pm} 0.82^{\rm bc}$	$67.97 {\pm} 0.33^{\rm bc}$	$62.27{\pm}0.58^{\rm bc}$	$0.068{\pm}0.009^{\text{abcd}}$	8.21±0.12 <sup>bc</sup>
			DI Water	31.03±0.351	$35.90{\pm}0.36^{k}$	$34.30{\pm}0.36^{\rm klm}$	$0.063{\pm}0.009^{\rm bcdef}$	$2.62{\pm}0.07^{\rm kim}$
			Soybean Oil	62.40±0.96ª	$74.97 \pm 1.77^{a}$	70.50±1.44ª	$0.037 {\pm} 0.003^{\rm ghij}$	9.86±0.29ª
		11.5	Air	$52.10{\pm}0.52^{\scriptscriptstyle cdef}$	$60.93{\pm}0.77^{\scriptscriptstyle efghi}$	$56.80{\pm}0.46^{\rm defg}$	$0.056{\pm}0.002^{{}_{\text{bcdefgh}}}$	$7.12{\pm}0.09^{\rm defg}$
			DI Water	$30.77 \pm 0.07^{1}$	$34.93{\pm}0.35^{k}$	$33.37{\pm}0.20^{\rm klm}$	$0.063{\pm}0.004^{\rm bcdef}$	$2.43{\pm}0.04^{\rm klm}$
			Soybean Oil	$54.40{\pm}0.49^{\scriptscriptstyle bcde}$	$66.30{\pm}0.23^{\scriptscriptstyle bcde}$	63.17±0.37 <sup>b</sup>	$0.032{\pm}0.004^{ij}$	8.39±0.07 <sup>b</sup>
		12	Air	$50.77{\pm}0.52^{\rm defg}$	$59.07{\pm}0.98^{\rm ghij}$	$55.10{\pm}0.64^{\rm fgh}$	$0.055{\pm}0.005^{{}_{bcdefghi}}$	$6.78{\pm}0.13^{\rm fgh}$
			DI Water	29.47±0.151	32.67±0.29 <sup>k</sup>	$31.57{\pm}0.33^{\text{lm}}$	$0.053{\pm}0.002^{{}_{bcdefghi}}$	$2.07{\pm}0.07^{\rm lm}$
			Soybean Oil	$50.67{\pm}0.24^{\rm defg}$	$62.40{\pm}0.68^{\text{cdefg}}$	59.40±0.76 <sup>bcde</sup>	0.033±0.001 <sup>ij</sup>	$7.64{\pm}0.15^{\text{bcde}}$

\* Values are mean  $\pm$  SD over three replicates. Within columns, means with different letters are significantly different (P < 0.05) for the interaction of layer, heating mode, electrode gap, and surrounding medium effects.
**Table 4.3** Minimum, maximum, and average temperature, heating uniformity index, and the heating rate at the top and middle layers of corn flour surrounded with air, DI water, and soybean oil under stationary heating mode for 5 min at the different surrounding medium levels (6.5, 7.1, and 8.8 cm) with an 11 cm electrode gap.

Layer	Surrounding Medium	Surrounding Medium Level (cm)	Min Temperature (°C)	Max Temperature (°C)	Average Temperature (°C)	Heating Uniformity Index	Heating Rate (°C/min)
Тор	Air	NA	43.20±1.46	59.03±0.46	48.83±0.30	$0.088 {\pm} 0.004$	5.53±0.06
	DI Water	6.5	39.20±1.55 <sup>e</sup>	$51.35{\pm}1.18^{\rm f}$	$47.60{\pm}0.90^{\rm f}$	$0.074{\pm}0.001^{b}$	5.10±0.18 <sup>e</sup>
		7.1	28.10±0.33 <sup>g</sup>	$33.15{\pm}0.37^{hi}$	$31.31{\pm}0.06^i$	$0.061{\pm}0.006^{bcd}$	$1.84{\pm}0.01^{h}$
		8.8	$30.77{\pm}0.15^{fg}$	$35.00{\pm}0.17^{hi}$	$33.37{\pm}0.30^{hi}$	$0.045{\pm}0.004^{cde}$	$2.43{\pm}0.06^{g}$
	Soybean Oil	6.5	45.25±1.67 <sup>d</sup>	56.25±0.61 <sup>e</sup>	51.00±0.08 <sup>e</sup>	$0.048{\pm}0.002^{cde}$	5.26±0.02 <sup>e</sup>
		7.1	$46.75{\pm}1.92^{d}$	$59.70{\pm}0.33^{d}$	$54.05{\pm}0.12^{d}$	$0.053{\pm}0.005^{bcde}$	$5.73{\pm}0.02^{d}$
		8.8	51.10±1.30°	$65.87 \pm 1.28^{bc}$	$58.40{\pm}0.80^{\circ}$	$0.064{\pm}0.003^{bcd}$	$7.44 \pm 0.16^{bc}$
Middle	Air	NA	55.00±0.36	68.20±0.61	61.77±0.47	$0.069{\pm}0.001$	8.11±0.09
	DI Water	6.5	$34.70{\pm}0.49^{\rm f}$	$44.65 \pm 0.53^{g}$	$41.10{\pm}0.24^{g}$	$0.099 \pm 0.009^{a}$	$3.80{\pm}0.05^{\rm f}$
		7.1	$28.15{\pm}0.29^{\rm g}$	$32.65{\pm}0.20^i$	$31.30{\pm}0.08^i$	$0.070{\pm}0.014^{bc}$	$1.84{\pm}0.02^{h}$
		8.8	$32.23{\pm}0.35^{\rm f}$	$36.30{\pm}0.67^{\rm h}$	$35.00{\pm}0.49^{\rm h}$	$0.043{\pm}0.007^{de}$	$2.76 \pm 0.10^{g}$
	Soybean Oil	6.5	$54.10 \pm 0.24^{bc}$	62.65±0.20 <sup>cd</sup>	59.95±0.61°	$0.034{\pm}0.005^{e}$	7.05±0.12°
		7.1	$55.55 \pm 1.35^{b}$	$67.65{\pm}0.61^{b}$	$63.90{\pm}0.90^{b}$	$0.040{\pm}0.005^{de}$	$7.70{\pm}0.18^{b}$
		8.8	$63.27{\pm}0.24^{a}$	75.83±1.23ª	$70.53{\pm}0.55^{a}$	$0.051{\pm}0.002^{bcde}$	9.87±0.11ª

\* Values are mean  $\pm$  SD over three replicates. Within columns, means with different letters are significantly different (P < 0.05) for the interaction of layer, surrounding medium and surrounding medium level effects.

Corn flour heated in the air could not be included in the statistical analysis due to the lack of surrounding medium-level effect. It was only shown in the table for comparison with the other treatments.

**Table 4.4** Minimum, maximum, and average temperature, heating uniformity index, and the heating rate at the top and middle layers of corn flour surrounded with air, DI water (at 8.8 cm level), and soybean oil (at 8.8 cm level) under continuous heating mode for 4, 5, and 6 min treatment with an 11 cm electrode gap.

Layer	Surrounding Medium	Heating Time (min)	Min Temperature (°C)	Max Temperature (°C)	Average Temperature (°C)	Heating Uniformity Index	Heating Rate (°C/min)
	Air	4	$44.10 \pm 0.87^{f}$	56.63±1.02 <sup>e</sup>	$48.77{\pm}0.82^{i}$	$0.057{\pm}0.001^{bcd}$	$6.89{\pm}0.20^{gh}$
		5	$47.50{\pm}0.50^{ef}$	$61.97{\pm}1.07^{d}$	$52.03{\pm}0.20^{hi}$	$0.063 {\pm} 0.003^{bc}$	$6.17{\pm}0.04^{hi}$
		6	49.23±0.22 <sup>e</sup>	$64.77 \pm 0.44^{cd}$	$55.30{\pm}0.42^{gh}$	$0.063{\pm}0.008^{bc}$	$5.68{\pm}0.07^{i}$
		4	$30.03{\pm}0.12^{g}$	$35.17{\pm}0.13^{fg}$	$33.67{\pm}0.12^{jk}$	$0.042{\pm}0.001^{bcd}$	$3.12{\pm}0.03^{jk}$
Тор	<b>DI Water</b>	5	$29.23{\pm}0.57^{g}$	$33.77{\pm}0.09^{g}$	$32.37{\pm}0.15^k$	$0.046 {\pm} 0.002^{bcd}$	$2.23{\pm}0.03^{1}$
		6	$32.23{\pm}0.32^{g}$	$38.33{\pm}0.71^{\rm f}$	$36.30{\pm}0.72^{jk}$	$0.045{\pm}0.004^{bcd}$	$2.52{\pm}0.12^{kl}$
		4	51.00±1.51°	$64.77 \pm 0.82^{cd}$	$58.80 \pm 1.01^{efg}$	$0.049 \pm 0.003^{bcd}$	$9.40{\pm}0.25^{bc}$
	Soybean Oil	5	50.27±1.17e	$66.67 \pm 0.54^{bc}$	$60.07{\pm}0.47^{ef}$	$0.047{\pm}0.005^{bcd}$	$7.77 {\pm} 0.09^{\text{ef}}$
		6	57.00±1.75°	$75.33{\pm}1.04^{a}$	$67.27 \pm 1.59^{bc}$	$0.050{\pm}0.001^{bcd}$	$7.68{\pm}0.27^{efg}$
		4	$51.63 {\pm} 0.55^{de}$	$61.00{\pm}1.11^{de}$	$56.83{\pm}0.82^{fg}$	$0.053{\pm}0.009^{bcd}$	$8.91 {\pm} 0.20^{cd}$
	Air	5	56.37±0.82°	$67.97 \pm 0.33^{bc}$	$62.27{\pm}0.58^{de}$	$0.068{\pm}0.009^{ab}$	$8.21 {\pm} 0.12^{de}$
		6	$58.47{\pm}0.78^{bc}$	$68.63 {\pm} 0.44^{bc}$	$64.30 \pm 0.12^{cd}$	$0.044{\pm}0.002^{bcd}$	$7.18{\pm}0.02^{\text{fg}}$
		4	$31.47{\pm}0.23^{g}$	$36.13{\pm}0.34^{\mathrm{fg}}$	$34.77{\pm}0.27^{jk}$	$0.051{\pm}0.004^{bcd}$	$3.39{\pm}0.07^j$
Middle	<b>DI Water</b>	5	$31.03{\pm}0.35^{g}$	$35.90{\pm}0.36^{\mathrm{fg}}$	$34.30{\pm}0.36^{jk}$	$0.063 \pm 0.009^{bc}$	$2.62{\pm}0.07^{jkl}$
		6	$31.80{\pm}0.36^{g}$	$38.87{\pm}0.83^{\rm f}$	$36.57{\pm}0.37^j$	$0.090{\pm}0.004^{a}$	$2.56{\pm}0.06^{kl}$
		4	$55.90{\pm}0.90^{cd}$	$69.87{\pm}0.26^{\text{b}}$	$66.17 \pm 0.55^{cd}$	$0.041{\pm}0.006^{cd}$	$11.24{\pm}0.14^{a}$
	Soybean Oil	5	$62.40{\pm}0.96^{ab}$	$74.97{\pm}1.77^{a}$	$70.50{\pm}1.44^{b}$	$0.037{\pm}0.003^{cd}$	$9.86{\pm}0.29^{b}$
		6	63.80±0.50ª	79.37±1.35 <sup>a</sup>	$74.70{\pm}1.40^{a}$	$0.031{\pm}0.005^{d}$	$8.92{\pm}0.23^{cd}$

\* Values are mean  $\pm$  SD over three replicates. Within columns, means with different letters are significantly different (P < 0.05) for the interaction of layer, heating mode, electrode gap and surrounding medium effects.

## Figures



**Figure 4.1** Experimental set-up drawing (a) side view; (b) top view.



Figure 4.2 Drawing of RF heating system with a conveying unit.



**Figure 4.3** Relationship between the rise in standard deviation and the rise in the mean of corn flour temperatures at the top layer surrounded with air, DI water (at 8.8 cm level), and soybean oil (at 8.8 cm level) under continuous heating mode for 4, 5 and 6 min treatment with an 11 cm electrode gap.



**Figure 4.4** Temperature distribution of corn flour surrounded with air, DI water (at 8.8 cm level), and soybean oil (at 8.8 cm level) under stationary and continuous RF heating with an 11 cm electrode gap and 5 min treatment time.



**Figure 4.5** Temperature distribution of corn flour surrounded with DI water and soybean oil with different levels (6.5, 7.1, and 8.8 cm) under stationary RF heating with an 11 cm electrode gap and 5 min treatment time.



**Figure 4.6** Temperature distribution of corn flour surrounded with soybean oil (at 8.8 cm level) under stationary and continuous RF heating with 11, 11.5, and 12 cm electrode gaps and 5 min treatment time.



**Figure 4.7** Temperature distribution of corn flour surrounded with soybean oil (at 8.8 cm level) under continuous RF heating with an 11 cm electrode gap and 4, 5, and 6 min treatment time.

### CHAPTER 5

# RADIO FREQUENCY ASSISTED THERMAL PROCESSING FOR PASTEURIZATION OF PACKAGED WHOLE MILK POWDER SURROUNDED BY

 $OIL^1$ 

<sup>&</sup>lt;sup>1</sup>Dag D., Singh R.K., Kong F. To be submitted to Food Control.

#### Abstract

The presence of Salmonella spp. in milk powders has caused several outbreaks resulting in serious health problems and even deaths as well as significant economic loss due to recalls. The objective of this study was to investigate the effect of RF heating on reduction of *Salmonella* spp. in packaged whole milk powder at a water activity of 0.35 surrounded by oil medium. The thermal destruction kinetics (decimal reduction time; Dvalue) of Salmonella spp. and Enterococcus faecium NRRL B-2354 (a potential surrogate of *Salmonella* spp. for low moisture foods) in whole milk powder were determined at 75, 80, and 85 °C using a thermal-death-time (TDT) cells, and the z-values (the temperature increase required to obtain a decimal reduction of the D-value) were calculated. The efficacy of the RF treatment was validated by heating the milk powder immersed in soybean oil by RF heating to 75 °C and holding it in a conventional oven for different time periods. A 5 log CFU/g reduction of Salmonella spp. in whole milk powder was achieved after RF heating followed by 2 h holding in the oven. The water activity of whole milk powder significantly increased while its moisture content was significantly decreased after RF heating followed by 2 h holding. A significant color change was observed for the treatment in which a 5 log CFU/g reduction of Salmonella spp. was achieved. The results indicate that Radio frequency heating combined with oil immersion can be used as a rapid and volumetric method for milk powders to achieve the target temperature but a holding time of a few hours will be needed for a post-process lethality treatment.

#### Introduction

Milk powder is generally considered shelf-stable due to low water activity (<0.70) at which most food pathogens including *Salmonella* spp. are not capable of multiplying. Even though the low water activity of milk powders can suppress bacterial growth and multiplication, Salmonella spp. can still survive for an extended period of time and become more heat resistant due to their high desiccation tolerance. Historically, several foodborne outbreaks have been reported due to the consumption of Salmonella-contaminated milk powders, such as dry milk powder and skim milk powder (El-Gazzar & Marth, 1992), powdered milk products (CDC, 1993), powdered infant formula milk (Angulo, Cahill, Wachsmuth, Costarrica, & Embarek, 2008; Brouard et al., 2007; Park et al., 2004; Rodríguez-Urrego et al., 2010; Rowe et al., 1987) and infant milk products (Jourdan-da Silva et al., 2018). Although raw milk used in milk powder production is subject to multiple thermal treatments such as pasteurization, concentration, and spray drying; microbial contamination from the environment, workers and equipment is still a problem in milk powder plants. Cross-contamination might occur due to poor sanitation practices, handling, distribution, and storage conditions (Podolak, Enache, Stone, Black, & Elliott, 2010; Wei et al., 2020). Also, if the pasteurization of raw milk is inadequate, Salmonella spp. in milk may survive in spray-drying conditions and will be retained in the final product.

Milk powders are frequently used as an ingredient in ready-to-eat foods such as confectionery products, drink mixes, seasoning, nutritional bars, and dry blend infant formula which are not subjected to further thermal treatment. Therefore, the consumption of those ready-to-eat foods formulated with *Salmonella*-contaminated milk powders might potentially cause a public health risk. Currently, there is no pasteurization step after spraydrying in the production of milk powders. The number of outbreaks and recalls related to the consumption of *Salmonella*-contaminated milk powders indicates that effective pasteurization of the milk powder is in need to prevent possible health problems which might be associated with the contaminated milk powders.

Radio frequency (RF) heating is promising pasteurization and sterilization treatment with the advantages of rapid and volumetric heating. During RF heating, the food sample is heated in an RF oven to reach the target temperature. The pasteurization/sterilization standards in terms of the requirement of lethality or log CFU/g reduction of microbial are mostly achieved by combining RF heating with other technologies such as hot air circulation and conventional ovens. In RF heating, dipole molecules such as water and ions such as salt and other minerals in food contribute to heat generation within the sample. A food sample is placed between two electrodes where an alternating field is applied. Heat is generated within the food due to frictions created by (1) dipole rotation (as a result of the continuous oscillation of dipole molecules along the changing electric field), and (2) ionic depolarization (as a result of the continuous movement of ions in the food toward the oppositely charged regions of the electric field) (Marra, Zhang, & Lyng, 2009; Michael et al., 2014; Piyasena, Dussault, Koutchma, Ramaswamy, & Awuah, 2003).

Unlike conventional heating, fewer changes in quality and sensory attributes can be achieved in RF heating due to the shorter heating times. Compared to conventional hot air and steam heating, RF heating results in a more uniform temperature distribution since it heats the sample volumetrically and independently from conduction. However, RF heating uniformity needs to be further improved to extend its application to pasteurization and sterilization. Temperature differences within the sample might affect the microbial inactivation significantly. For this reason, several techniques were implemented in the RF studies to improve the heating uniformity including surrounding the food sample with materials with comparable dielectric properties (Jiao, Tang, & Wang, 2014; Ozturk, Kong, Singh, Kuzy, & Li, 2017; Wang, Zhang, Gao, Tang, & Wang, 2014), intermittent mixing/stirring (Gao, Tang, Wang, Powers, & Wang, 2010; S. Wang et al., 2006), and adjusting electrode bending/configuration and gap (Tiwari, Wang, Tang, & Birla, 2011). In our previous study, it was shown that an improved RF heating uniformity with a higher heating rate could be achieved when corn flour was immersed in soybean oil. For commercial food applications, it is necessary to validate the effect of oil immersion in RF pasteurization of food powder by conducting microbial inactivation analysis.

The overall goal of this study was to evaluate the thermal inactivation of *Salmonella* spp. and *Enterococcus faecium* NRRL B-2354 (*E. faecium*) in whole milk powder immersed in oil. The specific objectives were to (1) evaluate *E. faecium* as a surrogate for *Salmonella* by conducting isothermal treatments and comparing the thermal resistance of the two bacteria; (2) validate the RF inactivation of *Salmonella* and *E. faecium* in whole milk powder with soybean oil as a surrounding medium; (3) investigate the influence of RF heating on the quality of whole milk powder.

#### **Materials & Methods**

#### <u>Materials</u>

Whole milk powder (Hoosier Hill Farm, Fort Wayne, Indiana, USA) and soybean oil (Great Value, Walmart) were purchased from a local supermarket and held at ambient temperature ( $23 \pm 2^{\circ}$ C). Peptone water, tryptic soy broth (TSB), trypticase soy agar (TSA), and yeast extract were bought from Becton, Dickinson, and Company (Sparks, MD, USA). Ammonium iron citrate and sodium thiosulfate were obtained from Sigma-Aldrich (St. Louis, MO, USA) and esculin hydrate was purchased from Acros Organics, (Morris, NJ). <u>Milk Powder Sample</u>

Whole milk powder was held in a Hotpack 435315 humidity chamber (SP Industries, Inc., Warminster, PA, USA) preset at 35% relative humidity at 40 °C. Moisture content and water activity at 40 °C (a<sub>w</sub>,40°C) were determined by a halogen moisture analyzer (HR73, Mettler Toledo Laboratory, and Weighing Technologies, Greifensee, Switzerland) and a dew point water activity meter Aqualab 4TE (Decagon, Pullman, WA, USA) respectively. The background microflora was enumerated from three randomly collected 1 g milk powder samples diluted in 0.1 % (w/v) peptone water, stomached (Seward Stomacher, 400 Lab Blender, Norfolk, UK) at 260 rpm for 5 min, plated on TSA, and incubated for 24 h at 37 °C.

#### **Bacterial Strains and Inoculation**

A cocktail of four different strains of *Salmonella enterica* (*Salmonella* Agona, Enteritidis Montevideo, and Tennessee) associated with different low moisture foodborne outbreaks were selected for milk powder inoculation. *E. faecium* was selected as the nonpathogenic surrogate for the validations. *E. faecium* and *Salmonella* Agona, Enteritidis, Montevideo, and Tennessee were obtained from Dr. Mark Harrison's laboratory at the University of Georgia and kept at -80 °C in TSB supplemented with 20% (v/v) glycerol until used.

#### Inoculum Preparation and Inoculation

For each strain, 1 ml of the frozen stock was transferred into 9 ml of TSB supplemented with 0.6% (w/v) yeast extract (TSBYE) and incubated for 24 h at 37 °C. E. faecium and Salmonella cocktail were subjected to two consecutive transfers (at 37 °C for 24 h) in 9 ml of TSBYE. For isolation, the culture was streaked onto TSA supplemented with 0.6% (w/v) yeast extract (TSAYE) plates using a 10 µL sterile loop, then plates were incubated upside down at 37 °C for 24 h. After incubation, a loopful (~10 µL) of one isolated colony was taken and transferred into TSBYE and then incubated at 37 °C for 24 h. Then, 0.5 ml was evenly spread on each of the 4 TSAYE plates and incubated at 37 °C for 24. The bacterial lawn on each TSAYE was harvested by adding 5 and 3 mL of 0.1% peptone water to the plate consecutively and scraping the agar surface gently with a sterile T-shaped plastic spreader. The slurry was then transferred into a 50 ml sterile conical centrifuge tube and centrifuged for 10 min at 1800x g (Eppendorf Centrifuge 5810, Eppendorf AG, Hamburg, Germany) to remove the media from bacterial suspension. The supernatant was discarded and the pellet was re-suspended in 10 ml 0.1% peptone water and centrifuged again. The final pellets were resuspended in 2 ml 0.1% peptone water, corresponding to approximately  $10^8$  to  $10^9$  CFU/ml pellet suspension.

The milk powder sample  $(10 \pm 0.1 \text{ g})$  which was pre-equilibrated in the environmental chamber to 0.35 a<sub>w</sub> at 40 °C was weighed and placed into a sterile Whirl-Pak bag in a biological safety cabinet. The concentrated pellet (1 ml) was hand-mixed into 10 g milk powder in a sterile stomacher bag until the pellet was evenly mixed. After hand-mixing, inoculated 10 g powder sample was used to further inoculate 90 g milk powder, which was then hand-mixed for another 2 min and stomached at 260 rpm for 5 min. Then,

five of 1 g samples were randomly selected and enumerated on TSA plates to confirm the uniformity of inoculum distribution. The inoculated milk powder (<2 mm thickness) was transferred onto a sanitized aluminum tray and placed into an environmental chamber preset at 35% relative humidity at 40 °C. The equilibration of milk powder to the water activity of 0.35 was achieved in 4 days. This equilibration period also allowed the bacteria to acclimatize and adapt to the low moisture environment.

#### Isothermal Heating

Thermal resistances of *E. faecium* and *Salmonella* cocktail in milk powder were determined at 3 different temperatures (75, 80, and 85 °C) using aluminum thermal-death-time (TDT) cells purchased from Washington State University, Pullman, WA (Chung, Birla, & Tang, 2008). The cells were filled with 0.45 g inoculated milk powder and subjected to isothermal heat treatment in a water bath (Model: 18802A, AquaBath, Barnstead Lab-Line, Dubuque, IA, USA) maintained at 75, 80, and 85 °C. The come-up times (CUT, the time needed for the sample to reach the target process temperature) for each temperature were measured by using cells filled with non-inoculated milk powder with a T-type thermocouple inserted in the center of the cell. Each isothermal treatment was conducted at the same time intervals starting after the CUT was achieved. Isothermal treated cells were removed from the water bath and immersed immediately in an ice-water bath for 90 s to stop thermal inactivation in the sample.

#### Enumeration

Inoculated sample from the TDT cells (from isothermal experiments) or thermally sealed packages (from RF heating experiments) was then transferred to a sterile Whirl-Pak bag, ten-fold serially diluted, and spread plated onto eTSA (TSAYE supplemented with 0.05% (w/v) ammonium iron citrate and 0.025% (w/v) esculin hydrate) for *E. faecium* and mTSA (TSAYE supplemented with 0.05% (w/v) ammonium iron citrate and 0.03% (w/v) sodium thiosulfate) for *Salmonella* cocktail and incubated for 24 h at 37 °C. Black colonies were enumerated as *E. faecium* on eTSA while colonies with a black center were enumerated as *Salmonella* spp on mTSA. The total bacterial reduction was obtained by subtracting the number of survivors to the treated sample (log N) from the untreated sample (N<sub>0</sub>). The holding time after RF heating which gave more than 5 log CFU/g reduction of *Salmonella* spp. was considered as the optimal pasteurization treatment and was selected for quality analysis of uninoculated samples.

#### Modeling of Microbial Reduction Kinetics in Isothermal Inactivation Study

The thermal inactivation kinetics of *E*. *faecium* and *Salmonella* spp. in whole milk powder were modeled with the log-linear model to obtain the D-value as follows:

$$\log\left(\frac{N}{N_0}\right) = -\frac{t}{D_T}$$
(5.1)

where N and N<sub>0</sub> are the populations at time t and 0 (CFU/g), respectively, t is the isothermal heating time (min), and  $D_T$  (min) is the time required to reduce the population by 10-fold at a specified temperature, T (°C).

Z-value which is defined as the temperature change (°C) needed to achieve a 1-log change in D (min) was calculated by plotting logarithmic D-values of *E. faecium* and *Salmonella* at 75, 80, and 85 °C against temperature which is called as TDT curve. The z-value was determined by taking the reciprocal of the slope of the TDT curve for each microorganism as follows:

$$z = \frac{T_2 - T_1}{\log D_{T_1} - \log D_{T_2}}$$
(5.2)

where  $D_{T1}$  and  $D_{T2}$  are the D-values at temperatures  $T_1$  and  $T_2$ , respectively.

#### <u>RF Heating of Whole Milk Powder</u>

A pilot-scale 6 kW, 27.12 MHz free-running oscillator RF machine (COMBI 6-S, Strayfield International, Wokingham, UK) with parallel-plate electrodes (L= 83 cm and W= 40 cm) was used as the source of RF energy in this study.

Whole milk powder (300 g in total) at  $30.0\pm0.4$  °C was placed in a Ziploc plastic food storage container with 11.43 cm (OD)  $\times 8.25 \text{ cm}$  (H) dimension given in the product specification provided by the company. The inner dimensions of the food storage container were measured as 10.5 cm (ID)  $\times 7.5 \text{ cm}$  (H). Both the food container and its lid were made of polypropylene. The plastic container having milk powder was then placed into a cylindrical polypropylene container with 22.5 cm OD and 21.5 cm ID at the top, 18 cm OD and 17 cm ID at the bottom, and 11.2 cm outer and 11 cm inner height. The space between two plastic containers was filled with soybean oil at approximately 30 °C. For another experimental setup, the space between two containers was left empty to compare the effect of air and soybean oil as a surrounding media. Two polystyrene tubes (one at the top layer and one at the middle layer) with 1 cm OD and 0.7 cm ID were inserted through predrilled holes to lift the sample container so that soybean oil has contact with the bottom of the plastic container. From these two openings, two fiber optic temperature probes (one at the top and one at the middle layer) were inserted into the sample container to record the temperature profile during RF heating. In the subsequent microbial inactivation experiments, the inoculated sample package was placed at the same location without a fiber optic sensor to estimate the inactivation of RF heating. The whole system was positioned at the center of the bottom electrode and heated for the predetermined time needed for the sample's cold spot to reach 75 °C.

For microbial validation experiments, the inoculated pack method described in (Liu et al., 2018) was used in this study. Briefly, the inoculated package was prepared by transferring  $10 \pm 0.1$  g whole milk powder into a sterile Whirl-Pak sample bag (75 mm x 125 mm x 0.057 mm) in a biosafety cabinet. After sealing the inoculated package using an impulse sealer (Model: PFS 300), it was placed at the pre-determined cold spot in a container filled with 290  $\pm$  0.1 g of milk powder so that the total mass was kept as 300 g. The sample heated in the RF oven was then held at 75 °C for 90 and 120 min in an incubator (ISOTEMP, Model: 516D, Fisher Scientific, Waltham, MA). As a final step, the whole milk powder was cooled to room temperature by placing the sample container into the ice bath to simulate the cooling process at the big scale. Enumeration was performed by randomly sampling 1 g of treated inoculated whole milk powder and stomaching with 9 mL of 0.1% peptone water, followed by plating 10-fold serial dilutions as previously described. Untreated milk powder conditioned in the environmental chamber was used as a control.

#### **Quality Analysis**

#### Water Activity and Moisture Content

For quality analysis, the water activity of milk powders at 25°C (a<sub>w</sub>,25°C) was determined by a dew point water activity meter Aqualab 4TE (Decagon, Pullman, WA, USA). Half of the aqua lab dishes were filled by sample and closed with its lid until measurement. The water activity meter was initially cleaned and calibrated according to the instructions written in the manual. The instrument was initially tested by the standards (distilled water and 8.57 M LiCl) at the range of water activity of the sample. For moisture content measurement, approximately 2 g of whole milk powder was placed on an

aluminum plate. The moisture content (%) calculated by a halogen moisture analyzer (HR73, Mettler Toledo Laboratory, and Weighing Technologies, Greifensee, Switzerland) was recorded in triplicates for each sample. Both the water activity meter and moisture analyzer were warmed up for at least 30 min before the measurements.

#### Color Measurement

Konica Minolta chromameter (Model CR-410 HS, Minolta, Tokyo, Japan) with illuminant C, 50 mm aperture size, wide-area illumination/0° viewing angle, 2° standard observer was used to measure the color parameters of whole milk powder. Initially, the calibration was done against a white calibration plate provided by the company.

Milk powders were placed in a petri dish to attain a flat surface for the measurements. Four random locations on the external surface of the powder were measured by the colorimeter. The color values were expressed as L\* (lightness: 100, white; 0, black), a\* (+, red; -, green), and b\* (+, yellow; -, blue) respectively. The total color difference ( $\Delta E$ ) was calculated by using the following equation where L<sub>0</sub>, a<sub>0</sub>, and b<sub>0</sub> represent reference values:

$$\Delta E = \sqrt{(a^* - a_0)^2 + (b^* - b_0)^2 + (L^* - L_0)^2}$$
(5.3)

To evaluate the effect of the RF heating on whole milk powder color, the total color difference ( $\Delta E$ ), chroma, and hue angle were calculated by equations (3), (4), and (5-6) respectively. (Sant'Anna, Gurak, Ferreira Marczak, & Tessaro, 2013).

Chroma = 
$$\sqrt{(a^*)^2 + (b^*)^2}$$
 (5.4)  
Hue angle = arctan(b\*/a\*) for quadrant I (+a\*,+b\*)

Hue angle =  $180 + \arctan(b^*/a^*)$  for quadrant II (-a\*,+b\*) and quadrant III (-a\*,-b\*)

(5.5)

Hue angle =  $360 + \arctan(b^*/a^*)$  for quadrant IV (+a\*,-b\*) (5.6)

#### Data Processing and Analysis

All measurements were conducted in triplicate and all results were reported as the mean value ± standard deviation. Excel (Microsoft Office, Redmond, WA, USA) was used in data processing and analysis. Analysis of variance and Tukey's pairwise comparison test with a significant level of 0.05 were performed with the JMP Pro 15 software package (SAS Institute, Cary, NC).

#### **Results & Discussion**

#### Heating Profiles

Fig. 5.1 shows the heating profiles of whole milk powder at the geometric and top center while heating in air and soybean oil. During RF treatment, the corners and edges absorb more electromagnetic energy due to the fringe effect. The sample at these regions are further overheated and become hot spots since the sample dielectric properties increase with an increase in temperature. The air in the RF cavity is not heated due to its dielectric properties; therefore, the sample loses heat to the surrounding air. This heat loss at the sample surfaces resulted in the top center of the sample being the cold spot in the present study as well as the several previous studies associated with the RF heating of food powders (Liu et al., 2018; Wei et al., 2018; Wei, Lau, Stratton, Irmak, & Subbiah, 2019).

Heating the sample container in a surrounding medium having comparable dielectric properties with the sample might provide an improved heating uniformity. Additionally, the liquid surrounding medium can be used to transport the sample packages

in the RF cavity. Previously, the heating uniformity and heating rate were extensively studied for corn flour heated in air and soybean oil (Dag, Singh, & Kong, 2021). It was concluded that a higher heating rate with the improved heating uniformity (supported by lower heating uniformity index and more even temperature distribution via thermal images) when the samples were heated in soybean oil compared to those heated in air.

As illustrated in Fig. 5.1, the heating rates varied with a heating type (RF vs. conventional heating) and surrounding media used in RF heating (air and soybean oil). Among the treatments, heating packaged whole milk powder in the RF oven when surrounded with soybean oil resulted in the fastest RF heating rate, thus the shortest comeup time. The top center of the milk powder reached 75 °C in approximately 21 and 17 min when heated in air and soybean oil, respectively. By dividing the temperature change (with the initial sample temperature of 30 °C) with heating time, the heating rates were calculated as 2.12 and 2.65 °C/min for samples heated in air and soybean oil, respectively. It can be concluded that the heating rate increased by 25% by placing packaged milk powder in soybean oil.

The temperatures at the geometric center were recorded as 87.53 and 87.66 °C for packaged milk powders heated in air and soybean oil, respectively, demonstrating that the presence of the soybean oil in the RF cavity did not influence the heating rate at the location where the sample was not in contact with oil. In addition to the heat loss at the top, the higher temperature at the geometric center might be also associated with the density dependence of the dielectric loss factor. The sample at the lower locations had a higher dielectric loss factor due to higher density as a result of compression by the sample above. The sample portions with a higher dielectric loss factor (meaning higher conversion of

electromagnetic energy to heat) would result in higher temperatures at those regions (Liu et al., 2018; Nelson, 1984). Although higher temperature at the geometric center is favorable in terms of microbial inactivation, it might cause a quality deterioration in milk powder. Continuous mixing during heating or decreasing the sample height might help to reduce the temperature differences throughout the sample.

The same amount of whole milk powder in the same designed package used in RF heating was also heated in the conventional oven for comparison. The come-up time for the whole milk powder container (in the air only) to reach 75 °C was recorded as 220 min. The come-up time was considerably reduced (90.5 %) when milk powder was heated by RF energy. In conventional heating, the fluctuations at the top layer due to the heat loss on the sample surface were observed during heating (Fig. 5.1.b). The temperature difference between the top (76.7 °C) and middle layer (75 °C) was less than 2 °C due to the conduction allowed by a longer heating time. The long heating treatment might cause quality changes in milk powders. Incorporation of a hot air circulation system into the RF oven may alleviate the problem as the cold top layer can be heated by hot air during RF heating decreasing the temperature difference between the top and middle layers

# The Thermal Resistance of *Enterococcus faecium* NRRL B-2354 and *Salmonella* spp. in Whole Milk Powder

Background microflora was detected as  $<10^2$  CFU/g which is below the standard acceptable range ( $<10^4$  CFU/g) of aerobic bacteria in whole milk powder (Michael et al., 2014). The population of *E. faecium* and *Salmonella* spp. in the inoculated sample was considerably higher than the background microflora; thus, it should have minor interference with target microorganism enumeration (Liu et al., 2018).

The validation of a potential surrogate microorganism for *Salmonella* spp. is crucial since there is a limitation of direct use of *Salmonella* spp. in the food processing plants due to its pathogenicity and possible cross-contamination to the production lines. *Enterococcus faecium* NRRL B-2354 which is a nonpathogenic microorganism with a high thermal resistance was identified and widely used as a surrogate for multiple *Salmonella* spp. in thermal processing of food powders including whole and nonfat milk powder (Wei, Agarwal, & Subbiah, 2021), wheat flour (Liu et al., 2018), corn flour (Ozturk et al., 2019), ground black pepper (Wei et al., 2019). Inactivation kinetics of *E. faecium* and *Salmonella* spp. in whole milk powder at a<sub>w</sub>,40°C = 0.35 were shown in Fig. 5.2. Survival data of both microorganisms fit well to the log-linear model with R<sup>2</sup>>0.95.

The log-linear model was further used to describe thermal resistances of both *E. faecium* and *Salmonella* spp. in whole milk powder at 75, 80, and 85 °C and plot the z-values (Fig 5.2 and 5.3). The D-values for *E. faecium* were 16.81, 12.03, and 5.61 and 11.36, 6.44, and 3.26 min at 75, 80, and 85 °C, respectively. These D-values of *Salmonella* spp. inoculated whole milk powder were comparable to values reported in the literature. In the study conducted by (Wei et al., 2020), D-values of whole and nonfat milk powder at 3 different a<sub>w</sub> (0.10, 020, and 0.30) were calculated for *Salmonella* spp. (cocktail containing *Salmonella* enterica subsp. enterica serovar Agona 447967, *Salmonella* Montevideo 488275, and *Salmonella* Mbandaka 698538, *Salmonella* Reading 180418 and *Salmonella* Tennessee K4643). Since a<sub>w</sub> has a great influence on the thermal resistance of microorganisms, D-values at the closest a<sub>w</sub> (0.30) to that used in this study (0.35) were compared. Wei et al., (2020) reported the D-values of *Salmonella* spp. inoculated whole milk powder at 0.30 a<sub>w</sub> as 16.97, 8.11, and 3.56 at 75, 80, 85 °C, respectively. The z-values

of *E. faecium* and *Salmonella* spp. in whole milk powder were 20.9 and 18.5 °C, respectively. The z-value of *Salmonella* spp. inoculated whole milk powder at 0.30 a<sub>w</sub> was previously reported as 14.75 °C. The difference in D and z-values in the studies calculated for the same food sample might be associated with the composition, water activity, *Salmonella* spp. used, inoculation type, inoculation protocols, isothermal treatments, etc. Inactivation of *Enterococcus faecium* NRRL B-2354 and *Salmonella* spp. by RF Heating

The inoculated pack method introduced by Liu et al., 2018 was followed in this study. Previously, Liu et al., 2018 verified the inoculated pack method with no significant difference (P > 0.05) in the temperatures recorded at the central points; 1) without a pack, 2) with a pack, and 3) on the pack surface. The larger package size with less than 10 mm thickness was preferred in the present study to cover a larger area on the container surface. To observe the effect of RF heating on microbial inactivation, E. faecium, and Salmonella spp. inoculated milk powders were cooled down to room temperature in an ice bath immediately after RF heating to 75 °C. Different from the previous studies, the whole container having 290 g uninoculated and 10 g inoculated sample was cooled down to room temperature gradually (Liu et al., 2018; Ozturk et al., 2019; Wei et al., 2018; Xu et al., 2018). Although rapid cooling by taking the inoculated sample bag into the ice bath after heating was preferred in the previous studies to calculate the inactivation provided by RF heating, the bacteria can be further damaged due to the thermal shock provided by fast cooling which might affect the overall bacterial population reduction results. In this study, a gradual cooling process was chosen to mimic the case that might happen in industrial applications. In a real scenario, the sublethally injured microorganisms can recover or repair themselves during the cooling step.

Table 5.1 shows the bacterial population reduction in *E. faecium* and *Salmonella* spp. inoculated milk powders: 1) without holding after CUT, 2) 90 min holding, and 3) 120 min holding at 75 °C in the isothermal oven. The temperature-rise period of RF heating resulted in  $0.16\pm0.12$  and  $0.79\pm0.0 \log$  CFU/g reductions of *E. faecium* and *Salmonella* spp. with initial inoculated populations of 8-9 log CFU/g after 4 days incubation at 35% relative humidity at 40 °C. The lethality achieved during the come-up time was considered as the start point for holding times (t=0) of 90 and 120 min.

According to D-values of *E. faecium* (16.81 min) and *Salmonella* spp. (11.36 min) at 75 °C, 5 log CFU/g reduction was expected after holding the sample at 75 °C for 84.05 and 56.8 min respectively. However, a longer holding time at 75 °C was required to achieve the desired inactivation level in milk powder. This might be explained by an increase in the thermal resistances (D-values) of *E. faecium* and *Salmonella* spp. during heating due to the decreased  $a_w$  as a result of moisture loss (Xu et al., 2019). Another reason could be the reductions were calculated from the sample placed on the least heated area. Higher lethality would be achieved from the sample located in different locations/layers in the container. For future work, it is highly recommended to evaluate the microbial inactivation in both cold and hot spots.

More than 5 log CFU/g reduction of *Salmonella* spp.  $(5.19\pm0.11)$  was achieved after holding milk powder at 75°C for 120 min while only  $1.99\pm0.13$  log CFU/g reduction of *E. faecium* was reported for the milk powder heated under the same conditions. The considerable difference between the bacterial population reduction in *E. faecium* and *Salmonella* spp. is associated with their thermal resistances. Although *E. faecium* was extensively studied and proposed as a potential surrogate for *Salmonella* inoculated food powders (Ozturk et al., 2019; Rachon, Peñaloza, & Gibbs, 2016; Wei et al., 2021, 2019; Xu et al., 2018), an alternative surrogate with a comparable D-value with the target pathogen is still in need to prevent overheating of sample causing quality losses.

#### Quality Analysis

#### Water Activity and Moisture Content

The a<sub>w</sub> of whole milk powder at different conditions is shown in Fig. 5.4. Due to the temperature-humidity range of the environmental chamber,  $a_w$  of 0.35 could be only reached at 40 °C; thus, all a<sub>w</sub> measurements were conducted at 40 °C for comparison. The initial a<sub>w</sub> of milk powder was 0.32±0.001 at 40 °C while the sample water activity was recorded as 0.34±0.001 after 4 days of equilibration at 35% relative humidity and 40 °C. The  $a_w$  of whole milk powder increased significantly to 0.36±0.007, 0.48±0.005 and 0.50±0.009 after 1) heating in RF oven to 75 °C, 2) heating in RF oven to 75 °C followed by 2 h holding at 75 °C in the conventional oven and 3) 210 min heating in the conventional oven to reach 75 °C, respectively. The increase in  $a_w$  of whole milk powder with heating was reported in our previous study (Dag, Singh, & Kong, 2019) as well as in the study conducted by (Baechler, Clerc, Ulrich, & Benet, 2005). The reason for an increase in the aw of whole milk powder with heating could be associated with the release of water from the amorphous lactose during crystallization (Vuataz, 2002). In the study conducted by Baechler et al., (2005), the effect of heating whole milk powder at  $a_w$  of 0.28 and 0.35 was studied and it was concluded that water released upon lactose crystallization remains in the matrix (since the powder was sealed during heating) and caused an increase in the overall aw of the sample. The milk powders become sticky before crystallization temperature. The observations of clumps in heated whole milk powder in this study as well as our previous study supports that crystallization could be a possible explanation for an increase in a<sub>w</sub>.

The moisture contents of the whole milk powder before and after RF and conventional heating are illustrated in Fig. 5.5. It should be noted that there was no significant difference in the moisture content of whole milk powder before and after conventional heating while there was a significant decrease with those heated in the RF oven (Fig. 5.5). The decrease in the moisture content of the whole milk powder after RF heating can be explained by the moisture migration to the bottom and edges during heating. A similar trend in moisture content change was previously reported for RF heated low moisture foods including non-fat milk powder (Dag et al., 2019), corn flour (Ozturk et al., 2017), powdered red and black pepper spices (Jeong & Kang, 2014) and black peppercorn (Wei et al., 2018). The relation between the moisture content and a<sub>w</sub> can be explained by whole milk powder moisture sorption isotherms indicating the increase in the a<sub>w</sub> with increasing temperature at the same moisture level (Langová & Štencl, 2014).

#### <u>Color</u>

Color is one of the most important quality attributes determining the acceptance of milk powder by consumers. Table 5.2 shows the effects of RF (with and without holding) and conventional heating on the color parameters (L\*, a\*, b\*,  $\Delta E$ , chroma, and hue) of whole milk powder. The results indicated that heating whole milk powder to 75 °C in RF oven had no significant effect (p>0.05) on brightness (L\* value), yellowness (b\* value) and chroma while had a significant effect on redness (a\* value) and hue compared to the non-treated samples. When the samples heated in RF and conventional to 75 °C (without holding), the sample treated in the conventional oven had a significant effect (p>0.05) on

color attributes. Holding the sample in a conventional oven for 2 h after RF heating (required to achieve a 5 log CFU/g reduction in Salmonella spp.) had a significant effect on all color parameters.  $\Delta E$  (total color change from the control sample) of whole milk powder was 2.58, 10.89 and 19.25 after 1) 1) heating in RF oven to 75 °C, 2) heating in RF oven to 75 °C followed by 2 h holding at 75 °C in the conventional oven and 3) 210 min heating in the conventional oven to reach 75 °C, respectively. Color differences in samples can be classified as very distinct ( $\Delta E > 3$ ), distinct (1.5< $\Delta E < 3$ ), and small difference  $(1.5 \le \Delta E)$  (Adekunte, Tiwari, Cullen, Scannell, & O'Donnell, 2010). Thus, holding the sample in a conventional oven after RF heating has caused a very distinct color change in whole milk powder. Chroma showing the strength and degree of saturation of the color increased significantly (p>0.05) when the milk powders were held in the conventional heating after RF treatment. The hue angle value represents the color type and in general, an angle of 0 or 360° represents a red hue, while angles of 90, 180, and 270° represent yellow, green, and blue hues, respectively (Karaaslan & Tuncer, 2008). Unheated and RF heated (with and without holding) milk powders showed more greenness while conventionally heated milk powder showed more redness. Heating whole milk powder by RF energy considerably shortened the heating time required for heating the milk powder to 75°C; thus, the color change in whole milk powder was reduced significantly.

#### Conclusion

RF heating of packaged whole milk powder immersed in soybean oil increased the heating rate by 25% compared to the sample surrounded by air. *E. faecium* can be considered as a surrogate of *Salmonella* spp. to validate RF heating for pasteurization of whole milk powder due to its higher thermal resistances than *Salmonella* spp. Heating the

cold spot (top layer) of the whole milk powder by RF energy to 75 °C followed by holding it in the oven for 2 h was shown to be an effective lethality step achieving >5 log CFU/g reduction of *Salmonella* spp. The RF treatment giving >5 log CFU/g reduction of *Salmonella* spp. resulted in increased a<sub>w</sub>, decreased moisture content, and remarkable color change in whole milk powder. Therefore, further studies are in need to investigate the optimum conditions (treatment type, target temperature, sample a<sub>w</sub>, and different RF design), to achieve a safe food product while maintaining the food quality of whole milk powder. In addition to holding the sample in the conventional oven after RF heating, other technologies in combination with RF heating should be studied to achieve high-quality shelf-stable food powders.

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## Tables

**Table 5.1.** Thermal inactivation of *Enterococcus Faecium* NRRL B-2354 and *Salmonella* spp. in whole milk powder after RF heating to 75 C and holding for different times in the conventional oven.

Ractoria Typa	Surrounding Modio	Holding Time	<b>Bacterial Population</b>	
Dacteria Type	Surrounding Media	(min)	Reduction (log)	
	Air	0	$0.11 \pm 0.05$	
Enterococcus Faecium	Soybean Oil	0	0.16±0.12	
NRRL B-2354		90	1.10±0.05	
		120	1.99±0.13	
	Air	0	0.81±0.10	
Salmonella	Soybean Oil	0	0.79±0.01	
Saimonella spp.		90	4.31±0.04	
		120	5.19±0.11	

**Table 5.2.** Color values of whole milk powder with a water activity of 0.35 at 40 °C; (A) Whole milk powder just after opening, (B) After holding 4 days in the environmental chamber, (C) After RF heating to 75 °C without holding, (D) After RF heating to 75 °C followed by 120 min holding in a conventional oven, (E) After heating in the conventional oven to 75 °C without holding.

	L*	a*	b*	ΔΕ	Chroma	Hue
A	101.68±0.81 <sup>a</sup>	-6.09±0.18 <sup>d</sup>	$18.81 \pm 0.84^{\circ}$	NA	19.77±0.85°	178.74±0.01ª
B	101.85±0.16 <sup>a</sup>	-5.89±0.12 <sup>d</sup>	18.96±0.94°	0.69±0.06°	19.85±0.93°	178.73±0.01ª
С	101.67±0.31ª	-4.84±0.45°	19.97±1.55°	$2.58{\pm}0.65^{\circ}$	20.55±1.39°	$178.67 \pm 0.04^{b}$
D	97.76±0.90 <sup>b</sup>	-2.53±0.06 <sup>b</sup>	28.31±0.33 <sup>b</sup>	10.89±0.84 <sup>b</sup>	28.42±0.33 <sup>b</sup>	178.52±0.01°
Е	96.41±1.67 <sup>b</sup>	0.15±0.03ª	36.18±0.59 <sup>a</sup>	19.25±1.39 <sup>a</sup>	36.18±0.59 <sup>a</sup>	$1.57{\pm}0.001^{d}$

\*Results were the mean values  $\pm$  standard deviation. Different letters in the same column indicated significant differences (p < 0.05).





• Air-Top Layer \* Air-Middle Layer • Oil-Top Layer • Oil-Middle Layer



**Figure 5.1.** The heating profiles of the non-inoculated whole milk powder when heated (a) in the RF oven while surrounded with air and soybean oil and (b) in the conventional oven.



**Figure 5.2.** Inactivation kinetic curves of (a) *E. faecium* and (b) *Salmonella* spp. in whole milk powder ( $a_w$ , 40°C = 0.35) at 75 °C, 80 °C and 85 °C.



**Figure 5.3.** The thermal death time (TDT) curve of (a) *E. faecium* and (b) *Salmonella* spp. in whole milk powder (a<sub>w</sub>, 40°C=0.35).



**Figure 5.4.** The water activity of whole milk powder with different treatments; (A) Untreated powder, (B) After holding 4 days in the environmental chamber, (C) After RF heating to 75 °C without holding, (D) After RF heating to 75 °C followed by 120 min holding in a conventional oven, (E) After heating in the conventional oven to 75 °C without holding.

Results were given as mean values  $\pm$  standard deviation. Different letters indicated significant differences (p < 0.05).



**Figure 5.5.** The moisture content of whole milk powder with different treatments; (A) Untreated powder, (B) After holding 4 days in the environmental chamber, (C) After RF heating to 75 °C without holding, (D) After RF heating to 75 °C followed by 120 min holding in a conventional oven, (E) After heating in the conventional oven to 75 °C without holding.

Results were given as mean values  $\pm$  standard deviation. Different letters indicated significant differences (p < 0.05).

## CHAPTER 6

## CONCLUSION

In the first part of this dissertation, the dielectric properties of the whole, nonfat milk powder, and their mixtures were measured at different temperatures (20-90 °C) and frequency (1-30 MHz). An increase in the dielectric properties of milk powders was observed with an increase in temperature and a decrease in frequency. The dielectric properties of the whole milk powder were higher than nonfat milk powder which might be due to the bulk density. A higher heating rate was achieved for milk powder with lower fat content. The electric field strength increased with the height of the boxes because more power was absorbed by the sample in the taller box. The electric field strength was lower for the samples that were heated in the cylindrical boxes. Electric field strength decreased overheating time for all milk powders. The solubility of whole milk powder increased while the solubility of the nonfat milk powder decreased and the solubility of the 1:1 mixture remained constant after RF heating.

Out of the three surrounding media (air, DI water, and soybean oil), the cornflour container with soybean oil showed improved RF heating uniformity. This was supported by lower heating uniformity indices and temperature distribution obtained by thermal images. Moreover, the heating rate increased which can be considered as an advantage for the industrial application of RF treatment. Conveying the cornflour container through the cavity further improved the heating uniformity of cornflour surrounded by soybean oil.

In the last section of the dissertation, the proposed system (RF heating of milk powder immersed in soybean oil) was validated by microbial inactivation analysis. It was concluded that *E. faecium* might be considered as a surrogate of *Salmonella* spp. for whole milk powder to validate RF heating for pasteurization. More than 5 log CFU/g reduction of Salmonella spp. in whole milk powder was achieved by RF heating the sample to 75 °C followed by holding it in the oven for 2 h. This treatment increased water activity decreased moisture content and caused a significant color change in whole milk powder. Therefore, further studies in which optimum conditions such as treatment type, target temperature, sample water activity, and different RF designs are recommended to achieve the safety standards while maintaining the food quality of whole milk powder. The difference between the maximum and minimum temperature should be further minimized to make RF heating acceptable for the pasteurization purpose. For this purpose, the combination of other techniques such as mixing and rotating with immersing the sample in liquids and/or the hurdle effect of RF technology with other pasteurization techniques are recommended for future studies.

In addition to the beforementioned suggestions which are specific to the study in this dissertation, future researches are recommended as follows with a long term goal to commercialize RF pasteurization technology:

1. There is a lack of information on the dielectric properties data of various food powders as a function of frequency, temperature, moisture content, water content, density, and composition. Comprehensive studies on the dielectric properties of food powders are in need to design an effective RF heating system. 2. The effect of frequency used in RF pasteurization was not investigated yet for food powders. The application of RF pasteurization at different frequencies (13.56, 27.12, and 40.68 MHz) could give valuable information about electric field distribution in RF heating. Moreover, most of the previous studies were conducted using free-oscillator RF systems. The studies with 50  $\Omega$  RF systems can be also useful due to their improved power control, heating uniformity, and stability.

3. It is well known that the electrode shape and configuration have a great influence on electric field distribution. Electric field distribution during RF heating can be investigated by computer simulations as well as experiments for different electrode geometries.

4. Most of the studies were carried out in a lab-scale RF system so far. RF pasteurization studies with the larger RF systems and sample size can be useful to estimate the efficiency and economics of the industrial RF system.

5. Collaborations between food scientists, food process engineers in food plants, RF engineers, and RF manufacturers are needed for systematic RF studies and a compatible integration of RF pasteurization system to the food industry.

6. Computer simulation is a quick, and cheap solution to obtain an understanding of RF pasteurization in food powders. More computer simulations are needed to understand the factors affecting RF heating uniformity.

 7. Food quality and sensory perception of the RF heated powders can be studied to develop an effective RF system by optimizing the factors influencing the quality of food powders.
 8. More studies need to be conducted on the microbial validation of RF heating for pasteurizing food powders. The study on the lethal kinetics of various pathogens including their spores is necessary to develop an effective RF pasteurization system.

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