IDENTIFICATION OF TEMPERATURE-DEPENDENT THERMAL PROPERTIES OF SOLID MATERIALS

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Abstract. *The thermal properties estimation, as the thermal diffusivity, α, and the thermal conductivity, λ, is extremely important in the identification of new materials, especially in the evaluation of insulation material performance at different level of temperature. However, the majority of the existent techniques determine these thermal properties in the room temperature. This work proposes an experimental technique to simultaneously obtain the thermal properties α and λ varying with temperature. The thermal model uses a transient one-dimensional heat transfer problem. Several classic methods have been used for determining α and λ of metallic and non-metallic materials, as the Hot Wire and Flash Methods. The two methods can be used for simultaneous determination of these properties, but usually the Hot Wire method is indicated for λ determination, while the Flash method for α determination. An alternative technique to obtain these properties simultaneously is based on the use of an input/output dynamical system. In this technique a phase angle objective function in the frequency domain is used for the estimation of α and a least square error objective function in the time domain is used for the estimation of λ. A polyvinyl chloride sample is exposed at different temperatures inside an oven and the properties are estimated with an additional heating of approximately 4.5 ^oC at frontal sample surface. Analyses of sensor location sensitivity are also presented.*

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

1. Introduction

Accurate modeling of thermal systems is becoming increasingly important. Designers are relying more on computer simulations to design complex thermal systems, with less dependence on costly experimental testing and validation. Consequently, materials thermal properties and estimation techniques are required to support simulation-based designs. A considerable amount of effort has been devoted for the fulfillment of the ever-growing demand for new materials with relevant application in engineering, especially in the evaluation of insulation material performance. In this case, the characterization of thermophysical properties as thermal diffusivity, α, and thermal conductivity, λ, is essential for the correct prediction of the thermal behavior of these materials. Besides when developing a new thermal-insulating material, one must perform a good deal of tests so that the heat-resistant properties of the material can be analyzed under various heating conditions corresponding to different operating conditions. In this manner, methods to estimate thermal properties should be general enough to treat temperature dependence. In this paper procedures are described for estimating temperature-dependent thermal properties (thermal diffusivity and thermal conductivity). Some experimental methods have been used for determining these properties. As the hot wire and flash methods. Blackwell (1954) presents the hot wire technique for the measurement of the thermal conductivity. The technique requires inserting a probe inside the sample, and this appear to be the main difficulty of the method when applied to solid materials. Variations of this method have been used in recent works for the thermal conductivity determination as a function of temperature. For example, in Miyamura and Susa (2002) for λ determination of liquid gallium and Luo *et al*. (2003) determine λ by solving IHCP in an infinite region. Parker *et al*., (1961) have developed one of the more 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 reach half of the maximum temperature rise. The use of flash method to measure α has been employed in countless papers as in Mardolcar (2002) in rocks at high temperature and Eriksson *et al*. (2002) in Liquid silicate melts. It should be observed that in both hot wire and flash methods only one property can be obtained with precision. Recently, many methods have been developed to determine the temperature-dependent thermal properties of materials and most studies employed the nonlinear least-squares formulation (Beck *et al*. 1985, Alifanov, 1994 and Dowding *et al*. 1999). This method minimizes the formulation from the sum of the squares of the difference between the experimental measurements and the calculated responses of the system. Although, some efforts should be done to avoid low sensitivity regions for obtaining both properties with confidence. In this work, the main objective is to develop an efficient experimental technique to determine simultaneously α and λ varying with temperature of non conductor solid materials. In order to obtain this objective the frequency and time domains are mixed to estimate the thermophysical properties simultaneously and in an independent way. The great advantage of this procedure is the easiness in data experimental manipulation. Several experiments that cover a range from 20 °C and 70 °C are analyzed. Results of α and λ are in good agreement for a Polyvinyl chloride (PVC) sample. In fact, the method proposed in this work represents an alternate form of simultaneous estimation of α and λ varying with temperature for materials of low thermal conductivity.

2. Theoretical fundamentals

2.1. Temperature model

In Figure 1, ϕ_1 represents the heat flux, θ_1 the upper surface temperature and θ_2 the lower surface temperature. The thermal model can be given by a one-dimensional model as shown in Fig. (1).

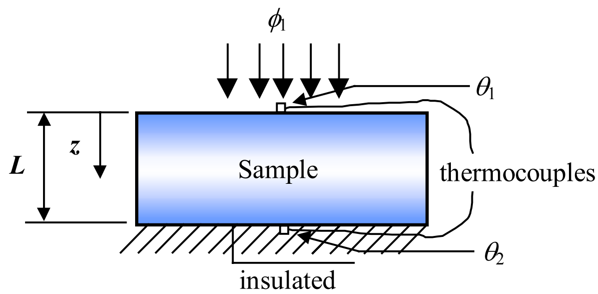


Figure 1. Thermal model proposed

The heat diffusion equation for the problem shown in Fig. 1 can be written as:

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

subjected to the following boundary conditions

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

$$\left.\frac{\partial \theta(z,t)}{\partial z}\right|_{z=L} = 0, \tag{3}$$

and initial condition

$$\theta(z,0) = \theta_0, \tag{4}$$

where $\theta(z,t) = T(z,t) - T_0$. The temperature solution is obtained through the numerical resolution of Eqs. (1-4).

2.2. Dynamical system

The technique proposed here uses the input/output dynamical system to obtain α (Fig. 2), where x is the input signal and y is the output.

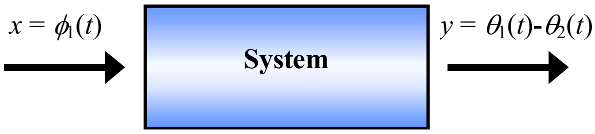


Figure 2. Input/output model

Using the convolution theorem (Bendat & Piersol, 1986), the impedance function can be identified in the frequency domain by

$$Z(f) = H(f) = \frac{\theta_1(f) - \theta_2(f)}{\phi_1(f)} = \frac{Y(f)}{X(f)} \tag{6}$$

where $Z(f)$ is the impedance function, that is equivalent to the frequency response function and $X(f)$ and $Y(f)$ are the input and output, respectively, in the transformed f plane. Their values are found by application of the finite Fourier transform of the data $x(t)$ and $y(t)$. The Fourier transforms are 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 $\boldsymbol{X}(f)$,

$$Z(f) = \frac{Y(f)X^*(f)}{X(f)X^*(f)} = \frac{S_{xy}(f)}{S_{xx}(f)} \tag{7}$$

where S_{xy} is the cross-spectral density of $x(t)$ and $y(t)$ and S_{xx} is the autoespectral density of $x(t)$. In Eqs.(1-4) it can be observed the frequency response $Z(f)$ is strongly dependent of the thermal properties, it means:

$$Z(f) = \frac{\theta_1(f) - \theta_2(f)}{\phi_1(f)} = \mathit{function}\ (\alpha, \lambda) \tag{8}$$

In polar form, $Z(f)$ can be written as

$$Z(f) = |Z(f)| e^{-j\varphi(f)} \tag{9}$$

where $|Z(f)| = |S_{xy}(f)| / S_{xx}(f)$, and $\varphi(f) = \varphi_{xy}(f)$ represent, respectively the modulus and the phase factor of $Z(f)$. The phase factor can be written by

$$\varphi(f) = \text{arctang}\left[\operatorname{Im} Z(f) / \operatorname{Re} Z(f)\right] \tag{10}$$

where Im$Z(f)$ e Re$Z(f)$ are the real and imaginary parts of Z. As well as, Borges *et al*. (2004) the phase angle of the impedance function $Z(f)$and the time evolution of superficial temperatures, $\theta_1(t)$ and $\theta_2(t)$ are the experimental basis to be used for estimation of thermal diffusivity and thermal conductivity respectively.

2.3. Thermal diffusivity estimation: frequency domain

The fact that the phase factor is just a function of the thermal diffusivity α is the great convenience of working in the frequency domain. The basic idea here is the observation that the delay between the experimental and theoretical temperature is an exclusive function of α. Therefore, the minimization of an objective function, S_p, based on the difference between of the experimental and calculated values of the phase is the way to determine the thermal diffusivity. Then, this function can be written by

$$S_p \sum_{i=1}^{Nf} \left(\varphi_e(i) - \varphi_t(i)\right)^2 \tag{11}$$

where φ_e and φ_t are the experimental and calculated values of the phase factor of Z respectively. The theoretical values of the phase factor are obtained from the identification of $Z(f)$ by Eq. (10). In this case the output $Y(f)$ is the Fourier transform of the difference $\theta_1(t)$ - $\theta_2(t)$ obtained by the numerical solution of Eqs. (1-4) by using the finite volume method. In fact, this procedure avoids the necessity of obtaining an explicit and analytical model of Z(*f*). In this work, the minimization of Eq. (11) is done by using the golden section method with polynomial approximation (Vanderplaats, 1984).

2.4. Thermal conductivity estimation: frequency domain

Once the thermal diffusivity value is obtained, an usual objective function based on temperature error can be used to estimate the thermal conductivity. In this case, there are no identifiability problems once just one variable is being estimated. Therefore, the variable λ will be the parameter that minimizes the least square function, S_{mq}, based on the difference between the calculated and experimental temperature defined by

$$S_{mq} = \sum_{j=1}^{s} \sum_{i=1}^{n} \left[\theta_e (i, j) - \theta_t (i, j)\right]^2 \tag{12}$$

where n is the total number of time measurements and s represents the number of the sensor. The optimization technique used to obtain λ is also the golden section method with polynomial approximation (Vanderplaats, 1984).

3. Sensor location, sensitivity analysis and sample dimensions

Once the method proposed in this work uses the frequency domain, it is necessary that the temperature signal, θ, goes to zero after the heating. In this case, two thermocouples must be used and the difference between these temperatures must go to zero. When two thermocouples are used only on the upper surface, for non conductor solid materials (in a two-dimensional or three-dimensional case), it is very difficult to obtain gradients with high enough values to allow any estimation (Fig. 3). To avoid this problem, thermocouples are used here at upper and lower surfaces. Figure 4 shows the sample temperature distribution that indicates a great gradient in the thickness direction

Another important way of analysis is the behavior of the sensitivity coefficients involved in the process. The first sensitivity coefficient analyzed was the $S_{\varphi,\alpha}$ that is defined as the first derivative of the phase angle with respect to the α parameter (Eq. 13). Figure 5 shows the behavior of $S_{\varphi,\alpha}$ in the frequency domain. It can be seen that for frequencies greater than 1.0 x 10^{-3} Hz, $S_{\varphi,\alpha}$ becomes constant and a little contribution is given for the estimation procedure. This fact reduces the analysis band and establishes the interest frequency in values less than 0.001 Hz. The other important coefficient is related with λ in the time domain. Figure 6 presents the $S_{\theta,\lambda}$ coefficient that is defined as the first derivative of the difference between the temperature model θ at lower and upper surfaces, with respect to λ. The high values of this coefficient shows the great advantage of thermal conductivity estimation in time domain. In Figure 6 for times greater than 1000 s the sensitivity coefficient becomes negligible. The boundary conditions used in the theoretical model must be guaranteed in the experimental apparatus. It means, the isolated condition at the reminiscent surface needs to be reached for the success of the estimation techniques. A good way to reach the isolation condition in the horizontal direction is the use of a large sample.

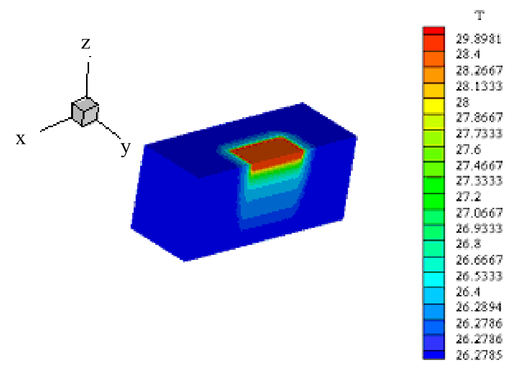


Figure 3. Temperature evolution in the frontal surface

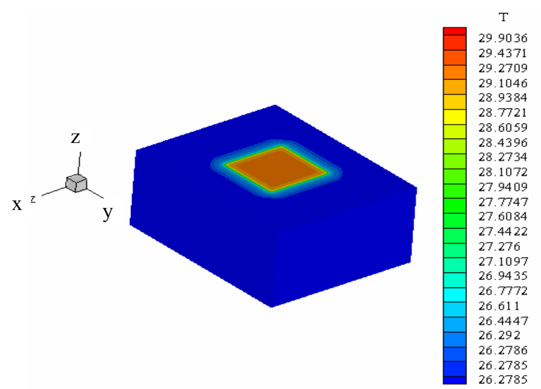


Figure 4. Temperature evolution in the width of the sample

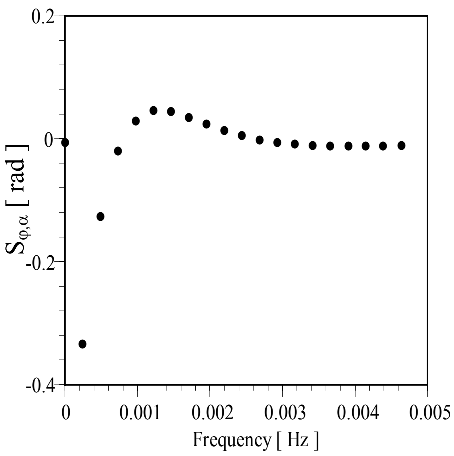


Figure 5. Sensitivity coefficient related to α

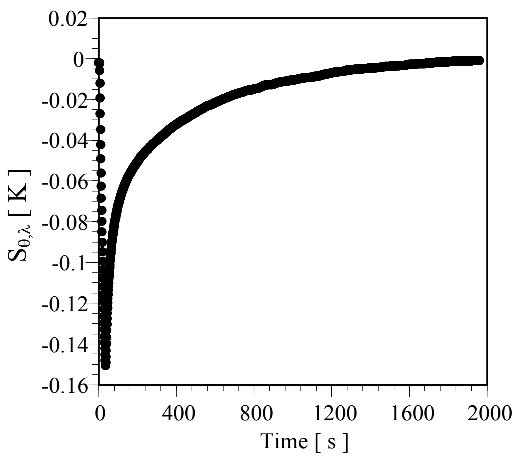


Figure 6. Sensitivity coefficient related to λ

$$S_{\varphi,\alpha} = \frac{\alpha}{\psi} \frac{\partial \varphi}{\partial \alpha} \tag{13}$$

$$S_{\theta,\lambda} = \frac{\lambda}{\theta_1 - \theta_2} \frac{\partial(\theta_1 - \theta_2)}{\partial \lambda} \tag{14}$$

4. Experimental apparatus

A polymer sample of Polyvinyl chloride (PVC) with thickness of 25 mm and lateral dimensions of 245 x 245 mm was used. These lateral dimensions were used in order to guarantee the sample is submitted to a unidirectional and uniform heat flux on its upper surface. Figure 7 shows the experimental apparatus where at time $t = 0$, the sample is in thermal equilibrium at T_0. The heat is supplied by a 10.5 Ω electrical resistance heater, with lateral dimensions of 100 x 100 mm and thickness of 0.2 mm. The heat flux are acquired by a transducer with lateral dimensions of 50 x 50 mm, thickness 0.2 mm, and constant time less than 10 ms. The transducer is based on the thermopile conception of multiple thermoelectric junction (made by electrolytic deposition) on a thin conductor sheet (Güths, 1994). The temperatures are measured using surface thermocouples (type T). The signals of heat flux and temperatures are acquired by a data acquisition system HP Series 75000 with voltmeter E1326B controlled by a personal computer. The temperature in the oven was controlled by a temperature controller Watlow 93.

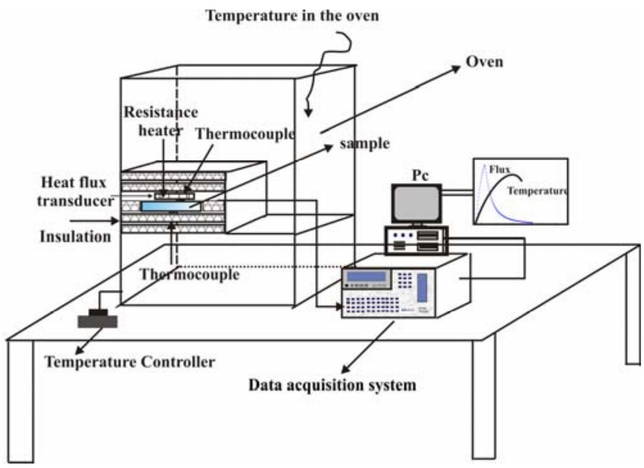


Figure 7. Experimental apparatus for estimating thermal properties α and λ

5. Results and discussion

Figures 8 and 9 show respectively the evolution of the input signal and the output signal in function of time for one of the experiments of the PVC sample. Eight cases of initial temperature with a range from 20 °C and 70 °C are analyzed. Twenty independent runs for each case of oven temperature were realized. For each experiment 4096 points were taken, where the time intervals, Δt, were 1.0 s. The time duration of heating, t_h, was approximately 30 s with a heat pulse generated by a 8.50 V (dc). Figure 10 shows the measured temperatures inside the oven for the case of average temperature equal to 56.56 ^oC.

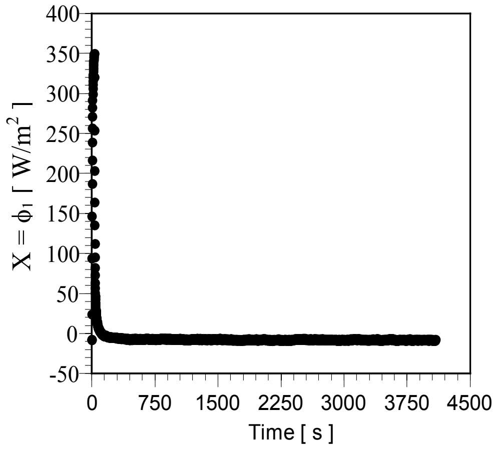


Figure 8. Evolution of the input signal

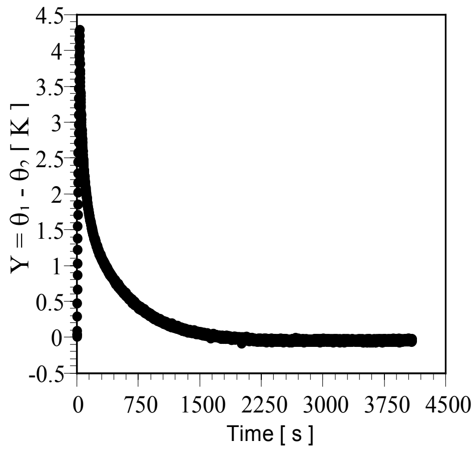


Figure 9. Evolution of the output signal

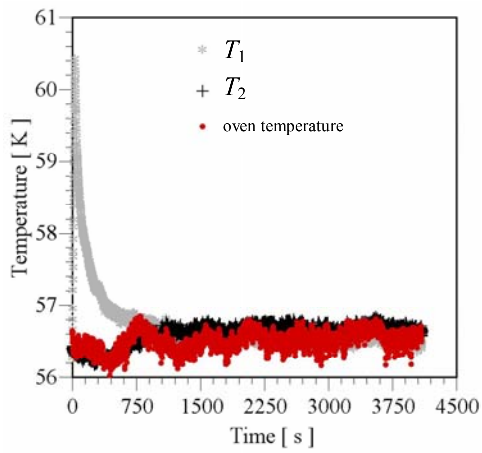
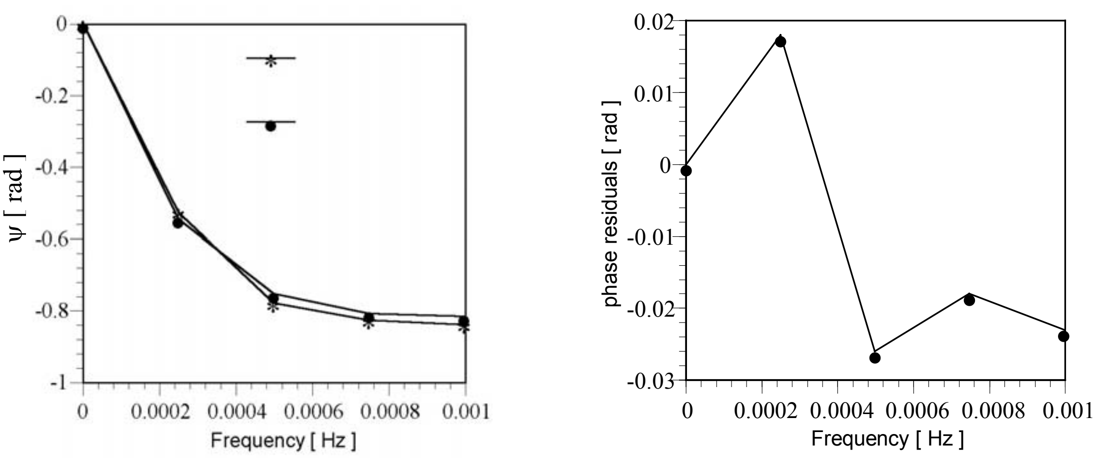


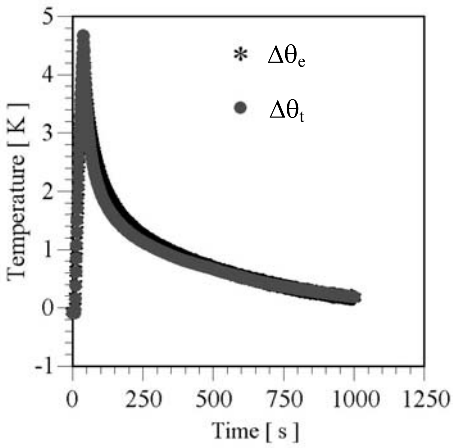
Figure 10. Experimental temperatures for the case of average oven temperature 56.56 ^oC

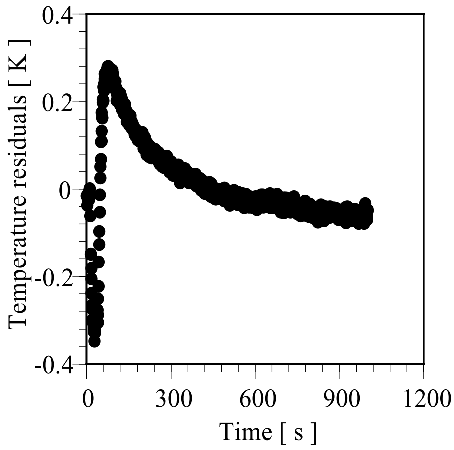
In Figure 11a a comparison between experimental and estimated phase factor is presented. It can be observed a very good agreement among them. The Figure 11b shows the residuals.



a) b)
Figure 11. Phase factor: a) experimental and calculated data b) residuals

A comparison between the experimental and estimated temperatures for $\alpha = 1.25 \times 10^{-07}$ m^2/s and $\lambda = 0.160$ W/m.K of one experiment (oven temperature 27.42 ^oC) is shown in Fig. 12a. Again a good agreement between the data can be observed. It can be noted that the residuals presented in Fig. 12b, are situated in the range of uncertainty measurement of thermocouples, that in this work is ± 0.3 K. Table 1 and Figure 13 present respectively the estimated values of α and λ for all cases. In Table 1 only the reference value of α for room temperature was found (Borges *et al*. 2004). It can be observed a good agreement between the values of this work and the literature for the thermal diffusivity (error less than 2%). A comparison of the estimated values with the literature (Touloukian *et al*. 1970) for the thermal conductivity is shown in Fig. 13.





a) b)

Figure 12. Temperature evolution: a) experimental and calculated data b) residual

Table 1. Statistics data to the averaged value of α, (initial value of $\alpha = 1.0 \times 10^{-8}$ m^2/s).

Average initial temperature (^oC)	$\alpha \times 10^{07}$ (m^2/s)	$\alpha \times 10^{07}$ (m^2/s) (Literature)	Error (%)
20.46	1.308	-	-
27.42	1.255	1.24	1.19
36.64	1.310	-	-
43.21	1.290	-	-
49.82	1.301	-	-
56.65	1.246	-	-
65.73	1.300	-	-

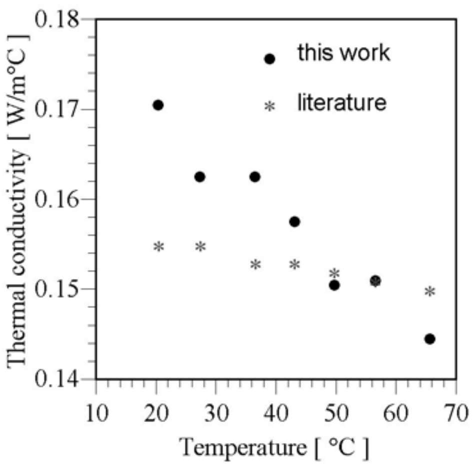


Figure 13. Thermal conductivity results

Although we cannot assure that the sample used here has the same compositions of plasticizer content and molecular weight with the Touloukian *et al*. (1970) work , it can be observed that in both cases the thermal conductivity decay with temperature and the agreement is reasonable.

6. Conclusions

The technique based on the use of an input/output dynamical system shows suitable to be applied to determine thermal properties varying with temperature. Its great advantage is that just a small temperature difference across the sample (as 4.5 ^oC) is high enough to estimate both properties simultaneously. This fact is necessary to identify the thermal properties in an representative average temperature. This technique can also be used for conductor applications.

7. Acknowledgements

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