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Measurement of Thermal Conductivity of Foods in Liquid and Solid Phase Using a Thermal Probe

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Abstract An instrument using thermal probe method was designed to measure thermal conductivity of liquid and solid foods. Thermal conductivity probe was designed with diameter to length ratio of 100 and diameter of 0.51 mm to minimize axial flow effect on thermal conductivity measurement. Thermal conductivities of distilled/deionized water, glycerin, and beef frankfurter meat were measured at 20-80°C. Mean thermal conductivity values of water showed less than 2.0% difference from several reference values without using time correction factor or probe calibration constant. For glycerin, difference was less than 0.7% from reference values at 20-50°C. Mean values of thermal conductivity for beef frankfurter meat ranged from 0.389 to 0.350 W/m·K at 20-80°C.

Keywords: conductivity, instrument, liquid, measurement, thermal probe

Introduction

Thermal properties of food products are key factors in the design of thermal processes such as cooling or heating for food preservation. With an accurate knowledge of thermal properties such as thermal conductivity and specific heat, the total amount of heat to be added or removed from a product in a specific process can be determined as well as the rate at which heat can be added or removed.

Methods for thermal conductivity measurement can be divided into two categories, those using a steady state and those using a transient state of heat transfer. The steady state methods include those using longitudinal heat flow methods generally for dry homogeneous materials in slab form and those using radial heat flow methods for loose materials, powder or granular materials. Among the steady state longitudinal heat flow methods, the guarded-hot-plate method (1) is regarded as the most accurate and most widely used method for materials with low moisture content such as insulation materials. However, steady state methods are not very effective for determining the thermal conductivity of materials with high moisture content due to moisture migration problems inside the materials with the development of steady state heat flow. On the other hand, among the transient methods, the line heat source method is the most common method used, particularly for liquid and solid materials. The transient hot-wire method is regarded as the most accurate method for the measurement of thermal conductivity of materials in liquid phase (2, 3). This method, however, is expensive and not suitable for solid materials.

Thermal conductivity probe method is a modification of the line heat source method and is used extensively for conductivity measurements of a number of non-food materials, including soil (4), silicon rubber (5), and liquid chemicals (6), in both liquid and solid states. Studies on thermal conductivity of agricultural products have included tomato juice (7), fruits and vegetables (8), beef (9), and potato (10).

The thermal probe method assumes one-dimensional heat conduction of an infinite cylindrical body with a line heat source at its center. However, due to the sample size and convenience of probe construction, many thermal probes have been designed with a probe length/diameter ratio (L/D) smaller than 50 and/or with size larger than 0.9 mm in diameter. In addition, because of these finite probe length and size restraints, a time correction method (11) or calibration factor is generally required for acceptable use of such probes. D'Eustachio and Schreiner (12) have suggested that using a thermal probe of very small size would probably eliminate the need for a time correction factor in most measurements. Hooper and Lepper (4) recommended L/D of 100 to minimize the effect of axial heat flow due to the finite length of the probe, whereas for foods in liquid phase or soft solid foods with a long shape, larger L/D and smaller diameter probe design is more desirable.

The probe constant (calibration factor) can be obtained by calibrating the thermal probe with the reference materials of a known conductivity such as glycerin (9, 13) or water-containing agar (10, 13, 14). Asher *et al.* (6) measured the absolute conductivity of liquids including water and reported accuracy better than 5% without using a probe calibration factor; however, their probe still has room for improvement by applying a smaller probe diameter, larger L/D, less air space inside the probe, and better homogeneity of the probe material throughout its length.

The objectives of this study were to develop a thermal conductivity probe with a small diameter and an L/D of 100 suitable for measuring the thermal conductivity of liquid and solid food materials, and to determine the

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effects of temperature on the thermal conductivity of beef frankfurters.

Materials and Methods

Thermal conductivity probe methods Theory of the thermal conductivity probe methods is based on the line heat source method, and assumes one-dimensional heat conduction of an infinite cylinder with a line heat source at its axial center. The body is initially at a uniform temperature and thermophysically homogeneous. At time zero, a constant rate of heat is generated and conducted only in the radial direction. The rise of temperature at any point near the line heat source will thus be a function of the thermal properties of the material including thermal conductivity. The differential equation of Fourier for this conduction process is:

$$\frac{\partial T}{\partial t}=\alpha\left(\frac{1}{r}\frac{\partial T}{\partial r}+\frac{\partial^2 T}{\partial r^2}\right) \quad (1)$$

Where r, t, α, and T denote radial distance from the heat source, time, thermal diffusivity, and temperature, respectively.

For a finite temperature rise at the line heat source, Van der Held and Van Drunen (11) reported Eq. 1. The first two terms of the solution is:

$$T=\frac{q'}{4\pi k}\left[-\ln\left(\frac{r^2}{4\alpha t}\right)-0.577216\right] \quad (2)$$

Where q', α, k, and r denote the heat input per unit length of the heat source, thermal diffusivity of the medium, thermal conductivity of the medium, and the radius of the line heat source, respectively. The rest of the solution is negligible compared to the first two terms for a very small value of $(r^2/4\alpha t)$.

From Eq. 2, a change in temperature at the surface of the line heat source between times t_1 and t_2 is reduced to the following equation:

$$T_2-T_1=\frac{q'}{4\pi k}\ln\left(\frac{t_2}{t_1}\right) \quad (3)$$

The heat input q' is generally calculated as I^2R per unit length of the heat source, where I is the input current in amps and R is the resistance of the heater wire in Ω/m. Because q' and k are constants, the temperature rise is a linear function of $ln(t)$, and k can be calculated by measuring the slope of a temperature-$ln(t)$ plot (Eq. 3).

Design of thermal conductivity probe Thermal probe developed in this study consisted of a thermal conductivity unit, a diffusivity unit, and a small cylindrical Teflon block as a holder (Fig. 1). The thermal conductivity unit consisted of a constantan wire for heating, a thermocouple, and stainless steel tube. A 52-mm long stainless steel hypodermic needle tube (0.51 mm OD and 0.1 mm thick wall; Popper & Sons, Inc., New Hyde Park, NY, USA) was used as sheathing material for thermal conductivity and thermal diffusivity units. The tip of the tube was

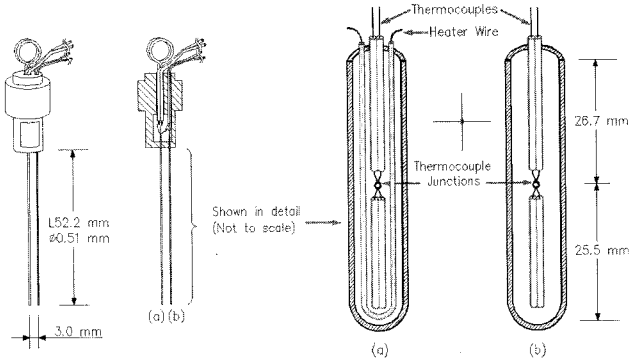

Fig. 1. Schematics and cross sections of the thermal probe: (a) thermal conductivity unit and (b) thermal diffusivity unit.

crimped and soldered with silver solder. Because the probe parts are made of stainless steel tubing, the thermal probe should be effective for measuring thermal conductivity of acid foods. The diameter to length ratio of the probe was set at 100 to minimize the effect of axial heat flow due to the finite length of the probe, following the recommendation of Hooper and Lepper (4). Teflon-insulated 40-gauge constantan wire with a resistance of 111.07 Ω/m (Physitemp Instrument Inc., Clifton, NJ, USA) was used as the heater wire. The heater wire was electrically insulated from the tubing and thermocouple. Teflon-insulated 44-gauge type-T thermocouples (Physitemp Instrument Inc., Clifton, NJ, USA) were used to measure the temperature rise of the thermal conductivity and thermal diffusivity units. These small thermocouples were used to minimize the time lag of temperature measurement. The thermocouple junctions were made at the center of the thermal unit (Fig. 1). Dummy thermocouples were extended beyond the junction to the tip of the probes to make the heat capacity of the thermal probe homogeneous throughout its length.

Thermal diffusivity unit was constructed with the same-sized stainless steel tubing and thermocouple materials as the thermal conductivity unit, and was mounted 3.0 mm from the axial center of the probe holder. The thermal diffusivity unit was designed to estimate the thermal diffusivity of the sample and to check the temperature gradient inside the sample before and during the conductivity measurement process.

A cross-section of the sample holder is shown in Fig. 2. An indentation in the same shape of the probe holder was cut inside the upper Teflon cylindrical block of the sample holder so that the thermal probe could be tightly inserted. The sample cell had 19.1 mm diameter, 54 mm long copper tube with 1.6 mm wall thickness. To further increase the convenience of sample loading and unloading, open-ended copper tubing was used in construction of the holder.

A schematic diagram of the thermal conductivity measurement system is shown in Fig. 3. The system temperature control device consisted of a 2-L glass jar water bath, a circulation pump, a stirrer, a circulation heater, and a CR7X measurement and control system (Campbell Scientific, Inc., Logan, UT, USA). At the inlet of the water bath, a T-connection tube was used to divert the direct flow of heated water from the sample cell. A twin blade

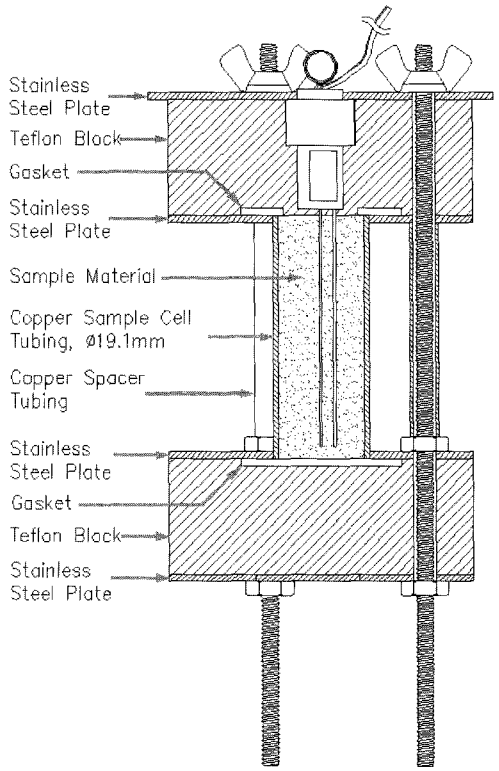

Fig. 2. Cross section of the sample holder.

stirrer was used to mix the water from the bottom and the top towards the center.

The sample cell was immersed 20 mm below the water level, and the top opening of the water bath was sealed and covered with insulation material to minimize heat loss through the upper part of the sample cell. The water bath, pump unit, and circulation heater were thermally insulated with glass wool and insulation board to minimize system temperature fluctuation.

System temperature was controlled with a pulse-width modulation method to minimize temperature fluctuations due to overheating. The duty cycle of heating was varied from 5 to 50% of a 6-s cycle according to the difference between the measured and the desired temperatures. Temperatures were measured at three points inside the water bath (top, middle, and bottom levels of the sample cell) and averaged to represent the controlled water bath temperature.

A CR7X data logger was used to store the measured data and to control the thermal conductivity measurement process and the water bath temperature. CR7X was interfaced to a microcomputer through SC32A (Campbell Scientific, Inc., Logan, UT, USA) RS232 interface module. The CR7X data logger was programmed, monitored, and controlled through the microcomputer using a PC208 (Campbell Scientific, Inc., Logan, UT, USA) software. A control circuit board was designed to drive relays used for switching stirrer motor, circulation motor, power to the probe, and circulation heater.

Thermal conductivity measurement As a standard reference material in liquid phase, distilled/deionized water was used for checking the accuracy of the probe. Thermal conductivity of water was measured from 21.8 to 81.9°C. As a second reference material for the thermal conductivity measurement verification, 99.5% pure glycerin (Fisher Scientific, Co., PA, USA) was used. Thermal conductivity of glycerin was measured from 20.1 to 50.1°C. As a solid food material, thermal conductivity of beef frankfurter meat was measured from 20 to 80°C. Oscar Mayer" brand Beef Franks obtained from a local grocery store was used as the test meat. Their mean moisture content and density were 53.7 ± 0.44% (wet basis) and 1033 ± 20 kg/m^3, respectively. Moisture content of the beef frankfurters was determined using a convection oven method at 75°C for 24 hr, and density was determined using a graduated cylinder and a balance. To determine the variations among packages, two samples each from three different packages were used at each temperature level. The effects of temperature and different packages on thermal conductivity of beef frankfurters were tested using General Linear Model Procedures (GLM) of SAS (16).

After a copper sample cell was loaded with a test sample material, the thermal probe was inserted into the indent in the top block and tightened with three wing nuts. When the water bath reached the desired experimental temperature, the sample holder was placed into the water bath. Temperatures of the thermal conductivity and diffusivity units were monitored until they were within ± 0.1°C of the water bath temperature. When the fluctuation of the probe temperature became less than ± 0.05°C, the measurement program was started.

At the start of the measurement program, the circulation

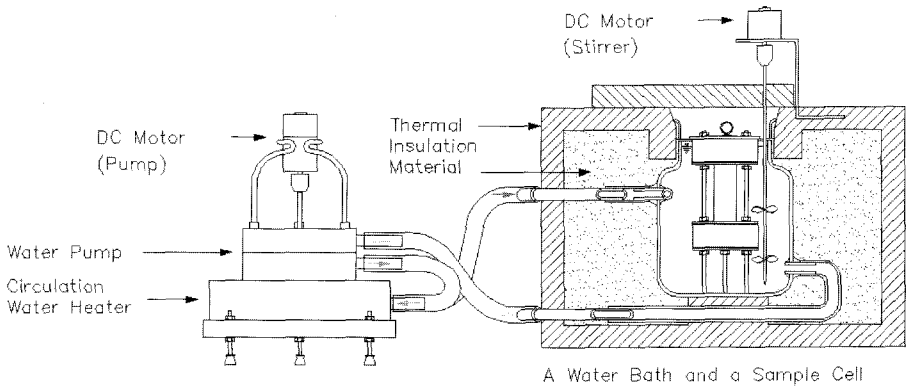

Fig. 3. Schematic diagram of the temperature control system.

pump and stirrer were turned off for 30 s to minimize the effects of vibration on the measurements of the loaded liquid sample. Subsequently, the probe heater was turned on for 10 s, and the temperature rise of the thermal probe, system temperature, and the current to the probe were recorded at 1-s intervals for 10 s. After the measurement, the stirrer and water pump were turned on for 200 s to allow the sample temperature to be equilibrated with that of the water bath. The process was repeated five times, and the average value was used to represent the thermal conductivity of each sample. Thermal conductivity of a sample was calculated using Eq. 3 based on the least square method.

The current level of the probe heater was determined to the nearest 0.01 mA with CR7X by measuring the voltage drop across an MP821 resistor (Caddock Electronics, Inc., Roseburg, OR, USA) connected in series with the probe heater. The resistance value of MP821 was measured as 1.000 ± 0.001 Ω using a Fluke 5100B calibrator and a Fluke 8086A Digital Multimeter (Fluke Corp., Everett, WA, USA). To determine the optimum current level for the probe, thermal conductivity of water at 20.0°C was measured from 20 to 150 mA. At a current level of 100 mA, the best reproducibility of conductivity measurements was observed. A current level of 100 mA was used for all tests, and the variation of the measured current level during any experiment was less than 0.01 mA. For the current level of 100 mA, the power dissipated by the probe was 2.33 W/m. An HP6236B triple output power supply (Hewlett Packard Company, Palo Alto, CA, USA) was used as the power supply to the probe.

A typical temperature-*ln*(t) profile of the thermal probe in water is shown in Fig. 4. Results showed that the slope of the temperature rise vs. *ln*(t) of the diffusivity unit began to decrease and leveled off after 12 s from the start of heating, which is an indication of the start of convection currents near the conductivity probe. Therefore, the heating time of the conductivity unit was set at 10 s for all experimental tests.

The method used to determine the liner section of the temperature rise vs. *ln*(t) curve for the conductivity unit was based on the following assumption. The pattern of the temperature rise of the conductivity unit can be divided into three sections: initial non-linear temperature rise, linear temperature rise, and finally additional non-linear temperature rise. Due to the finite dimensions of the probe, the first section is related to the effects of preheating of the probe itself or heat capacity of the probe. The second section is ascribed to the period at which all heat inputs to the probe is conducted only in the radial direction of the sample material. As stated earlier, the third section is

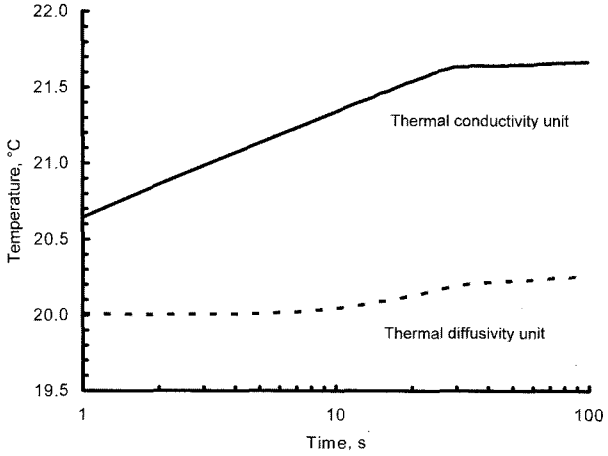

Fig. 4. Temperature profile of thermal conductivity uint and thermal diffusivity unit in water.

believed to be the effect of the convection current or axial heat flow effects caused by the finite length of the conductivity probe. Only the latter two stages of the probe temperature rise in water can be observed, because the first non-linear temperature rise phase was not very pronounced (Fig. 4).

A series of linear regression analyses was performed to determine the starting and end points of the linear section. This preliminary analysis produced the most consistent results with the starting point at 2 s. Starting from 2 s, the point which yielded the maximum R^2 value was chosen as the end point. The R^2 values of these linear regressions on the linear temperature rise varied from 0.99976 to 0.99990 for water and from 0.99998 to 0.99999 for glycerin.

Results and Discussion

Accuracy of the thermal conductivity measurements Measured values of thermal conductivity of distilled/deionized water from 21.8 to 81.9°C are shown in Table 1. The conductivity of water ranged from 0.604 W/m·K at 21.8°C to 0.662 W/m·K at 81.9°C with the standard deviations of each measurement less than 0.007 W/m·K. Measured values were in excellent agreement with the reference values published by Ramires *et al.* (3). Differences between the measured values and the reference values were less than 1.2% without using either a time correction factor or a probe calibration constant for the thermal probe.

During the experiment, the temperature of the thermal diffusivity unit did not rise higher than 0.1°C until 10 s had elapsed from the time the heater was energized (Fig. 4).

Table 1. Measured and reference values of thermal conductivity of water

Temperature (°C)	Measured* (W/m·K)	Std. deviation (W/m·K)	Reference** (W/m·K)	Difference (%)
21.8	0.604	0.00274	0.601	0.5
41.9	0.632	0.00472	0.632	0.0
61.9	0.647	0.00433	0.655	-1.2
81.9	0.662	0.00667	0.668	-0.9

*Average values of five measurements
**Ramires et al.(3)

This indicated that a sample diameter of 19.1 mm was large enough to satisfy the condition of the line heat source method.

Measured values of thermal conductivity of glycerin from 20.1 to 50.1°C are shown in Table 2. Thermal conductivity of glycerin ranged from 0.284 W/m·K at 20.1°C to 0.289 W/m·K at 50.1°C. The standard deviations of each measurement were less than 0.0004 W/m·K. Measured values were in excellent agreement with the reference values published by Eckert and Drake (15). The differences between the measured values and reference values were less than 0.7% without using either a time correction factor or a probe calibration constant for the thermal probe. Differences between the measured and the published conductivity of glycerin increased with increasing temperature, possibly due to the moisture absorption by glycerin during experiments, although no noticeable sign of leakage was observed.

The standard deviations of glycerin conductivities were less than 0.0004 W/m·K, while those of water were less than 0.0067 W/m·K. Because the viscosity of glycerin is higher than that of water, glycerin has less convection current effects. This could be the reason why the measurement of glycerin showed more consistent results than those of water as shown by the standard deviations of conductivity measurements.

Thermal conductivity of beef frankfurter Measured values of thermal conductivity of beef frankfurter meat from 20.0 to 80.0°C are shown in Table 3. The thermal conductivity values were between 0.350 and 0.389 W/m·K. Statistical analysis results showed no significant difference in conductivities among packages, whereas significant differences in conductivities were observed at different temperatures. As the temperature increased, the thermal conductivity values decreased (Fig. 5). Water drip inside the sample cell was observed at high temperatures, as similarly reported by Baghe-Khandan and Okos (9). Water loss could be the cause of decrease in the thermal conductivity values with increasing temperature.

Standard deviations for beef frankfurter meat were less than 0.0137 W/m·K, which were higher than those of glycerin, although no convection current effects were observed. This could have been caused by the non-homogeneous property of beef frankfurter meat, whereas glycerin is a homogeneous material. Because the size of the probe diameter was very small, non-homogeneous components such as meat fiber or fat near the measurement point could have a big influence on thermal conductivity measurements, which requires the measurement of a large number of samples to assure good representation of thermal conductivity value of a non-homogeneous material.

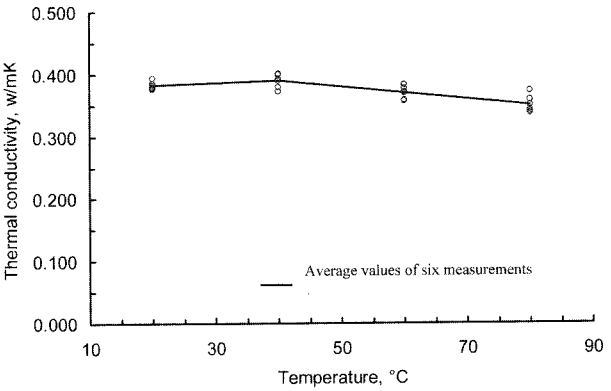

Fig. 5. Measured thermal conductivity values of beef frankfurter.

Table 3. Measured values of thermal conductivity of beef frankfurter meat

Temperature (°C)	Measured* (W/m·K)	Std. deviation (W/m·K)
20.0	0.383	0.00632
40.0	0.389	0.01135
60.0	0.369	0.01028
80.0	0.350	0.01370

*Average values of five measurements

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Table 2. Measured and reference values of thermal conductivity of glycerin

Temperature (°C)	Measured* (W/m·K)	Std. deviation (W/m·K)	Reference** (W/m·K)	Difference (%)
20.1	0.284	0.00000	0.286	-0.7
35.1	0.286	0.00043	0.286	0.0
50.1	0.289	0.00043	0.287	0.7

*Average values of five measurements
**Eckert and Drake (15)

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