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THE FLASH METHOD TO THE MEASUREMENT OF THE THERMAL PROPERTIES OF YOGURT

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Abstract. *The study proposes the use of the well – known flash method to measurement thermal proprieties in natural yogurt. The new experimental procedure was developed to sandwich liquid between a metal plate and a sample holder and measured the evaluating of the temperature on the front face, from which they obtained the thermal diffusivity and conductivity. The experimental procedure has been conducted in micro-flash apparatus, model LFA 457. The desingn of container is described in detail and the result for thermal properties was carrid out wit yogurt samples purchased in the local market. The experiments were performed 5 repetitions for each sample of yogurt. A good agreement is found between the experimental results obtained and the thermal condutivity and thermal diffusivity indicated in literature for yogurt.*

Keywords*: Thermal conductivity; Thermal diffusivity; FLASH method; yogurt.*

1. INTRODUCTION

Thermal properties of foods have recently received an increased attention because of its importance in the modeling of heat and mass transfers, and of the improvements of equipments and processes. Previously, heat transfer analysis during heating or cooling of food products was done with constant values of thermal properties. These studies were very simplified and inaccurate. Current techniques allow researchers to determine more accurately the thermal properties of foods, especially in irregular geometry varying with time, temperature and position of the product. Actually, the variation of temperature of a food product may cause significant variations in their chemical and physical properties (QUEIROZ, 2001).

The FLASH technique, which was firstly proposed by Parker et al. (1961), has become one of the most widely used method of measurement of the thermal characteristics of materials (thermal diffusivity and thermal conductivity). It presents several advantages in comparison to others classical methods of measurement. Indeed, as the measurement is achieved during a transient heating, its duration is noticeably lower than the one done with the classical guarded hot-plate method based on steady-state measurements. Moreover, the size of the sample required is relatively limited and a small amount of material is sufficient.

The FLASH method was first developed for measurements on isotropic purely conductive solid samples and it is, at present, mainly used on this kind of materials. Therefore, the use of this method to measure the thermal diffusivity of liquid or doughy materials is more problematic as it is not possible to obtain a rigid sample on which the FLASH measurement is applied to. Moreover, the accurate measurement of thermal transport in liquids is a particularly difficult task due primarily to two major issues: convective heat transport is difficult to eliminate and radiation may be important in transparent liquids. That is the reason why, for such materials, peculiar methods must be used to measure the thermal diffusivity. These include steady-state methods such as the method of coaxial cylinder and the method of parallel plates, or transient methods, mainly the hotwire method. However, some particular conditions have to be performed in these methods in order to avoid the problems previously mentioned for liquids due to radiation and convection.

Despite this, some authors have already tried to adapt the FLASH method to measurement on liquids. As a matter of fact, it may be a desirable method for measurement of the conductivity of liquids since the effect of convective heat transport could be greatly reduced by using a horizontally mounted specimen with the heat pulse impinging on the top. Schriempf (1972) was the first to develop a peculiar apparatus dedicated to measurement on liquids and applied it successfully to liquid mercury. He used a container made of insulating material. The surface of liquid was covered with a transparent plate of quartz. He measured the temperature rise at the back surface of the liquid as in the original method proposed by Parker et al. (1961). However, its method is not adapted to liquids with low thermal conductivity since the heat flow through the container is no more negligible and thus, the heat flow is no more one-dimensional. Farooq et al. (1981) proposed a similar approach based on a three-layered cell using an original sample holder made of outside layers brazed to a ring-shaped central spacer. They manage to estimate the thermal conductivity of water. Maeda et al. (1996) also proposed a special cell in which the liquid in sandwiched between an upper and a lower platinum crucible, to provide a three-layered sandwich. They applied a curve-fitting method using a three-layer analysis with a correction for the radiative component based on the transparent body assumption.

The objective of this work was the manufacture of a suitable container of known size. It was experimentally realized in a micro-flash apparatus, model LFA 457. This study can find numerous applications for measurements of many materials such as biological tissues (muscle, organs, blood) or food materials (meat, yogurt, cheese, food gels) whenever others traditional measurement methods are not suitable.

2. MATERIALS AND METHODS

2.1. Design of sample holder

Classical FLASH measurements could not be applied to liquid or pasty materials given that it requires a sample with given and immoveable dimension and shape. As a result, it was necessary to develop sample containers in which the tested sample could be introduced and its thermal diffusivity measured. The previous studies mentioned in the introduction that used the classical FLASH method have surmounted this problem by developing original receptacles allowing to contain the medium during the measurement. We have built on this idea by constructing sample containers in which the sample tested could be introduced and its thermal diffusivity measured.

Each container is made of a Brass hollow cylinder closed at its tops and bottoms by circular brass slabs (see Fig. 1). The external radius of the brass cylinder is R = 0,00515 m which corresponds to the standard radius of classical sample tested. We choose to use brass for the material composing the hollow cylinder as its thermophysical properties: thermal conductivity of brass (k_{brass}=110 W/m/K), density of brass (ρ_{bras}=8530 kg/m^3), specific heat of brass (C_{bras}=380J/kg/K), and thermal diffusivity of brass (α_{brass} ≈33,9x10^{-6}m^2/s).

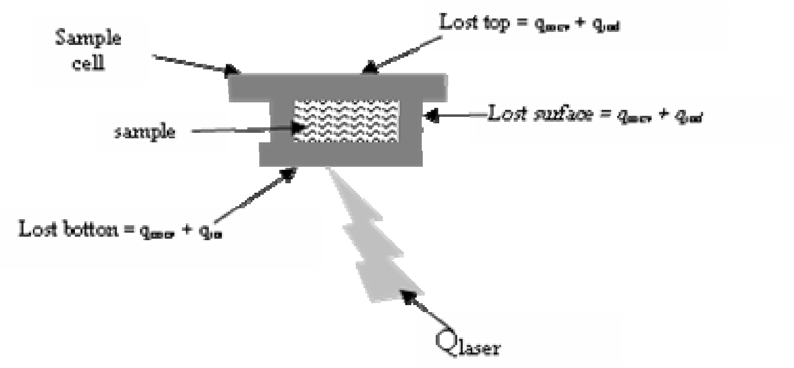

Figure 1. Representation the balance of energy in the cell of brass

2.2. Governing Equations

In our case, the bodies are made up of layers of different materials, and the solution of a heat transfer problem in such a medium requires the solution of the heat transfer problem in each layer. This, in turn, requires the specification of the boundary conditions at each *interface.* The boundary conditions at an interface are based on the requirements that (1) two bodies in contact must have the *same temperature* at the area of contact and (2) an interface (which is a surface) cannot store any energy, and thus the *heat flux* on the two sides of an interface *must be the same.* The thermal balance in a material submitted to a transient heat transfer is governed by the energy equation relating the variation of the local temperature to the heat flux divergence.

$$\rho C_p \frac{\partial T}{\partial t} = -\Delta\left(\vec{q}_{cond}\right) \qquad (1)$$

where:

ρ - density (kg m^{-3})
C_p - specific heat (J kg^{-1} K^{-1})
T - temperature (K)
t - time (s)
and $\vec{q}_{cond}$ conductive heat flux density (W m^{-2})

In our case, the medium in which the heat transfer occurs (sample container) is composed of three parts (brass, sample and brass) with different thermophysical properties. However, all the materials composing the sample container could be considered as opaque in the I.R. and thus, the radiative heat transfer can be neglected. As regards the convective heat transfer in the case of liquid materials, if we compute, for example, the Rayleigh number in water for a maximum temperature rise of the front side equal to 0.2 K, we find $Ra \approx 500$. But, for a Rayleigh number less than 1400, one can consider that natural convection phenomenon is negligible. Consequently, the convective heat transfer could also be neglected in all the part of the sample. Thus, the heat transfer is purely conductive in the entire sample. In order to simplify the resolution of the energy equation, one can remark that our sample container is one-dimensional and thus, the temperature distribution in the sample is a function of the axial z. We assume also that the thermal conductivity and the thermal diffusivity are temperature independent and uniform within each layer. In a rectangular coordinate system the mathematical model in dimensionless form is written as follows:

$$\rho C_p \frac{\partial T}{\partial t} = k \frac{\partial^2 T}{\partial z^2} \tag{2}$$

As regards the time boundary conditions, the medium is at a uniform temperature before the beginning of the heating. This, temperature is the external (T_{ext}) temperature (T) and then:

$$\forall r \text{ and } z\,,\; T(z,0) = T_{ext} \tag{3}$$

During the pulse irradiation of the sample by the FLASH (duration τ), the front face of the sample collects a heating power per surface unit $\dot{Q}$. In our numerical model, we assume that this radiative energy is entirely absorbed after a small penetration length χ and that the heat is uniformly generated along its path in the brass. Thus, the heat generation could be treated as an internal heat source. If we assume that the heating power is uniform on all the surface, we have, for the region $0 < z < \chi$; $0 < r < R_{\max}$:

$$\rho C_{pbrass} \frac{\partial T_{bras}(z,t)}{\partial t} \chi = \dot{Q} \text{ for } 0 < z < \tau \tag{4}$$

Given that the sample is composed of several parts with different thermophysical properties, several boundary conditions also occur at the interface between the different parts:

- At the horizontal interfaces between the brass and the sample ($z = e$ and $z = L$ with $r < R_{brass}$), if we neglect the thermal resistance, the conductive heat fluxes balance leads to:

$$\left(k_{brass} \frac{\partial T_{brass}}{\partial z} \right)_{z=e^-} = \left(k_{sample} \frac{\partial T_{sam}}{\partial z} \right)_{z=e^+} \text{ and } \left(k_{brass} \frac{\partial T_{brass}}{\partial z} \right)_{z=(L-e)^+} = \left(k_{sam} \frac{\partial T_{sam}}{\partial z} \right)_{z=(L-e)^-} \tag{5}$$

where k_{brass} and k_{sam} are respectively the thermal conductivity the brass and the sample ($Wm^{-1}K^{-1}$).
The analysis of the experimental data is performed with Proteus® software, provided by Netzsch, which includes different mathematical models, such as: Adiabatic (Parker et al, 1961.) and (Cowan, 1963). This paper, the model Cowan that taken into account pulse correction is definitively default as a suitable model for the intermediate analysis. However this not a limit for you as of course can use other models in the analysis.

2.3. Laser Flash Apparatus

By far the most popular and widely used instrument for measuring thermal diffusivity is the laser flash apparatus. Compared with the direct measurement of thermal conductivity, this method has the advantage of simple test piece configuration, small test piece size, applicability for a wide range of diffusivity values, great accuracy and reproducibility. Because very little time is needed for one single measurement wide temperature ranges can be covered in a few hours.

The construction, with a vertical alignment of the laser and the infrared sensor, makes a simple, horizontal insertion of the test piece possible; short distances minimize the loss of laser energy and temperature signal.

The front face of a cylindrically shaped test piece is homogeneously heated by an unfocused laser pulse. On the rear face of the test piece the temperature increase is measured as a function of time. The mathematical analysis of this temperature/time function allows the determination of the thermal diffusivity.

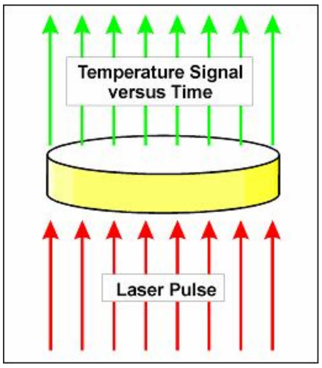

Figure 2. Representation of the principle of Laser Flash

In this study, the measurements are conducted on a LFA 457 Laser flash apparatus, with integrated solid state laser ND-GGG (1.06 m) produces a beam of intensity 10–30 J with a duration of less than 0.5 ms. The rear-face temperature of the sample is detected through infrared detector head with InSb, N_2 cooled, Ge lens and aperture with holes N_2 dewar for 24h operating time and focused on the rear face of the sample. The control, measurement and evaluation is realized by system controller TASC 414/4 for data acquisition and temperature control with the following function units (NETZSCH)

- Single analogous input, resolution +/- 2.000.000 digits;
- Inputs galvanically isolated;
- Preamplifier with range switches;
- Analogous PID temperature controller with STC features, ambient temperature compensation and safety against breakage of thermocouples; and
- Interface for computer IEEE 488 and USB.

Software and Computer System

The software permits both manual and fully automatic control of the experimental process and also the evaluation of measuring results. Integrated mathematical model functions enable thermal diffusivity and the thermal resistance between layered and sandwich test pieces to be calculated. Proteus software for Laser Flash Apparatus LFA 427 I LFA 457 for instrument control, data acquisition, storage and evaluation for MS Windows, 32 bit, with the following features:

- graphical operating surface, pull-down menu technique, online help, mouse support
- multitasking of measurement and analysis
- control of the test run interactively or fully automatically following pre-selected parameters, optimization of the signals included
- signal optimization (amplifier, analog filter)
- digital filter
- calculation of thermal diffusivity for different marginal conditions/models, with pulse width correction: adiabatically; Cowan; radiation correction; Cape-Lehman; 2 and 3 layer models
- storage of the measuring data and measuring results in a database system (hardware requirements: TA system controller TASC 41414 with LFA board, Pentium PC as of 1 GHz.

3. EXPERIENCES AND RESULTS

In this study, the “Triple layers with heat loss correction” method has been chosen. The front and rear lid of the container has been sprayed with a graphite paint with high emissivity in order for the lid to absorb and emitter enough energy. The “Fig. 3” show a typical schema of the unknown layer physically positioned on middle relative to the laser**,** i.e. that the unknown layer can also be in the middle of the sandwich or faced to the laser.

The sample was placed in the brass crucible a cylindrical shape of 10,3 mm in diameter and 1,06 mm in thickness. This brass container was placed in a chamber and the measurement was carried out under air atmosphere. The front and rear lid of the container was coated with a graphite spray to increase the signal to noise ratio. The sample was heated up to 300 K in tube furnace with resistor heater of Kanthal, fibre insulation, inbuilt protective tube of fused silica, control thermocouple type S. The infrared detectors measure the temperature responses bottom surfaces after laser irradiation.

"Figure 3" shows a typical plot of the experimental temperature response and the calculated theoretical curve. From the figure we show that they coincide and the temperature response time is longer due to the difficult of heat diffusion and large specific heat of yogurt.

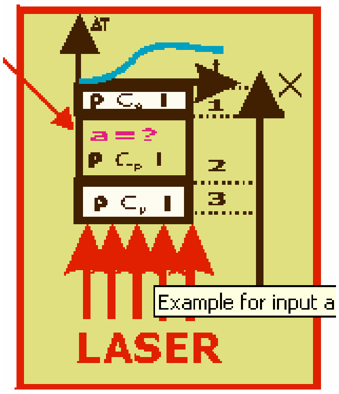

Figure 3. Arrangement and nomenclature for 3(three) layer analysis defined in measurement

In order, we conducted several thermal diffusivity measurements on three different commercial yogurt easily purchased at markets. The yogurt is composed of 90% of water and its thermal properties have been investigated experimentally by Kim and Bhowmik (1997) for different moisture content concentration and different temperature. The thermal diffusivity of the yogurt tested is regrouped in "Table 1".

The thermal diffusivity estimated by the Flash Method, values using the specific heat and density available in the literature. This procedure aims, test the viability of the flash method to measure thermal diffusivity of dairy products. For this purpose, the thermal diffusivity of yogurt was estimated using the average values of specific heat from 1597 to 3520 J / KgK (Afonso et. al., 2003) and the density from 1050 to 1033 kg/m3 (Tavman et. al., 1999). The results of these estimations are shown in Table 1.

The results for thermal diffusivity are in the range of 1.59×10^{-7} - 1.77×10^{-7} m^2 / s. These results were compared with values for yogurts available in the literature, 1:07 $\times 10^{-7}$ - 1:55 $\times 10^{-7}$ m^2 / s (Kim et. al. 1997).

Table 1. Experimental results of thermal diffusivity of yoghurt

Temp (°C)	Diffusivity (mm^2/s)	Std_Dev	Model	Laser_voltage (V)	Pulse_width (ms)
24.50	0.159	2.7784×10^{-3}	3L heatl + pc.	2690.0	0.50
25.39	0.170	2.5022×10^{-3}	3L heatl + pc.	2690.0	0.50
25.80	0.177	2.3494×10^{-3}	3L heatl + pc.	2690.0	0.50
25.97	0.169	2.5232×10^{-3}	3L heatl + pc.	2690.0	0.50

In "Figure 4 we plot the model for the adiabatic temperature of T = 25.39 °C of yogurt. We can see that from the moment when the radiation beam of the laser focuses on the anterior surface of the sample, a process of diffusion of heat by conduction takes place in the sample on the side bet. Thus, the temperature signal of the opposite side begins to increase exponentially until it reaches a maximum and constant for some period of time. It can be observed that after 6000 ms, the temperature signal of the back surface to decrease due to loss of heat to the environment (oven). This difference is due to the fact that the theoretical model does not consider the loss, giving the temperature signal to remain constant, which is not physically real, where we noticed a drop in the values of temperature.

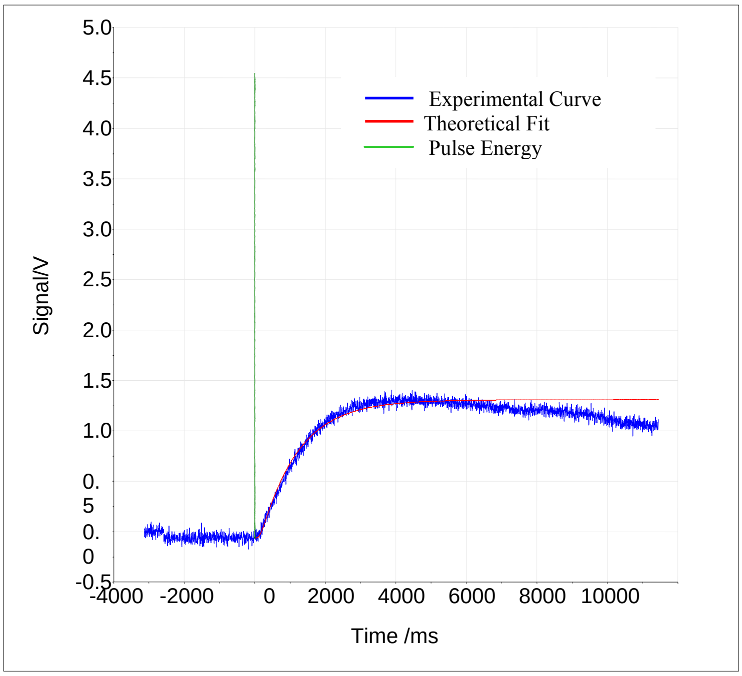

Figure 4. The evolution of temperature of the rear surface obtained experimentally for the yogurt tested.

4. CONCLUSION

We have used the flash method to measurement of the thermal conductivity of commercial yogurt. This case, requires the fabrication of a suitable sample container with known dimensions and properties in which the sample is introduced to this kind of materials for which it is not possible to obtain homogeneous rigid samples with given and immoveable dimensions and shape.

The FLASH method concerns the computation of the diffusivity from the experimental thermogram. Indeed, we have recourse to an identification procedure based multilayer model of the transient heat transfer in the axisymmetric composite sample. The overall measurement uncertainty comes mainly from the uncertainties in the sample thickness, the thermal contact resistance, the thermophysical properties of the three layers, and the inhomogeneity of the laser pulse as well as the asymmetry of the heat transfer coefficient. The inverse method allows us to evaluate the error of estimation. In our case, the uncertainties are in the order of magnitude of 10^{-3}. In sense of estimation, thermal properties are accurately known. The results obtained by our method are in good agreement with literature data. Using the parameter estimation process and the sandwiched structure, this technique is convenient to measure the thermal diffusivity of liquid. Then, flash method to be very practical for the measurement of thermal characteristics of materials used in various technological food industry (meat, yogurt, cheese, food gels). A typical advantage of this method is that a very small sample is needed with a very rapid measurement process.

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