

ESTIMATION OF THERMAL EFFUSIVITY OF POLYMERS USING THE THERMAL IMPEDANCE METHOD

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Abstract. *Thermal impedance is a way of defining the characteristics of thermal systems. It is a function that represents the relation between the frequency components of temperature and heat flux. From the experimental point of view, it is determined simply by measuring the heat flux and the temperature, simultaneously. These signals are measured only on the frontal surface of the sample. The experimental technique proposed here is very well adapted for making in situ measurements. In this work a one-dimensional semi-infinite thermal model is used. For the semi-infinite model, just the thermal effusivity, b , can be estimated. The thermal effusivity is estimated for three polymers: Polyvinyl chloride (PVC), Polymethyl methacrylate (PMMA) and Polyethylene (PE). An objective function representing the difference between experimental and theoretical values of the modulus of the thermal impedance function is minimized. The optimization technique BFGS is used with the golden section technique followed by a polynomial approximation for minimizing this objective function. For all cases studied in this work the thermal effusivity is in good agreement with literature. The difference is less than 3.6 % for PVC and PMMA. In addition an uncertainty analysis is also presented.*

Keywords: *thermal properties estimation, heat conduction, optimization, experimental methods, thermal impedance.*

1. INTRODUCTION

Information on thermal transport properties (thermal effusivity, b , thermal conductivity, λ and thermal diffusivity, α) has become increasingly important in a wide range of engineering fields, especially in the evaluation of insulation material performance. In this sense a lot of experimental techniques have been developed for the determination of these properties. These experimental techniques that allow determining the thermal properties values are based upon an identical principle: a signal is produced on the entrance face of a studied material sample, and the thermal response is then recorded at the same face or at another point on the material. This signal is generally an impulse, a periodic function or a step function. Some experimental methods have been used for determining these properties such as the hot wire, flash and photoacoustic methods.

Blackwell (1954) presented the hot wire technique for the measurement of the thermal conductivity. This technique requires inserting a probe inside the sample, and this appears to be the main difficulty of the method when applied to solid materials (it is a destructive method). Another restriction is the use of this method in metallic materials, due to the problems with contact resistance. Moreover, their high thermal conductivity would greatly reduce the maximum time of measurement. Variations of this method have been used in recent works for the thermal conductivity determination. For example, the λ determination of liquid gallium in Miyamura and Susa (2002) and in Luo *et al.* (2003) by solving inverse heat conduction problems (IHCP) in an infinite region. Parker *et al.* (1961) have developed one of the most employed methods for measuring α of solid materials. This method involves exposing a thin slab of the material to a very short pulse of radiant (or other form) energy. The thermal diffusivity is determined through the identification of the time when the rear surface of the sample reaches half of the maximum temperature rise. The use of the flash method to measure α has been employed in countless papers, for instance, in Mardolcar (2002) for rocks at high temperature, in Eriksson *et al.* (2002) for liquid silicate melts and in Santos *et al.* (2005) for polymers. However, it should be observed that in the flash method the costs with the experimental apparatus are very expensive. The Photoacoustic method for measuring the thermal effusivity of solids samples based on the theory of layered samples was proposed by Benedetto and Spagnolo (1988). The method consists of the measurement of the phase of the sound pressure generated with a front-surface illumination, as a function of frequency. The sample surface is painted by a black spray whose thermal properties have been previously determined. This allows moreover to eliminate the stray light contribution, which may be important in the case of reflecting samples, as metals with polished surface, and to improve the signal-to-noise ratio, also with low exciting power. Moreover the technique has the advantage of requiring neither laborious sample preparation nor specific ad hoc manufacturing. A possible limit is that the method can not be applied to samples for which the deposition of a surface layer may change the sample properties.

In this work, impulse signals are used in an experimental technique based on the concept of the thermal impedance. The thermal impedance method requires simultaneous measurements of the variations in heat flux and temperature of the measured surface. The sensor, placed on the system to be characterized, induces a disturbance. For a slow change in thermal characteristics and for low frequencies, the disturbance due the sensor is negligible (Defer *et al.*, 1998). The thermal impedance concept has been used in several works (Defer and Duthoit, 1994; Guimarães *et al.*, 1995; Krapez,

2000; Antczak *et al.*, 2003; Jannot and Meukam, 2004; Borges *et al.*, 2006). The estimation of thermophysical properties using thermal impedance method in materials, assumed to be semi-infinite, is very well adapted for making in situ measurements. This method is particularly suitable for studying civil engineering materials. However, for the case of semi-infinite thermal model the medium only depends on its thermal effusivity. In this case, the effusivity is the only parameter that can be identified. It is also possible to determine the thermal conductivity or thermal diffusivity, but one of these values must be known.

The purpose of this paper is to determine the thermal effusivity for three polymers: Polyvinyl chloride (PVC), Polymethyl methacrylate (PMMA) and Polyethylene (PE). For this, a one-dimensional semi-infinite thermal model is used. An objective function representing the difference between experimental and theoretical values of the modulus of the thermal impedance function is minimized to obtain the effusivity. In order to minimize this objective function the optimization technique BFGS is used with the Golden Section technique followed by a polynomial approximation. The thermal effusivity results are in good agreement with literature. For all the cases studied the difference is less than 3.6 % for PVC and PMMA. In the case of PE the difference is higher because the number of experiments used was only 1024 points. An error analysis is also conducted to show the improved accuracy of the results of b obtained in the present study. In fact, the method proposed in this work represents an alternate form to estimate b , it can be used for a large range of thicknesses, and adapted for *in situ* application.

2. THEORETICAL ASPECTS

2.1. Temperature model

The thermal model is given by a one-dimensional semi-infinite model as shown in Fig. 1, where ϕ_1 represents the heat flux and θ_1 the upper surface temperature.

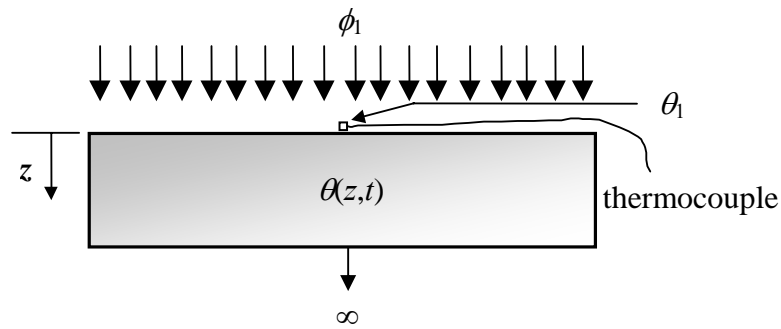


Figure 1. Thermal model proposed.

The one-dimensional diffusion equation can be given by:

$$\frac{\partial^2 \theta(z,t)}{\partial z^2} = \frac{1}{\alpha} \frac{\partial \theta(z,t)}{\partial t}, \quad (1)$$

subjected to the following boundary conditions:

$$-\lambda \left. \frac{\partial \theta(z,t)}{\partial z} \right|_{z=0} = \phi_1(t), \quad (2)$$

$$\theta(z \rightarrow \infty, t) = 0, \quad (3)$$

and the initial condition

$$\theta(z,0) = \theta_0, \quad (4)$$

where $\theta(z,t) = T(z,t) - T_0$. In this work, the thermal problem (Eqs. 1-4) is solved in the frequency domain by using the idea of the input/output dynamical system as presented below.

2.2. Theoretical thermal impedance

The technique proposed here uses the input/output dynamical system (Fig. 2), where x and y are the input and output signals, respectively.

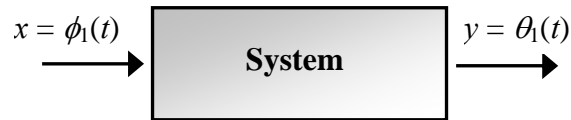


Figure 2. Input/output model.

For a semi-finite medium (Fig. 1) the theoretical thermal impedance is given by

$$Z(f) = \frac{1}{b\sqrt{j2\pi f}}, \quad (5)$$

where b is the thermal effusivity ($b = \lambda/\sqrt{\alpha}$) that is the ability to exchange heat with the environment by the material, f is the frequency and j is the imaginary unit.

2.3. Experimental thermal impedance

By analogy with electrical or mechanical systems, the experimental thermal impedance, Z_e , of a conducting system can be defined in the access plane by the following equation

$$Z_e(f) = H(f) = \frac{\Theta_1(f)}{\Phi_1(f)} = \frac{Y(f)}{X(f)}, \quad (6)$$

where $\Theta_1(f)$ and $\Phi_1(f)$ represent respectively, the Fourier transforms of $\theta_1(t)$ and $\phi_1(t)$. $Z_e(f)$ is equivalent to the frequency response function and $X(f)$ and $Y(f)$ are the input and output, in the transformed f plane respectively. Their values are found by the application of the Fast Fourier transform of the data $x(t)$ and $y(t)$. The Fourier transforms were performed numerically by using the Cooley-Tukey algorithms (Discrete Fast Fourier Transform) (Bendat & Piersol, 1986). A more stable impedance function can be obtained by multiplying Eq. (6) by the complex conjugate of $X(f)$ given by:

$$Z_e(f) = \frac{Y(f)X^*(f)}{X(f)X^*(f)} = \frac{S_{xy}(f)}{S_{xx}(f)}, \quad (7)$$

where S_{xy} is the cross-spectral density of $x(t)$ and $y(t)$ and S_{xx} is the autospectral density of $x(t)$.

2.4. Thermal effusivity estimation

The minimization of an objective function, Obj , based on the difference between experimental and calculated values of the modulus of Z is one way to determine the thermal effusivity. This function can be written by:

$$Obj = \sum_{i=1}^{nf} (|Z_e(i)| - |Z(i)|)^2, \quad (8)$$

where i represents the discrete frequency measurements, nf is the number of frequency measurements used in the optimization process and $|Z_e|$ and $|Z|$ are the experimental and calculated values of the modulus of Z . The sequential unconstrained optimization technique BFGS (Broydon-Fletcher-Goldfarb-Shanno) is used to minimize the objective function (Eq. 8). In addition, the Golden Section method is used in the one-dimensional search, followed by a cubic polynomial interpolation (Vanderplaats, 1984). The package Design Optimization Tools (DOT) (Vanderplaats, 1995) is used to apply the optimization technique.

3. EXPERIMENTAL PROCEDURE

Figure 3 shows the experimental apparatus used to determine the thermal effusivity of polymers materials. Three polymers samples were used: Polyvinyl chloride (PVC), Polymethyl methacrylate (PMMA) and Polyethylene (PE), all the samples have thickness of 50 mm and lateral dimensions of 305 x 305 mm. These lateral dimensions were used in

order to guarantee that the sample could be submitted to a unidirectional and uniform heat flux on its upper surface, where at time $t = 0$ (T_0), the sample is in thermal equilibrium. The heat is supplied by a 22Ω electrical resistance heater, covered with silicone rubber, with lateral dimensions of 305×305 mm and of 1.4 mm thickness. A heat flux transducer with lateral dimensions of 50×50 mm, thickness of 0.1 mm, and constant time less than 10 ms measures the heat flux input (Leclercq and They, 1983). The transducer is based on the thermopile conception of multiple thermoelectric junction (made by electrolytic deposition) on a thin conductor sheet. The electromotive force (emf) measured is proportional to the heat flux crossing the measured area. The evolution of temperature with time at the upper frontal boundary surface is measured using a surface thermocouple (k type). The signals of temperature and heat flux are acquired by a data acquisition system HP Series 75000 with a voltmeter E1326B controlled by a personal computer.

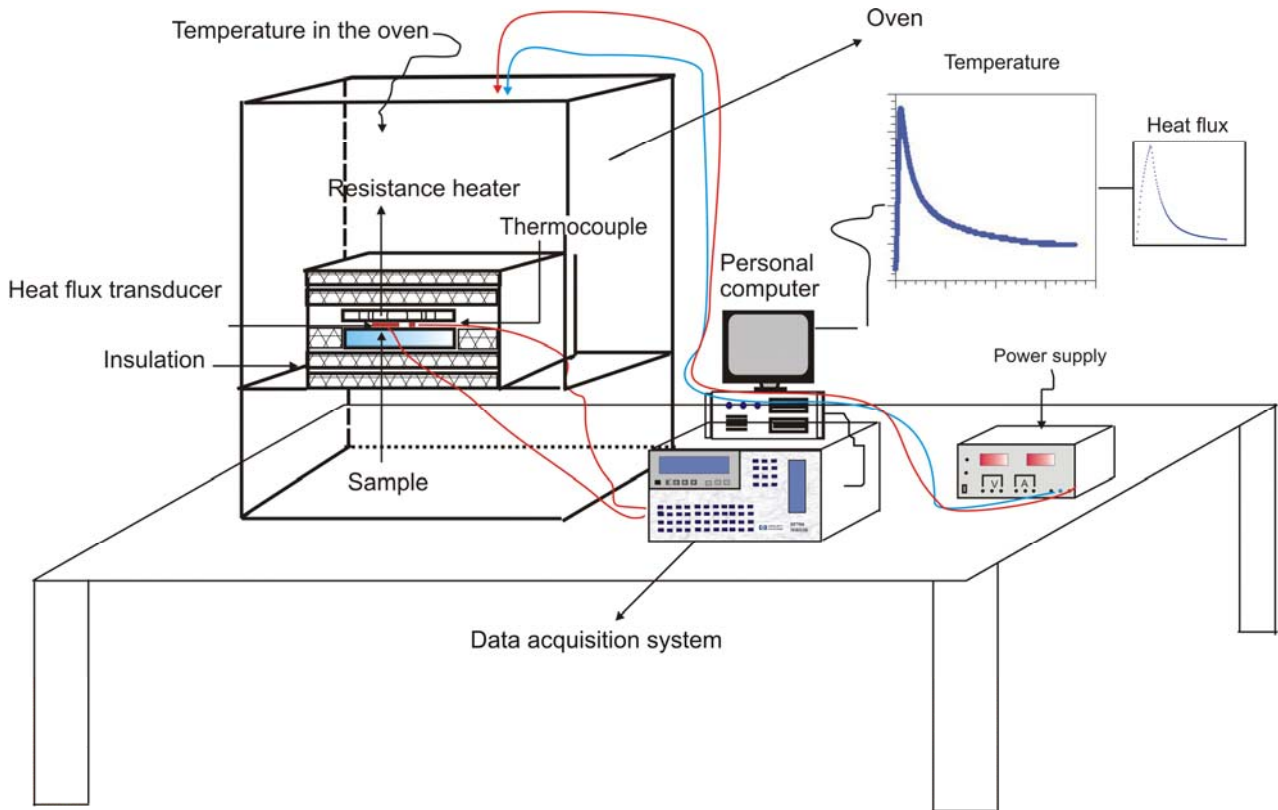


Figure 3. Scheme of the experimental apparatus.

4. DEFINITION OF THE EXPERIMENTAL PARAMETERS

4.1. Comparison between finite and semi-finite thermal models

As already mentioned, the semi-infinite model must be used for the determination of b . In this work, the used samples are finites with a thickness of $L = 50$ mm. However, under certain conditions of time the thermal behavior of a finite medium of thickness L can be considered identical to the semi-infinite medium (Beck *et al.*, 1992). In addition, this behavior tends to be the same as larger the thickness of the medium and smaller the time of heat diffusion. In order to verify this condition, a comparison between a finite model and a semi-infinite model is done for the calculated temperatures on the frontal surface (Fig. 4a). The used finite model is a one-dimensional model with heat flux imposed on the frontal surface and isolated on the other surface. In this case, the signal of heat flux supplied to each one of the samples, studied in this work, is used for the calculation of these temperatures. Figure 4a presents this comparison for the sample of PVC. For this study the values of thermal properties $\alpha = 1.28 \times 10^{-7} \text{ m}^2/\text{s}$ and $\lambda = 0.156 \text{ Wm/K}$ from Lima e Silva *et al.* (2003) were used. However, a small discrepancy occurs, it can be noted that the residuals presented in Fig. 4b are situated in the range of uncertainty measurement of thermocouples, which in this work is ± 0.3 K. Thus, the use of the semi-infinite thermal model is guaranteed. The same results were obtained for the PMMA and PE samples.

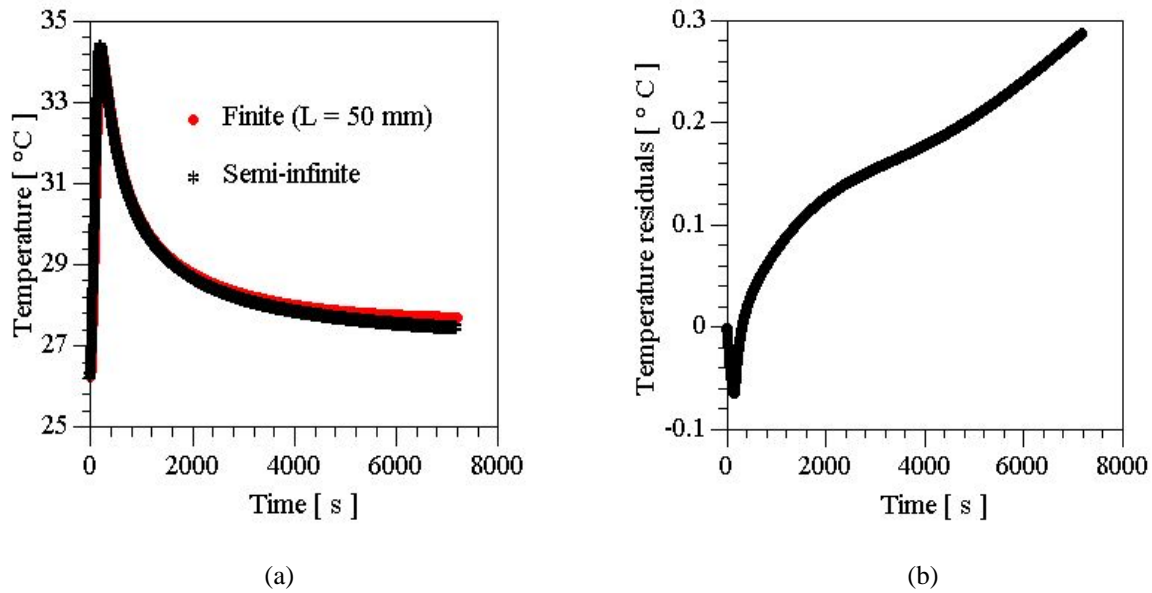


Figure 4. a) Comparison of the temperature evolution for the finite and semi-infinite models b) Residuals of temperature.

4.2. Sensitivity analysis

Another important way of analysis is to study the behavior of the sensitivity coefficient involved in the process. The analyzed sensitivity coefficient S_b is defined as the first derivative of the modulus of Z with respect to the parameter b . Figures 5a, 5b and 5c show the behavior of S_b in the frequency domain for each sample. It can be seen that for frequencies greater than 2.0×10^{-3} Hz, S_b becomes constant and a little contribution is given to the estimation procedure for all cases. In this case, fourteen points for PVC, nine points for PMMA and fourteen points for PE were used to determine b .

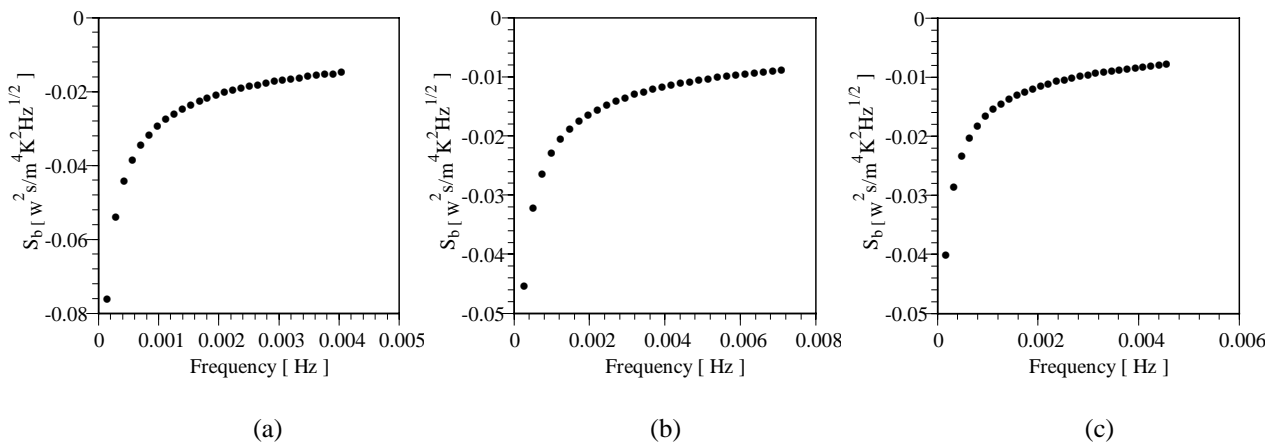
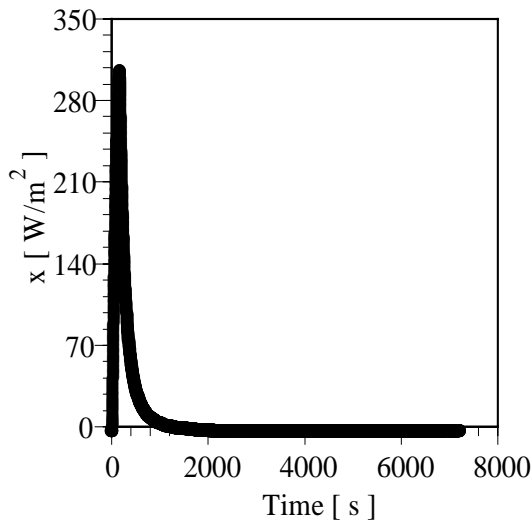


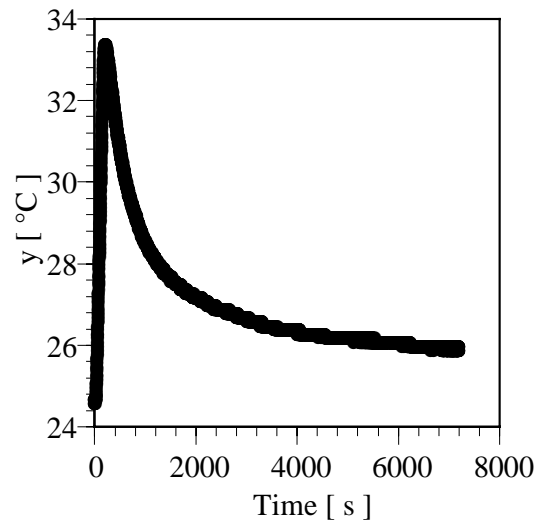
Figure 5. Sensitivity coefficients related to b for a) PVC, b) PMMA and c) PE

5. RESULTS

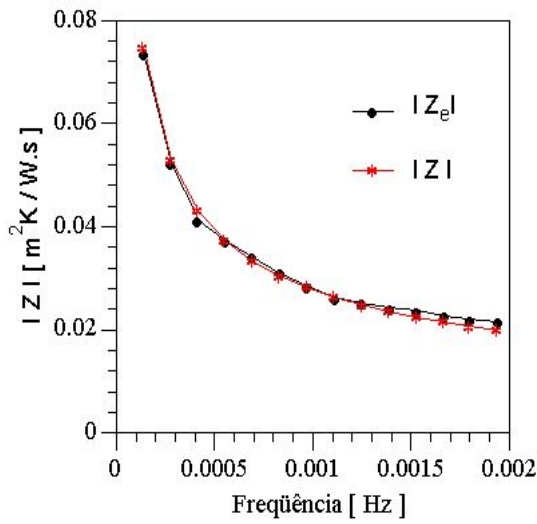
Fifty independent runs for PVC, forty for PMMA and twenty for PE were realized. The number of points used for each sample was 8192 for PVC, 4096 for PMMA and 1024 for PE. The time steps, Δt , were 0.8793 s for PVC, 1 s for PMMA and 6.242 s for PE. The global time of heating, t_h , for PVC and PMMA was approximately 150 s and for PE it was approximately 90 s. The generated heat pulse was 40 V (dc) supplied for the three materials. Figures 6a and 6b present, respectively, the heat flux and temperature signals in the upper surface of the PVC sample. As the heat flux and temperature curves for the PMMA and PE samples have the same behavior of the PVC, these curves are presented for PVC sample only. In Figure 7a a comparison between estimated and experimental modulus of the impedance function for PVC is presented. It can be observed a very good agreement. Figure 7b shows the residuals. The same agreement presented in Fig. 7b was found for the samples of PMMA and PE. Figures 8 to 10 present the values obtained for b for all samples studied.



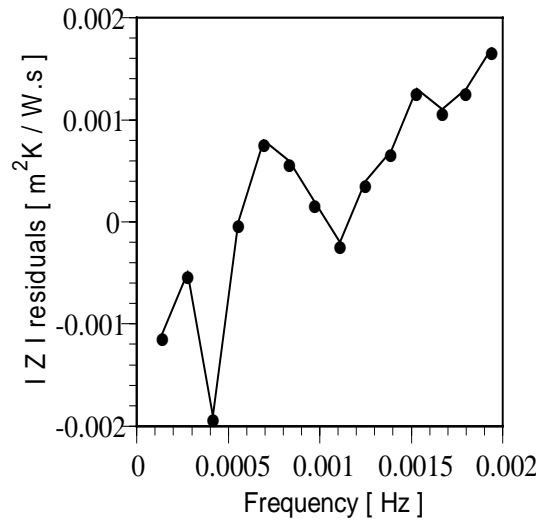
(a)



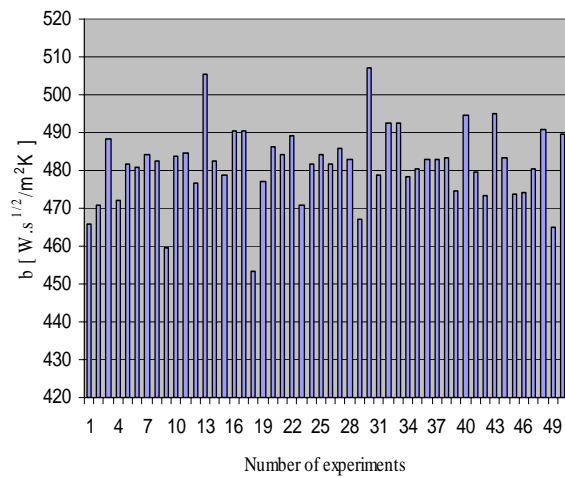
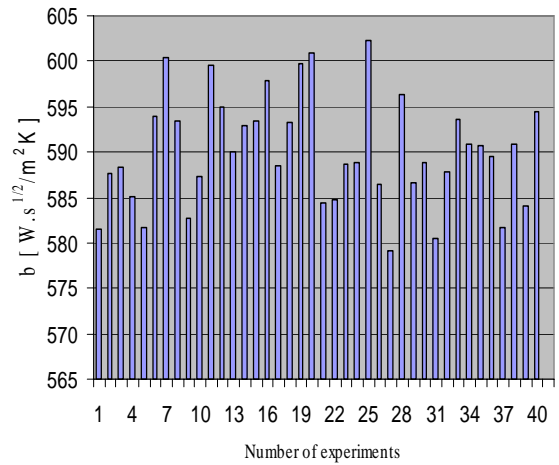
(b)

Figure 6. a) Evolution of the input signal $x(t) = \phi_1(t)$ b) Evolution of the output signal $y(t) = \theta_1(t)$ 

(a)



(b)

Figure 7. a) Experimental and calculated modulus of Z b) Residuals of $|Z|$ Figure 8. Histogram of b for PVCFigure 9. Histogram of b for PMMA

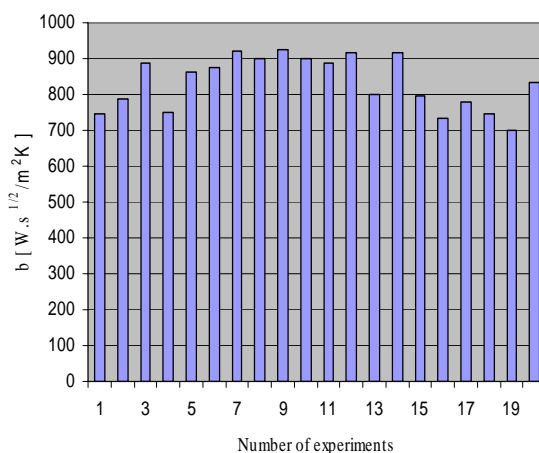


Figure 10. Histogram of b for PE

When analyzing a series of measurements some data can present uncertainties that interfere in the process, causing inexact values (Taylor, 1997). The experiments for each sample were repeated many times in order to reduce these uncertainties, as presented in Figs. 8 to 10. Thus, the value of b found for each sample is an average of the values presented in Figs. 8 to 10. This mean value of b was obtained using a confidence interval of 99.7 %. Table 1 presents the estimated mean value and standard deviation of b for the three samples studied. In this table, the values of b from literature were obtained from Defer *et al.* (2001), for PVC, from Gustavsson *et al.* (2005) for PMMA and from Jannot and Meukam (2004) for PE. It can be observed that the results presented good agreement with the respective reference values.

Table 1. Statistic data to the average values estimated for b .

Sample	b (W.s ^{1/2} /m ² K)	Literature (W.s ^{1/2} /m ² K)	Difference (%)
PVC	480.94 ± 0.93 %	498	3.55
PMMA	589.73 ± 0.50 %	573	2.83
PE	828.74 ± 5.76 %	888.6	7.22

6. UNCERTAINTY ANALYSIS

6.1. Introduction

Experiences have shown that no measurement, however carefully made, can be completely free of uncertainties. Since most of science depends on measurements, it is crucially important to be able to evaluate these uncertainties and to keep them to a minimum. Thus, errors are not mistakes and you cannot avoid them by being very careful, only make them as small as possible. In measurement processes of physical largenesses as temperature and heat flux, the validation of the measured values is based on the comparison of an unknown variable with a reference value. However, several factors interfere in the process, causing inexact values. The deviations are the result of inherent errors to the measurement technique, the reference standard value and the resolution of the measurement equipment. In this sense, any measured value is inexact and must be dealt with care (Taylor, 1997). Once the thermal effusivity determination is the result of these measurements it is natural that their values are in the same form (inaccurate). For the determination of b with accuracy, the errors must be minimized. The causes of these errors are many. In the case of thermal properties determination, the most common ones are: errors in the restrictions of the theoretical model, errors in temperature and heat flux measurements due to calibration, time response and thermal contact resistance in the sensors and the measurement uncertainty in the data acquisition of the signals. In addition, numerical errors can also be produced when calculating the distributions and using the Fast Fourier Transform and they must be taken into account.

6.2. Uncertainty analysis in the determination of b

The used procedure for the estimation of error in the determination of b is based on the propagation calculus of the uncertainties in the measurement of the original variables (heat flux and temperature) and the numerical calculus of the Fast Fourier Transform in these signals. Once the objective function, Obj , is based on the difference between experimental and calculated values of the modulus of Z , in this work the hypothesis of linear propagation is used. The hypothesis of one-dimensional heat flux is qualitatively analyzed through the numerical simulation. The results of the simulation assure the hypothesis of one-directional heat flux. These simulations were done inside the experimental conditions used in this work as: materials of low conductivity (polymers), sample with dimensions of 305 x 305 x 50 mm and the total time of heating of approximately 150 s for the three samples (Lima e Silva, 2000). Bias errors due to the thermal contact between the sensors and samples were analyzed by Guimarães (1993). The results obtained by Guimarães (1993) assure the hypothesis of perfect thermal contact between the sensors and the sample. In this sense, the measurement uncertainty in the original variables is estimated as the partial uncertainties in the data acquisition system and in the calibration of the temperature and heat flux sensors. From the theory of linear propagation errors (Taylor, 1997), the uncertainty of the temperature can be calculated from the uncertainties of the data acquisition system and temperature sensor (thermocouple) as:

$$U_{\theta}^2 = U_{data\ aquis}^2 + U_{therm}^2, \quad (9)$$

Also from the theory of linear propagation errors, the uncertainty of the heat flux is obtained from the uncertainties of the data acquisition system and heat flux sensor (heat transducer) as:

$$U_{\phi}^2 = U_{data\ aquis}^2 + U_{heat\ trans}^2, \quad (10)$$

Thus, the uncertainty in the objective function Obj can be obtained through the analysis of the uncertainty propagation in the temperature (Eq. 9), in the heat flux (Eq. 10) and in the numerical errors produced when using the Fast Fourier Transform. The calculus of the FFT is done over temperature and heat flux. In this case the uncertainty in the Obj can be given by

$$U_{Obj}^2 = U_{\theta}^2 + U_{\phi}^2 + U_{FFT\ \theta}^2 + U_{FFT\ \phi}^2. \quad (11)$$

The used data acquisition is a Data Acquisition/Control HP 75000 produced by Hewlett Packard with a voltmeter E1326B controlled by a personal computer. Excepting the relative uncertainty to the sensors, all the other elements of measurement, as the voltmeter, cables and functional modules, operating in the range of temperature between 20 and 35 °C, auto-zero on with NPLC = 1, range of 125 mV and with warm-up of one hour, have an uncertainty estimated in

$$U_{data\ aquis} = 0.45\%. \quad (12)$$

The thermocouple uncertainty can be obtained from the uncertainty of the temperature controller bath (Thermometry Calibration System) manufactured by ERTCO, with maximum fluctuation of ± 0.1 °C and mean temperature of 30.5 °C.

$$U_{therm} = 0.66\%. \quad (13)$$

The uncertainty of the heat transducer can be estimated from the previous calibration, considering the uncertainties in the tension, current and area of measurement, that is,

$$U_{heat\ trans} = U_V^2 + U_I^2 + U_S^2 = 1.85\%. \quad (14)$$

The uncertainty in the Fast Fourier Transform is obtained from the comparison of the calculated temperatures numerically (using FFT) with analytically (Lima e Silva *et al.*, 1998) and was estimated in

$$U_{FFT} = 0.03\%. \quad (15)$$

For the objective function of impedance the uncertainty was obtained by substituting Eqs. (12-15) in Eq. (11) as

$$U_{Obj} = 2.07\%, \quad (16)$$

Thus, from the hypothesis of linear propagation of errors, the uncertainty in the estimated thermal effusivity is given by:

$$U_b = U_{Obj} = 2.07 \%. \quad (17)$$

It can be observed that the estimated value for the uncertainty of b is conservative. In situations as in the determination of the difference of experimental temperature, the random errors of measurement tend to annul in the average. This fact also contributes for a lower real uncertainty for the calibration of the heat transducer. However, the uncertainty presented in this work should only serve as a reference value. Some aspects, as the possible current leakage in the calibration of the heat flux transducer were not analyzed. In addition the influence of the time response in the heat flux transducer and thermocouple (less than 0.01 s) was also despised. It can be observed that the sampling interval of the signals were of 0.879 s for PVC, 1.0 s for PMMA and 6.243 for PE. In spite of this, the values of b obtained for PVC, PMMA and PE are placed inside the band of uncertainty specified for the reference values. These results indicate that the estimated uncertainty for the analyses presented above is sufficiently representative.

7. CONCLUSIONS

This work showed that thermal impedance could be used as a means of non-destructive testing and that it provides quantitative information on a conductive system. The experimental procedure is very simple with inexpensive devices used for the measurements. It is easy to implement and involves simply placing sensors of temperature and heat flux on the surface of the material. It can be observed that the results present good agreement with the respective reference values. This technique can also be used for conductor applications.

8. ACKNOWLEDGEMENTS

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