CHAPTER III

MATERIALS AND METHODS

3.1 The ohmic heating apparatus

3.1.1 Design of the ohmic heating cell

3.1.1.1 Factors for consideration

A static ohmic heater was designed and built by modifying the ohmic cell from Kanchanakitsakul (1999). The prototype was made of a glass pipe fitted with blackelite electrode holders. Slight leakage was found during heating at high temperature (more than 100°C) because the glass surface could not possibly sealed with the electrode surface as stated in the recommendation. In this study, the designed ohmic cell must be able to stand temperature up to 125°C under pressure without leakage and minimize heat loss to environment. The material selected to make an ohmic cell was Teflon (polytetrafluoroethylene) which had the required properties of low thermal conductivity, high melting point, high tensile strength and chemically inert (Callister, 1997). Titanium electrodes was chosen by its properties of inert, high corrosion resistance, and high melting point. The electrode holders were designed to fit precisely with the heating chamber, then the electrode surfaces would be exposed within the cross-sectional area of the chamber. The thermocouple ports were carefully designed to prevent the leakage and endure the increase pressure due to high temperature up to 125°C by drilling a cone-shape hole of 2 mm to 1 mm diameter and fitted with a silicone bead. The needle-type thermocouple was then inserted through the silicone bead via the thermocouple port to avoid the leakage.

3.1.1.2 Experimental device

The static ohmic heaters were consisted of cylindrical sample chambers made of teflon drilled with 2.65 cm inside diameter and thermocouple openings.Two titanium electrodes were inserted at both ends of the chambers. Two sample chambers were designed for solid and liquid samples with sample length between the electrodes of 1.5 cm for solid sample and 5.5 cm for liquid sample. Schematic drawings of the ohmic heating devices were shown in Figure 3.1 and 3.2. One thermocouple port was located at the center for solid chamber (Figure 3.1) and three ports, at the center and between the center and each side of the electrodes for liquid chamber (Figure 3.2). T-type thermocouple of 0.3 mm diameter, was used to measure the temperature of the sample, and was calibrated against the standard thermometer.

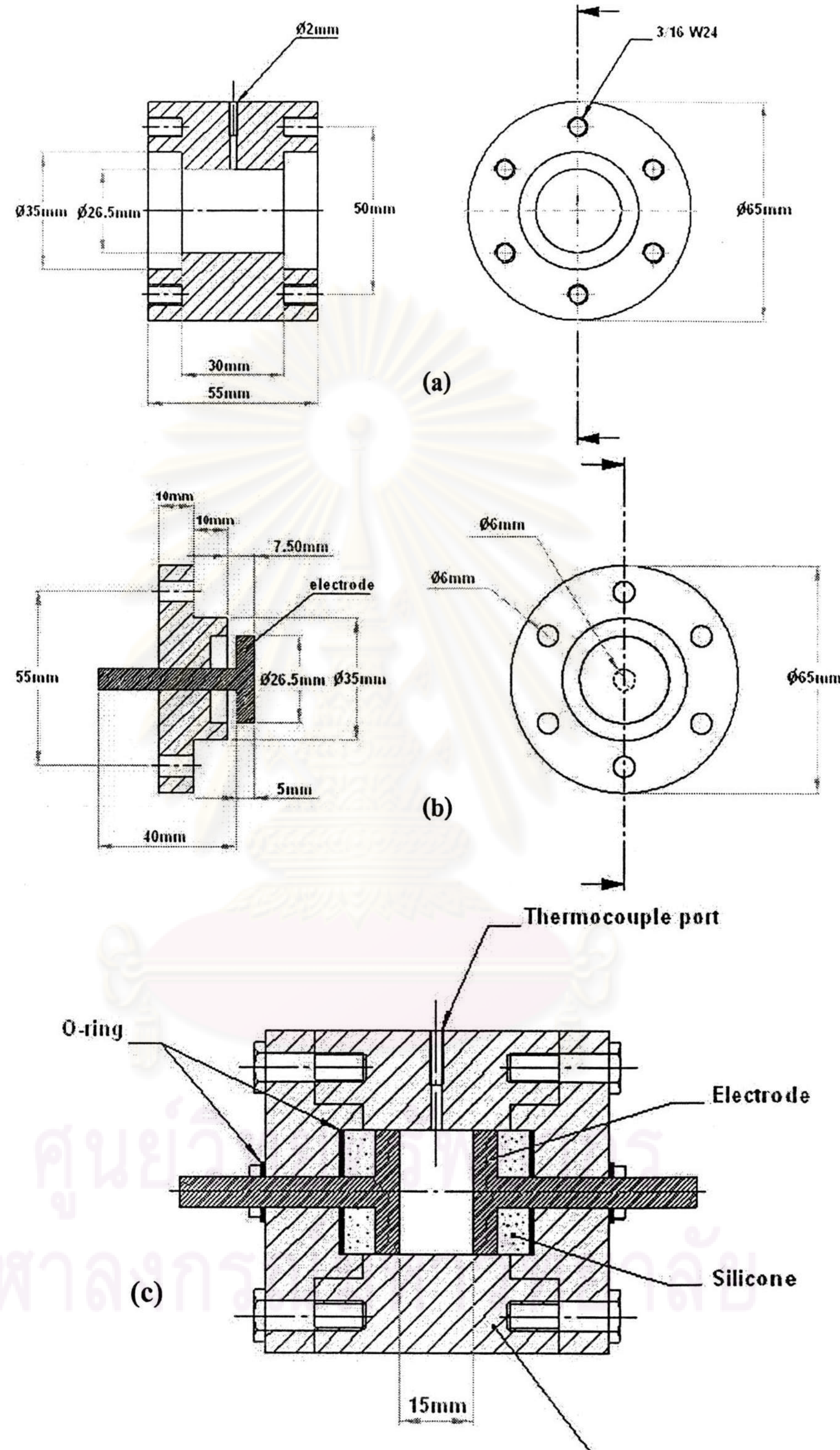

Figure 3.1 Schematic drawing of ohmic heating device for solid sample:
(a) sample chamber; (b) electrode holder: (c) ohmic heating cell

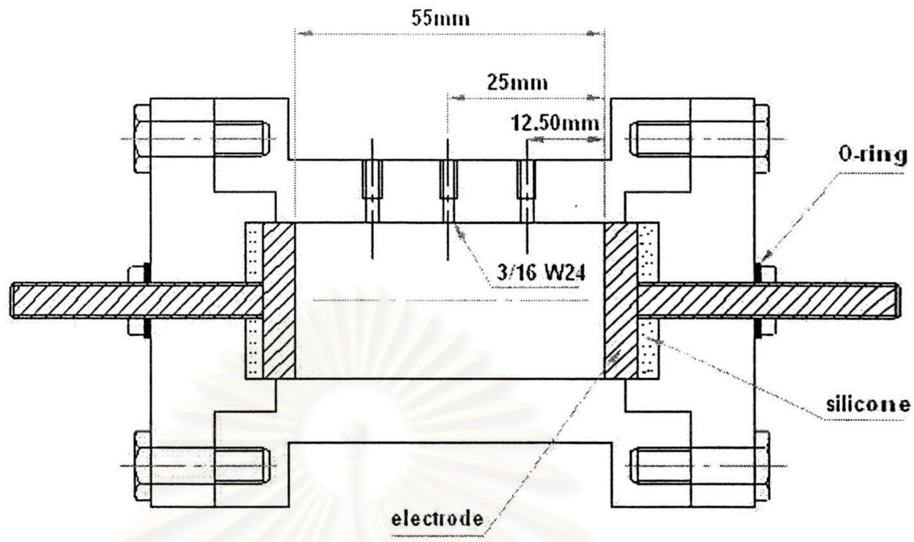

Figure 3.2 Schematic drawing of ohmic heating cell for liquid sample

3.1.2 Experimental setup

Schematic diagram of ohmic heating apparatus was shown in Figure 3.3. Alternate current at 50 Hz. and 220 V from main power supply was adjusted to the desired voltage gradient by a variable transformer (YOKOHAMA TSB-7.5). To study the effect of frequency, the apparatus was set up as shown in Figure 3.4. It consisted of an oscillator (KENWOOD AG-203), amplifier (Power Amplifier 500 W), line transformer(NPE L-600) and variable transformer. Voltage and current transducers were used to measure voltage across and current through the sample. Constant voltage was manually maintained during heating. A datalogger program linked to a microcomputer was used to monitor time, temperature, current, and voltage at 5 second interval.

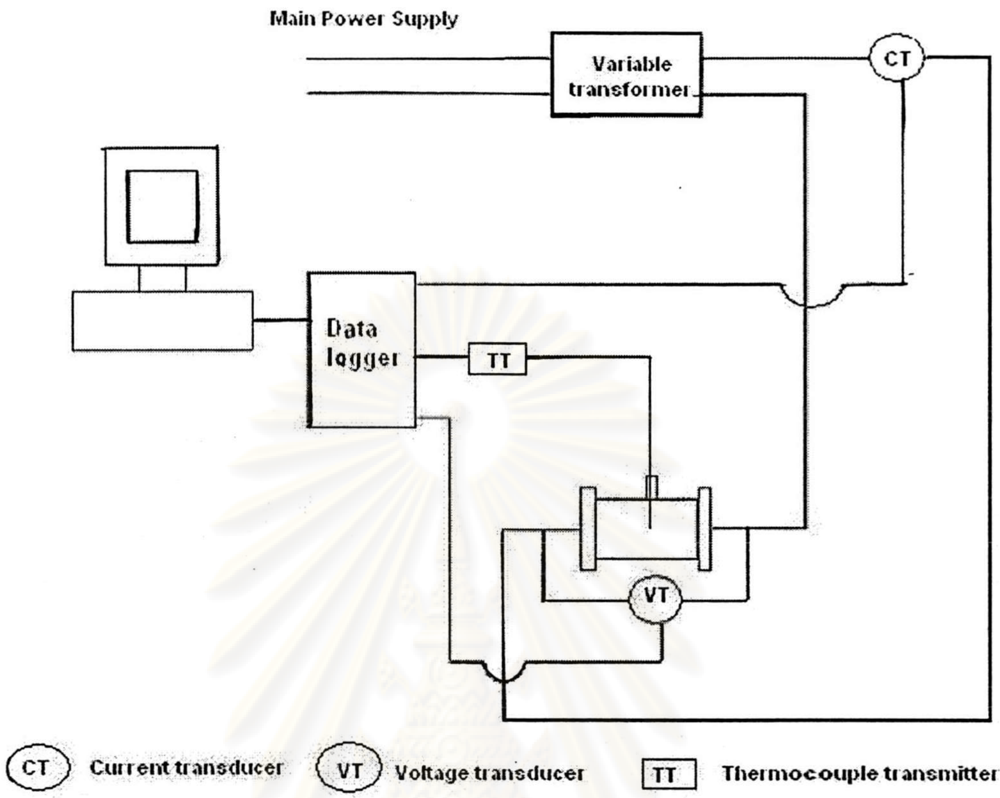

Figure 3.3 Schematic diagram of ohmic heating device

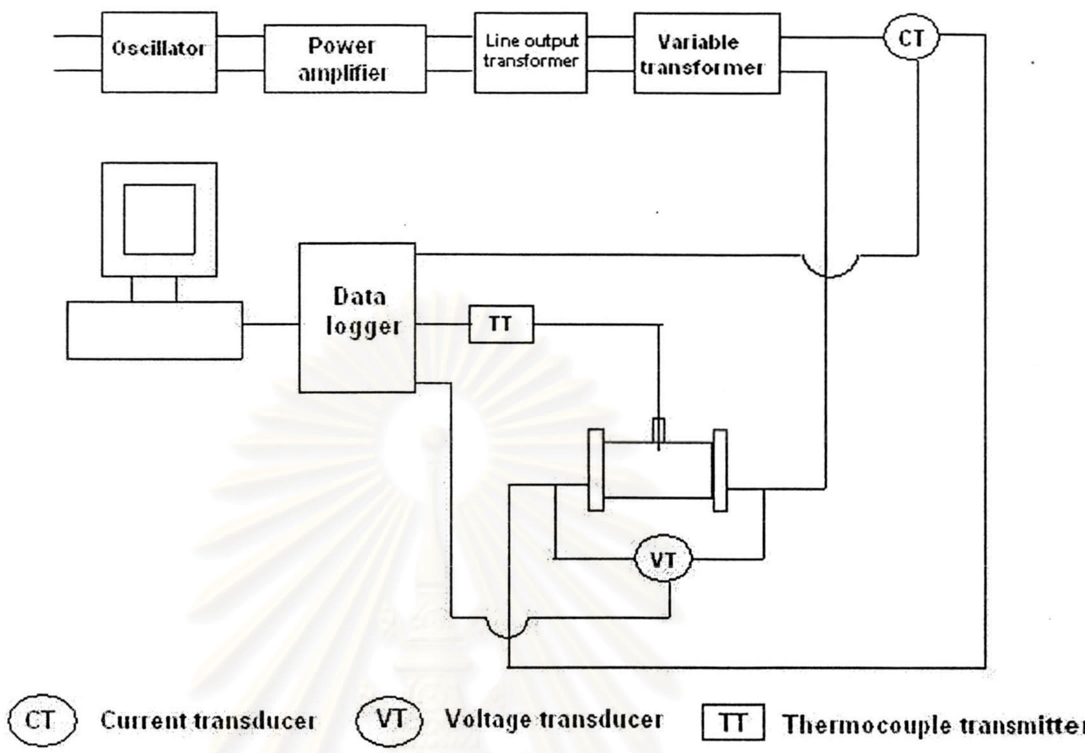

Figure 3.4 Schematic diagram of ohmic heating device to generate high frequency alternate current

3.1.3 Test for accuracy of the device

The accuracy of the device was tested by determining the electrical conductivity of the reference salt solutions: 0.1M NaCl, 0.02 M NaCl and 0.1M monosodium phosphate (NaH_2PO_4) by the method described in 3.2, using 4-6 V and 50 Hz. The measured values were compared with those reported in the literature (Palaniappan and Sastry, 1991a,b).

3.2 Electrical conductivity measurement

Samples were placed into the sample chamber and the electrodes were fitted at both ends of the chamber. Considerable care was taken to maintain full contact between the sample and the electrodes so a uniform heating rate was assumed. The thermocouples were inserted through the thermocouple ports at the geometric center of the sample. The samples were subjected to constant voltage to heat the samples from 25 to 125°C. Temperature, voltage, and current of the sample were continuously measured and recorded on a datalogger program linked to a microcomputer at 5 second interval.

Electrical conductivities were calculated using the equation described by Palaniappan and Sastry (1991a,b):

$$\sigma = (I/V)(L/A) \qquad \text{.....................................(3.1)}$$

The relationship between electrical conductivity and temperature was plotted and a trend line was determined by linear regression.

3.3 Factors affecting the electrical conductivity of solid foods

3.3.1 Solid sample preparation

Potato and white radish were selected as vegetable samples in this study regarding the differences in starchy and fibrous structures. Lean pork and surimi were selected as animal samples corresponding to the fibrous and homogeneous structures. Potato, white radish, and lean pork were purchased from the local market and were kept in 4°C before used. Frozen Threadfin bream surimi having 6% sugar and 0.2% tetrasodium pyrophosphate as cryoprotectants was purchased from a local surimi manufacturer and kept at -20°C until required.

The solid foods studied in this experiment were pretreated. Vegetables were blanched and soaked in salt solution while animal samples were cooked. The pretreatment by blanching and cooking were performed to simulate the industrial practice that most product would have been either thermally pretreated for enzyme inactivation, color fixing or cooking prior to sterilization. Soaking in salt solution was performed to increase the electrolytic content within vegetable tissues and resulted in the higher σ. In an ohmic heating, it was reported that the most desirable situation was that the σ of fluid and solid particles were equal in order to heat at the same rate (Zoltai and Swearingen, 1996). Since most vegetables had lower σ than liquids in particulate foods, an increase in σ could be achieved by salt diffusion (Wang and Sastry, 1993; Palaniappan and Sastry, 1991b). For pork, soaking in salt solution was not necessary since it was found that pork meat and chicken meat had a faster heating rate than brine which may be due to the higher electrolytes in meat (Halden *et al.*, 1990). Therefore, the blanched and soaked vegetables and cooked pork and surimi represented the solid samples in this study.

Vegetable samples were peeled and bored at the core perpendicular to vascular tissue into a cylinder of 2.65 cm diameter and 1.5 cm length to fit precisely within the ohmic cell. The samples were then blanched in steam for 5 min and soaked for 15 min in 0.75% NaCl solution, and then drained.

The surimi paste was prepared by thawing the frozen Threadfin Bream surimi at room temperature (~28°C) for 2 h. The thawed surimi was cut into small pieces (about 3 cm cubes), chopped 1.5 min in a Moulinex mixer model I171 at high speed, 1%

sodium chloride was added to the sample and the mixture was then mixed for another 1.5 min at high speed. The surimi paste was stuffed into a PVC mould (2.65 cm inside diameter x 1.5 cm length). Both ends of the mould were covered with stainless steel sheets and tightly wrapped. The sample was then cooked in water bath by two step heating method (Suwansakornkul, Kongpan, and Oka, 1999) i.e., 20 min at 40°C and 20 min at 90°C before cooling in ice slush for 15 min. The cooked surimi was then brought out of the mould.

The lean pork was cut perpendicular to the muscular bundles into cylinder with the same dimension as raw vegetables. The pork was placed into the mould and cooked in boiling water for 10 minutes. Cooking time for pork was 10 minutes which was determined from preliminary experiments for the sample's center temperature to reach 90°C.

All samples were kept in polyethylene plastic bag and stored at 4°C until required. It was brought to room temperature until the core center temperature reached 25°C before starting the ohmic heating.

3.3.2 Effect of voltage and frequency on electrical conductivity of solid foods

For solid samples, the effects of voltage and frequency were investigated. Three levels of voltage at 10.5-24 V (corresponding to 7-16 V/cm) and 50 Hz. and three levels of frequency at 50-1000 Hz. and 10 V/cm were applied to the pretreated samples. The electrical conductivity of the sample of each treatment was measured during heating from 25°C to 125°C. Since structural variation existed, 4-6 replicates were performed for each sample. The linear relationships between electrical conductivity and temperature were determined according to equation 3.2.

$$\sigma = \sigma_{25} + m\sigma_{25} (T - 25) \quad \text{..............................(3.2)}$$

Since the relationship between σ and temperature was linear and regressed as σ_{25} and $m\sigma_{25}$, both parameters were tested for significant difference by one-way ANOVA using SPSS program at 95% confidence level. Non significant effect of the factors studied was interpreted only the case that no significant differences were obtained from both parameters.

3.4 Factors affecting the electrical conductivity of liquid foods

3.4.1 Liquid sample preparation

For liquid samples, the model system was 1% NaCl (except for studying the effect of salt concentration). To determine the effect of sugar concentration, sucrose was added to the salt solution and thoroughly mixed prior by putting into the ohmic cell. For the study of the effect of starch concentration, the mixture of starch and salt solution was fully gelatinized to 80°C by heating on hot plate with magnetic stirrer and cooled to 25°C in ice slush. It was ensured that the gelatinization occured completely since heating until 80°C was far more higher than the normal gelatinization temperature range of 56-66 °C as reported by Lisinska and Leszezynski (1989). It was also designed to cover the gelatinization temperature which would slightly increase due to salt (Beleia, Miller, and Horsney, 1996; Chinachoti *et al.*, 1991a).

3.4.2 Effect of investigated factors on electrical conductivity of liquid foods

For liquid samples, the factors considered were:

(a) voltage at 100, 200 and 260 V(corresponding to voltage gradient of 18.2, 36.4,and 47.3 V/cm) at 50 Hz.

(b) frequency at 50, 500 , and 1000 Hz. at 9.1 V/cm

(c) salt concentration: 0.5, 1,and 1.5% NaCl at 50 Hz. and 9.1 V/cm

(d) added starch concentration: 0, 2, 5, 10, and 15 % potato starch at 50 Hz. and 9.1 V/cm

(e) added sucrose concentration: 0, 2, 4, 6, and 8% sucrose at 50 Hz. and 9.1 V/cm

All experiments were based on completely randomized design and were performed in triplicate. The electrical conductivity of the sample of each treatment was measured during heating from 25°C to 125°C. The relationships between electrical conductivity and temperature were determined as equation 3.2. Both the σ_{25} and $m\sigma_{25}$ were test for significant difference by one-way ANOVA using SPSS program at 95% confidence level.

3.5 Correlation between ingredients and electrical conductivity of model liquid

3.5.1 Starch paste preparation

Native potato starch from Sudstarke'-GMBH (GERMANY) was used. Three levels of salt (0.5, 0.75,1% w/w), sucrose (2, 6, 12% w/w), and potato starch (2, 4, 8% w/w) were selected within the range of commercial thick soup ingredient investigated. All ingredients used were of AR grade.

The experimental design was 3x3x3 factorial and was conducted in duplicate. The relevant ingredients were weighed and thoroughly mixed with water. The mixtures were then heated on hot plate with regular agitation by magnetic stirrer until 80°C and cooled to 25°C in ice slush. The starch pastes were then immediately put into the ohmic cell and heated up to 125°C using alternate current of 50 Hz. at applied voltage of 50 V, giving the voltage gradient of 9.1 V/cm.

3.5.2 Data analysis

A statistical program (Minitab version 13.2) was used to develop and validate the empirical model using stepwise regression. Ten variables, including four main effects temperature (T), salt content (Sa), sugar content (Su), and starch content (St), six interactive effects (TSa, TSu, TSt, SaSu, SaSt, SuSt) were initially analyzed for statistical significance. Residual plot of the model was evaluated to assure that the assumption of constant variance and normality were satisfied.

3.6 Effective electrical conductivity and heat distribution of solid-liquid food mixture

3.6.1 Sample preparation

The mixture of 0.7cm solid cubes in 0.75% salt-4% starch-2% sugar liquid was prepared with solid volume fraction of 0.2, 0.4, and 0.6. The starch paste was prepared as mentioned in section 3.5.1. Potato, white radish, and surimi were selected to be solid particles in the mixture. Potato and white radish were cut into pieces of about

1cm thick perpendicular to vascular bundles, blanched in steam for 5 minutes and then cut into 0.7cm cubes. As the difference in electrical conductivities of liquid and solid affected the heating behavior, the solid conductivities were varied as high and low. The solid cubes soaked in 0.75% salt solution (solid: liquid = 1:4) for 24 hours would represent high conductivity (Wang and Sastry, 1993) while the unsoaked solid cubes were the low conductivity. The cooked surimi was prepared as mentioned in section 3.2.1, and then cut into 0.7cm cubes. Electrical conductivity of each solid sample was also measured individually every batch.

3.6.2 Experimental setup

One side of electrode was fitted to the ohmic heating chamber. The thermocouples were inserted into the thermocouple ports. The thermocouple was arranged to measure the solid temperature by inserting into the center of a cube. Considerable care was required in this operation since the significant damage of sample due to the thermocouple could result in leakage of liquid medium into the thermocouple vicinity which greatly affected the experimental outcome. Another thermocouple was left free of contact to the particle to monitor liquid temperature. The rest of solid samples with desired volume fraction were put into the liquid, mixed, and immediately poured into the chamber. Another side of electrode was then fitted to the chamber and sealed. All cubes were in randomly scatter orientation. The mixture of various solid samples and volume fractions were randomly prepared. For the mixture of two-type solids in liquid, equal fractions of each solid particles were used. The mixture studied were:

1. unsalted potato in liquid,
2. salted potato in liquid,
3. unsalted white radish in liquid,
4. salted white radish in liquid,
5. surimi in liquid,
6. mixture of unsalted potato and unsalted white radish in liquid,
7. mixture of unsalted potato and surimi in liquid,
8. mixture of salted potato and surimi in liquid,
9. mixture of unsalted white radish and surimi in liquid,

10. mixture of salted white radish and surimi in liquid, and
11. mixture of salted potato and salted white radish in liquid.

The experiment was conducted in duplicate. Samples were heated using constant voltage gradient at 15 V/cm. Temperature of liquid and particles, as well as voltage and current were measured every five seconds. After each run, samples were examined for mechanical damage and for accurate placement of thermocouples.

The temperatures of solid and liquid of each run were plotted against time. The effective electrical conductivity of the mixture was calculated from the measured voltage and current of the system using equation 3.1 and was plotted against liquid temperature.

3.7 Relationship between effective electrical conductivity and electrical conductivities of both phases

The relationship between effective electrical conductivity and electrical conductivity of each component was determined by considering that components in the heater were arranged as a set of equivalent resistances in series according to the circuit analogy concept model proposed by Sastry and Palaniappan (1992a) (section 2.4.1.1). For the mixture of one type of solid in liquid, the effective electrical conductivity was calculated from the total resistance (equation 2.11) and equation 2.4. The σ-T equations of model liquid and solids obtained from section 3.5 and section 3.6.1 were used. The temperatures were then substituted intervally from 25 to 125°C to calculate the effective electrical conductivity values at each temperature. The calculated values were compared to those measured from the experiment, and computed for the differences. Similarly, for the two types of solids in liquid, the effective electrical conductivity was determined from equations 2.22 and 2.4. Samples of calculation were shown in Appendix F.

3.8 Mathematical models for temperature prediction

In this study, the following assumptions had been made to simplify the problem.

- particles were cube-shaped and of average size (Sastry and Palaniappan, 1992a)
- all particle was in uniform distribution within the liquid and was considered to be equivalent to electrical circuit (Sastry and Palaniappan, 1992b)
- heat generation was uniform throughout the sample (Fryer *et al*, 1993)
- heat transfer was rapid between phases (Fryer *et al*, 1993)
- heat loss to the surroundings was negligible (Fryer *et al*, 1993; Sastry and Palaniappan, 1992b; Fu and Hsienh, 1999)
- no orientation effects was considered due to cubic-shape particles (Sastry and Palaniappan, 1992b)
- thermophysical properties of the component were independent of temperature (Fryer *et al*, 1993; Sastry and Palaniappan, 1992b; Fu and Hsieh, 1999)
- cold spot of particle was located at the center of particle (Fu and Hsieh, 1999).

Therefore, simple models of analytical solution could be applied to solve the problem. The electrical field distribution was solved based on circuit-analogy concept to obtain the effective electrical conductivity (Sastry and Palaniappan, 1992b)

The heat generation was calculated based on equation 2.29. The infinite thermal conduction approach assumed that conduction and convection heat transfer occurred infinitely fast (Fryer *et al.*, 1993), i.e. the liquid temperatures were uniform. All the liquid thus heated at the same rate. The temperature of the system was then calculated from equation 2.30.

The parameters used in this equation were electric field strength of 1500V/m, initial temperature at 25°C, and the properties of mixture as presented in Table 3.1. The properties of mixture were averaged from the property of each component (Appendix C) and the weight fraction in the combination. Density was calculated from weight and volume measurement. Specific heat was estimated from equation 2.9 and moisture content determination (AOAC, 1995).

The predicted temperatures were plotted against time and compared to the temperature of slowest heating phase measured from the experiment. The differences between the predicted temperature and slowest-heating temperature at each time interval were calculated.

Table 3.1 The properties of mixture for temperature prediction

Mixtures	Density (kg/m^3) at volume fraction			Specific heat (J/kg^oC) at volume fraction		
	0.2	0.4	0.6	0.2	0.4	0.6
Potato in liquid*	1045	1056	1068	3897	3833	3768
White radish in liquid	1018	1005	990	3974	3986	3999
Surimi in liquid	1029	1026	1022	3868	3774	3679
Potato and white radish in liquid	1032	1030	1029	3936	3910	3883
Potato and surimi in liquid	1037	1041	1045	3883	3803	3724
White radish and surimi in liquid	1024	1015	1006	3921	3880	3839

* 0.75% salt -2 % sugar -4 % potato starch

3.9 The efficiency of energy conversion from electrical energy to thermal energy

The system power input (P) as given by VI in equation 2.2, was calculated at each time. The electrical energy increment (dQ_{et}) at each time interval (Δt, 5 s) was calculated by:

$$dQ_{et} = P*\Delta t = VI*\Delta t \quad \text{.........................(3.3)}$$

The electrical energy input (Q_e) was calculated from the accumulation of energy by integrating the energy increments over time increased, as:

$$Q_e = \int_0^t dQ_{et} \quad \text{.........................(3.4)}$$

The electrical energy inputs were plotted against time.

For the thermal energy, the increasing in temperatures of each phase during 5 second intervals (dT) were calculated by $T_2 - T_1$. The thermal energy increment (dQ_t) of each phase at 5-second interval was calculated from equation 3.5.

$$dQ_t = mC_p dT \quad \text{.........................(3.5)}$$

The specific heat of samples used in equation 3.5 were obtained from Table C1 in Appendix C. Similarly, the thermal energy (Q_t) was calculated from the accumulation of energy increments over time increase as:

$$Q_t = \sum_{t=i}^{j} dQ_t \quad \text{.........................(3.6)}$$

The total thermal energy was computed by summation of the thermal energy of all phases and was plotted against time. The electrical (Q_e) and thermal energy (Q_t) was compared . Small differences indicated the limited heat loss to the environment.