

A simple approach to introduce photothermal techniques basic principles for thermal diffusivity measurement



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Abstract

We report an approach to introduce the students to the field of the photothermal techniques. For this, we describe an experiment about the measurement of the periodical oscillations of the temperature in a body heated with a periodic heat source. These thermal oscillations can be called thermal waves. We show how, by means of the measurement of the phase difference and attenuation of the thermal waves with the penetration distance in the body, we can determine their thermal diffusivity. This is a result in which some of the photothermal techniques are based for applications related with the thermal characterization of materials. In particular we use the technique of pyroelectric detection to show this purpose.

Keywords: Photothermal, thermal diffusivity, heat, temperature.

Resumen

Presentamos una forma de introducir a los estudiantes en la temática de las técnicas fototérmicas, para lo cual describimos un experimento sobre la medición de las oscilaciones periódicas de la temperatura de un cuerpo calentado con una fuente de calor periódica variante en el tiempo. Estas oscilaciones de temperatura pueden denominarse ondas térmicas. Mostramos como, a partir de la medición de la diferencia de fase y la atenuación de estas ondas térmicas con la distancia de penetración en el cuerpo, podemos determinar su difusividad térmica. Este es un resultado en el cual se basan algunas de las técnicas fototérmicas para aplicaciones relacionadas con la caracterización térmica de los materiales. En particular, utilizamos la técnica de detección piroeléctrica para mostrar este propósito.

Palabras clave: Fototermal, difusividad termal, calor, temperatura.

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I. INTRODUCTION

The photothermal (PT) techniques [1] are a group of experimental methods based in a common principle of heating a sample with periodical (harmonic) or pulsed radiation (often visible light) and on measuring directly or indirectly the induced temperature changes. These are related with characteristic sample's properties that can be recovered by designing a proper experiment. Among these properties, we should mention the sample's absorption coefficient at the impinging radiation wavelength [2], the parameters involved in the incident radiation energy into heat conversion process (for example the quantum efficiency of this process) [3, 4], and thermal properties such as the thermal diffusivity, α [5], and the thermal effusivity, ε [6, 7]. One of the most important fields of applications of the PT techniques is the thermal

characterization of materials. Among the mentioned parameters, the thermal diffusivity is the most widely studied using PT approaches, and several experimental configurations have been designed for its determination [8, 9, 10]. In the last years we have witnessed the widespread and routine use of the PT techniques for this purpose in many research laboratories worldwide. Thus, we think that it is necessary the familiarization of physics students and teachers in high-schools, colleges and universities with their basic principles and related phenomena, in the same way as they familiarize with other more traditional instruments, which they can use to look inside their surrounding world.

In this article we will show how an inexpensive and simple experiment can be useful to introduce the students, in a straightforward way, to the basic principles of the PT techniques for thermal diffusivity measurement in materials.

II. THEORETICAL ASPECTS

For the sake of simplicity, we will limit our analysis to one of the simplest experimental situations that one can think for the measurement of the thermal diffusivity of a solid sample. Thus, we will consider one homogeneous and isotropic semi-infinite sample that is uniformly illuminated (in such a way that the next one dimensional approach will be valid) by means of a monochromatic electromagnetic radiation beam modulated in intensity in a periodical sinusoidal form. The energy of the incident radiation is fully absorbed at the sample's surface and totally transformed into heat.

The temperature distribution $T(x, t)$ within the solid can be obtained by solving the homogeneous heat diffusion equation [11]

$$\frac{\partial^2 T(x, t)}{\partial x^2} - \frac{1}{\alpha} \frac{\partial T(x, t)}{\partial t} = 0, \quad x > 0, t > 0, \quad (1)$$

with the boundary condition (from the Fourier's Law)

$$-k_t \left. \frac{\partial T(x, t)}{\partial x} \right|_{x=0} = \text{Re} \left[\frac{I_o}{2} \exp(i\omega t) \right], \quad (2)$$

where x is the spatial coordinate, t is the time, ω is the light angular modulation frequency, I_o the light intensity flux, $k_t = \varepsilon\alpha^{1/2}$ is the thermal conductivity and $i = (-1)^{1/2}$ is the imaginary constant. The Eq. (2) expresses that the thermal energy generated at the surface of the solid is dissipated into its bulk by diffusion.

The solution of physical interest of the problem for applications in PT techniques is the one related to the time dependent component. If we separate this component from the spatial distribution, the temperature can be expressed as:

$$T(x, t) = \text{Re}[\Theta(x)\exp(i\omega t)]. \quad (3)$$

Substituting in Eq. (1) we obtain

$$\frac{d^2 \Theta(x)}{dx^2} - q^2 \Theta(x) = 0, \quad (4)$$

where

$$q = \sqrt{\frac{i\omega}{\alpha}} = (1+i) \sqrt{\frac{\omega}{2\alpha}} = \frac{(1+i)}{\mu}, \quad (5)$$

and

$$\mu = \sqrt{\frac{2\alpha}{\omega}}. \quad (6)$$

So, we have transformed a partial differential equation, Eq. (1), in a simpler ordinary differential equation with constant coefficients, given by Eq. (4). Using the condition (2) the general solution of Eq. (4) has the form

$$\Theta(x) = \frac{I_o}{2\varepsilon\sqrt{\omega}} \exp\left(-\frac{x}{\mu}\right) \exp\left[-i\left(\frac{x}{\mu} + \frac{\pi}{4}\right)\right]. \quad (7)$$

From Eqs. (7) and (3) we can obtain the temperature field $T(x, t)$ as:

$$\begin{aligned} T(x, t) &= \frac{I_o}{2\varepsilon\sqrt{\omega}} \exp\left(-\frac{x}{\mu}\right) \cos\left(\omega t - \frac{x}{\mu} - \frac{\pi}{4}\right), \\ &= T_s \exp\left(-\frac{x}{\mu}\right) \cos(\omega t - \phi) = T_0 \cos(\omega t - \phi). \end{aligned} \quad (8)$$

Because of the wave-like features of this equation, it is often denoted as a thermal or temperature wave. It is similar to the equation describing a plane wave of wavelength $\lambda = 2\pi\mu = 2\pi/k$, where $k = 1/\mu$ can be defined as a wave number, being equal to both the real and imaginary parts of the parameter q in Eq. (5).

The factor $T_0 = T_s \exp(-x/\mu)$ in the Eq. (8) shows that thermal waves are attenuated, and that the parameter μ represents the distance at which the wave amplitude decays e times from its value (T_s) at the surface of the periodical heated body. Due to this reason μ is called the thermal diffusion length or damping coefficient. As can be seen from Eq. (6), it depends on the thermal diffusivity and on the modulation frequency. Between the light excitation (see the right hand side of Eq. (2)) and the thermal response of the sample, Eq. (8), there is a phase-lag given by the term $\phi = x/\mu + \pi/4$ in the complex exponent. Note that the phase lag is dependent on the thermal diffusion length, and hence, on the modulation frequency. It depends on the thermal diffusivity too.

Thermal waves propagate with phase velocity $v_{ph} = \omega/k = (2\alpha\omega)^{1/2}$. Since Eq. (4) is a linear ordinary differential equation describing the motion of a thermal wave, then the superposition of solutions will be also a solution of it (we have approximate the temperature distribution by just the first harmonic of that superposition because the higher harmonics damp out more quickly, due to the damping coefficient increase with frequency). This superposition represents a group of waves with angular frequencies in the interval between ω and $\omega+d\omega$ travelling in space as "packets" with a group velocity $v_g = (dk/d\omega)^{-1} = 2v_f$.

We can further assume that thermal waves experiment reflection and transmission at interfaces between media with dissimilar thermal properties, like other kind of waves does. One can demonstrate [1] that for a thermal wave propagating from region 1 to region 2 the reflection and transmission coefficients can be calculated (for normal incidence) by

$$R_{12} = (1-b_{12})/(1+b_{12}) \quad \text{and} \quad T_{12} = 2/(1+b_{12}), \quad (9)$$

respectively. The parameter b_{12} is the ratio of the thermal effusivities of the involved materials:

$$b_{12} = \varepsilon_1/\varepsilon_2. \quad (10)$$

Thus, we see from Eq. (8) that thermal effusivity determines the wave amplitude at the surface of the sample (namely at $x=0$), as well as the thermal wave behaviour at interfaces (Eqs. (9)). Hence, we see that although thermal effusivity is, without a doubt, a bulk characteristic of a given sample, it is really a property involved only in processes taking place at surfaces. As a consequence, thermal effusivity measurements are mainly based in normalization procedures in which the investigated sample is placed in intimate contact with a test sample in such a way that a two layer system is formed. Then, reflection and transmission of thermal waves at the interface takes place, thus involving the thermal effusivity ratio given by Eq. (10) [12]. This is a very interesting theme that is behind the scope of the present work, which will be focused to the measurement of the thermal diffusivity, a parameter that determines the temporal and spatial form of the thermal wave field inside a material, its attenuation and its propagation velocity, and for whose determination no normalization artifact will be necessary. Several other aspects concerning the principal features of thermal waves can be found elsewhere [1, 6, 13, 14].

III. THERMAL DIFFUSIVITY MEASUREMENT: A SIMPLE APPROACH

From Eq. (8), we can obtain the following relationships for the amplitude and for the phase-lag of the temperature field:

$$\begin{aligned} \ln T_0 &= \ln [I_0 / (2\epsilon\omega^{1/2})] - x/\mu, \\ &= \ln [I_0 / (2\epsilon\omega^{1/2})] - x(2\omega/\alpha)^{1/2}, \quad (11) \\ &= \ln [I_0 / (2\epsilon\omega^{1/2})] - m x, \end{aligned}$$

and

$$\phi = -x/\mu - \pi/4 = -x(2\omega/\alpha)^{1/2} - \pi/4 = -m x - \pi/4. \quad (12)$$

These equations are the basis of a method devised about two centuries before by Fourier for the measurement of thermal diffusivity in soils [15] and independently by Angstrom for the measurement of the same parameter in a solid in a form of a rod [16]. Both methods will be described later but they are almost the same: A periodical heating is applied on an end of the sample, while its other end is kept at a constant temperature, then, from the measurement of the amplitude and/or the phase of a thermal wave at different distances from the heated surface the thermal diffusivity should be determined from the slopes, m , of the curves of $\ln T_0$ Vs. x and ϕ Vs. x as

$$\alpha = 2 \omega / m^2. \quad (13)$$

An experiment based in this scheme and on Fourier's proposal of thermal wave propagation in soils was discussed by McIntosh and Sharratt [17] and more recently by Marin *et al* [18]: The surface of the earth is exposed to more or less regular daily temperature fluctuations, owing

to different causes, which differ appreciably from day to day, from season to season and from region to region. As a consequence, air and soil temperatures generally exhibit a diurnal cycle with a period $T=2\pi/\omega$ of near 24 hours. The temperature variations with time and with depth below the surface can be described using equations like Eq. (8).

A measurement device have been developed in our laboratory [19] using eight LM35 [20] solid state temperature sensors located along a measurement probe in a form of a bar at 5 cm intervals in such a manner that, when it is introduced into the soil, the first sensor remains fixed at a distance of 5 cm above the soil's surface to measure the air temperature, whereas the others are useful for temperature sensing at different depths ranging from the surface to 30 cm. The transducers transform the sensed temperatures values into analogue voltages that are then supplied to a home-made Analog-Digital Converter (ADC) to adequate data for binary acquisition. The Digital Data Outputs from the ADC are then multiplexed by two digital switches in order to send 2 packages of 4 bits through a parallel port (DB25 Connector) to a Computer. The ADC is supplied with a Power Supply of 5 V DC and an oscillator to supply a constant frequency used for the conversion time. The schematic diagram of this process is shown in Fig. 1.

The data acquisition control and processing software was developed using Visual Basic (VB) [21], which facilitates the development of graphical programs. We decided to use VB instead of other programming softwares like LabView [22] because it's cheaper and it let us build executable and setup files even using educational versions of the software. Another advantage is that VB requires less capacity of RAM Memory than LabView to be executed correctly.

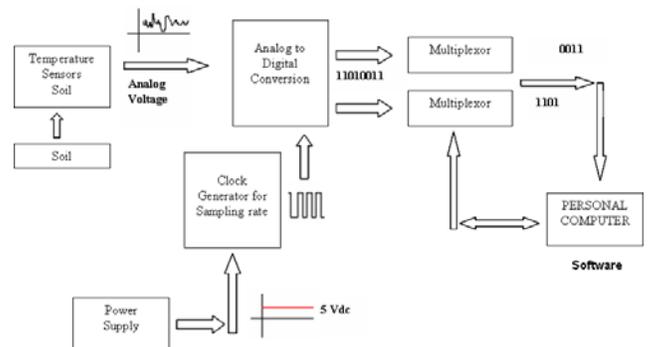


FIGURE 1. Block Diagram of the hardware interface.

The user interface of our program is shown in Fig. 2. It includes controls to select the total measurement time and the time interval between successive captured values, indicators to show the actual temperature as measured by a previously selected sensor and a table to display the temperature vs. time curves, among others. The measurement results can be then imported from the program used for data processing, in our case MICROCAL-ORIGIN 07 [23]. More details about the

measurement set-up, including hard - and software are given elsewhere [19].

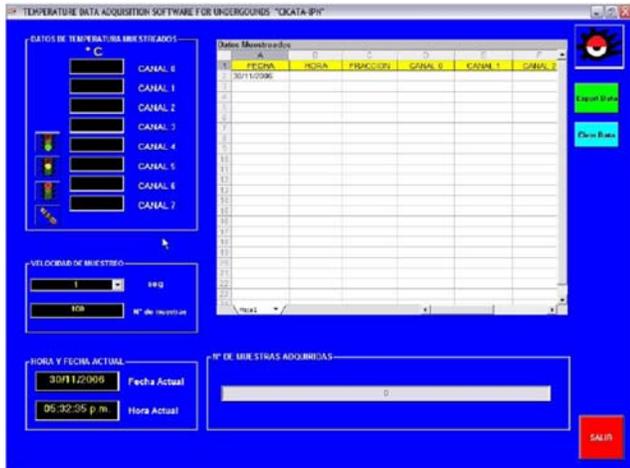


FIGURE 2. User graphical interface.

Fig. 3 shows temperature vs. time curves measured at different depths beneath the soil surface during 24 hours at a clear winter day in México City. The time interval between successive measurements was 5 minutes. One can easily see the attenuation of the temperature wave (*i.e.* the reduction in the wave amplitude) as well as the phase shift between the maximum and minimum values of the curves with depth.

One can also observe from the figure that the variations in temperature are approximately sinusoidal, but that obvious differences exist: Although the period of the temperature oscillation must be of 24 h, the maximum and minimum of the air temperature do not occur half of an oscillation (about 12 hours) apart. The principal cause of this behavior is the deviation of the heat source from the assumed harmonic function of time.

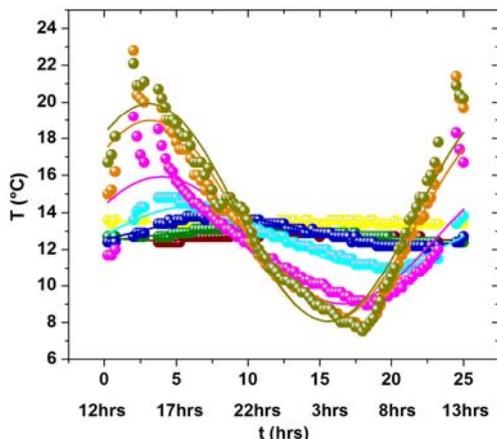


FIGURE 3. Temperature Vs time curves measured at different depths beneath the soil surface during 24 hours. The amplitude diminishes with measurement depth. The first sensor was at a distance of 5 cm above the soil's surface, whereas the others were located at different depths with a separation distance of 5 cm among nearby neighbours and the last one located to 30 cm from the surface soil.

Also temperature convection effects that can take place near the soil's surface, which have not been considered in our theoretical model, can be partially the cause of the deviations from the predicted harmonic behavior. This is more evident in the curves collected with the upper sensors nearest the surface, where the dispersion of the experimental points from the theoretical predictions is greater. Veleva *et al.* [24] have also reported before about this kind of deviations, although these authors do not comment about possible causes and limit their analysis to modeling the time evolution of the temperature using linear and parabolic functions for night and day time respectively.

Other authors [17, 18] do not show marked deviations from the harmonic sinusoidal behavior, but they used greater time intervals (1 h) between measurements that can partially overlap the described non-harmonic behavior.

On the other hand, for measurements performed at the greater depths, namely at 25 and 30 cm below the surface, these deviations are not present because the role played by convection is negligible. Also, one can believe that soil is a heterogeneous thermal system, whose non-uniformity arises from the dependence of its thermal properties mainly on water content and bulk density (porosity), which vary strongly with depth near the soil air interface due to evaporation, drainage, transpiration by plants, etc. At soil depths far below the surface, the dependence of these, and hence the thermal parameters, on the above processes decrease until it vanishes. But, in spite of these aspects that should be discussed with students to gain in their understanding about the analyzed physical situation, this type of measurements can constitute a good exercise to realize our objectives.

The solid curves in Fig. 3 are the best fits of the Eq. (8) to the data, leaving T_0 , ω and ϕ as adjustable parameters. The mean value of the angular frequency obtained values, for each depth, x , was $\omega=(0.284\pm 0.009) \text{ h}^{-1}$, from which the period of the temperature oscillation was calculated as $T=2\pi/\omega=(22.085\pm 0.009) \text{ h}$. This value is very closer to the expected value for this magnitude, *i.e.*, one day. Therefore, our sinusoidal approximation can be considered as a good assumption. Because at depths above 20 cm the amplitude of the temperature oscillations become so small that reliable values of the fit parameters can not be obtained, we have not consider these curves in the following analysis.

In Fig. 4 the logarithm of the difference between maximum and minimum temperature values, *i.e.*, the wave amplitudes, is represented as a function of the distance, x , at which the measurements were performed. From this semi-log plot one becomes a straight line from whose slope, as follows from Eq. (11), we obtained, using Eq. (13), the thermal diffusivity as $\alpha=2\omega/m^2 = (1.8 \pm 0.1) \text{ cm}^2/\text{s}$, in good agreement with previous reported values for soils [25]. It is worth to notice again that this value is an effective value for the thermal diffusivity of soil, actually a depth dependent parameter. For the angular frequency we have taken the above determined value from the fits of Fig. 3.

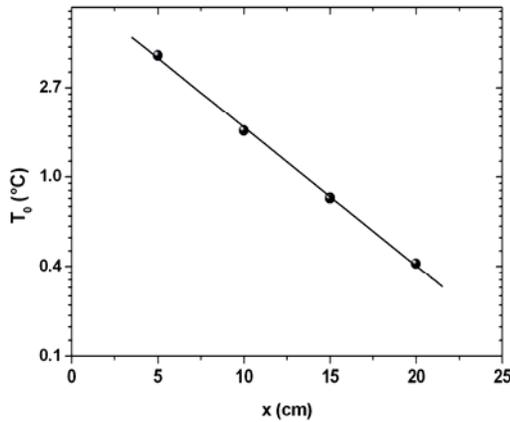


FIGURE 4. Logarithm of the thermal wave amplitudes as a function of the distance x at which the measurements were performed. The solid line is linear fit obtained using the least-squares method.

To use the phase shift dependence of the temperature oscillations with depth, to determine soil’s thermal diffusivity, we show in Fig. 5 the product of the modulation frequency and the time for which the minimum in the temperature Vs. time curves appears in Fig. 3 (this is a good measure of the phase difference) as a function of the corresponding depths. Note also that the sign of the phase is not important, so the slope m can be positive. Following Eq. (12), the slope, m , of the straight line given by the best least squares linear fit lead to the value of the thermal diffusivity in a way similar as from the amplitude vs. x curves. We obtained $\alpha=2\omega/m^2 = (1.8 \pm 0.2) \text{ cm}^2/\text{s}$, a similar value as that obtained from the thermal wave amplitude.

A similar experiment, but based in Angstrom method, has been reported by Bodas *et al* [27]: Here the thermal waves were generated in a thin bar of Copper by periodically heating at one end using a soldering iron embedded in the sample and connected to a circuit made on timers and switches.

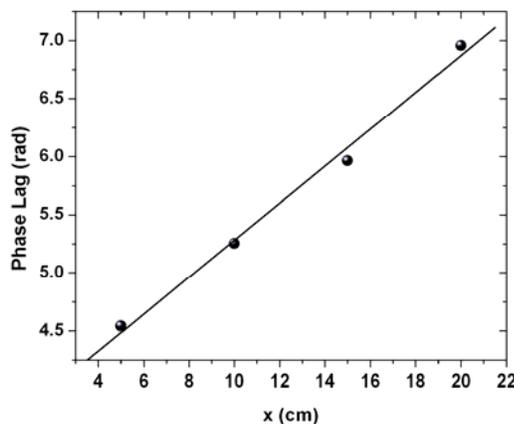


FIGURE 5. The phase difference as a function of the depth at which the measurements were performed. The solid line is the best least squares linear fit.

The temperature changes along the bar were measured at different distances from the heating point using also small thermistors. Although the period of the temperature oscillations can be made lower, the results and the data processing procedure are almost similar as those presented above for the Fourier’s approach, which is simpler because it uses a natural existing thermal wave source and propagation media. On the other hand, the measurement of thermal properties of soil constitutes a very interesting problem because their knowledge can be of great importance in agricultural and environmental problems involving, for example, soil degradation.

IV. THERMAL DIFFUSIVITY AS MEASUREMENT BY PT METHODS

The introduction of sensors inside an investigated sample without damaging it is not always possible, and some times the sample’s physical dimensions are so small that this is an impossible task. But the solution to this problem can be found again in the thermal wave’s attenuation and phase lag. We see from Eqs. (11) and (12) that the same dependence on the distance x also exists for the square root of the modulation frequency and this is the basis behind some photothermal approaches for thermal diffusivity measurements: One can perform measurements of the thermal wave amplitude and phase as a function of the square root of the modulation frequency at a given x , for example at the sample’s rear surface opposite from its heated face, i.e., at $x=L$, where L is the sample’s thickness. In other words, for $x=L$ Eqs. (11) and (12) can be rewritten as:

$$\begin{aligned} \ln(T_0\omega^{1/2}) &= \ln[I_0/(2\varepsilon)] - L/\mu, \\ &= \ln[I_0/(2\varepsilon)] - L(2\omega/\alpha)^{1/2}, \\ &= \ln[I_0/(2\varepsilon)] - m' \omega^{1/2}, \end{aligned} \tag{14}$$

and

$$\phi = -L/\mu - \pi/4 = -L(2\omega/\alpha)^{1/2} - \pi/4 = -m' \omega^{1/2} - \pi/4, \tag{15}$$

where $m' = L(2/\alpha)^{1/2}$. Thus, the thermal diffusivity can be calculated straightforward from the slopes m' of the linear curves $\ln(T_0\omega^{1/2})$ vs. $\omega^{1/2}$ and ϕ Vs. $\omega^{1/2}$, if the sample’s thickness L is well known.

To show an example for this methodology, we performed a photothermal measurement of the thermal diffusivity of a 450 μm thick semiconductor Silicon sample using a temperature pyroelectric (PE) [27, 28] sensor. The experiment consists basically in a PE (in our case a metalized pyroelectric PVDF -Polyvinylidene Difluoride foil of 25 μm of thickness) placed in good thermal contact with the sample using a very thin layer of conducting thermal paste. A laser beam modulated using a mechanical chopper impinges onto a sample surface. Superficial light absorption was achieved by the use of a Ar-ion laser beam (514 nm, 100 mW), whose photons energy is much greater than the band gap energy of the Si crystal. The thermal waves generated in the sample due to

the absorption of light propagates into the sensor and the resulting PE voltage signal is measured in amplitude and phase using a dual phase digital Lock-in amplifier (LIA) synchronized at the modulation frequency. A LIA can be defined as an instrument that permits the measurement of AC signals modulated at a given frequency, f , in an environment of very low signal to noise ratio. The main function of LIA is then to single out the component of the signal at f and reject noise signals at other frequencies [29].

Measurements were performed automatically using a PC. In the frequency range of our experiment the PE sensor showed a flat response so that the use of Eq. (14) is allowed (Care must be taken in the case of the amplitude measurements as a function of the modulation frequency with the fact that the used temperature sensor can have a non-flat frequency response and in this case Eq. (11a) must be used with prudence. This is actually not a problem with the phase lag. Due to this reason photothermal techniques for thermal diffusivity determinations use often the phase lag dependence on the modulation frequency for this purpose).

In the Fig. 6 we show the obtained results. The solid lines are the best least squares linear fit from whose slopes the thermal diffusivity was determined.

For the mean value from measurements performed using both the amplitude (Fig. 6 (a)) and phase (b) channels we obtained $\alpha=2(L/m^2)^2 = (0.18 \pm 0.05) \text{ cm}^2/\text{s}$, also in good agreement with reported values [30]. Students can perform similar experiments using samples of other materials.

V. CONCLUSIONS

In this work we have described how technically simple, low cost, and instructive from a pedagogic point of view laboratory experiments designed for the measurement of one of the key parameters governing the heat propagation, the thermal diffusivity, can be used to introduce the students to the field of photothermal science and techniques.

We feel that the approach presented here is plausible and satisfactory to introduce and motivate students in the photothermal science and techniques, and their applications, without involving excessively sophisticated mathematical analysis and/or meticulous experimental techniques.

This approach can be presented to students since the first years of study, when familiarize with the heat conduction phenomenon. For example, the well recognized text book of Alonso and Finn [30] discusses the here analyzed problem of the temperature distribution of a body with a periodical heat source at its surface, showing annual and daily temperature versus time curves measured at different distances from the surface at the Mid Atlantic Ocean (see example 24.4 in [31]), which are similar to those presented in Fig. 2 for a soil. To our knowledge, this is the only general physics text book where this important problem, which can also serve as motivation to talk about a non-conventional wave like phenomenon, is discussed. Students can go deeper inside this theme in more advanced

courses on experimental physics at undergraduate and graduate level.

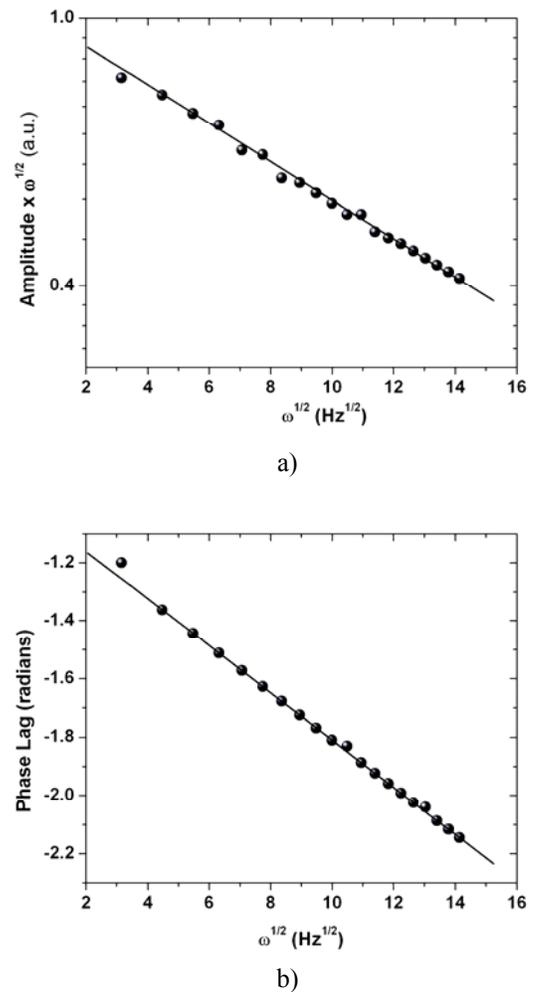


FIGURE 6. Results of the photopyroelectric measurements performed in amplitude (a) and phase channel (b) as a function of the square root of the modulation frequency.

This form of introducing photothermal techniques has been used successfully, for example, during the postgraduate courses given by some of the authors (EM and AC) as part of the studium curricula of the Postgraduate Program of Advanced Technology at CICATA-Legaria, IPN México [32].

The here presented results also demonstrate once again how a discovery done at in a certain historical moment (we refers us to the Fourier's and Angstrom's approaches discussed in Section 3) can be rediscovered many years later giving rise to exciting applications, in this case those of the PT techniques for thermal diffusivity measurements.

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