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## INFLUENCE OF TEMPERATURE AND ADDITIVES ON THE ADSORPTION KINETICS OF FOOD EMULSIFIERS

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#### ABSTRACT

The influence of temperature (6, 25, and 50C) and additive substances (salt, sugar and vinegar) on the adsorption kinetics of typical food emulsifiers, egg yolk and whey protein concentrate (WPC) were studied using the Wilhelmy plate/burst membrane method to monitor the interfacial tension (IT) at the water soya oil interface. The effect of solutes on the adsorption kinetics depended on emulsifier type. The presence of salt and higher temperatures reduced the IT of egg yolk solutions, while the IT of WPC solutions increased with salt addition. The presence of sugar and vinegar generally increased the IT of both emulsifiers while increased temperature caused the equilibrium IT of both emulsifiers to decrease and attain equilibrium more quickly.

#### INTRODUCTION

Commercial salad dressings and mayonnaise are oil-in-water emulsions. The stability and consistency of these dispersed systems are important properties in the food industry. During emulsification, the dispersed phase is broken into small droplets by mechanical energy. This creates new interfaces, which have to be stabilized within short times. Unstabilized droplets collide and coalesce thereby reducing the efficiency of the process. In order to avoid recoalescence of newly-formed droplets, emulsifiers with fast adsorption kinetics have to be used. The adsorption kinetics of an emulsifier is the rate at which the new interfaces are stabilized by adsorbing emulsifier molecules.

Stabilizers often referred to as emulsifiers or surface active agents are used to lower the interfacial tension (IT) between the liquid interfaces, thus reducing the pressure gradient required to disrupt the droplets when forming an emulsion (Elizalde *et al.* 1988; Toro and Regenstein 1989).

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Proteins act as emulsifiers by lowering the IT between water and oil. Adsorption of proteins at the fluid interfaces provide stability against phase separation. The greater the decrease in IT, the greater the emulsifying activity of the proteins (Elizalde *et al.* 1991). IT is an important property of liquid/liquid systems. It serves as an index of the relative forces of intermolecular attraction and acts as a measure of the free energy per unit area of the interface, and also provides information about the emulsifying ability of the system (Gaonkar 1989).

Protein structure and hence functionality (e.g, emulsifying properties) are affected by many factors in the surrounding environment, such as pH, ionic strength, temperature, presence of other compounds, etc. (Toro and Regenstein 1989; Turgeon et al. 1992). Solutes can affect the water uptake of protein, and also modify their surface charges, thus affecting the IT. The presence of electrolytes such as salts have been shown to affect the charge stabilization between droplets in an oil-in-water emulsion (Elizalde et al. 1991). The chloride ions increase the exposure of hydrophobic side chains which accelerate adsorption and unfolding on the interface. Salt enhanced the stability of oil-in-water emulsions by disrupting the egg yolk granules, leaving more active sites exposed at the interface thereby lowering the IT (Carrillo and Kokini 1988). Gaonkar (1992) reported that at a given temperature, IT decreased in the presence of NaCl which was suggested to be due to the increase in surface activity of polar impurities present in commercial soya oil used in the study. The impurities were salted out and concentrated at the oil/water interface thus lowering the IT. The author also reported that IT decreased with increasing temperature in the presence of salt.

The pH affects charge and electrostatic balance within and between proteins. Below and above the iso-electric point (pI), proteins have a positive or negative charge which enhances their solubility. As the pH approaches the pI of the protein, the charge tends to zero and the attractive forces predominate and molecules tend to associate and aggregate easily (Nakumara *et al.* 1982; Li *et al.* 1985; Turgeon *et al.* 1992). Therefore, to prevent protein aggregation, it is necessary to avoid using emulsifiers in the iso-electric point range.

Food emulsions such as salad dressing and mayonnaise are developed under different conditions of pH, temperature, and ionic strength. It is therefore necessary to investigate how these conditions affect the emulsifying properties of different proteins. The objective of this research therefore, was to study the influence of temperature, pH, and solutes (NaCl, and sugar) on the emulsifying properties of egg yolk and whey protein concentrate (WPC) solutions by monitoring the IT of the emulsion mixture with time.

All methods of measuring adsorption kinetics at a surface or interface are based on the measurement of surface or interfacial tension which is a function of the age of the interface. The numerous available methods can be grouped as static or dynamic methods. Some of these methods have one or more of the following disadvantages. (1) Measurement of adsorption kinetics at the interface is not continuous. (2) For some of the methods, measurement is possible only at the surface. (3) Measurement can take place only for a limited period (Anbarci and Armbruster 1987).

The formation of an emulsifier free surface/interface and a properly defined zero point are important in the measurement of surface/interfacial tension. The following methods are applicable to both surface/air or liquid-liquid interface. These include De-Nouy ring, Pendant-Drop apparatus, and Wilhelmy plate methods.

With the De-Nouy rings, IT can only be measured 15-30s after the experiment had begun. This method is most suitable for emulsifiers with slow adsorption kinetics. In addition, the establishment of an emulsifier free interface at the beginning of measurement is not certain, such that the definition of a zero time is inaccurate. The Pendant-drop apparatus, on the other hand, permits measurement from the start of the experiment. The zero point is the time the first droplet breaks off. However, this method requires the use of very expensive and precise optical and photographic equipment. The Wilhelmy plate coupled to a tensiometer measures the adsorption kinetics continuously to equilibrium. The method is suitable for measuring adsorption kinetics of fast emulsifier and can easily be reproduced. The burst membrane provides a well defined zero-point. The Wilhelmy plate allows the interfacial area to remain constant during measurement. This method is widely used because of its simple experimental design, fast operational capability, more straight forward data manipulation and ease of automation (Ganonkar 1989). Detailed description of the principles and operations of the above methods are in literature (Anbarci and Armbruster 1987; Dickenson and Stainsby 1988).

#### MATERIALS AND METHODS

Commercial soybean oil was employed in this study as it is commonly used in the manufacture of food emulsions. The oil was purchased from Pflanenöl Lesieur Gmbh, Kleinblitterdorf. All eggs used were from a single strain of Lohne-Riessel hens. The eggs were stored in a refrigerator at 4C for a maximum of five days until broken. The yolks were separated using the method of Lu and Baker (1987). The vitteline membrane was ruptured and the liquid yolk was collected in a beaker. The yolks from four eggs were pooled, mixed and used immediately. To prepare the egg yolk emulsifying solution, 37.5g =10 CMC (Critical Micelle Concentration) of the yolk was mixed in one liter of water. Demineralized water was used in all preparations.

Commercial whey protein concentrate (60% protein, 4% ash, 5.5% moisture, 6% fat, and 20% lactose content) with trade name Lacprodan-60 was obtained from Kalichemie AG, Hannover. The emulsifying solution of WPC was

obtained by mixing 24g = 10 CMC of WPC in one liter of water. The NaCl used was of reagent grade (E. Merck, Darmstadt). Table sugar (Fa. Südzucker AG, Waghäusel) and table vinegar (Feldman AG, Karlsruhe) were used.

Formulations and levels of ingredients used in this study are as commonly found in commercial salad dressings and mayonnaise (Carrillo and Kokini 1988). The levels of salt used were 0.5, 1.0, 1.5 and 2.0% and levels of sugar used were 2.0, 5.0 and 7.0%. In order to vary the pH of the emulsifying solutions, vinegar was used at the following concentrations 5.0, 4.0, 3.0, 2.0 and 1.0%. The undiluted vinegar was 5% acid. To obtain the various pH levels, the vinegar solution was diluted with varying quantities of water. The pH of the various vinegar solutions were determined using a pH meter. The temperature levels used were 6, 25 and 50C to avoid the denaturation of the protein molecules.

The IT between oil and the aqueous phase was measured on a LAUDA Tensiometer (Fa.LAUDA, Germany) using the bursting membrane with a Wilhelmy Plate. The Wilhelmy plate (4cm circumference) consist of an alloy of platinum and irradium. IT was recorded continuously by connecting a recorder (Model SE 130, BBC Goerz, MetraWatt) to the tensiometer. The sample holder made of plexi glass (Fig.1) has the bottom part filled with the emulsifier solution and also contains a magnetic stirrer. Commercial balloon (the bursting membrane) is expanded over the bottom glass eliminating air bubbles to ensure that it bursts instantaneously and completely when ruptured. The balloons (with the necks cut off to allow for proper expansion) were soaked in Isopropanol to remove all impurities. The balloons were rinsed with demineralized water before use. The O-ring acts as a joint between the two parts of the sample holder and



FIG. 1. EXPERIMENTAL LAYOUT FOR MEASURING THE ADSORPTION KINETICS OF EMULSIFIERS BY THE BURST MEMBRANE METHOD

also prevents leakage of liquid. A constant volume of water was measured onto the balloon and the surface tension was determined. Soya oil was carefully added to the water with the aid of a precision dispensing pipette in order not to disrupt the lamella created by the plate between oil and water. The oil and aqueous phases were maintained at the required temperature throughout the duration of the experiment. During measurement, temperature was maintained by placing the sample holder in a thermostated container with water circulating through it connected to a water bath at the required temperatures.

The solutions were mixed constantly until the timing started. Timing of the experiment began when the membrane was punctured. The balloons were ruptured using a sterile needle (Neopoint 20 G  $\times$  1½) attached to a metal holder. IT measurements were repeated in triplicate to ascertain the reproducibility between runs ( $\pm 0.15$  mN/m). A two-way analysis of variance was carried out on the data using the MSTAT-C software (Nissen 1990). The experimental layout is shown in Fig. 1.

#### **RESULTS AND DISCUSSION**

Table 1 shows the pH values obtained for the different concentrations of vinegar in preparing the acidified emulsifier solutions. For both egg yolk and WPC mixtures, the pH decreased with increase in vinegar concentration but the rate of decrease was affected by the type of protein molecules. At all concentrations, the pH of egg yolk solutions were lower than those of WPC.

Surface tension at the air/water interface was determined at the beginning of each experimental run and was 72.5 mN/m at 25C. However, the value varied slightly at 6C and 50C respectively, indicating the absence of surface impurities in the water.

Vinegar concentration. (%)	Egg yolk (10CMC)	WPC (10CMC)
0	6.9	6.0
1	3.3	3.6
2	3.1	3.4
3	3.0	3.3
4	2.9	3.2
5	2.9	3.1

TABLE 1. pH VALUES OF EGG YOLK AND WPC SOLUTIONS ON ADDING VINEGAR AT DIFFERENT CONCENTRATIONS

The effects of variation of salt concentration on the emulsifying properties of egg yolk at different temperatures are shown in Fig. 2a-c.



FIG. 2. INFLUENCE OF TEMPERATURE AND SALT CONCENTRATION ON THE ADSORPTION KINETICS OF EGG YOLK AT THE WATER/SOYA OIL INTERFACE

Irrespective of the temperature, the presence of salt improved the adsorption kinetics of egg volk molecules. Initial decrease in the IT occurred within the first 300 s of measurement. In addition a faster stabilization of the system resulted by attainment of equilibrium faster than the salt-free egg yolk solution. Variation of salt concentration did not have a significant (P < 0.05) influence on the IT. At 6C, the influence of salt addition to egg yolk solution was not significant (P < 0.05) in reducing IT. Although IT was lower for salted egg yolk solutions at the beginning of the experiment, by 2000s, however, the salt-free (0%) egg yolk solution had lower IT. The decrease in IT observed in the presence of salt is in agreement with the results of previous investigations. Carrillo and Kokini (1988) reported that salt enhanced the stability of oil-inwater emulsions by disrupting the egg yolk granules and leaving more active sites exposed at the interface. Gaonkar (1992) reported that the IT of oil-inwater systems decreased in the presence of salt. This decrease in IT was attributed to increase in the surface activity of polar impurities which were salted out and then concentrated at the interface. It has been suggested that salt may have induced some conformational changes in the proteins, thereby releasing the hydrophobic regions for a more effective interaction with oil (Turgeon et al. 1992). In general, increasing the temperature decreased the range of equilibrium IT (EO-IT) and speeded EO-IT attainment. Gaonkar (1992) also reported a decrease in IT with temperature increase in the presence of salt. The extent of reduction in the IT due to the presence of NaCl depended on temperature. The higher the temperature, the greater the IT reduction. This was attributed to the change in the solubility of polar impurities with temperature and salt.

The adsorption kinetics of WPC solutions were not significantly (P < 0.05) influenced by the presence of salt up to the 1% level (Fig. 3a-c). The effect of salt on the IT of WPC solutions at 50C (Fig.3c) increased the IT at all times of measurement. Increasing the salt level beyond 1% resulted in an increase in IT, and the higher the salt concentration, the higher the IT of the solution. The effect of temperature on IT of the solutions was noted. Increasing the temperature greatly reduced the EQ-IT values. Furthermore, temperature affected the rate of decline of the IT of the solutions. The higher the temperature, the longer the time required for the solutions to attain equilibrium state.

The general increase in IT of WPC solutions in the presence of salt is the opposite of that reported for egg yolk solutions. This is probably due to sample variability, i.e. the type of protein molecules in WPC may be different from those of egg yolk. Solutes affect the unfolding nature of proteins differently, hence the different response to adsorption kinetics which is reflected by the IT measurement (Li-Chan *et al.* 1985). It has been suggested that the effect of salt is influenced by the type and source of protein molecules. Montero and Borderias (1991) reported that adding NaCl in amounts up to 2% of

-10000



FIG. 3. INFLUENCE OF TEMPERATURE AND SALT CONCENTRATION ON THE ADSORPTION KINETICS OF WPC AT THE WATER/SOYA OIL INTERFACE

solubilized collagenous material from the skin of Hake fish had no effect on the emulsifying capacity, while the emulsion capacity of collagenous material from Hake muscle decreased for all quantities of NaCl added.

For both egg yolk and WPC solutions, adsorption kinetics at the interface was affected by temperature and the addition of sugar (Fig. 4a-c and 5a-c, respectively). The effect of sugar was temperature dependent. The presence of sugar increased IT at all times compared to the 0% solution and caused the solutions to attain their EQ-IT more quickly at 6 and 50C. EQ-ITs for solutions containing sugar were also significantly (P<0.05) higher than that for 0% solution at 6 and 25C (Fig. 4a,b). At 6 and 50C, increasing sugar concentration of the emulsifiers did not improve their adsorption kinetics as the IT increased. At 25C, solutions containing 7% sugar had lower IT but the extent of reduction was influenced by emulsifier type. In the egg yolk solution (7% sugar), IT was significantly lower than at other concentrations studied and was approximate to that of the sugar free solution. IT of 7% sugar solution in WPC, though reduced, was not significantly (P<0.05) different from the IT of other sugar concentration in WPC at 25C.

During experimentation, with sugar addition it was observed that egg yolk particles settled with time, leaving a clear solution. The rate of sedimentation of the egg volk particles was influenced by both temperature and sugar concentration. The sedimentation of the egg yolk molecules reduced the amount of proteins available at the interface to interact with both oil and water molecules. Several authors have reported that protein acted as a mediator in the formation of a stable emulsion by unfolding at the interface to associate with both water and oil. An interfacial bilayer was formed whose thickness depended not only on the water and oil adsorption capacity of the protein, but also on the protein concentration (Mendoza and Sherman 1991; Turgeon et al. 1991; Elizalde et al. 1991). Hence, when the interfacial bilayer protein concentration dropped with time (through sedimentation), the emulsifying ability of the bilayer was reduced, which may account for the high IT values obtained. Generally increasing the temperature of the solutions affected their adsorption kinetics resulting in lower EQ-IT values. The time required to attain equilibrium was longer with temperature increase for WPC and shorter for egg yolk solutions. These results suggest that stabilization of the interface is a function of proteins and temperature among others. In Fig. 4a-c for example, the EQ-IT range from 5-13 mN/m at 6C was reduced to 4-11 mN/m at 25C and finally to 1-3 mN/m at 50C.

Figures 6a-c show the influence of vinegar (pH) on the IT of egg yolk solutions at different temperatures. For measurements at 6 and 25C (Fig. 6a,b) the presence of vinegar initially lowered the IT compared to the 0% vinegar solution, but by 1800 s the values had levelled off while the IT of the 0% solution continued to decrease. The addition of vinegar to egg yolk solutions at



FIG. 4. INFLUENCE OF TEMPERATURE AND SUGAR CONCENTRATION ON THE ADSORPTION KINETICS OF EGG YOLK AT THE WATER/SOYA OIL INTERFACE



FIG. 5. INFLUENCE OF TEMPERATURE AND SUGAR CONCENTRATION ON THE ADSORPTION KINETICS OF WPC AT THE WATER/SOYA OIL INTERFACE



FIG. 6. INFLUENCE OF TEMPERATURE AND VINEGAR CONCENTRATION ON THE ADSORPTION KINETICS OF EGG YOLK AT THE WATER/SOYA OIL INTERFACE



FIG. 7. INFLUENCE OF TEMPERATURE AND VINEGAR CONCENTRATION ON THE ADSORPTION KINETICS OF WPC AT THE WATER/SOYA OIL INTERFACE

all levels and times of measurement increased the IT of the solutions compared to that of the 0% solution at 50C (Fig. 6c). In general no significant (P < 0.05) influence on the IT of the solutions was observed when the pH level was varied at 50C. Increasing the temperature from 6 to 25C resulted in a decrease in the IT of the egg yolk solution in the presence of vinegar for respective vinegar concentrations but further temperature increase to 50C increased the IT of the mixtures. This fluctuating behavior of IT values with pH variation was also reported by Chung and Ferrier (1992).

The results of IT of WPC solutions containing varying vinegar concentrations determined at the different temperatures are shown in Fig. 7a-c. The higher the vinegar concentration at 6 and 50C, the higher the IT value of the WPC solutions (Fig. 7c). The addition of vinegar increased the IT of the solutions and caused them to reach their EQ-IT very quickly.

In this study, WPC solutions containing no vinegar had lower IT compared to the acidified solutions. It has been reported that although whey proteins are flexible at pH 7, they are rigid and resistant to unfolding at pH 3, causing them to have a more compact structure, resulting in a high IT (Shimizu *et al.* 1985; Phillips *et al.* 1990). It is suggested that some structural changes of whey proteins at the acidic pH occurs which affects its adsorption and emulsifying properties.

#### CONCLUSION

From the results obtained, the addition of salt improved the adsorption kinetics of egg yolk molecules by reducing the tension at the interface. The effect of sugar is dependent on both temperature and protein type. The range of EQ-IT values reduced with temperature increase with salt and sugar additives. Addition of vinegar to WPC solutions did not improve adsorption kinetics but reduced stabilization time.

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## THE EFFECT OF EDIBLE FILM ON OIL UPTAKE AND MOISTURE RETENTION OF A DEEP-FAT FRIED POULTRY PRODUCT

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#### ABSTRACT

The recent trend in reducing the fat content in fried foods is leading to the development of low-fat products using batter and breading formulations from protein and other hydrocolloids. The functionality of many of these edible ingredients on moisture retention and fat barrier properties during deep-fat frying is not clearly understood. The objective of this study is to evaluate the effectiveness of hydroxypropyl methylcellulose edible film coating on chicken balls for moisture retention and fat reduction during deep-fat frying. Two sizes (35 and 47 mm) of uncoated and coated chicken balls were fried in peanut oil at 175C for five different frying periods ranging from 30 to 180s for small and 30 to 300s for large balls. The moisture and fat content from the surface layer and core were determined. In comparison to the control, moisture retention up to 16.4% and fat reduction up to 33.7% were observed.

#### **INTRODUCTION**

Deep-fat frying is a widely used method for preparing foods with an attractive and tasty surface. The soft and moist interior along with the porous outer crispy crust provides increased palatability to foods (Varela 1988). In the United States, the small fried-food business is flourishing. Among fried products, fried chicken, often called America's comfort food, is a six billion dollar industry. During frying, the energy needed for cooking is supplied by the

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hot oil and uptake of fat by the food occurs. This fat could contribute to the consumption of calories by the consumers. In recent years, interest in reducing the oil uptake in fried foods has increased. The Surgeon General's Report on Nutrition and Health included a dietary recommendation to reduce the fat consumption by 25%, so that less than 30% of calories consumed are from fat, especially saturated fats (O'Donnell 1994). Industry is responding to these challenges from the government as well as the consumers by introducing low-fat products. Many of these approaches rely on empirical tests to develop batter and breading formulations from protein and other hydrocolloids. Irrespective of different claims, the independent functionality of many of these edible ingredients on moisture retention and fat barrier properties during deep-fat frying is not clear.

Many researchers focused their attention to understand the relationship between oil uptake and moisture loss during frying (Gamble *et al.* 1987, McDonough *et al.* 1993; and Saguy and Pinthus 1995). However, limited research has been conducted to understand transport phenomena associated with deep-fat frying of foods (Rao and Delaney 1995; Singh 1995 and Moreira *et al.* 1995). The work done on deep-fat frying so far has been primarily limited to non-fatty products (such as potato) and the mechanism of fat transport in deep-fat fried products of animal origin (i.e., products initially containing fat) has not been extensively investigated. An understanding of fat transfer during frying of meat products is of interest to processors as consumers become more concerned about the amount of fat they consume (Ateba and Mittal 1994).

Edible coatings prepared from ingredients such as polysaccharides, proteins, lipids or their combinations have been used as a part of batters and breading to improve coating performance (Kester and Fennema 1986; El-Nokaly and Hiller 1991; Stark and Gross 1992; Feeney *et al.* 1993). Carboxymethyl cellulose (CMC) has shown to significantly improve the breading adhesion (Suderman *et al.* 1981). Meyers (1990) has discussed the functionality of HPMC and MC as barriers in the breading and batters. The use of proteins (such as milk powder and egg albumen) as an ingredient in the fried product is also known, yet their functionality as edible coatings have not been established (Cunningham 1989). Fan and Arce (1986) reported the surface application of amylose to reduce fat uptake during frying. However, most of the information regarding the role of these ingredients used in edible coatings is proprietary in nature (Saguy and Pinthus 1995). Also, the functionality of edible films as moisture and fat barriers during frying is not fully understood.

A preliminary experiment was conducted in our laboratory on intact pieces of chicken breast ( $50.8 \times 37.6 \times 12.5$  mm) coated with different edible films (hydroxypropyl methyl cellulose (HPMC), commercial ether derivative of HPMC (E-15) and corn zein (CZ)). The coated samples were deep-fat fried for 3.5 min in peanut oil maintained at 200C. Among the films tested, HPMC was

identified as the best based on oil barrier (27% reduction in comparison to control) and moisture retention (6% more retention than control) properties during deep-fat frying of chicken pieces. Some of the limitations of this study include smaller sample size and variation in oil-bath temperature. Further, the initial fat content and distribution of the fat within the samples were not controlled. Hence, in the present study, ground chicken was used with uniformly distributed and known initial content of fat. Based on the preliminary experiments, HPMC was selected as the edible film.

The long term objective of this project is to study the role of edible films/coatings in enhancing the quality of deep-fat fried products by retaining the moisture and reducing the fat absorption during frying. The specific objectives of this study were: to study the feasibility of deep-fat frying of edible film coated food products and to evaluate the influence of frying time and product size on moisture retention and fat barrier capabilities of an edible film coating during deep-fat frying.

#### MATERIALS AND METHODS

#### **Chicken Ball Preparation**

Fresh chicken breasts were obtained from a local poultry processor (Wayne Poultry, Pendergrass, GA) and the surface fat was removed following visual inspection. The meat was ground using a Toledo Chopper (Model A-200, Toledo Chopper, Toledo, OH) equipped with 6 mm die. To the ground mass, 1.5% salt was added for protein extraction and mixed at low speed for 2 min at 2C using a mixer (Model A-200, Hobart Mfg., Co., Troy, OH). Then, sodium tripolyphosphate (0.5%) was added as a binding agent. The mixture was kept at -20C until further use. Prior to experiments, the meat was thawed at 0C for 36 h. Two commercial meat ball shapers (of sizes 47 and 35 mm inner diameter) were used for making the chicken balls. The average mass of large and small chicken balls were  $56.2 \pm 0.7$  g and  $24.2 \pm 0.4$  g, respectively.

#### **Edible Coating**

Hydroxypropyl methylcellulose (HPMC) edible film solution was prepared by adapting the formulation of Park and Chinnan (1995). Nine grams of HPMC (grade: KW, Aldrich Chemical Company Inc., Milwaukee, WI) was added to 200 g of distilled water maintained at 45C. After complete dissolution, 200 g of 95% food grade ethyl alcohol (190 proof, Florida Distillers Co., Lake Alfred, FL) was added and thoroughly mixed. Then 2 mL of polyethylene glycol (grade: KV, Aldrich Chemical Company Inc., Milwaukee, WI) was added as a plasticizer. The rheological characteristics of the HPMC solution were estimated using a rotational viscometer (Model HATD, Brookfield Engineering Laboratories, Stoughton, MA) equipped with a disc spindle (#2 H). The power law model:  $\tau = K\dot{\gamma}^n$ , where,  $\tau$  is the shear stress and  $\dot{\gamma}$  is the shear rate, was employed. The coefficients K (consistency coefficient) and n (flow behavior index) of the model were estimated from experimental data. These coefficients would provide an insight to the applicability of the edible coatings for spraying.

Chicken ball was coated by placing it on a rotating (15 rev/min) platform and spraying (using a Popular Mechanics spray gun - PM50595, Beckley, WV) it with the film solution  $(3.1 \times 10^4 \text{ kg/m}^2/\text{s})$  for one minute. After air drying for 2 min, the procedure was repeated to ensure uniform coating of the film on the ball. The increase in mass of chicken balls due to the coating was of the order of 0.06 to 0.1 g for small and large balls, respectively. Hence, attempts to measure the film thickness on the chicken balls based on mass difference did not yield satisfactory results due to adherence of the product to the surface of the balance. Subsequently we estimated the film thickness using a flat surface under similar experimental conditions. This approach yielded an estimated film thickness of 0.05 mm ( $\approx 2$  mil).

#### **Deep-Fat Fryer**

A specially designed laboratory model deep-fat fryer  $(0.76 \times 0.31 \times 0.12 \text{ m})$  was used for frying chicken samples (Fig. 1). The temperature of the oil bath was controlled using a microprocessor based temperature controller (CN7600, Omega Engineering, Stamford, CT). The fryer was also provided with an agitator mechanism to ensure uniform temperature distribution within the bath. Peanut oil at 175C was used as the frying medium. A specially designed hollow helical shaped ball holder (Fig. 1) made from thin copper tubing, was used for transferring the chicken balls in and out of the fryer. The ball holder had negligible contact area and it did not form its impression on the meat surface. In addition, the heat gained by conduction via tubing was negligible compared to the convective heat gained from the frying medium.

#### Frying

Experiments were conducted for uncoated and coated balls. The mass of the chicken ball prior to frying was recorded. The chicken balls were allowed to equilibrate with the ambient temperature ( $\approx 20$ C) before frying. The chicken balls were fried for five different frying periods (for large balls: 30, 60, 120, 210, and 300 s; and for small balls: 30, 60, 90, 120, and 180 s). All the experiments were carried out in triplicates. The frying experiments lasted for about 3 h. The maximum frying period for respective sizes was selected so that at the end of frying, the geometric center of the meat ball reached 70C or above.



FIG. 1. SCHEMATIC DIAGRAM OF THE EXPERIMENTAL SETUP

A typical experimental procedure consisted of introducing the meat ball with the maximum frying period (300 s for large balls or 180 s for small balls) first into the frying medium followed by balls with successive frying periods. The procedure was repeated until the last ball (30 s) was placed into the medium. This approach prevented the sudden drop in the frying medium temperature and enabled us to maintain isothermal conditions during frying. At the end of frying, the balls were taken out in the reverse order they were placed in. A maximum of 6 s error was made between removing test samples from the frying medium.

The temperature history of the samples was measured by introducing a chicken ball into the fryer simultaneously with the sample to be cooked for maximum time (e.g., 300 s for large balls). The temperature at the surface and at the geometric center were monitored (with sampling frequency of 1 s) using a T type 30 gage thermocouple (TT-T-30, Omega Engineering, Stamford, CT) attached to a data logger (Hotmux, DCC Corporation, Pennsauken, NJ). The temperature of the oil in the vicinity was also monitored. Samples of frying oil were collected every hour for quality assessment. The oil quality was determined by measuring absorbance spectra between 300 and 800 nm using a spectrophotometer (Model 8451A, Hewlett-Packard Co., Santa Clara, CA). No appreciable change in oil quality was observed.

#### Analysis

After frying, the balls were air cooled for 30 min and excessive surface oil was gently wiped off using a tissue paper. The meat balls were then immediately

weighed, placed in freezer bags and frozen at -20C until further analysis. The product mass loss was calculated as the percent mass difference between uncooked and cooked samples. The moisture and fat content of the fried chicken balls were determined as follows. The chicken ball was cut into four quarters. The surface layer, identified by visual inspection, was scraped off and collected. The mass of surface layer and core portion was recorded and their moisture content was determined using a freeze dryer (Research Freeze dryer, Virtis Company, Gardiner, NY). Freeze drying was chosen over the traditional oven drying method as it enhances the porous structure of the samples and facilitates the complete fat extraction during subsequent fat analysis (Ateba and Mittal 1994). The fat content of the freeze-dried samples were determined using AOAC method 960.39 (AOAC 1990). The moisture retention and fat reduction capabilities of edible coating in relative to the control (uncoated samples) were estimated as the percent of percent moisture and fat content differences between uncoated and coated samples, respectively.

#### **Statistical Analysis**

Statistical analyses were performed using General Linear Model Program to test the effects of edible coating, frying time, product size on product moisture and fat content, mass loss and temperature. Duncan multiple range test was used to estimate the significant differences among the means at the 5% probability level (SAS 1988).

#### **RESULTS AND DISCUSSION**

#### **Initial Properties**

The proximate composition of the raw ground chicken was: 72.80% moisture, 25.64% protein, 0.62% fat and 0.94% ash. The initial pH of the mixture was 5.82. The consistency coefficient (K) and the flow behavior index (n) of the power law model for HPMC were estimated as 34.2 Pa.s<sup>n</sup> and 0.94, respectively.

#### **Temperature History**

Sample temperature history of a chicken ball is given in Fig. 2. The oil temperature during frying was maintained at  $175 \pm 0.5$ C. As expected, the surface temperature rose rapidly initially and had a plateau at around an average temperature of 110C at the end of frying. This was attributed to the removal of moisture from the surface by evaporation. However, within the range of experimental conditions studied, the surface temperature never reached above 130C. This could be attributed to the continuous evaporation of moisture that



DURING FRYING

migrated from the center. The mean surface temperature at the end of frying was 122.0, 109.3, 109.5 and 102.0C for large uncoated, large coated, small uncoated, and small coated chicken balls, respectively. The change in center temperature was minimal during the initial period and increased faster during the later stages of frying. At the end of frying, the product geometric center reached above 70C. Differences between surface temperatures of coated and uncoated chicken balls were attributed to the retained moisture in coated chicken balls. Due to difference in surface area and frying time, large balls had higher surface temperatures than small chicken balls (p < 0.05).

#### Mass Loss History

Sample mass loss histories of small coated and uncoated balls are given in Fig. 3. At the end of frying, the overall average mass loss for small coated ball (14.4%) was less than that of control (18.8%) (p < 0.05). Similarly, an average mass loss of 12.6% was observed for large coated balls and 15.2% for large uncoated balls (p < 0.05). During frying, in addition to serving as a moisture and fat barrier, the edible barrier could have acted as protective layer reducing the loss of any of surface fibers to the frying medium. The differences in mass loss between large and small balls could be attributed to the initial mass.



FIG. 3. OVERALL MASS LOSS HISTORY FOR SMALL CHICKEN BALLS

#### **Change in Moisture Content**

During initial stages of frying (up to 60 s for large ball and 30 s for small ball), the surface layer lost more moisture (Fig. 4). At later stages of frying, the balls were still losing moisture during frying, but at reduced rate. As expected, the change in moisture content in the surface layer was more rapid than that of the interior core. At the end of frying, the moisture content in the surface and core of large coated balls was 44.2% and 55.1% (w.b.), respectively. For small coated chicken balls, the moisture content in the surface and core was 51.8% and 64.5% (w.b.), respectively.

After 30 s of frying, the surface moisture of the coated balls was less than that of the control (Fig. 5). However, as frying continued, uncoated balls rapidly lost moisture resulting in higher moisture loss for uncoated chicken balls in comparison to that of coated balls (p < 0.05). At the end of frying, the moisture content (on w.b.) of the coated and the uncoated large chicken balls were: 44.2% vs 41.8% (w.b.) for surface and 55.1% vs 48.7% (w.b.) for core. The surface layer of coated balls retained 5.7% to 8.7% more moisture than that of control, whereas, about 13.1% to 16.4% extra moisture retention was observed in the core region of coated balls in comparison to that of uncoated samples (p < 0.05).



FIG. 5. EFFECT OF COATING ON MOISTURE CONTENT FOR LARGE CHICKEN BALLS

The following visual observations were made during simultaneous frying of coated and uncoated balls. Initially the coated balls produced more bubbles in comparison to uncoated balls (up to 30 s). As frying progressed, the trend reversed, the uncoated ball formed a large envelope of bubbles suggesting the ball was losing more moisture to the frying medium. These observations also explain the difference in pattern of moisture loss between coated and uncoated balls (Fig. 5).

#### **Change in Fat Content**

Figure 6 illustrates the change in fat content of surface layer and core portions of large and small size coated meat balls. The surface layer of the chicken balls absorbed fat rapidly up to 90 s of frying and then reached a steady state value. Similar trends were observed in the small coated balls also. Reduction of fat content in core region was observed after 300 s of frying for large balls and 180 s of frying for smaller size balls. Since the fried poultry samples were air-cooled before the analyses, the data do not represent the instantaneous fat content of the samples. However, they represent the amount of fat retained in the product after cooking for specified time period.



For a large chicken ball, up to 60 s of frying, the fat absorption pattern for coated and uncoated balls was similar (with uncoated absorbing more fat). However, after 60 s, the rate of fat absorption by the uncoated samples was higher than that of coated samples (Fig. 7). In the surface layer, 13.1 to 17.9% reduction in fat was observed for coated balls in comparison to the control (p < 0.05). Similarly a fat reduction of 26.2 to 33.7% was observed in the core (p < 0.05).



FIG. 7. EFFECT OF COATING ON FAT CONTENT FOR LARGE CHICKEN BALLS

A major functional property of HPMC film is the capability to form thermal gels. The methyl groups on HPMC molecules undergo intermolecular association with adjacent HPMC molecules at temperatures above the incipient gelation temperature (IGT). The IGT is the temperature at which intermolecular association begins to occur very rapidly. Above the IGT, dramatic viscosity increases occur with increasing temperature, to the point where the solution gels (Meyers 1990). For HPMC, temperature is in the range of 50 to 90C (depending on degree of substitution, concentration, etc.). The surface layer temperature of large coated ball rapidly increased and reached about 93C after 40 s frying (Fig. 2) implying that the gel formation had occurred by this time. This gel layer

might have controlled the transfer of moisture and fat between the product and the frying medium. On the other hand, uncoated ball continuously lost moisture and might have absorbed the fat.

#### CONCLUSIONS

Relative to control, the coating of edible films on chicken balls reduced the fat absorption in the surface layer (up to 17.9% reduction) and the core (33.7% reduction). The coated samples also showed improved moisture retention (up to 8.7% in surface layer and 16.4% in the core) in comparison to that of control. The moisture retention and fat reduction capabilities of HPMC film were probably due to its ability to form a thermal gel to protect their content during frying.

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# THERMOPHYSICAL PROPERTIES OF FRESH AND ROASTED COFFEE POWDERS<sup>1</sup>

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### ABSTRACT

The present work deals with experimental determination of bulk specific heat, bulk thermal conductivity, bulk and true density and moisture content of Columbian and Mexican coffee bean powders. Specific heat was determined by DSC, thermal conductivity by modified Fitch apparatus, density by stereopycnometer and moisture contents by standard vacuum oven method. Specific heat values showed marginal rise over the temperature range (45 to 150C) studied. Thermal conductivity values, determined in the temperature range of 20-60C were not found to have any definite trend with rise in moisture in the experimental range of 4.8 to 9.8% moisture (dry basis). Bulk density of powders varied appreciably (1.298 to 0.882 g.cm<sup>-3</sup>), while the change was negligible for true density (1.361 to 1.092 g.cm<sup>-3</sup>) with the degree of roasting. Statistical analysis of the data shows the reliability of the experimental determinations. Suitable correlations were developed to determine bulk specific heat and bulk density.

#### **INTRODUCTION**

The total coffee production in the world today is 6.3 million tons per year, out of which, the US alone consumes 1.2 million tons. The coffee in the US is mostly imported from Brazil, Columbia and Mexico at a total cost of \$1900 million (Anon. 1993). Coffee roasting has been a major food industry in the US, although it has received considerable attention only recently because of the possible pollution problems associated with roasters.

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According to a recent market survey (Anon. 1994), opacity or smoke emissions are the major concerns for the coffee roasters. It is expected that they will shortly be switching over to a better design for burners and control for emissions. Although it is an old industry, only recently the EPA has categorically identified some volatile organic compounds (VOC) from the coffee roasters which pollute the atmosphere. Nitrous oxides, carbon monoxide, particulates and odor are other objectionable and harmful effluents from the coffee roasters which are attracting the attention of researchers.

Thermophysical properties of coffee beans are important for any elaborate study, design or operation of the roasters. In order to improve the operation of existing roasters by retrofit combustion system or to design a new system all together will need thermophysical properties of the beans as the basic data. Considerable literature on determination of specific heat and thermal conductivity of different vegetables, fruits, nuts, raisins and some beans are available, but not for coffee beans. Specific heat and thermal conductivity of coffee beans have not been reported probably because of their hardness and irregular shape and size, not suitable for either DSC or Probe method or modified Fitch method.

Since the US markets use coffee mainly from Brazil, Mexico and Columbia, the main objective was to determine thermophysical properties of typical coffee beans from these countries. Due to limitations of the experimental setups, beans could not be taken for studies directly. Beans from Brazil could not be obtained at the time of experiments, therefore, the present work describes elaborate experimental determination of the following thermal and allied physical properties of Mexican and Columbian coffee (genus *Coffea* of the *Rubiaceae* family) powders: (1) bulk specific heat, (2) bulk thermal conductivity, (3) true and bulk density and (4) moisture content. It is expected that the data obtained in the present investigation can be used for design of equipments and processes in the coffee industries.

# MATERIALS AND METHODS

#### **Bulk Specific Heat**

**Principle of DSC Measurements**. The heat flow H to the sample is equal to the difference between the two heat flows  $Q_s$  and  $Q_r$ 

$$H = Q_{r} - Q_{r} \tag{1}$$

Here subscripts s and r refer to the sample and reference, respectively. According to the thermal analogy of Ohms law,

$$Q = (T_2 - T_1) / R_{th}$$
 (2)

i.e., the heat flow is proportional to the driving force (temperature difference,  $T_2-T_1$ ) and inversely proportional to the thermal resistance  $R_{th}$ . Applied to the DSC cell it follows that

$$H = Q_{s} - Q_{r} = \left(\frac{T_{c} - T_{s}}{R_{th}}\right) - \left(\frac{T_{c} - T_{r}}{R_{th}}\right)$$
(3)

where  $T_c$  is the furnace temperature.

Because of symmetry, the two  $T_c$  and the two  $R_{th}$  values are identical, and

$$H = \frac{(T_s - T_r)}{R_{th}}$$
(4)

As the temperature difference  $\Delta T = T_s - T_r$  is measured by the sensor thermocouple, it follows from the thermocouple equation that

$$\Delta U = S \Delta T \tag{5}$$

where S is the sensitivity of the thermocouple (microvolts/degree C).  $\Delta U$  is the difference in the electrical signal from source and the reference (microvolts).

Thus

$$H = \frac{\Delta U}{R_{tb}S}$$
(6)

 $R_{th}$ ,S=E is called calorimetric sensitivity, which can be divided into a temperature dependent relative term ( $E_{rel}$ ) and a temperature independent term ( $E_{in}$ ). For the cell being used in the present setup,  $E = E_{rel}$ , $E_{in}$ . Thus finally,

$$H = \frac{\Delta U}{E_{in} \cdot E_{rel}}$$
(7)

The temperature dependence of  $E_{rel}$  is contained in the TA processor as a hard programmed polynomial,

$$\mathbf{E}_{\rm rel} = \mathbf{A} + \mathbf{B}\mathbf{T} + \mathbf{C}\mathbf{T}^2 \tag{8}$$

where A, B, C are regression constants. On the other hand,  $E_{in}$  is determined by calibration, which is about 11  $\mu$ V/mW for the standard sensor in the present case.

The specific heat at constant pressure,  $C_p$  of a sample can be determined as a function of temperature by DSC. This function can either be presented graphically or printed out in a table.

The heat flow to the sample, measured at a constant heating rate, is directly proportional to the specific heat of the substance. In order to obtain precise  $C_p$  values, a correction must be applied for the additional heat flow arising from difference in weight of sample pan and reference pan. With the help of preliminary blank experiment, the correction is automatically made. The weights of both sample and blank pan enter into the calculation as does the Cp function for aluminum. At the same time, the blank correction compensates for any asymmetry in the measuring system.

**Experimental Procedure**. The coffee beans (fresh, roasted for 3, 6, 9, 12, 13.5 and 15 min) were supplied from a gourmet roaster in Rockford, IL. The beans were received in sealed plastic packets. They were ground to fine powders in a blender. The powders sieved between numbers 35 and 40 USA standard testing sieves (-32 +35 Tyler equivalent mesh,  $425-500\mu$ m size) were retained for the testing purposes. The powders kept in sealed plastic packets were stored away from moisture. Specific heat measurements were done only for the coffee powders, since beans could not be used in the present setup of DSC.

Differential Scanning Calorimeter (DSC) (Mettler, Model: DSC30) with Mettler TC11 TA Processor hooked to an IBM personal computer and Epson FX-850 printer and Hewlett Packard 7475A plotter was used for determining bulk specific heat of coffee powder samples. The DSC was checked for its accuracy of temperature measurement by test runs at a heating rate of 1C/min and using indium, zinc and lead as standards. The 2-5 mg samples were taken and sealed in aluminum pans. The sample pan was measured against a reference pan in all DSC measurements. An empty pan, similar in weight and shape to the sample pan with perforated lid was used as a reference.

After opening the DSC cell, the sample pan was placed in the left hand measurement position so that the center pin of the pan was located in the hole surrounded by the thermocouples. The reference pan was inserted in the right hand measurement position. As soon as the pan was inserted, the furnace lid was placed over the furnace opening. The cell cover was closed and locked. A heating rate of 10C/min was used for all the coffee powder samples.

The data logger was started to record specific heat as a function of temperature. It was observed that the aluminum pans ruptured beyond 150C due to excessive pressure developed in the pans because of moisture in the powders. The specific heat determinations were, thus not possible beyond 150C.

#### **Bulk Thermal Conductivity**

The present work deals with the bulk thermal conductivity determination of coffee powders using a modified Fitch method.

Theory and Assumptions. The quasi-steady conduction heat transfer through the sample yields the following simplified equation (Mohsenin 1980; Rahman 1991)

$$\frac{A.K(T_i - T_f)}{L} = M_c.C_{p_c}.\frac{dT}{dt}$$
(9)

with the initial condition

at 
$$t=0, T = T_o$$

the solution to Eq. (9) is

$$\ln \left(\frac{T_o - T_f}{T_i - T_f}\right) = \frac{A.K.t}{L.M_c.Cp_c}$$
(10)

where: = heat transfer area perpendicular to the direction of heat flow A  $(m^2)$ Κ = thermal conductivity of sample  $(Wm.^{-1}.C^{-1})$ T. sample temperature at any time (°C) = T<sub>o</sub> = initial sample temperature ( $^{\circ}C$ )  $T_f =$ temperature of the source (°C) L = sample thickness (m)  $M_c = mass of copper plug (kg)$  $C_{pc}$  = specific heat of copper plug (J.kg<sup>-1</sup> C<sup>-1</sup>) time (s) ==

Obviously, the thermal conductivity of the sample (K) can be calculated by determining the slope of the straight line plot of  $\ln \left(\frac{T_o - T_f}{T_i - T_f}\right)$  vs t.

The precision of the result depends upon proper application of Eq. (9). If quasi steady-state conditions are maintained, the equation gives satisfactory results.

**Construction and Details of the Apparatus**. The device is a modification of the Fitch apparatus (Mohsenin 1980; Rahman 1991; Zuritz *et al.* 1989). It consists of a constant temperature, insulated container with a specially designed stopper and an insulated copper plug. A schematic diagram of the device is as shown in Fig. 1. The main container, insulated from sides and top and due to large thermal mass, acts as a heat reservoir to heat the source copper rod and maintain it at a constant temperature throughout the experimental run. The copper plug at the bottom acts as the heat sink. This design ensures a sufficiently small heat transfer surface for particle heat transfer. Polystyrene



FIG. 1. MODIFIED FITCH APPARATUS

discs provided as guards insulate the copper rod source and copper plug sink from the surroundings. Thermocouple wires were installed in the copper rod about 2 mm from the exposed end for measurement of source temperature. The exposed end of the copper plug was machined to 15 mm diameter and thermocouple wires were soldered to it to measure the temperature of the sink. Three screw-holes in the copper plug insulation were provided to keep the hexagonal aluminum support and the whole set up in position during the experimental runs. This apparatus was designed for samples with about 15 mm diameter. For samples with smaller diameters, smaller plugs should be designed and installed (Singh and Singh 1994).

**Experimental Procedure**. The apparatus was checked for its performance by determining thermal conductivity of some known samples, viz., glass, carrot, potato and apple.

A sample was first prepared carefully by cutting it in the form of a circular disk. The diameter of the samples was always 14-15 mm and the thickness 2-3 mm. The sample was kept between the source rod and the copper plug taking special care not to crush it. The temperature of the sample and the copper plug was allowed to stabilize to a constant value. Hot water was poured into the container which was quickly closed at the top. The temperature of the copper rod quickly attained a constant value. The data logger was started to record the temperature at time intervals of 10 s.

After 4-5 min, the data logging was stopped and the temperature of the source and sample were printed out. The calculations were performed and a plot of  $\ln[(T_f - T_o)/(T_f - T_i)]$  vs t was prepared. The data points were fitted with linear regression analysis. From the slope of these plots and knowing the values of specific heat and mass of copper plug, the thermal conductivities of these samples were determined. It was found that the apparatus gave thermal conductivities of these samples within  $\pm 5\%$ .

In order to determine the bulk thermal conductivity of coffee powders, samples were taken in the groove provided above the copper plug (about 2 mm deep). The procedure was repeated as for standard samples and temperature-time history curves were plotted. Every care was taken to keep the coffee powder uniformly distributed over the copper plug, to make a smooth surface for heat transfer.

#### **True and Bulk Density**

The true density of coffee powders was determined by stereopycnometer. The equipment is basically designed to measure the volume of solid objects. This is accomplished by employing Archimedes principle of fluid displacement to determine the volume. The displaced fluid in the present case was helium which can penetrate the finest pores to assure maximum accuracy. Helium, having small atomic dimensions, assures penetration into crevices and pores approaching one Angstrom  $(10^{-10} \text{ m})$ .

**Theory**. Assuming that the empty and sealed sample cell volume is  $V_c$  and by opening the vent toggle valve the system is brought to ambient pressure ( $P_a$ ) after being purged with helium. By ideal gas law:

$$\mathbf{P}_{\mathbf{a}} \cdot \mathbf{V}_{\mathbf{c}} = \mathbf{n} \cdot \mathbf{R} \mathbf{T}_{\mathbf{a}} \tag{11}$$

where, n is the number of moles of gas occupying volume  $V_c$  at pressure  $P_a$ .  $T_a$  is the ambient temperature and R is the gas constant.

When a solid sample of volume  $V_p$  is placed in the sample cell, the above equation modifies to

$$P_{a}(V_{c}-V_{p}) = n_{1}.R.T_{a}$$

$$(12)$$

when pressurized to about 17 psi  $(1.1951 \text{ kg. cm}^{-2})$  above ambient, the state of the system is given by

$$P_2(V_c - V_p) = n_2.RT_a$$
 (13)

where  $P_2$  indicates a pressure above ambient and  $n_2$  represents the total number of moles of gas contained in the sample cell.

When the rotary value is turned to connect the added volume  $V_A$  to that of the cell, the pressure will fall to a lower value  $P_3$ , given by

$$P_{3}(V_{c}-V_{p}+V_{A}) = n_{2}RT_{a}+n_{a}.RT_{a}$$
 (14)

where  $n_a$  is the moles of gas contained in the added volume at ambient pressure. Finally, when  $P_a$  is made to read zero, i.e. all pressure measurements become relative to  $P_a$  which is zeroed prior to pressurizing, Eq. 11-14 may be manipulated to give

$$V_{p} = V_{c} + \frac{V_{A}}{1 - P_{2}/P_{3}}$$
(15)

which is the working equation for the stereopycnometer.

**Experimental Procedure**. Standard method was followed to measure true volume of coffee powders by stereopycnometer using helium as the displaced fluid. The true volume of the powders calculated by Eq. (15) was used for the determination of true or particle density. Bulk density was determined by accurate measurement of bulk volume of a sample of known mass using measuring cylinder.

## **Moisture Content**

Moisture content of coffee powders was determined by standard method.

**Experimental Procedure**. The samples were taken in an aluminum dish of known weight and weighed carefully. The difference gave the weight of the sample. The sample in the dish was kept overnight in vacuum oven maintained at 70C. At the end, the dish was taken out of the oven, kept in a desiccator, allowed to cool for about 30 min and then again weighed. This gave the weight of the dry sample. The moisture was calculated on dry or wet basis.

## **RESULTS AND DISCUSSION**

#### **Specific Heat**

Table 1 summarizes the average values of specific heat for different coffee powders in a temperature range of 45 to 150C. The values in the table show a definite trend of marginal rise with temperature. A linear regression of the data gave following correlations:

$$C_p = 0.007554 T + 0.9210 (R^2 = 0.973)$$
 (16)

for Mexican coffee powders, and

$$C_p = 0.006459 \text{ T} + 1.2729 (R^2 = 0.976)$$
 (17)

for Columbian coffee powders.

The above correlations are similar to correlations given by Andrieu *et al.* (1989) for pure durum wheat starch (slope = 0.005737 and intercept = 1.328); by Rask (1989) for dried dough (slope = 0.00486 and intercept = 1.114); Choi and Okos (1983) for insoluble solids of tomato juice (slope = 0.003284 and intercept = 1.16602); and Noel and Ring (1992) for dry amylopectin (slope = 0.00571 and intercept = 1.050).

## **Thermal Conductivity**

It is to be noted from Eq. (10) that the slopes of the temperature-time history plots are given by

slope = 
$$\frac{A.K}{L.M_c.C_{p_c}}$$
 (18)

or,

$$K = M_{c} \cdot C_{p_{c}} \cdot \left(\frac{L}{A}\right) \text{ .slope}$$
(19)

since,  $M_{\rm c}$  and  $C_{\rm pc}$  are constant for all the runs, it can be written as

$$K = const.(L/A).slope$$
 (20)

(00)

in other words,

$$\frac{K_{coffee}}{K_{standard}} = \frac{L_{coff}}{L_{stand}} \cdot \frac{A_{stand}}{A_{coff}} \cdot \frac{(slope)_{coff}}{(slope)_{stand}}$$
(21)

# TABLE 1.

# SPECIFIC HEAT OF COFFEE POWDERS AT DIFFERENT TEMPERATURES

		Mexican Bean				
Sample	Specific Heat (J/g K)					
Temp. °C	Mexican Fres	h Ro	asted 6 min	Roast	ed 9 min	Roasted 15 min
	1.10					1.00
45	1.19		1.34		1.10	1.38
50	1.25		1.47		1.19	1.47
60	1.27		1.49		1.34	1.63
70	1.33		1.52		1.32	1.60
80	1.41		1.56		1.40	1.56
90	1.54		1.61		1.46	1.73
100	1.65		1.66		1.52	1.81
110	1.71		1.74		1.59	1.83
120	1.82		1.80		1.64	1.85
130	1.98		1.89		1.75	1.84
140	2.22		2.00		1.90	2.04
150			2.46		1.99	1.90
		Columbian Bean				
			Specific	Heat (J/g K)		
Sample	Columbian	Roasted	Roasted	Roasted	Roasted	Roasted
Temp. °C	fresh	3 min	6 min	9 min	12 min	13.5 min
45	1 77	1 64	1.62	1.70	1.29	1 33
50	1.80	1 71	1.69	1 79	1 36	1 39
60	1.80	1 77	1.71	1.81	1 45	1 48
70	1.85	1.86	1 75	1.86	1 46	1 48
80	1.00	1.00	1.80	1.80	1.52	1.52
90	1.97	2.18	1.87	1.86	1.60	1.61
100	2.05	2.10	1.07	2 01	1.68	1.68
110	2.05	2.05	2.13	2.01	1.00	1.75
120	2.15	2.20	2.15	2.10	1.74	1.75
120			2.24	2.22	1.87	1.78
140			2.80	2.51	2.04	1.80
140				2.44	2.04	1.00

40

Thus, the bulk thermal conductivity of coffee powders can be directly calculated, knowing the thermal conductivity of standard samples (glass, carrot, potato and apple). This method gives the bulk thermal conductivity value within 5 min with simple calculation, especially if the thickness and area of the sample are similar to the standard, the ratio of thermal conductivities will simply be the ratio of the slopes of the temperature-time history curves (Eq. 21). This eliminates any error caused by the apparatus. As long as the experiments with standard and coffee samples are conducted under identical conditions, the method gives excellent results.

A typical temperature-time plot for fresh Mexican coffee powder is presented in Fig. 2. Linear regression of the data gave slope = 0.00128, intercept = 0.0178 and  $R^2 = 0.996$ . The data for other samples were also regressed and found to have values of  $R^2$  always more than 0.975. From the slope of these plots, the values of bulk thermal conductivity were determined as discussed earlier.



FIG. 2. A TYPICAL TEMPERATURE-TIME HISTORY PLOT FOR FRESH MEXICAN COFFEE POWDER

Tables 2 and 3 present average values of bulk thermal conductivity of standard samples and coffee samples respectively in the temperature range of 20-60C. It can be observed from Table 3 that the values of bulk thermal conductivity do not have any specific trend and a correlation could not be obtained between thermal conductivity and moisture. The values are very close to each other. Statistical analysis of the data show that the standard deviation for Columbian coffee powders is 0.00705 with percent coefficient of variation = 5.42%. The values can be improved to 0.00509 and 3.91% respectively, if the last value of thermal conductivity (0.117) was omitted. The standard deviation for Mexican coffee powders was found to be 0.00556 with coefficient of variation of 4.38%. Thus the maximum deviation of the bulk thermal conductivity values from the mean varied to within  $\pm/-5.5\%$ .

It is to be noted that the values of bulk thermal conductivity are low, and they could not be compared with any other values, since there is no literature on thermal conductivity of coffee beans/powders. Values of thermal conductivity of 0.1 to 0.2 Wm<sup>-1</sup> C<sup>-1</sup> for kidney beans (Zuritz *et al.* 1989) and 0.15 to 0.32 Wm<sup>-1</sup> C<sup>-1</sup> for corn kernels (Kustermann *et al.* 1981) have been reported.

## Moisture

The moisture content of different samples has been given in Table 3. The moisture of coffee powders was found to vary between 9.8% for fresh samples to 4.8% for the maximum roasted (15 min) samples. This small variation in moisture was not found to affect the overall values of bulk specific heat and bulk thermal conductivity to an appreciable extent.

Material	Moisture Content % Wet Basis	Average (3 samples) Thermal Conductivity (Wm <sup>-1</sup> C <sup>-1</sup> )
glass	-	1.05
carrot	89	0.52
apple	83	0.42
potato	81	0.54

 TABLE 2.

 AVERAGE THERMAL CONDUCTIVITY OF STANDARD SAMPLES

Specification	Thermal Conductivity (Wm <sup>-1</sup> C <sup>-1</sup> )	Moisture (% dry basis)
Columbian		
fresh	0.136	9.50
roasted 3 min	0.128	8.60
roasted 6 min	0.131	7.20
roasted 9 min	0.135	6.40
roasted 12 min	0.138	5.80
roasted 13.5 min	0.117	5.20
Standard deviation: 0	.00509 (neglecting last value, 0.117)	
Coefficient of variation	on: 3.91% (neglecting last value, 0.11)	7)
Mexican	on: 3.91% (neglecting last value, 0.11)	7)
Mexican fresh	0.123	9.80
Mexican fresh roasted 3 min	0.123 0.120	7) 9.80 8.20
Mexican fresh roasted 3 min roasted 6 min	0.123 0.120 0.131	7) 9.80 8.20 7.40
Mexican fresh roasted 3 min roasted 6 min roasted 9 min	0.123 0.120 0.131 0.122	7) 9.80 8.20 7.40 6.80
Mexican fresh roasted 3 min roasted 6 min roasted 9 min roasted 12 min	0.123 0.120 0.131 0.122 0.135	7) 9.80 8.20 7.40 6.80 5.20
Mexican fresh roasted 3 min roasted 6 min roasted 9 min roasted 12 min roasted 15 min	0.123 0.120 0.131 0.122 0.135 0.131	7) 9.80 8.20 7.40 6.80 5.20 4.80
Mexican fresh roasted 3 min roasted 6 min roasted 9 min roasted 12 min roasted 15 min	0.123 0.120 0.131 0.122 0.135 0.131	7) 9.80 8.20 7.40 6.80 5.20 4.80
Mexican fresh roasted 3 min roasted 6 min roasted 9 min roasted 12 min roasted 15 min	0.123 0.120 0.131 0.122 0.135 0.131	7) 9.80 8.20 7.40 6.80 5.20 4.80

TABLE 3. BULK THERMAL CONDUCTIVITY OF COFFEE POWDERS

# **True and Bulk Density**

Table 4 gives the average values of true and bulk density of coffee powders. It is to be noted that the true and bulk densities both vary linearly with moisture. Linear regression of the data gave the following correlations for determination of true density from moisture:

$$\rho = 0.051 \text{ X} + 0.8721 (\text{R}^2 = 0.950)$$
(22)

for Mexican coffee powders, and

$$o = 0.0628 X + 0.7624 (R^2 = 0.988)$$
(23)

for Columbian coffee powders.

Similarly, for the determination of bulk density, following two correlations were developed by linear regression of the data:

$$\rho_{\rm b} = 0.0848 \ {\rm X} + 0.496 \ ({\rm R}^2 = 0.900) \tag{24}$$

for Mexican coffee powders, and

$$\rho_{\rm b} = 0.1034 \text{ X} + 0.350 (\text{R}^2 = 0.970) \tag{25}$$

for Columbian powders.

Similar correlations were developed by Hsu *et al.* (1991) for pistachios. While, they also reported an increase in bulk density with moisture, they found the true density to decrease with an increase in moisture, an observation contradicting the present findings. The reason might have been the wide range of moisture (8.2% to 35.0%, wet basis) in their study. The decrease in true volume compared to the bulk volume was probably more significant than the decrease in weight, causing the true density of pistachios to increase with the decrease in moisture.

TABLE 4. TRUE AND BULK DENSITY AND BULK THERMAL DIFFUSIVITY OF COFFEE POWDERS

	Density	Density (g.cm <sup>-3</sup> )		
Material	Bulk	True	$\times 10^4$	
Mexican				
fresh	1.290	1.361	8.29	
roasted 3 min	1.280	1.311		
roasted 6 min	1.120	1.263	9.66	
roasted 9 min	1.010	1.190	9.66	
roasted 12 min	0.978	1.162		
roasted 15 min	0.872	1.099	12.01	
Columbian				
fresh	1.298	1.355	6.16	
roasted 3 min	1.285	1.302	6.55	
roasted 6 min	1.110	1.235	7.61	
roasted 9 min	0.980	1.152	8.50	
roasted 12 min	0.965	1.122	12.43	
roasted 13.5 min	0.882	1.092	10.61	

#### **Bulk Thermal Diffusivity**

The bulk thermal diffusivity of Mexican and Columbian coffee powders was calculated from experimentally obtained values of bulk thermal conductivity, bulk specific heat and bulk density as,

$$\alpha = K/\rho_b C_p \tag{26}$$

where, K = bulk thermal conductivity

 $\rho_{\rm b}$  = bulk density

 $C_p$  = bulk specific heat

 $\alpha$  = bulk thermal diffusivity

Since the above equation needs average values of specific heat in the temperature range of 20-60C (the range of temperature for thermal conductivity determination), the regression equations were used to determine the average value of specific heat. The values of bulk thermal diffusivity so obtained are also included in Table 4. Because thermal diffusivity is indirectly obtained from three thermophysical properties (K,  $C_p$  and  $\rho_b$ ), the trends exhibited by these properties are inherent in the values of thermal diffusivity. Table 4 shows that the values of thermal diffusivity do not have a specific trend, although, neglecting values corresponding to 12 min roasting and beyond, a mean value of  $8.06 \times 10^{-4}$  cm<sup>2</sup>/s (with standard deviation =  $1.279 \times 10^{-4}$  and coefficient of variation = 15.8%) could be obtained. A value of  $8 \times 10^{-4}$  cm<sup>2</sup>/s for bakery products by Rask (1989) and a value of  $9.5 \times 10^{-4}$  cm<sup>2</sup>/s for tomato solids by Choi and Okos (1983) was reported. Choi and Okos (1983) also reported percentage standard error of 29.5% in their values.

# ERROR ANALYSIS OF EXPERIMENTAL MEASUREMENTS

#### **Specific Heat**

Equation (7) can be rewritten to give bulk specific heat  $C_p$  as

$$C_{p} = S/E_{in}E_{rel}$$
(27)

The error in C<sub>p</sub> can be calculated by

$$dC_{p} = ((\partial C_{p}/\partial S.dS)^{2} + (\partial C_{p}/\partial E_{in}.dE_{in})^{2})^{1/2}$$
(28)

Realizing the fact that  $E_{rel}$  is given by Eq. (8) and A, B, and C are regression constants determined by the DSC automatically,  $E_{rel}$  was not included in the error analysis. The influence coefficients and the percentage error in  $C_p$  from Eq. (28) can be calculated by the values given in Table 5. This table is typical for Mexican coffee powder at 45C and gives 1.50% error. The error varies between 0.89% (minimum) and 1.50% (maximum) for other powders.

Parameter	value	Measurement Method	Measurement Error
Darameter	Value	Magguramant Mathod	Magguramant Ernen
	MEASUREMEN	T ERRORS IN BULK SPEC	IFIC HEAT
	VALUES, MEASUREME	ENT METHODS AND MAX	IMUM ESTIMATED
		TABLE 5.	

4				Ĩ
S	20 µ V/K	DSC	$0.1 \ \mu V/K$	
$\mathbf{E}_{in}$	11 $\mu$ V/m W	DSC	$0.1 \ \mu \ V/mW$	
T	5C	Thermocouple (DSC)	0.1C	

# **Thermal Conductivity**

Equation (10) can be rewritten as

$$K = M_c Cp_c L/At *ln((T_o - T_f)/(T_i - T_f))$$
(29)

The error in K can be calculated by

$$d\mathbf{k} = ((\partial \mathbf{K}/\partial \mathbf{M}_{c} d\mathbf{M}_{c})^{2} + (\partial \mathbf{K}/\partial \mathbf{C} \mathbf{p}_{c})^{2} + (\partial \mathbf{K}/\partial \mathbf{L} d\mathbf{L})^{2} + (\partial \mathbf{K}/\partial \mathbf{A} d\mathbf{A})^{2} + (\partial \mathbf{K}/\partial t dt)^{2} + (\partial \mathbf{K}/\partial \Delta \mathbf{T}_{o} d\Delta \mathbf{T}_{o})^{2} + (\partial \mathbf{K}/\partial \Delta \mathbf{T}_{i} d\Delta \mathbf{T}_{i})^{2})^{1/2}$$
(30)

Where

$$\Delta T_{o} = T_{o} - T_{f} \tag{31}$$

and

$$\Delta T_i = T_i - T_f \tag{32}$$

The influence coefficients and the percentage error in dK from Eq. (30) can be calculated with the values given in Table 6. The values of  $\Delta T_o$ ,  $\Delta T_i$  and t change with the samples. This table is typical for Mexican fresh coffee. The percentage error for this sample from Eq. (30) is 7.8%. The values for other samples varied between 6.0 to 8.2%.

TABLE 6. VALUES, MEASUREMENT METHODS AND MAXIMUM ESTIMATED MEASUREMENT ERRORS OF PARAMETERS FOR THERMAL CONDUCTIVITY OF MEXICAN FRESH COFFEE POWDERS

Parameter	Value	Measurement Method	Maxm. Measurement Error
Mc	0.0321 kg	Precision balance	10 <sup>-6</sup> kg
C <sub>pc</sub>	385 J kg <sup>-1</sup> C <sup>-1</sup>	DSC	8.5Jkg <sup>1</sup> C <sup>-1</sup>
A	1.767X10 <sup>-4</sup> m <sup>2</sup>	Micrometer	$10^{-7}m^2$
L	2X10 <sup>-3</sup> m	Micrometer	10 <sup>-5</sup> m
ΔΤο	25C	Thermocouples	0.1C
ΔΤί	21.5C	Thermocouples	0.1C
t	100 s	Datalogger	10 <sup>-6</sup> s

# **True and Bulk Density**

Equation (15) is the working equation for the determination of true volume of particles. The error in  $V_p$  can be calculated by

$$\frac{dV_p}{(\partial V_p/\partial P_2.dP_2)^2} + (\partial V_p/\partial V_A.dV_A)^2 + (\partial V_p/\partial P_2.dP_2)^2 + (\partial V_p/\partial P_3.dp_3)^{2}$$
(33)

The influence coefficients and percentage error in  $dV_p$  can be calculated with the values given in Table 7. The values of P<sub>2</sub> and P<sub>3</sub> change with the samples. This table is a typical one giving values for Mexican coffee powder (fresh). The error for this powder in true particle volume determination is 14.2%. The error for other samples varies between 12.4% to 19.6%.

TABLE 7. VALUES, MEASUREMENT METHODS AND MAXIMUM ESTIMATED ERRORS IN TRUE AND BULK DENSITY MEASUREMENTS FOR MEXICAN FRESH COFFEE POWDERS

Parameter	Value	Measurement Method	Max. Measurement Error	
Vc	37.66 cm <sup>3</sup>	Pycnometer constant	0.05 cm <sup>3</sup>	
V,	78.47 cm <sup>3</sup>	Pycnometer constant	$0.05 \text{ cm}^3$	
$P_2$	17.00 psi	Pycnometer	0.01 psi	
P <sub>3</sub>	15.00 psi	Pycnometer	0.01 psi	
Vb	16.50 cm <sup>3</sup>	Measuring cylinder	$0.50 \text{ cm}^3$	
m	21.285 g	Precision balance	0.01 g	

The bulk density was calculated by simply dividing mass of a sample by its bulk volume. The error introduced in the measurement of bulk density can be calculated by

$$d\rho_{\rm b} = ((\partial \rho_{\rm b}/\partial m.dm)^2 + (\partial \rho_{\rm b}/\partial V_{\rm b}.dV_{\rm b})^2)^{1/2}$$
(34)

The influence coefficients and the percentage error in  $d\rho_b$  can be calculated with the values given in Table 7. The values of mass, m, and bulk volume,  $V_b$  change with the samples. The values in this table are for fresh Mexican coffee powder. The error in bulk density for this powder is 3.06%. The error for other powders vary between 2.60% to 6.02%.

## CONCLUSIONS

Bulk specific heat, bulk thermal conductivity, bulk density, bulk thermal diffusivity and moisture content of Columbian and Mexican coffee powders were determined experimentally. Detailed statistical analysis of the data was done.

Useful correlations were developed for determination of bulk specific heat at a temperature in the range of 45 to 150C, true density and bulk density as a function of moisture in the range of 4.8 to 9.8% moisture (dry basis). No correlation was found for thermal conductivity and thermal diffusivity variation with moisture in the observed range. The error analysis of the data showed that the experimental values had good precision. The values of the thermophysical properties reported in the present investigation should be useful for better design of coffee roasters and equipments/processes dealing with coffee powders.

# NOMENCLATURE

Α	Heat transfer area in Eq. $(9)$ $(m^2)$
C <sub>p</sub>	Bulk specific heat at constant pressure (Jg <sup>-1</sup> C <sup>-1</sup> )
E	Calorimetric sensitivity ( $E = Ein.Erel$ )
$\mathbf{E}_{in}$	Constant (11 $\mu$ Vm W <sup>-1</sup> )
E <sub>rel</sub>	$A+BT+CT^2$ (A, B and C are regression constants)
Н	Heat flow rate (Js <sup>-1</sup> )
Κ	Bulk thermal conductivity (Wm <sup>-1</sup> C <sup>-1</sup> )
L	Sample thickness (m)
m	Mass of sample (kg)
M <sub>c</sub>	Mass of copper plug (kg)
n, n <sub>1</sub> , n <sub>2</sub>	Number of moles of gas
n <sub>a</sub>	Number of moles of gas in added volume
$P_2, P_3$	Pressure in pycnometer (Kpa)
Pa	Ambient pressure (Kpa)
Q	Heat flow rate (Js <sup>-1</sup> )
R	Universal gas constant
R <sub>th</sub>	Thermal resistance (KsJ <sup>-1</sup> )
S	Sensitivity of thermocouple (mVK <sup>-1</sup> )
Т	Temperature (K)
t	Time (s)
$\Delta T$	Temperature difference (K)
ΔU	Difference in electrical signal (mV)
V <sub>c</sub>	Sample cell volume (m <sup>3</sup> )
V <sub>p</sub>	Sample volume (m <sup>3</sup> )
X	Moisture content (% dry basis)
α	Thermal diffusivity (m <sup>2</sup> s <sup>-1</sup> )
ρ	True density (kg m <sup>-3</sup> )
$ ho_{ m b}$	Bulk density (kg m <sup>-3</sup> )

# Subscripts/Superscripts

1	Position 1
2	Position 2
а	Ambient
b	Bulk
с	Copper in Eq. (9)
с	Furnace in Eq. (3)
coff	Coffee
f	Final
f	Source in Eq. (9)
i	Initial, Any time
0	Initial
r	Reference
s	Sample
stand	Standard
th	Thermal

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# PREDICTING INTERNAL TEMPERATURE RESPONSE TO CONDUCTION-HEATING OF ODD-SHAPED SOLIDS

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# ABSTRACT

Heat penetration experiments were carried out in a hot water bath with small blocks of a solid polycarbonate material cut into various shapes (cylinder, cube and oval) to obtain heat penetration factors for each shape. These factors, along with characteristic dimensions from the odd shapes were then used in the finite cylinder heat transfer model to predict accurately the temperature at the center of each block in response to measured dynamic bath temperatures. Results showed sufficiently good agreement between predicted and measured center temperature profiles to justify use of the finite cylinder model for rapid evaluation of process deviations in thermal processing of foods in odd-shaped containers.

## INTRODUCTION

Numerical heat transfer models capable of simulating the thermal processing of canned foods have gained increasing importance with the realization of their potential application to intelligent on-line computer control of retorts in the food canning industry (Teixeira and Shoemaker 1989; Teixeira 1989). Teixeira *et al.* (1975) were the first to demonstrate that a powerful feature of numerical solutions by finite difference was the ability to accurately predict internal

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temperatures in response to any dynamic temperature change that might occur at the container surface. The use of such a model for on-line correction of process deviations was first proposed by Teixeira and Manson (1982), and later demonstrated by Datta *et al.* (1986). In order to overcome the limitation to strict conduction heat transfer, Bichier *et al.* (1994) modified the Teixeira model to deal effectively with the thermal processing of canned foods under mechanical agitation, but considered only finite cylinder shaped food containers.

The recent introduction of shelf stable prepared foods in novel-shaped lunch bowls and dinner trays along with more established one-half steam table trays and flexible retort pouches has prompted growing interest in the application of heat transfer models capable of simulating thermal processes in such odd-shaped containers. Manson *et al.* (1974) were the first to demonstrate the use of "equivalent cylinders" in a finite cylinder model to simulate thermal processes in pear-shaped containers, and hinted that this "equivalent cylinder" concept could apply to any nonstandard geometry.

The purpose of this paper is to show how a numerical model for simulating the thermal processing of canned foods in traditional cylindrical containers can be used without further modification to accurately predict the product temperature at the center or cold spot of any odd-shaped containers in response to either constant or dynamic retort temperatures.

#### THEORY

Existing heat transfer models, whether based on analytical or numerical solutions to the second order partial differential equation that describes heat conduction in a finite cylinder, make use of the heat penetration factor  $(f_h)$ determined from heat penetration tests along with cylindrical container dimensions of height and diameter to compute a working value of thermal diffusivity that allows the model to accurately predict center temperatures in response to boundary conditions (retort temperature). These models should be expected to perform equally well on any container shape when f<sub>h</sub> for an oddshaped container is used along with characteristic dimensions of a "phantom" finite cylinder to represent the actual container used in the heat transfer model. In this way, the model would compute a working value of thermal diffusivity that, although fictitious, when used with the assumed "phantom" cylinder dimensions, would allow accurate prediction of product center temperature in the actual odd-shaped container. This concept is similar to the "equivalent cylinder" concept employed by Manson et al. (1974) who simulated a pearshaped container with a finite cylinder model.

This working value of apparent thermal diffusivity ( $\alpha$ ) becomes the crucial factor in model performance because it dictates the overall rate of heat transfer from the retort environment at the boundary to the location at which the heat

#### **ODD-SHAPED SOLIDS**

penetration test data were taken (usually the container cold spot or geometric center). By entering into the finite cylinder model the heat penetration factor  $(f_h)$  for the cold spot in an odd-shaped container along with the radius and height of a fictitious or "phantom" circular cylinder, a working value of thermal diffusivity will be computed for use in the finite cylinder model that will cause the cold spot of the "phantom" cylinder in the model to respond to dynamic boundary conditions in precisely the same way as it would in the true odd-shaped container from which the heat penetration data were obtained.

The phantom cylinder dimensions could be arbitrary, but selection should reflect reasonable approximation of equivalent thermal capacitance. The shortest dimension is usually considered the thickness or height of the "phantom" circular cylinder (Manson *et al.* 1974), while the longest dimension or arithmetic average of the remaining shortest and longest dimensions represents the characteristic diameter of the "phantom" circular cylinder. Fig. 1 shows equivalent "phantom" circular cylinders for cube and oval-shaped blocks, along with the equation used to determine the working value of  $\alpha$  from cylinder dimensions and  $f_h$ .

## MATERIALS AND METHODS

In order to test the above theory, heat penetration experiments were carried out in a hot water bath with small blocks of a polycarbonate material (Lexan<sup>®</sup>) cut into three shapes; cylinder (height 25.2mm, diameter 25.4mm), oval (major axis 65.1 mm, minor axis 38.3 mm, thickness 26.1 mm), and cube ( $25.4 \times 25.6 \times 25.4$  mm). Insulated 36-gage thermocouples were inserted at the center of each block. Prior to each test, the blocks were immersed in 4C water to equilibrate at a uniform initial internal temperature. Once the water bath had reached a constant controlled temperature of approximately 98C, the blocks were promptly immersed into the hot water bath. Heating was continued until the center temperature of the slowest heating block (i.e. oval shape) reached within 5C of the water bath temperature. The test blocks were then promptly immersed in cold water at 4C to cool.

Water bath and block center temperatures were recorded using a computerized data acquisition system. The recorded data were analyzed with an appropriate spreadsheet software to calculate  $f_h$  and apparent thermal diffusivity. Heat penetration tests were replicated three times. To vary the block surface temperature, the entire rack which supported the test blocks was quickly transferred from one water bath at one temperature to another at a different temperature at specified time intervals followed by a return to the original water bath to recover the original boundary temperature. This transfer was repeated twice to simulate double process temperature deviations during a heating period. It may be argued that the equation shown in Fig. 1 for estimating thermal diffusivity from heating curve slope and container dimensions is valid only when an infinite surface heat transfer coefficient can be assumed, such as under steam heating in retort processing, and should not be used for heating in a water bath. This argument is true when an accurate value of actual thermal diffusivity is needed as a thermal property of the material being heated.

**EQUIVALENT "PHANTOM"** TRUE SHAPE CIRCULAR CYLINDER From which heat penetration parameter  $(f_{h})$ For which phantom thermal diffusivity " $\alpha$ " is determined at geometric center. is calculated: 0.398  $\alpha = \frac{0.12}{(1/R^2 + .427/H^2)f_h}$ CUBE a R = a/2H = a/2а h (b + c)/2а OVAL R = (b + c)/4H = a/2

FIG. 1. SCHEME FOR CONVERTING ANY ODD-SHAPED FOOD CONTAINER INTO AN EQUIVALENT "PHANTOM" RIGHT CIRCULAR CYLINDER FOR USE IN NUMERICAL HEAT TRANSFER MODEL OF A FINITE CYLINDER

In this application, however, the value obtained is more of an "apparent" thermal diffusivity that may also take into account the thermal capacitance of the can wall and small impedances to heat transfer at the boundary in addition to

internal product resistance. Thus, if a heat penetration test is conducted under the same heating conditions to be used in processing, the resulting slope of the semilog heat penetration curve will produce an apparent thermal diffusivity that enables the model to predict the correct internal temperature response to conditions outside the container boundary in the heat exchange medium. For this reason, heat penetration data obtained from water bath heating experiments should not be extrapolated to steam heating in retorts.

The f<sub>k</sub>-values obtained from the constant temperature heat penetration tests for each block were then used in the finite cylinder numerical heat transfer model with "phantom" cylindrical dimensions to predict the temperature at the center of each block in response to the measured constant and dynamic bath temperatures. Both measured and predicted center temperature profiles were plotted on computer-generated graphs for visual comparison. The f<sub>b</sub> and j<sub>b</sub> values of each heating curve were determined from the slope and intercept of the straight line portion by a least square regression analysis. The j<sub>c</sub> value was determined by trial and error search with the simulation model as described by Manson (1992) and Bichier et al. (1995), while the cooling factor (f<sub>c</sub>) was assumed equal to the heating factor  $(f_b)$ . Additionally, process lethalities using the predicted and monitored sample temperature histories at the cold spot were calculated and compared for each block shape. For this purpose, a reference temperature of 100C with a Z-value of 10C was used for the lethal rate calculation in order to produce values appropriate for water bath heating at temperatures under 100C.

#### RESULTS

Heat penetration factors  $(f_h, j_h, j_c)$  are summarized in Table 1. Thermal diffusivities calculated from the  $f_h$  for the cylinder block agreed within 5% of the true thermal diffusivity of the polycarbonate as reported by the manufacturer,  $(1.1 \times 10^{-7} \text{ m}^2/\text{s})$ . This close agreement confirmed that heat transfer resistance at the block wall-water bath interface was negligible. Also, heat conduction along the thermocouple wires within the blocks had little effect on the  $f_h$ , but was likely responsible for the unexpectedly low values of heating and cooling lag factors ( $j_h$  and  $j_c$ ). Normally, the lag factor for pure conduction heat transfer at the geometric center of such blocks should be approximately 2.0. The values reported in Table 1 suggest that the center temperature profiles obtained from thermocouple measurements will show a shorter lag time in response to boundary condition changes than would be predicted on the basis of pure conduction heat transfer through the polycarbonate alone for these blocks.

SHAPE FACTORS	CUBE	OVAL	CYLINDER
DIMENSIONS	2.54 cm (all sides)	height = $2.54$ cm major = $6.51$ cm minor = $3.83$ cm	diameter = $2.54 \text{ cm}$ height = $2.54 \text{ cm}$
$f_h = f_c$	6.5 min	12.3 min	6.1 min
j <sub>h</sub>	1.3	1.3	1.1
j <sub>c</sub>	1.5	1.8	1.4
r <sup>2</sup>	0.998	0.998	0.994

TABLE 1. HEAT PENETRATION FACTORS FOR VARIOUS SHAPED BLOCKS OF POLYCARBONATE

Measured center temperature profiles of all three blocks in response to constant water bath temperature heating are shown in Fig. 2. Figures 3, 4 and 5 compare model predicted with experimentally measured center temperature profiles in the cylinder, cube, and oval-shaped block, respectively. Good agreement between measured and predicted profiles is readily apparent by visual inspection in all three cases. This is especially true in the temperature range above 85C that would account for most of the accumulated lethality during thermal processing. Maximum temperature errors found between measured and



FIG. 2. MEASURED TEMPERATURE PROFILES AT GEOMETRIC CENTER OF VARIOUS-SHAPED POLYCARBONATE BLOCKS HEATED UNDER CONSTANT WATER BATH TEMPERATURE



FIG. 3. EXPERIMENTALLY MEASURED AND MODEL-PREDICTED TEMPERATURE PROFILES AT THE GEOMETRIC CENTER OF A CYLINDER-SHAPED POLY-CARBONATE BLOCK HEATED UNDER CONSTANT BOUNDARY TEMPERATURE



FIG. 4. EXPERIMENTALLY MEASURED AND MODEL PREDICTED TEMPERATURE PROFILES AT THE GEOMETRIC CENTER OF A CUBE-SHAPED POLYCARBONATE BLOCK HEATED UNDER CONSTANT BOUNDARY TEMPERATURE



FIG. 5. EXPERIMENTALLY MEASURED AND MODEL PREDICTED TEMPERATURE PROFILES AT THE GEOMETRIC CENTER OF AN OVAL-SHAPED POLYCARBONATE BLOCK HEATED UNDER CONSTANT BOUNDARY TEMPERATURE

predicted temperature profiles for each block under constant temperature heating along with lethalities calculated from each profile are given in Table 2.

Similar results were obtained under dynamic bath temperature heating in which the tray of blocks was quickly moved from one water bath to another held at different temperatures. This was done twice during heating to simulate a thermal process experiencing two process temperature deviations. The measured center temperature profiles in response to this double deviation process are shown in Fig. 6 for all three blocks, while the comparison of predicted with measured center temperature profiles is shown in Figs. 7, 8 and 9 for each block, respectively. Maximum temperature errors between measured and predicted profiles, along with lethality values from the measured and predicted temperature profiles for each block under these dynamic heating conditions are summarized in Table 3. Again, there is reasonably good agreement between measured and predicted results for all three blocks, although the agreement was closer with the oval and cylinder than with the cube.

The lack of perfect agreement stems from the limitation that the model assumes pure conduction heat transfer in a finite cylinder and responds to sudden boundary condition changes with a thermal lag factor (j value) of 2.04 for the

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# TABLE 2.

MAXIMUM TEMPERATURE ERROR AND PROCESS LETHALITIES FOR POLYCARBONATE BLOCKS HEATED UNDER CONSTANT WATER BATH TEMPERATURE (FIGS. 3, 4 AND 5)

Block Shape	Maximum Temperature Error	Process Lethality (min @ 100C for $Z = 10C$ )		
	in Range Above 85C (°C)	Experimentally Measured	Model Predicted	Difference (%)
Cylinder (Fig. 3)	0.55	9.1	9.3	2.2
Cube (Fig. 4)	1.05	5.1	5.2	2.0
Oval (Fig. 5)	0.77	3.0	3.1	3.3



FIG. 6. MEASURED TEMPERATURE PROFILES AT THE GEOMETRIC CENTER OF VARIOUS-SHAPED POLYCARBONATE BLOCKS HEATED UNDER VARIABLE BOUNDARY TEMPERATURE WITH DOUBLE DEVIATIONS IN WATER BATH TEMPERATURE HISTORY



FIG. 7. EXPERIMENTALLY MEASURED AND MODEL PREDICTED TEMPERATURE PROFILES AT THE GEOMETRIC CENTER OF A CYLINDER-SHAPED POLY-CARBONATE BLOCK HEATED UNDER VARIABLE BOUNDARY TEMPERATURE WITH DOUBLE DEVIATIONS IN WATER BATH TEMPERATURE HISTORY



FIG. 8. EXPERIMENTALLY MEASURED AND MODEL PREDICTED TEMPERATURE PROFILES AT THE GEOMETRIC CENTER OF A CUBE-SHAPED POLYCARBONATE BLOCK HEATED UNDER VARIABLE BOUNDARY TEMPERATURE WITH DOUBLE DEVIATIONS IN WATER BATH TEMPERATURE HISTORY



FIG. 9. EXPERIMENTALLY MEASURED AND MODEL PREDICTED, TEMPERATURE PROFILES AT THE GEOMETRIC CENTER OF AN OVAL-SHAPED POLYCARBONATE BLOCK HEATED UNDER VARIABLE BOUNDARY TEMPERATURE WITH DOUBLE DEVIATIONS IN WATER BATH TEMPERATURE HISTORY

TABLE 3.
MAXIMUM TEMPERATURE ERROR AND PROCESS LETHALITIES FOR
POLYCARBONATE BLOCKS HEATED UNDER DYNAMIC WATER BATH
TEMPERATURES WITH DOUBLE DEVIATIONS (FIG. 7, 8 AND 9)

Block	Maximum	Process Lethality			
Shape	Temperature Error	$(\min @ 100C \text{ for } Z = 10C)$			
	in Range Above 85C	Experimentally	Model	Difference	
	(°C)	Measured	Predicted	(%)	
Cylinder	0.39	2.75	2.78	0.9	
(Fig. 7)					
Cube	0.94	1.80	2.10	16.5	
(Fig. 8)			Ge.		
Oval	0.55	0.67	0.70	4.5	
(Fig. 9)					

geometric center of a finite cylinder. As shown in Table 1, the blocks used in these experiments revealed much lower j-values (lag times) explaining why measured center temperatures responded more quickly to the unexpected boundary temperature deviations than shown by the response predicted by the model. Of course, the actual lag factors ( $j_n$  and  $j_c$ ) are used in the model to adjust the lag time response at the beginning of planned heating and cooling which explains the near perfect agreement found under constant temperature heating. However, these factors are not brought into play when responding to unexpected deviations during the heating process allowing the normal response governed by pure conduction to prevail.

## CONCLUSION

These results show that a numerical heat transfer model for a finite cylinder such as that of Teixeira *et al.* (1969) can be used with reasonable success to predict cold spot temperature in containers of any shape, so long as the model is used to predict the temperature at the same internal location that was used to produce the heat penetration factors entered in the model. In applications where the nonuniform temperature distribution between container boundary and center is needed, shape-specific models in three dimensional space should be used instead. The method for using the equivalent "phantom" cylinder model on oddshaped containers described in this study can be applied to the calculation of thermal processes in odd-shaped containers, the evaluation of process deviations in odd-shaped containers, and for intelligent on-line computer control of retorts for automated thermal processing of odd-shaped containers; so long as process performance is based upon single point lethality criteria.

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## SELECTION OF VARIABLE RETORT TEMPERATURE PROCESSES FOR CANNED SALMON

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#### ABSTRACT

Variable retort temperature processes (VRT), in which retort temperature is a function of time, were developed for sterilization of Pacific salmon in 307  $\times$  115 steel cans, with objectives of minimizing loss of quality criteria such as surface quality or thiamine, or minimizing process time, all while maintaining constant center-point lethality  $(F_{o})$ . A finite difference computer model of conduction within a finite cylinder was used to test different temperature-time profiles. Processes were constrained to include steam vent schedules and to exclude temperature decrease during the heating process as well as temperature modulation during cooling, thereby improving compatibility with typical salmon canning facilities. Rho, the fraction of total lethality accumulated at steam-off time, was found to be a function of final unaccomplished temperature, retort temperature, salmon thermal diffusivity and container geometry. The search for favorable processes was aided by Random Centroid Optimization (RCO). Minimum surface cook in constant retort temperature (CRT) processes varied with retort temperature and z of surface quality, but the best VRT process was consistently better than the best CRT process. VRT reduced operator's process time from 64 min to 54 min and maintained equal  $F_{o}$  and surface quality. Thiamine losses were reduced from 19.6% loss by CRT to 16.8% loss by VRT.

#### **INTRODUCTION**

Almost all commercial retort processes for canned foods employ one retort temperature throughout the cook, making these constant retort temperature (CRT) processes. However, variable retort temperature processes (VRT) have been suggested as alternatives, especially for conduction heating foods (Teixeira *et al.* 1975; Banga *et al.* 1991; Almonacid-Merino *et al.* 1993). Benefits of VRT

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may include improved nutrient and flavor retention, reduced heat damage to product surface, lower energy costs or shorter process times. Because a large number of different VRT's are possible for a given product, selection of an optimum process is most easily found with a computerized experimental search technique. Given that retort experiments take several hours to complete, computer models of transient heating have been used as the medium for carrying out optimization trials of VRT processes. Kinetic equations describing the impact of time and temperature on the product allow the experimenter to evaluate bacterial lethality and product quality in the computer-simulated processes. Acceptable processes must achieve target lethality at the slowest heating spot, together with sufficiently low surface cook, or maximum nutrient retention, or minimum process time, etc. depending upon the aim of the experimenter. The VRT experimenter must identify parameter ranges that describe retort temperature as a function of time, then search the range for the best process. Search techniques have included the complex method (Almonacid-Merino et al. 1993), quasi-Newton multivariate optimization (Noronha et al. 1993), and Integrated Controlled Random Search for Dynamic Systems, a non-linear programming algorithm (Banga et al. 1991).

Some theoretically optimum VRT processes may be incompatible with present retort technology because they stipulate too rapid heating or cooling of the retort vessel, too high a retort temperature, etc. Also, steam retorts must be vented at the beginning of the process to remove air before uniform heating rates can be achieved within the load. Vent schedules are defined by a minimum vent time and a minimum achieved retort temperature, usually  $\geq 220F(104C)$ . Once a pure steam condition is obtained, temperature can be controlled with commercial computerized controller systems, but retort temperatures below about 220F (104C) have very little lethal effect on microbial spores and cannot be reliably maintained. Most canneries are not equipped to adjust cooling water temperature, making variable temperatures in the cooling cycle problematic. Banga et al. (1991) reported beneficial VRT processes which required control of retort temperatures throughout heating and cooling cycles. Almonacid et al. (1993) defined feasible VRT processes as those without negative steam consumption throughout the process and also reported significant benefits. Noronha et al. (1993) reported a VRT process that allowed a 28% reduction in process time of cylindrical cans with equivalent quality retention. No studies have reported the performance of VRT processes which included venting, nor VRT ramps restricted to the heating side of the thermal process.

This paper presents an efficient procedure for selecting VRT processes for conduction-heating canned foods, based on a finite difference model of transient conductive heat penetration. Random-Centroid Optimization (RCO) was used to search for the best process (Dou *et al.* 1993). The search was restricted to a subset of VRT's in which vent times and cooling cycles were compatible with

typical salmon cannery equipment. The objective was to determine whether canned salmon quality could be improved or process times decreased within these practical constraints.

#### **METHODS**

#### **Heat Penetration of Canned Salmon**

Pink salmon (*Oncorynchus gorbuscha*) was packed into  $307 \times 115$  cans, fitted with Ecklund nonprojecting thermocouple fittings, steam retorted, and center temperature histories were collected as previously described (Durance and Collins 1991). Average heating rate index ( $f_h$ ) and cooling rate index ( $f_c$ ) of cans were obtained from temperature histories of 15 cans. After correction for heat conduction along the thermocouple fitting (Ecklund 1956) temperature histories of cans were compared with a computer simulation of the same process.

#### Model of Unsteady-state Conduction Heating

Heat transfer within a cylindrical can during commercial retort sterilization was estimated with a two-dimensional finite difference model, as previously described (Sandberg *et al.* 1994) with modifications. Thermal diffusivity ( $\alpha$ ) of the food material was calculated as follows

$$\alpha = (0.398) / \{f_{\rm h} (1/r^2 + 0.427/b^2)\}$$

where  $f_h$  is the heating rate index determined in a retort trial, *r* is the radius of the can and *b* is the half-height. Thermal diffusivity of the cooling phase was adjusted by a factor of 1.112, to correct for changes in thermal properties of the salmon with cooking. The factor was chosen such that simulation of a CRT retort process yielded the same contribution to lethality in the cooling phase as the average contribution found in 15 heat penetration profiles from a retort trial. The model also controlled initial retort temperature, steam temperature, cooling water temperature, initial product temperature and final product temperature. Surface heat transfer coefficients for heating and cooling were 10,000 and 800 W/m<sup>2</sup> K, respectively. Steam-off condition was based on the accumulated center-point lethality at the time of steam-off. Output included temperature histories at container surface and center-point, as well as accumulated center-point lethality at the end of cooling ( $F_o$ ) and accumulated surface lethality at the end of cooling ( $F_{surface}$ ).

#### Lethality and Quality Kinetics

Bacterial death and thermal destruction of quality attributes were considered first order reactions with respect to time such that

$$t = D (\log a - \log b)$$

where t was time at a particular lethal temperature, a and b were bacterial numbers or quality indices at time 0 and time t respectively, and D was the time associated with a tenfold decrease in bacterial number or quality index. Rate of lethality or quality loss (L) was defined as

$$L = 10^{(T-Tr)/z}$$

where T is the temperature at the point of interest, Tr is the reference temperature (121.1C) and z is the temperature interval associated with a tenfold change in D. Accumulated bacterial lethality at the geometric center of the can  $(F_o)$ , average accumulated quality loss  $(F_{z,average})$  and accumulated quality loss at the product surface  $(F_{z,surface})$  were defined as

$$F = \Sigma L\Delta t$$

The z of *Clostridium botulinum* (10C) was used for bacterial quality, that of thiamine (26.4C) (Ramaswamy and Ghazala 1990) for average nutrient loss, and that of appearance of canned fish (23C) (Ohlsson 1980) for surface quality. Average quality loss ( $F_{z,average}$ ) of cylindrical cans was calculated by summing the quality loss of each ring-shaped element multiplied by its fractional volume.

 $F_{average, z} = \Sigma (L_{ij} * volume of element_{ij}) \div total volume$ 

L<sub>ii</sub> is lethal rate or destruction rate at the point of interest.

#### Rho (p)

Fraction of bacterial lethality that occurs in the heating side of thermal processing or  $\rho$  was estimated, to allow the experimenter to end heating at the correct time and achieve the target F<sub>o</sub> at the end of the cooling phase. Relationship of  $\rho$  to retort temperature (RT from 120 to 130C), final unaccomplished temperature (g = RT-T<sub>f</sub>; 1 ≤ g ≤ 12; T<sub>f</sub> = center-point temperature at time of steam-off), and thermal diffusivity ( $\alpha$ ) of the can contents ( $\alpha$  from 1 × 10<sup>-7</sup> to 2.2 × 10<sup>-7</sup> m<sup>2</sup>/s) was estimated with repeated computer simulations. A quadratic equation to predict  $\rho$  was developed by multiple regression.

#### **Computer Simulation of CRT and VRT Processes**

CRT processes were defined by a 6 min vent, during which retort temperature rose linearly to the nominal RT, a period of constant RT and a cooling cycle. Retort temperature during cooling decreased over 7 min, from the final RT to the cooling water temperature of 10C, then remained constant until can center-point reached 90C. VRT processes included a 6 min vent to 104C which was the vent time necessary to ensure a pure steam environment in the test retort. The shape of the subsequent retort temperature versus time profile was defined by four coordinate pairs on the profile,  $(0.25t_{VRT}, RT1)$ ,  $(0.50t_{VRT}, RT1)$ 

RT2), (0.75t<sub>VRT</sub>, RT3) and (t<sub>VRT</sub>, RT4). Straight line segments between such points can be made to approximated curvilinear temperature profiles. The five variables; total time of variable retort temperature heating (t<sub>vrt</sub>) and values of the four retort temperatures (RT1, RT2, etc.) were adjusted in each computer simulation experiment as directed by the Random Centroid Optimization search procedure. The search was further constrained such that temperatures increased throughout the cook (i.e. RT1 < RT2 < RT3 < RT4). In some trials, the total t<sub>vrt</sub> was not utilized, because sufficient center-point lethality was accumulated prior to that time. If process time exceeded vent + t<sub>vrt</sub>, then RT4 was maintained until accumulated bacterial lethality equalled the target F<sub>0</sub> of 8.1 min multiplied by  $\rho$ , at which point cooling was begun.

#### **Random Centroid Optimization (RCO)**

RCO was employed as a search algorithm to find optimum VRT (Dou *et al.* 1993). The five variable optimization factors were  $t_{VRT}$ , RT1, RT2, RT3, and RT4. Initial variable ranges within which the program searched for optima were 35 to 120 min for  $t_{vrt}$  and 104C to 130C for RT. Retort temperature ramps were further constrained such that RT1 < RT2 < RT3 < RT4. The first cycle of optimization included 15 random design experiments and two centroid experiments, after which the results were mapped as described in Dou *et al.* (1993). In some cases, variable ranges were adjusted after the first cycle. One or two subsequent cycles were typically required to reach an optimum which could not be improved substantially with further experiments.

#### **RESULTS AND DISCUSSION**

#### Verification of Computer Model

Heating rate index ( $f_h$ ) of salmon in 307 × 115 cans was 31.0 min, while  $f_c$  was 27.9 min. The finite difference computer simulation of the retort cook, based on these indices and effective can dimensions of 82 mm diameter and 45 mm height, generated a center-point temperature history very similar to those recorded in the actual retort trial (Fig. 1). Retort trials of VRT processes also yielded center-point temperature histories very similar to those predicted by VRT computer simulations (data not shown). Therefore, the model was considered sufficiently accurate for process optimization purposes.

#### Estimation of $\rho$

Accurate prediction of  $\rho$ , the fraction of total  $F_0$  which occurs prior to steam-off, greatly reduced the number of experiments required for computer optimization of VRT processes. If  $\rho$  is unknown, many simulations of each VRT process must be performed in order to arrive at a suitable process time to give



FIG. 1. CENTER POINT TEMPERATURE HISTORIES OF 307  $\times$  115 CANS OF SALMON COMPARED TO FINITE DIFFERENCE COMPUTER SIMULATION OF THE SAME PRODUCT

the target total  $F_0$ , while one is sufficient if  $\rho$  is known. Stumbo (1973) estimated  $\rho$  as a function of final unaccomplished temperature (g), the difference between maximum retort temperature and the center-point temperature of the product at steam-off. We found this estimate insufficiently accurate for our purposes. Computer simulation experiments indicated  $\rho$  was also a function of  $\alpha$  (Fig. 2a), retort temperature (Fig. 2b), container geometry and container size. As the container was constant in our study, the later two factors are neglected here. The following equation was developed to predict  $\rho$  for conduction in a 307  $\times$  115 can:

$$\rho = 0.422 + e^{(0.233g - 0.831)} - 0.019g + 0.0007RT + 0.102\alpha + 0.0170\alpha^2$$

Multiple  $R^2$  was 0.99999, n = 649. This equation was used in subsequent computer simulations to find steam-off times that would yield  $F_0$  of 8.1  $\pm$  0.1 min. Although this equation may not be generalized to different container sizes, it is valid for conduction foods in this can, such as other salmon packs.

#### **Random Centroid Optimization (RCO)**

RCO was found to be an efficient means of searching for optimum VRT conditions. VRT heating curves were defined by 5 factors that were adjusted simultaneously in simulation experiments. About 35-50 experiments were required to conclusively determine an optimum. As well as the main optimization objective, constraints or conditions were included in the experiments. For example, a VRT profile which resulted in a minimum process time to achieve  $F_0 = 8.1$  min, with the constraint that surface cook must not exceed a certain



Thermal Diffusivity

FIG. 2. (A) IMPACT OF UNACCOMPLISHED TEMPERATURE (g) AND THERMAL DIFFUSIVITY ( $\alpha$ ) ON RHO ( $\rho$ ) OF CONDUCTION FOODS IN 307 × 115 CANS AT CONSTANT RETORT TEMPERATURE OF 120C (248F)



**Thermal Diffusivity** 

FIG. 2 (B) IMPACT OF THERMAL DIFFUSIVITY ( $\alpha$ ) AND RETORT TEMPERATURE ON RHO ( $\rho$ ) OF CONDUCTION FOODS IN 307 × 115 CANS AT CONSTANT UNACCOMPLISHED TEMPERATURE (g) OF 8C (14.4F)

value was found simply by assigning an arbitrarily high score to any experiment that yielded an unacceptable surface cook. This tactic forced the search away from the region of high surface cook while searching for short processes. Similar tactics were employed to include other constraints in the optimizations.

#### **Minimizing Surface Cook**

Conduction products processed to adequate center lethality inevitably receive excessive surface cooks. Surface cook (F<sub>surface</sub>) for a product can be estimated from kinetic equations as indicated in the methods section, if the z which defines the temperature sensitivity of the critical quality attribute is known. A CRT process which yields minimum surface cook exists for each combination of container, product and surface z. This is illustrated in Fig. 3a where values of  $F_{surface}$  of salmon in 307  $\times$  115 cans are presented for different values of z. The optimum RT varies from 113C to 119C, depending upon the z of surface quality. The usual commercial approach to problems of surface scorching of heat sensitive canned foods is to adjust the CRT retort temperature to give the best quality. The hypothesis that an appropriate VRT would result in better surface quality was tested in this study. RCO optimization was employed to determine VRT processes for this product which minimized F<sub>surface</sub> based on z of 20, 23, 27, 30 and 33C (Fig. 3b). In each case,  $F_0$  was maintained at 8.1  $\pm$  0.1 min. Optimum VRT processes consistently achieved lower surface cook than even the best CRT process.

The z of canned salmon surface appearance has not been reported, although Ohlsson (1980) found a surface z of 23C for another fish product. At z=23, the VRT optimum resulted in a surface cook of 39 min while the best CRT process (RT = 115C) had a surface cook of 46 min. The best variable retort temperature profiles are presented in Fig. 4. Surface quality changes that were more sensitive to temperature change (smaller z) benefited from more gradual increases, lower temperature maxima and showed more improvement with VRT than less temperature sensitive the canned food, the greater the potential benefits of VRT processing.

#### **Minimizing Process Time**

Salmon in 307 × 115 cans is typically processed close to 118.3C (245F). When operator's process time (P<sub>t</sub>) was 64 min (plus vent time) and CRT was 118.3C,  $F_0$  of 8.0 min and  $F_{surface z=23C}$  of 48.5 min were achieved. RCO was applied to find a VRT which would reduce P<sub>t</sub> while maintaining the  $F_0$  at least as high and  $F_{surface}$  no higher than the CRT process. Thirty-seven computer simulation experiments were completed in the RCO-directed search for the best



(a)
 (b)
 FIG. 3. (A) ACCUMULATED SURFACE COOK (F<sub>SURFACE</sub>) OF CANNED SALMON
 AS INFLUENCED BY RETORT TEMPERATURE; F<sub>SURFACE</sub> WAS CALCULATED FOR Z
 OF SURFACE QUALITY FROM 20 TO 33C. (B) ACCUMULATED SURFACE
 COOK (F<sub>SURFACE</sub>) OF OPTIMIZED VRT PROCESSES FOR CANNED SALMON
 Different optimal processes were found for surface quality attributes
 characterized by different z's.



FIG. 4. VRT RETORT TEMPERATURE PROFILES FOUND TO YIELD MINIMUM SURFACE COOK ( $F_{surface}$ ) OF CANNED SALMON

process, as summarized in Table 1. The optimum (Fig. 4) with  $F_0$  of 8.07 and  $F_{surface'z=23C}$  of 48.4 min required  $P_t$  of 53.4 min plus vent, 16% shorter than the CRT process.

#### **Minimizing Thiamine Loss**

Thermal kinetics of thiamine loss are first order with z = 26.4C and  $D_{121.1C}$ = 394 min (Ramaswamy and Ghazala 1989). In our simulation, the CRT process described above would destroy 19.6% of thiamine in canned salmon. A VRT process designed to minimize  $F_{z=26.4C}$  averaged throughout the can was found in our model to predict only 16.8% loss of thiamine (Fig. 5). This optimum variable temperature profile was similar in general shape to ramp and step profiles previously reported to yield best thiamine retention in a computer model of a 307  $\times$  407 can (Teixeira et al. 1975). However, while the VRT improvement was a marginal 2% in the previous study, in this study a 3% improvement was found. The difference may be attributed to two factors. The smaller can used in this study is more favorable to thiamine retention, as previously predicted (Teixeira et al. 1975). Second, maximum retort temperature of the earlier study was 129.5C, 4C higher than the maximum of the present optimum process for thiamine in salmon. Such high retort temperatures were found to be unfavorable to thiamine retention. In fact the two finite difference models gave similar results when provided with the same product and process parameters. For example the CRT reference process described in the earlier study as vielding 43% thiamine retention, resulted in 46% retention in our model.



O MIN VENT. OPTIMUM IN BOLD TYPE										
Taial	D	Val	nables	DTO	DTA	Б	Results	D.		
I riai	Ramp Time	RII	RI2	RI3	RI4	Fo	FS	Pt		
	(min)	(°C)	(°C)	(°C)	(°C)	(min)	(min)	(min)		
1	66.3	109.6	112 5	118.9	126.6	8 13	42 5	73 4		
2	118 1	114 1	116.5	121	128.1	8.03	40.1	84 1		
3	93.5	110.6	116.3	128.9	129.2	8 14	42.8	74.2		
4	111.6	110.0	122.3	120.7	129.2	8 15	46.2	66.2		
5	78.8	104 4	124.5	129.5	129.4	8 12	46.3	63.1		
6	39.9	105.8	106.6	109.7	126.8	8.09	47.0	63 3		
7	84.5	104.7	108.8	115.1	128.2	8.11	42.6	90.5		
8	72.6	109.5	117.7	124.2	128.6	8.16	42.9	67.7		
9	69.6	109.3	117.9	123.5	128.5	8.18	43.1	67.0		
10	70.1	109.3	118.7	124.2	128.8	8.14	43.3	66.0		
11	70.1	107.9	118.1	123.4	128.7	8.09	42.6	67.4		
12	60.2	105.8	112.2	126.3	129.7	8.10	46.8	63.8		
13	81.1	106.8	111.9	121.7	127.8	8.12	41.0	79.3		
14	63.2	108.9	122.3	125.0	127.5	8.03	45.8	60.6		
15	81.9	106.9	123.7	127.5	129.5	8.13	44.6	64.4		
16	72.0	104.4	110.7	123.6	125.8	8.14	41.5	74.1		
17	88.7	107.6	120.7	121.5	126.2	8.09	39.8	73.0		
18	50.8	106.1	111.3	121.6	128.3	8.10	46.6	62.7		
19	73.7	108.7	120.2	122.6	127.7	8.02	41.8	67.5		
20	88.3	104.7	115.4	129.0	129.3	8.07	43.5	74.0		
21	60.4	106.4	116.1	123.2	128.6	8.12	44.5	64.7		
22	57.4	106.4	115.0	122.6	128.5	8.10	45.0	64.1		
23	56.9	106.4	114.9	122.5	128.4	8.08	45.2	64.0		
24	56.9	106.4	114.9	122.5	128.4	8.10	45.2	64.0		
25	60.9	106.5	114.6	126.2	126.3	8.18	45.0	64.0		
26	70.8	108.7	111.7	127.9	129.2	8.14	45.1	67.8		
27	77.2	106.4	116.6	127.5	127.7	8.08	43.8	68.6		
28	69.7	104.9	120.2	127.1	129.3	8.12	45.4	63.6		
29	53.3	108.7	114.2	127.4	127.7	8.07	48.4	59.4		
30	56.8	109.3	120.2	121.4	129.5	8.05	46.1	61.7		
31	74.4	110.0	117.3	121.6	128.6	8.01	41.4	70.8		
32	73.8	109.8	124.7	127.8	127.9	8.11	47.2	59.8		
33	58.9	104.7	124.1	124.9	127.6	8.13	47.6	59.4		
34	62.2	107.4	120.2	125.2	128.3	8.16	45.6	61.9		
35	59.9	107.9	119.6	124.8	128.1	8.06	45.6	61.5		
36	61.2	108.1	120.7	125.2	128.1	8.12	46.0	61.0		
37	61.0	108.2	120.8	125.2	128.1	8.13	46.0	60.9		

# $\label{eq:table 1.} TABLE 1.$ OPTIMIZATION EXPERIMENTS TO MINIMIZE PROCESS TIME OF CANNED SALMON WITH $F_s \leq 48.5$ MIN AND 8 MIN $\leq F_0 \leq 8.2$ MIN. PROCESS TIMES INCLUDE 6 MIN VENT. OPTIMUM IN BOLD TYPE

#### CONCLUSIONS

RCO, when combined with a finite difference model of conduction heating, provided a convenient, efficient method for choosing optimal VRT thermal

processes. Furthermore, VRT processing, even when designed to reflect equipment restrictions of typical salmon canneries, proved able to reduce surface overcook, reduce thiamine loss or decrease processing time by 15% or more, while maintaining bacterial lethality of the total process. In terms of surface damage to heat sensitive products, VRT provided greater benefit than the best CRT process.

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# ELECTRIC FIELD EFFECTS ON THE THERMAL CONDUCTIVITY OF MILK CHOCOLATE DETERMINED USING THE "MIRROR IMAGE METHOD"

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#### ABSTRACT

Electrorheological fluids exhibit changes in flow properties when exposed to an electric field. Generally, these fluids display an increase in apparent viscosity and a greater yield stress in the presence of a voltage. Another attribute of some electrorheological fluids is the ability to enhance heat transfer, allowing for greater control over heating and cooling unit operations. This paper examined the effects of an applied voltage on the thermal conductivity of molten milk chocolate, a material known to be an electrorheological fluid. The method used to measure the thermal property was based on the "mirror image" concept. In this technique, identical disks sandwich a heat source providing a constant heat flux directed evenly between the two sides. Knowing the heat flux and the boundary temperatures of the sample gap, an analytical solution was used to calculate the thermal conductivity and specific heat. An applied electric field of up to 450 V mm<sup>-1</sup> at 60 Hz was found to have no significant effect on the heat transfer properties of milk chocolate. The thermal conductivity was found to have an average value of 0.163 W  $m^{-1} K^{-1}$  over a temperature range varying from 25 to 60C. The measurement technique, however, proved to be a rapid method for determining thermal properties that may be useful in investigating other liquid foods.

#### INTRODUCTION

Thermal conductivity (k) is a very important property because heat transfer calculations are often essential for proper design and analysis of food processes.

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In food systems, k depends mostly on product composition, but there are other factors which may influence thermal conductivity, such as void spaces, homogeneity, particle orientation, and temperature (Sweat 1986; Rahman 1992; Cuevas and Cheryan 1979). Since biological materials, like liquid foods, are not homogeneous and may vary in cellular structure, composition, and air content, variation in measured conductivity is large (Mohsenin 1980). Hence, many food engineers find it more convenient to estimate k using mathematical correlations (Cuevas and Cheryan 1979). Several models exist which predict the conductivity of foods based upon composition, such as the Choi and Okos model (Sweat 1986):

$$\mathbf{k} = 0.61 \mathbf{X}_{\mathbf{w}} + 0.20 \mathbf{X}_{\mathbf{p}} + 0.205 \mathbf{X}_{\mathbf{c}} + 0.175 \mathbf{X}_{\mathbf{f}} + 0.135 \mathbf{X}_{\mathbf{a}}$$
(1)

where the subscripts indicate the mass fractions of water (w), protein (p), carbohydrate (c), fat (f), and ash (a). Equation 1 is a best fit expression between predicted and experimental data points. Much has been written which reviews models and methods for thermal property measurement of foods (Mohsenin 1980; Baroncini *et al.* 1979; Cuevas and Cheryan 1979; Ziegler and Rizvi 1985; Zuritz *et al.* 1989; Rahman 1992; Sweat 1986; Choi and Okos 1986).

The complex nature of milk chocolate makes it difficult to use theoretical models and necessitates the use of experimental data to determine conductivity. Milk chocolate is a multi-component system incorporating a suspension of particles consisting of sugar, cocoa, and milk solids in a continuous fat phase of cocoa butter (Chevalley 1974). Milk chocolate has a high concentration of solids, greater than 60% of the material is nonfat solids. The water component of a food is generally the most important factor when determining conductivity (Rahman 1992); however, this is not the case with milk chocolate since it only contains between 0.5 and 1.5% moisture (Minifie 1980).

Electrorheological (ER) fluids are materials which change physical properties upon the application of an electric field. These unique fluids are typically composed of polarizable particles suspended in an insulating oil. Generally, an ER fluid displays a greater yield stress and an increased apparent viscosity over an unelectrified sample, resulting from the formation of microstructures in the dispersed phase. These microstructures are thought to be particles linking together to form chains, which in turn orient in parallel with the applied electric field (Halsey 1992; Block *et al.* 1990). It is the microstructures which potentially increase the directional thermal conductivity of ER fluids, providing a direct path for energy transfer through the material. The fibrillar particulate structures of an electrified ER sample consisting of zeolite particles in silicone oil has been shown by Zhang and Lloyd (1993) to have increased thermal conductivity by 55%. Certain biological materials, such as corn starch and vegetable oils are well established ER fluids (Block and Kelly 1988). Milk

chocolate has recently been identified as an ER fluid (Daubert and Steffe 1996). Milk proteins are believed to be responsible for polarizing the solid phase of milk chocolate when exposed to an electric field. Controlling the heat transfer properties of fluid foods with an electric field can have a significant impact on a food process design.

The overall goal of this paper was to investigate the potential for electrically enhanced heat transfer in milk chocolate. The specific objectives of this research were: (1) to describe the "mirror image method" (MIM) used to measure thermal conductivity of fluids exposed to electric fields; and (2) to evaluate electric field effects on the thermal conductivity of molten milk chocolate.

#### MATERIALS AND METHODS

#### **Mirror Image Method (MIM)**

Thermal conductivity measurements were made using an apparatus designed to measure thermal conductivity (k) and volume specific heat  $(\rho c_p)$  simultaneously, using a parameter estimation technique. The method was based on a technique we call the "mirror image method" based on a concept proposed by Danielson and Sidles (1969). This technique assumes that when identical disks sandwich a heat source, a constant heat flux (q) directed to the disks is split equally between the two sides. Outside disks are well insulated with 2.0 cm wooden blocks to permit the assumption of an adiabatic boundary condition. To further simplify calculations, disk thickness had to be small in comparison to the disk diameter to allow for the one-dimensional heat transfer assumption. The system configuration is discussed further in later sections.

Zhang and Lloyd (1993) calibrated the MIM apparatus (the same one used in this research) with water to ensure that the estimated thermal conductivity was accurate. The authors found that the procedure produced thermal conductivities differing by 4.77% from published data, validating the technique as an acceptable method for liquid thermal conductivity measurement.

#### **Mathematical Model**

The differential equation describing one-dimensional heat transfer is

$$\frac{\partial \mathbf{T}}{\partial t} = \alpha \frac{\partial^2 \mathbf{T}}{\partial z^2} \tag{2}$$

where  $\alpha = k \rho^{-1} c_p^{-1}$ . From the general differential equation, the one-dimensional analytical solution for a flat plate design insulated on one side and heated by a steady heat flux on the other is given by Beck *et al.* (1992) as

$$T^{*} = t^{*} + \frac{1}{3} - z^{*} + \frac{1}{2} (z^{*})^{2} - \frac{2}{\pi^{2}} \sum_{n=1}^{\infty} \frac{[\exp(-n^{2}\pi^{2}t^{*})]\cos((n\pi z^{*})}{n^{2}}$$
(3)

with the dimensionless numbers defined as:

$$T^{\star} = \left(\frac{\alpha}{qL}\right)(T - T_{o}) \tag{4}$$

$$t^* = \frac{\alpha t}{L^2} \tag{5}$$

$$z^* = \frac{z}{L} \tag{6}$$

Equation (3) is valid for the MIM with the following assumptions:

- (1) negligible contact resistance,
- (2) homogeneous sample,
- (3) one dimensional heat transfer,
- (4) negligible heat transfer at the edges (adiabatic),
- (5) constant heat flux,
- (6) negligible convection.

In an enclosed system, like the one used in this procedure, with a highly viscous material, the energy transport from conduction dominates over the natural convection mechanism, justifying the final assumption (Bajorek and Lloyd 1982; Zhong *et al.* 1983; Nicolette *et al.* 1985). The analytical solution incorporates the assumptions as initial and boundary conditions

$$T^{*}(z^{*},t^{*}=0)=0$$
 (7)

$$-\frac{\partial T^{*}}{\partial z^{*}}(z^{*}=0,t^{*})=1$$
(8)

$$\frac{\partial T^*}{\partial z^*}(z^*=1,t^*) = 0 \tag{9}$$

In addition,  $T^*$  from Eq. (3) requires a known value of the heat flux (q), defined as

$$q = \frac{V^2}{RA}$$
(10)

The solution for a one-dimensional, transient heat transfer problem requires the temperature to be a function of time and position. Knowing the transient

boundary temperatures and the heat rate into the sample fluid, the thermal properties may be estimated.

#### **Mirror Image Method (MIM) Plates**

A disk series on one side of the heater consists of an aluminum plate, teflon O-ring gasket serving as the sample fluid reservoir, copper plate, followed by another teflon O-ring gasket. An identical disk series mirrors the opposing side of the heater. Disks constructed from aluminum and copper were selected due to their availability; other highly conductive materials could be substituted for these materials. Each disk was approximately 7.62 cm in diameter. The teflon gasket reservoir included a small aperture permitting the injection of fluid into the system. Reservoir thickness (L) was 0.3 cm. The heater was placed between the disk sets, and the complete series was clamped vertically based on diameter between two wooden blocks. The purpose of the final O-rings and the wooden blocks were to insulate the system, thereby satisfying the adiabatic boundary condition. Figure 1 illustrates the plate series.



FIG. 1. MIRROR IMAGE METHOD DISK CONFIGURATION
(A) Aluminum Plate, 0.1 cm; (B) Teflon O-Ring Sample Reservoir, 0.3 cm; (C) Copper Plate, 0.1 cm; (D) Teflon O-Ring Insulator, 0.3 cm; (E) Wooden Block, 2.0 cm.

#### **Overall Apparatus**

To liquefy the solid Hershey milk chocolate bars, a Brookfield Model EX-100 water bath was used. Once the liquid chocolate was deposited into the MIM sample reservoir, the constant heat flux boundary condition was satisfied by turning on a Hewlett Packard Model 6024A DC power supply. This power supply sent 12.5 volts to the electric heating disk (OMEGA-FLUX electric heater made of a layer of etched copper between two thin layers of electrical insulation material with an internal resistance of 10.0  $\Omega$ ) at the center of the MIM plates. To monitor the sample temperature, copper-constantan thermocouples (20 gage) were cemented at the center of the fluid side of each metal plate (z = 0 cm and z = L = 0.3 cm). The metal plates acted as the fluid border as well as electrodes for the electric field. Fluid temperatures at the electrodes were recorded by a Fluke Model 2625A data acquisition system. The ER electric field requirement was supplied by a transformer operating under extremely low current conditions (less than 1 mA). The low current condition precluded significant ohmic heating in the sample. The voltage source operated at 60 Hz. Electric frequency is known to have a large impact on ER behavior; however, for low frequencies (50-60 Hz), practically DC, the response does not vary with frequency (Block and Kelly 1988; Jordan and Shaw 1989; Block et al. 1990). After testing, data was downloaded from the Fluke data acquisition system into a personal computer for parameter estimation. Figure 2 depicts the overall experimental setup.



FIG. 2. MIRROR IMAGE METHOD EXPERIMENTAL SETUP

#### **Testing Procedure**

Two chocolate bars were melted at 40C using the water bath. Once the chocolate was liquefied, approximately 6 mL of molten chocolate were injected into the sample reservoir of the MIM plates using a heated hypodermic syringe. At this point, the milk chocolate temperature was monitored with the data acquisition system to insure that the thermocouples were not damaged or dislodged during the injection procedure. During this stage of the experiment, it was common for milk chocolate temperatures to decrease from the 40C initial temperature. A constant temperature across the sample was required as an initial condition for each test, validated by monitoring temperatures at each thermocouple location. Formal data collection was initiated once this condition was met.

The data acquisition system was activated to scan and record temperatures at three second intervals. At time zero, the high voltage amplifier was activated, sending a predetermined electric field of 0, 300, or 450 volts mm<sup>-1</sup> at 60 Hz to the ER fluid food. The next step was to apply the constant heat flux boundary condition by activating the power supply to the heating disk. The sample boundary temperatures were scanned for approximately three minutes with data collection continuing to occur at three second intervals. Milk chocolate temperatures generally varied between 25 and 60C during each test.

Once the experiment had run for approximately 3 min, power to the apparatus was turned off in the following order: the heating disk power supply, the high voltage power amplifier, and finally the data acquisition system. Raw data, consisting of temperatures at the fluid boundaries and the times when sampling occurred, were stored for later analysis.

#### **Data Analysis**

The heat flux value was required for calculations. To solve for q, the parameters for Eq. 10 were specified as follows: V, the voltage to the heater, was equal to 12.5 volts; R, the resistance of the heater, was equal to 10.0 ohms; and A, the total surface area of the two sided area was equal to  $9.12 \times 10-3$  m<sup>2</sup>. Substituting these values into Eq. (10) gave the total flux a numerical value of 1713 W m<sup>2</sup>. However, since only half the total flux is exposed to each side of the "mirror," q equals 856 W m<sup>-2</sup>.

Equation (3) was programmed into Microsoft Excel software and the Excel Solver function was implemented. Solver is used to find solutions to problems having numerous variables and constraints. Initial estimates for thermal conductivity and volumetric specific heat (k and  $\rho c_p$ ), along with recorded temperatures and the heat flux, allowed Solver to perform an iterative search for estimation of the thermal parameters at each thermocouple location. Initial values for thermal properties were selected based upon milk chocolate

composition. For a single iteration, differences between the actual and theoretical temperatures at each boundary were determined, then squared, and finally added together for all scanned times to produce a summation of the squared error value (SS), where

$$SS = \sum_{t=0} \left[ \left( T_{actual} - T_{theory} \right)_{z=0}^{2} + \left( T_{actual} - T_{theory} \right)_{z=L}^{2} \right]$$
(11)

The search adjusted values of k and  $\rho c_p$  until the constraint achieving the lowest SS was found. The thermal parameters satisfying the constraint were selected as optimal values for k and  $\rho c_p$ .

#### Statistics

A common statistical test was employed to compare the sample means of the conductivities determined with, and without, a voltage. The t distribution test statistic was used at a 95% confidence interval to determine whether the applied electric field had a significant influence on the thermal conductivity (Hicks 1993).

#### **RESULTS AND DISCUSSION**

A "mirror image" concept was used to predict the thermal conductivities of milk chocolate with and without the presence of an electric field. A total number of eighteen tests were performed (Table 1). Of the 18 experiments, eleven tests were run without an applied field, yielding an average conductivity of 0.158 W m<sup>-1</sup> K<sup>-1</sup> (standard deviation = 0.027 W m<sup>-1</sup> K<sup>-1</sup>). Five tests were operated with 300 volts mm<sup>-1</sup> across the chocolate sample, giving an average conductivity of 0.175 W m<sup>-1</sup> K<sup>-1</sup> (standard deviation = 0.013 W m<sup>-1</sup> K<sup>-1</sup>). Further measurements were collected at 450 volts mm<sup>-1</sup>, yielding an average conductivity of 0.159 W m<sup>-1</sup> K<sup>-1</sup>. More tests were performed without an electric field to establish the procedure.

The sample means for the conductivities at 0 and 300 volts mm<sup>-1</sup> were 0.158 and 0.175 W m<sup>-1</sup> K<sup>-1</sup>, respectively. The t distribution test statistic, with a 95% confidence interval and 14 degrees of freedom, was equal to 2.14. For our sample averages,  $t_{test} = 1.32$ . Since  $t_{test}$  was less than 2.14, the voltage was considered to have an insignificant effect on the thermal conductivity. Therefore, the mean value of k was calculated from all 18 experiments, 0.163 W m<sup>-1</sup> K<sup>-1</sup> (standard deviation = 0.023 W m<sup>-1</sup> K<sup>-1</sup>). A possible explanation for the lack of impact an electric field has on the directional heat transfer is that milk chocolate has a very high volume fraction of particulates, greater than 60% nonfat solids. Only a portion of milk chocolate solids polarize and form ER microstructures, while the other solids remain randomly dispersed throughout the continuous lipid

phase. The nonpolar particulates may be responsible for interfering with energy transfer through the milk chocolate. Although an electric field affects the rheology of milk chocolate, the microstructures do not provide noticeable increases in the rate of heat transfer.

	0 Volts mm <sup>-1</sup>	300 Volts mm <sup>-1</sup>	450 Volts mm <sup>-1</sup>
	$\begin{array}{c} 0.150\\ 0.150\\ 0.151\\ 0.118\\ 0.127\\ 0.127\\ 0.127\\ 0.194\\ 0.184\\ 0.177\\ 0.185\\ 0.180\\ \end{array}$	0.163 0.159 0.185 0.185 0.185	0.160 0.158
n	11	5	2
$\bar{\mathbf{Y}}$	0.158	0.175	
S	0.027	0.013	

TABLE 1. THERMAL CONDUCTIVITY (W m<sup>-1</sup> K<sup>-1</sup>) OF MILK CHOCOLATE EXPOSED TO VARYING ELECTRIC FIELDS

Published values for the thermal conductivity for milk chocolate were not found. Equation (1) produced a conductivity of 0.195 W m<sup>-1</sup> K<sup>-1</sup>. This value gives approximately 16% error between the estimated value and the number calculated with the compositional model. Compositional and chemical variations in food systems as well as procedural inconsistencies may be responsible for the deviation.

The recommended method (Sweat 1986) for determining thermal diffusivity,  $\alpha$ , is to calculate it from experimentally derived values of conductivity (k), specific heat (c<sub>p</sub>), and mass density ( $\rho$ ), where

$$\alpha = \frac{k}{\rho c_p}$$
(12)

According to Zhang and Lloyd (1993) the specific heat and density do not appear to be affected by the application of an electric field. The MIM produced specific heats ranging from 1.85 kJ kg<sup>-1</sup> K<sup>-1</sup> to 2.63 kJ kg<sup>-1</sup> K<sup>-1</sup> with an average value of 2.14 kJ kg<sup>-1</sup> K<sup>-1</sup> (standard deviation = 0.27 kJ kg<sup>-1</sup> K<sup>-1</sup>). Density of milk chocolate, determined as 1.24 g mL<sup>-1</sup>, lead to an average thermal

diffusivity of  $6.32 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$  (standard deviation =  $1.46 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$ ) for the milk chocolate. Chocolate diffusivity data were not found in the published literature, but comparison to known diffusivity values of other food materials (such as  $7.90 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$  for butter between 30-60C) reported by Choi and Okos (1986) show that the estimated value is reasonable.

Figure 3 shows a typical comparison of actual and theoretical temperatures at two thermocouple locations (z = 0 and z = L) for a single experiment, confirming the predictive model accurately represents the actual time - temperature relationship occurring in the sample. Optimal heat transfer parameters for this single test were 0.184 W m<sup>-1</sup> K<sup>-1</sup> and 1.96 kJ kg<sup>-1</sup> K<sup>-1</sup> for k and c<sub>p</sub>, giving a near approximation of temperature history at each thermocouple location. Figure 4 displays residuals between theoretical and actual temperatures at each boundary of the chocolate sample. When the differences are the smallest at each location, the predicted heat transfer properties more accurately reflect the experimental values. From this plot, the theoretical and experimental data fit best between 40 and 140 s, with the average temperature residual less than 0.5C. Within this time frame (Fig. 3) the heat transfer is nearly linear and more time-independent, simplifying the chore of parameter estimation. The residuals for each thermocouple generally showed increases after t = 140 s, confirming possible heat loss and deviation from the ideal one-dimensional model.



FIG. 3. TRANSIENT BOUNDARY TEMPERATURES OF MILK CHOCOLATE: COMPARISON OF ACTUAL AND THEORETICAL TEMPERATURES



FIG. 4. MILK CHOCOLATE BOUNDARY TEMPERATURE DIFFERENCES BETWEEN ACTUAL AND THEORETICAL VALUES

#### CONCLUSIONS

An electric field of up to 450 volts mm<sup>-1</sup> at 60 Hz did not produce a statistically significant enhancement of heat transfer in milk chocolate, an electrorheological fluid food. The average conductivity and diffusivity of molten milk chocolate over a temperature range of 25-60C was 0.163 W m<sup>-1</sup> K<sup>-1</sup> and  $6.32 \times 10^{-8}$  m<sup>2</sup> s<sup>-1</sup>, respectively. The "Mirror Image Method" proved to be a quick and practical technique for measuring heat transfer parameters of fluid foods with, or without, exposure to an electric field.

#### NOMENCLATURE

- k thermal conductivity, W m<sup>-1</sup> K<sup>-1</sup>
- X mass fraction, dimensionless
- $\rho$  mass density, g mL<sup>-1</sup>
- c<sub>p</sub> specific heat, J kg<sup>-1</sup> K<sup>-1</sup>
- q heat flux, W m<sup>-2</sup>
- T temperature, °C
- T\* dimensionless temperature
- t time, s

- t\* dimensionless time
- z position, m
- z\* dimensionless position
- $\alpha$  thermal diffusivity, m<sup>2</sup> s<sup>-1</sup>
- n iteration number, dimensionless

V voltage, V

- R resistance, ohms
- A area, m<sup>2</sup>
- L fluid thickness, m
- SS sum of squares

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