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# Photopyroelectric Technique, in the Thermally Thin Regime, for Thermal Effusivity Measurements of Liquids

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**Abstract** A photopyroelectric methodology using pyroelectric devices in the thermally thin regime, for the measurement of the thermal effusivity of liquids, is described. A commercial buzzer, taking advantage of the pyroelectric activity of the ceramic material (PZT in this case), is used to achieve this goal. Equivalence for thermal effusivity measurements between this photopyroelectric methodology and a previously reported photoacoustic technique, in the front configuration, is established. The thermal effusivity of six liquids was measured using this device, and very good agreement was found with corresponding values reported in the literature.

**Keywords** Photopyroelectric · Thermal · Effusivity · Liquids · PZT

## 1 Introduction

Several photothermal techniques have been reported for the measurement of the thermal effusivity of liquids [1–3]. Some of them, involving photoacoustic (PA) techniques in the front PA configuration, are adequate for the measurement of this thermal property for any type of liquids [1–5]. Other ones, involving

photopyroelectric (PPE) techniques in the transmission and front configurations [6–9], are adequate only for thermal effusivity measurements of liquids that will not damage the pyroelectric sensor because the sensor's surface is exposed to the liquid sample. With the exception of a PPE technique in the transmission configuration, with a pyroelectric sensor in the thermally thick regime [6], reported techniques involving the front configuration [7, 8] require the sensor's behavior inside an intermediate thermally thick regime to allow heat exchange with the liquid sample. In this paper, a commercial pyroelectric sensor (lead zirconate titanate (PZT)-based buzzer) is shown to be adequate for the implementation of a pyroelectric technique, in the front PPE configuration and in the sensor's thermally thin regime, for the measurement of the thermal effusivity of liquids. Since this commercial device is attached with a thin metallic foil on top, the presented pyroelectric technique is adequate for any type of liquid sample, as long as it does not damage the metallic foil. Considering this commercial device as a three-layered material, the analytical solution for a generalized one-dimensional heat diffusion problem through three layers is presented and used for this photopyroelectric technique for general liquids. What is shown, in particular, is the equivalence, in terms of the analytical treatment, between this new PPE methodology and a previously reported PA technique [3–5]. This PPE methodology was applied for thermal effusivity measurements of various pure reference substances (distilled water, methyl alcohol, and glycerol, among others), obtaining thermal effusivity values in good agreement with previously reported ones.

## 2 Theory

Commercial buzzers consist of a PZT ceramic between two electrodes, one a very thin layer of electrical conductive paste and the other a thin metallic foil. The corresponding

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mathematical model, for the purpose of the photothermal technique presented in this work, consists of a three-layered system, similar to the one shown in Fig. 1. In this model, modulated monochromatic radiation (intensity  $I_0$ ), coming from a semi-infinite transparent medium  $g$  with angular modulation frequency  $\omega = 2\pi f$ , is absorbed on the surface of medium  $m$ , with optical absorption coefficients  $\beta_m$ . The corresponding differential equations for the one-dimensional heat diffusion problem are shown in Eq. (1).

$$\begin{aligned} \frac{\partial^2 T_g}{\partial x^2} - \frac{1}{\alpha_g} \frac{\partial T_g}{\partial t} &= 0, & l_n + l_m \leq x \\ \frac{\partial^2 T_m}{\partial x^2} - \frac{1}{\alpha_m} \frac{\partial T_m}{\partial t} &= -\frac{\beta_m I_0}{k_m} \delta(x - l_n - l_m) e^{i\omega t}, & l_n \leq x < l_n + l_m \\ \frac{\partial^2 T_n}{\partial x^2} - \frac{1}{\alpha_n} \frac{\partial T_n}{\partial t} &= 0, & 0 \leq x < l_n \\ \frac{\partial^2 T_w}{\partial x^2} - \frac{1}{\alpha_w} \frac{\partial T_w}{\partial t} &= 0, & -l_w \leq x < 0 \\ \frac{\partial^2 T_s}{\partial x^2} - \frac{1}{\alpha_s} \frac{\partial T_s}{\partial t} &= 0, & -\infty < x \leq -l_w \end{aligned} \quad (1)$$

$T_j, j = g, m, n, w, s$ , refers to the temperature profile inside medium  $j$ , with  $k_j$ ,  $\alpha_j$ , and  $l_j$  representing its thermal conductivity, thermal diffusivity, and thickness, respectively. Pyroelectric signal is proportional to the average temperature of the pyroelectric sensor for which this system is solved with the usual boundary conditions of heat flux and temperature continuity at the interfaces  $g$ - $m$ ,  $m$ - $n$ ,  $n$ - $w$ , and  $w$ - $s$  and the physical requirement of finite solutions as  $x \rightarrow \pm\infty$ . It is possible to show that the average temperature inside medium  $n$  (the pyroelectric sensor) is calculated by Eq. (2).

$$T(f) = \frac{2}{l_n \sigma_n k_n \sigma_n} \frac{I_0}{D} \frac{[R(e^{\sigma_n l_n} - 1) + Q(e^{-\sigma_n l_n} - 1)]}{D}. \quad (2)$$

The pyroelectric signal can then be written as

$$V(f) = G(f) \frac{2}{l_n \sigma_n k_n \sigma_n} \frac{I_0}{D} \frac{[R(e^{\sigma_n l_n} - 1) + Q(e^{-\sigma_n l_n} - 1)]}{D}, \quad (3)$$

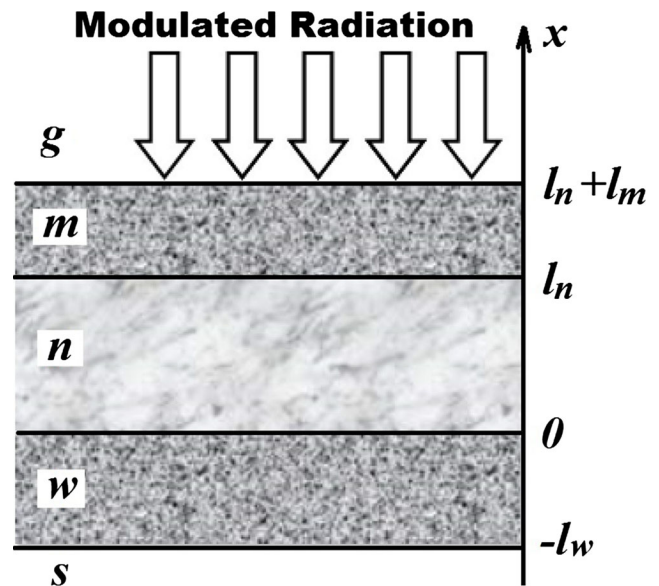
where

$$D = Q[(1 + b_{gm})(1 - b_{mn})e^{\sigma_m l_m} e^{-\sigma_n l_n} + (1 - b_{gm})(1 + b_{mn})e^{-\sigma_m l_m} e^{-\sigma_n l_n}] + R[(1 + b_{gm})(1 + b_{mn})e^{\sigma_m l_m} e^{\sigma_n l_n} + (1 - b_{gm})(1 - b_{mn})e^{-\sigma_m l_m} e^{\sigma_n l_n}],$$

and

$$Q = (1 + b_{ws})(1 - b_{nw})e^{\sigma_w l_w} + (1 - b_{ws})(1 + b_{nw})e^{-\sigma_w l_w}, \\ R = (1 + b_{ws})(1 + b_{nw})e^{\sigma_w l_w} + (1 - b_{ws})(1 - b_{nw})e^{-\sigma_w l_w}.$$

In these equations,  $\sigma_j = (1 + i)a_j$ ,  $j = m, n, w$ , where  $a_j = (\pi f / \alpha_j)^{1/2}$  and  $b_{jh} = e_j / e_h$  is the ratio of thermal effusivities for media  $j$  and  $h$ . The function  $G(f)$ , usually called the transfer



**Fig. 1** Schematic representation of the one-dimensional mathematical model, with surface absorption, of the commercial piezo buzzer for thermal effusivity measurements.  $g$  gas (air);  $m$  electrode of conductive paste, with surface absorption coefficient  $\beta_m$ ;  $n$  pyroelectric material (PZT);  $w$  metal foil; and  $s$  liquid sample

function, represents the frequency response of the sensor and the electronics. If medium  $m$  is very thin (one of the electrodes consists of a very thin layer, less than 1  $\mu\text{m}$ , of conductive paste), then, for the usual values of thermal diffusivity,  $|\sigma_m l_m| \ll 1$  and  $e^{\pm \sigma_m l_m} \approx 1$ , and the denominator in Eq. (3) simplifies as a consequence to

$$D = 2[R(1 + b_{gn})e^{\sigma_n l_n} + Q(1 - b_{gn})e^{-\sigma_n l_n}]. \quad (3a)$$

The pyroelectric signal can be written as

$$V(f) = G(f) \frac{1}{l_n \sigma_n k_n \sigma_n} \frac{I_0}{D} \frac{[R(e^{\sigma_n l_n} - 1) + Q(e^{-\sigma_n l_n} - 1)]}{[R(1 + b_{gn})e^{\sigma_n l_n} + Q(1 - b_{gn})e^{-\sigma_n l_n}]}. \quad (4)$$

The commercial buzzer used in this work is attached with a thin metallic foil of about 50  $\mu\text{m}$  (medium  $w$  in this case). Metallic materials usually have large thermal diffusivities; for instance, if a thermal diffusivity of 0.2  $\text{cm}^2/\text{s}$  is assumed for the metallic foil in this case and for modulation frequencies in a range of 1 to 6 Hz,  $|\sigma_w l_w| \leq 0.069$  and it can also be considered that  $e^{\pm \sigma_w l_w} \approx 1$ , for which  $Q = (1 - b_{ns})$  and  $R = (1 + b_{ns})$ .

Under these assumptions, Eq. (4) can be written in the very simple form as

$$V(f) = G(f) \frac{1}{l_n \sigma_n k_n \sigma_n} \frac{I_0}{D} \frac{[(1 + b_{ns})(e^{\sigma_n l_n} - 1) + (1 - b_{ns})(e^{-\sigma_n l_n} - 1)]}{[(1 + b_{ns})(1 + b_{gn})e^{\sigma_n l_n} + (1 - b_{ns})(1 - b_{gn})e^{-\sigma_n l_n}]}. \quad (5)$$

This equation is essentially the same as the one already used by Longuemart and co-workers [8] in the

implementation of a PPE technique in the front configuration for thermal effusivity measurements, assuming a pyroelectric sensor in the thermally thick regime. This equation simplifies further if medium  $g$  is considered as a gas, air for example, since in this case,  $e_g \ll e_n$  and  $b_{gn} \ll 1$ , and Eq. (5) is written as

$$V(f) = G(f) \frac{1}{l_n \sigma_n k_n \sigma_n} \frac{I_0 [(1 + b_{ns})(e^{\sigma_n l_n} - 1) + (1 - b_{ns})(e^{-\sigma_n l_n} - 1)]}{[(1 + b_{ns})e^{\sigma_n l_n} + (1 - b_{ns})e^{-\sigma_n l_n}]}. \quad (5a)$$

This is essentially the expression reported by Zammit and co-workers [9], and it is suggested for thermal effusivity measurements. Even though it is possible to use this last equation for thermal effusivity measurements, it simplifies in a more useful manner if the pyroelectric material is considered in the thermally thin regime  $|\sigma_n l_n| \ll 1$ . In such a way,  $e^{\pm \sigma_n l_n} \approx 1 \pm \sigma_n l_n$  (this assumption is well justified for the PZT ceramic, with a thickness of about 40  $\mu\text{m}$ , for the commercial buzzer used in this work but not for other pyroelectric materials, such as PVDF) [10], and the pyroelectric signal can be written as

$$\begin{aligned} V^{Ti}(f) &= G(f) \frac{1}{l_n \sigma_n} \frac{I_0}{k_n \sigma_n} \frac{b_{ns} \sigma_n l_n}{[1 + b_{ns} \sigma_n l_n]} \\ &= G(f) \frac{1}{l_n \sigma_n} \frac{I_0}{k_n \sigma_n} \frac{\sigma_n l_n}{[b_{sn} + \sigma_n l_n]}. \end{aligned} \quad (6)$$

## 2.1 Self-Normalized Photopyroelectric Signal

The best manner of eliminating the transfer function,  $G(f)$ , is to take a PPE reference signal for self-normalization. This can be found by taking the medium  $s$  as  $g$  (air) in Eq. (6); this reference signal takes the form

$$V_R^{Ti}(f) = G(f) \frac{1}{l_n \sigma_n} \frac{I_0}{k_n \sigma_n}. \quad (6a)$$

The self-normalized photopyroelectric signal, defined as  $V_{SN}^{Ti}(f) = \frac{V^{Ti}(f)}{V_R^{Ti}(f)}$ , becomes

$$V_{SN}^{Ti}(f) = \frac{\sigma_n l_n}{b_{sn} + \sigma_n l_n}. \quad (7)$$

This last equation is exactly the one obtained for a photoacoustic technique already reported for thermal effusivity measurement, in the thermally thin regime of

the light absorber [3, 4]. The amplitude  $|R(f)|$  and phase  $\Phi(f)$  of this equation can be written as

$$|R(f)| = \frac{1}{\sqrt{1 + 2\left(\frac{P_s}{\sqrt{f}}\right) + 2\left(\frac{P_s}{\sqrt{f}}\right)^2}}, \quad (8)$$

$$\tan(\Phi(f)) = \frac{\frac{P_s}{\sqrt{f}}}{1 + \frac{P_s}{\sqrt{f}}}, \quad (9)$$

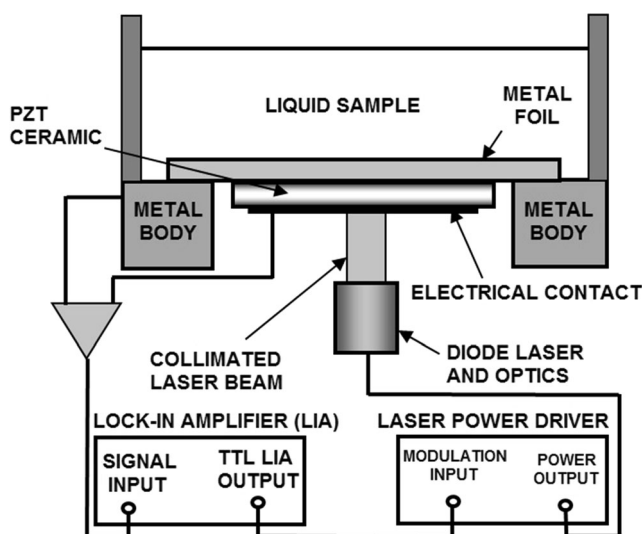
where

$$P_s = \frac{\sqrt{\alpha_n} e_s}{2\sqrt{\pi} l_n e_n}.$$

The thermal effusivity for medium  $s$  can be obtained from these two equations. The experimental procedure involves taking the PPE signal in the front configuration as a function of the modulation frequency in two different conditions, with and without the liquid sample (medium  $s$  in Fig. 1). The complex signal's ratio is calculated (Eq. (7)), and the experimental self-normalized amplitude or phase is fitted to Eq. (8) or (9), respectively. The corresponding thermal effusivity for the sample  $s$  ( $e_s$ ) can be obtained by means of the fitting parameter  $P_s$ , providing that the corresponding parameters for the absorber ( $l_n$ ,  $e_n$ , and  $\alpha_n$ ) are known. These parameters are, however, in general unknown; therefore, for practical reasons, it is more useful to obtain the corresponding parameter,  $P_R$ , for a liquid reference sample of known thermal effusivity (for instance, distilled water). The sample's thermal effusivity can be obtained in this case as  $e_s = (P_s/P_R)e_R$ , where  $e_R$  is the thermal effusivity of the reference sample.

## 3 Experimental Details

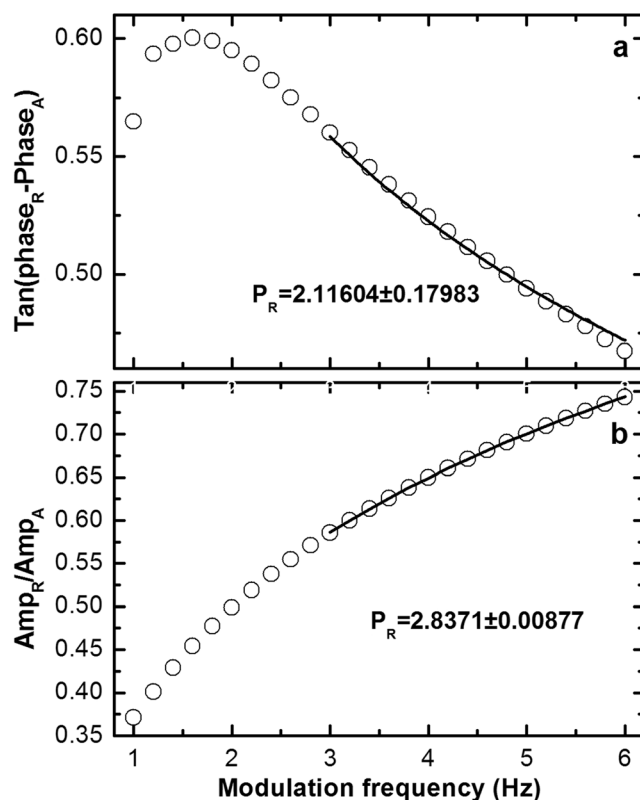
The experimental setup (Fig. 2) consisted of a commercial piezo buzzer (PUI Audio, model AB2022A, 80000 pF), 100  $\mu\text{m}$  thick (50  $\mu\text{m}$  of which consists of a thin metallic foil), with PZT as the sensor. A semiconductor diode laser (Sanyo DL7147-201, 658-nm wavelength), whose intensity was modulated by means of a laser-current power supply (Thorlabs, model LDC202C), controlled by the TTL output of the lock-in amplifier (Stanford Research Systems, model SR830), was used as a light source. The PPE signal was pre-amplified (Stanford Research Systems, SR540) and sent to the lock-in amplifier. Pyroelectric signals for self-normalization (empty sample's container), the reference sample (distilled water), and the liquid sample of interest were taken in a range of 1 to 6 Hz in steps of 0.2 Hz, setting the lock-in amplifier's time constant at 1 Hz.



**Fig. 2** Schematic cross section of the front pyroelectric experimental setup, for thermal effusivity measurements of liquids

## 4 Results

Since measurements were carried out at low modulation frequencies, experimental criteria needed to be set to avoid analysis in a modulation frequency range where experimental deviations from theory could be presented (such as three-dimensional effects). The tangent of the self-normalized phase was used for this purpose since the analysis of this function (Eq. (9)) predicts monotonic decreasing behavior and no inflexion points for the corresponding plot [3, 4]. Figure 3 (Fig. 3a for phase and Fig. 3b for amplitude) shows the self-normalized pyroelectric signal, as a function of the modulation frequency, for the reference sample (distilled water). Figure 3a shows the tangent of the self-normalized phase, and based on the theoretical expectations, a modulation frequency range from 3 to 6 Hz was taken for the analysis. Solid lines in Fig. 3a, b are the best fits to Eqs. (8) and (9), respectively; the values for the parameters  $P_R$  for the self-normalized amplitude and phase ( $P_{R,Amp}$  and  $P_{R,Ph}$ , respectively) are on the same plots and recorded in Table 1. Figure 4 (Fig. 4a for phase and Fig. 4b for amplitude) shows the corresponding pyroelectric signal for the glycerol sample. The analysis was carried out in the same modulation frequency range as the one previously defined for the reference sample (3 to 6 Hz) for parameter  $P_S$  for this sample. Table 1 summarizes the set of experimental parameters obtained for all samples from the analysis of the self-normalized amplitudes (column 2) and self-normalized phases (column 4). The analysis was carried out using commercial software Origin 6.1™; the reported uncertainties on these parameters are the ones directly obtained from that software. The corresponding thermal effusivities obtained from these parameters, according to the expression  $e_s = (P_S/P_R)e_R$ , are summarized, in parentheses, in Table 1, column 3, for the case of the self-normalized amplitude, and column 5, for the case of the self-normalized phase



**Fig. 3** Photopyroelectric (PPE) signal, as function of the modulation frequency, for the reference liquid sample (distilled water). **a** Tangent of the self-normalized PPE phase. **b** Self-normalized PPE amplitude. Solid lines correspond to best fits to Eqs. (8) (amplitude) and (9) (phase) for obtaining parameter  $P_R$ . The corresponding values are shown on the same plot

(thermal effusivity for the reference sample,  $e_R$ , was taken as  $e_R = 0.1588 \text{ W s}^{1/2} \text{ cm}^{-1} \text{ K}^{-1}$ ) [3, 4]; the reported uncertainties were obtained using the usual formula for error propagation. It is evident that these values of thermal effusivity are consistently smaller than the ones reported in the literature (Table 1, column 6). This can be attributed to the fact that, between the PZT ceramic and the metallic foil in commercial buzzers, there is a small amount of adhesive and this, working as an extra thermal impedance, produces “apparent” thermal effusivities, which are smaller than the real ones. The analytical effect of this extra layer should require a more general mathematical model; however, it is easier to introduce an instrumental factor to take into account the experimental effect of this extra layer, making the analysis simpler; furthermore, this instrumental factor should be redundant if pyroelectric sensors synthesized directly on a metallic foil are available. The instrumental factor here was taken as the ratio of the apparent thermal effusivity for methanol (Table 1, in parentheses in column 3) to the corresponding reported value (Table 1, in column 6). This value was 1.2 and is close to the average of instrumental factors calculated for the three pure substances (methanol, ethanol, and glycerol) listed in Table 1. This ratio shows more variability for the other three samples (essential oils), which is expected since these are



**Table 1** Thermal effusivities obtained by means of the pyroelectric technique described in this work, for some liquid samples

Liquid sample	$P_{S,Amp}$	$e_{amp}$ (Ws <sup>1/2</sup> cm <sup>-1</sup> K <sup>-1</sup> )	$P_{S,Ph}$	$e_{ph}$ (Ws <sup>1/2</sup> cm <sup>-1</sup> K <sup>-1</sup> )	$e_R$ (Ws <sup>1/2</sup> cm <sup>-1</sup> K <sup>-1</sup> )
Ethanol	0.87757 ±0.00648	(0.0491±0.0005) 0.0589±0.0006	0.59317 ±0.01056	(0.0445±0.005) 0.0534±0.006	0.0567 [1]
Methanol	0.94481 ±0.00863	(0.0529±0.0006) 0.0635±0.0007	0.55848 ±0.01167	(0.0419±0.004) 0.0503±0.005	0.0636 [1]
Glycerol	1.35310 ±0.00529	(0.0757±0.0005) 0.0946±0.0006	0.95028 ±0.00335	(0.0713±0.006) 0.0856±0.007	0.0934 [1]
Essential lime oil	0.60313 ±0.00204	(0.0338±0.0002) 0.0406±0.0002	0.39708 ±0.00251	(0.0298±0.003) 0.0358±0.004	0.049 [5]
Essential mandarin oil	0.55274 ±0.00247	(0.0309±0.0002) 0.0371±0.0002	0.35323 ±0.00183	(0.0265±0.002) 0.0318±0.002	0.048 [5]
Essential orange oil	0.74226 ±0.00277	(0.0415±0.0003) 0.0498±0.0004	0.42935 ±0.00478	(0.0322±0.003) 0.0386±0.004	0.048 [5]

$e_{amp}$  ( $e_{ph}$ ) refers to the thermal effusivity from the self-normalized amplitude (phase) and  $e_R$  to literature values. The corresponding parameters for the reference sample (distilled water) were  $P_{R,Amp} = 2.8371 \pm 0.00877$ , from the self-normalized amplitude, and  $P_{R,Ph} = 2.11604 \pm 0.17983$ , from the self-normalized phase. Thermal effusivity values in parentheses correspond to those ones without any instrumental factor correction; the corrected values are summarized right below

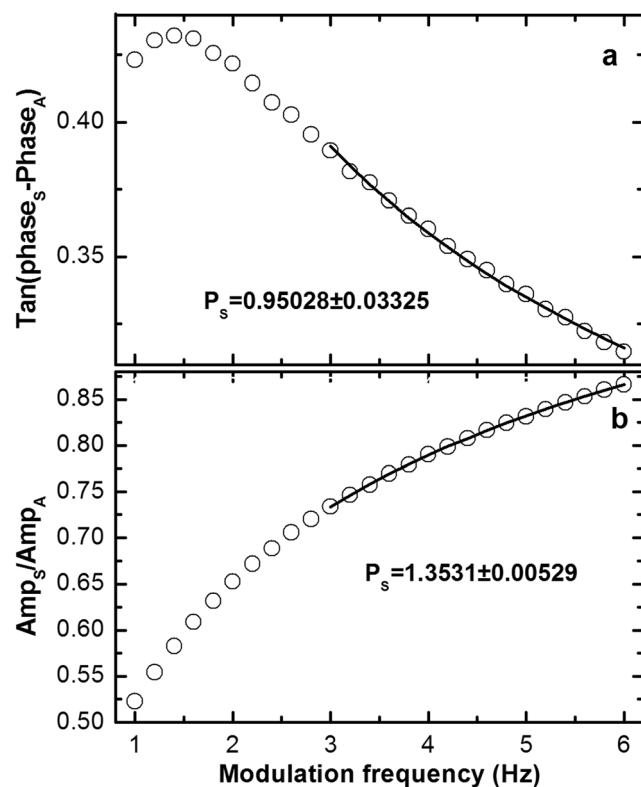
complex substances with high variability in chemical composition (volatile substances and other substances subject to oxidative processes, for instance) which depends, among other factors, on time of storage and the conditions of management; this

results in thermal effusivity values that could change strongly with time; there is a difference with pure substances whose thermal effusivity values are more stable. This fact could also explain the relatively strong difference in thermal effusivities reported here for mandarin oil as compared with the respective thermal property for other two essential oils, whose values were reported previously as being very similar to each other.

The corrected thermal effusivity values by this instrumental factor, shown right below the apparent ones (in parentheses in Table 1), are in close agreement with the ones reported in the literature, and this is especially true for the thermal effusivity values obtained from the self-normalized amplitude; the values corresponding to the self-normalized phase do not share such close agreement. It can be concluded from this that phase is more sensitive to the effect of the extra layer and can be used in this case as experimental criterion for validation purposes, as has been described earlier.

## 5 Conclusions

The presented photopyroelectric technique (in the thermally thin regime for the sensor) applies equally well if other pyroelectric sensors, with no metallic foil, are used; there is no expected instrumental factor in this case, but there is a limitation as to the type of liquid, which must be harmless to the sensor. The pyroelectric sensor requires small thickness and relative large thermal diffusivity for this. For pyroelectric sensors designed in a laboratory [11], there is always the possibility of attaching a protective material (like a thin silicon slab) with adequate physical properties to improve the applicability of the sensor to the type of liquid to be studied and to extend the modulation frequency range for the analysis.



**Fig. 4** Photopyroelectric (PPE) signal, as function of the modulation frequency, for glycerol. **a** Tangent of the self-normalized PPE phase. **b** Self-normalized PPE amplitude. Solid lines correspond to best fits to Eqs. (8) (amplitude) and (9) (phase) for obtaining parameter  $P_s$  for this sample. The corresponding values are shown on the same plot

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