



# Self-normalized inverse photopyroelectric technique with viscous front window for thermal effusivity measurements in liquids



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## ABSTRACT

In this paper, the front detection (also called inverse configuration) photopyroelectric method for thermal effusivity measurement in liquids is improved by adding a viscous liquid to the front face of the pyroelectric sensor, on which the intensity modulated excitation light beam impinges, while the sample is attached to the rear side of the sensor. This viscous liquid will act as an optical transparent window. This configuration avoids problems related with the piezoelectric contribution to the photopyroelectric signal at high modulation frequencies that can be present in conventional approaches due to thermoelastic waves generated in the heated sample or by thermally induced deformations of the detector material itself. The theoretical and experimental principles behind the proposed method are presented and its usefulness is demonstrated with measurements on test samples.

## 1. Introduction

Knowledge of thermal effusivity,  $e$ , is very important because it determines the thermal behavior of materials at surfaces and interfaces heated by non-stationary heat sources [1]. In the particular case of liquids, the photopyroelectric (PPE) technique [2] in the front detection configuration (FPPE) [3] has been emerged as one of the most useful methods for measurements of  $e$  (the FPPE is also called the inverse configuration or i-PPE technique by some authors [4]). In this technique, the sample is located at the rear side of the pyroelectric (PE) sensor, while an intensity modulated light beam impinges on the PE front face. In this way, the sample acts as a heat sink, while the PE sensor acts as both, a source of thermal waves propagating through the PE-sample system, and as the detector of them. The FPPE signal is usually measured in amplitude and phase as a function of the light modulation frequency,  $f$ , using phase sensitive synchronous detection, i.e. using a Lock-in amplifier synchronized at  $f$ . To obtain the thermal effusivity a fit of the experimental data using theoretical expressions is doing [5]. Since the first published works on this subject [3,6,7], several methodologies have been proposed to account for the frequency dependent instrumental function. In the most approaches normalization procedures are used aided by measurements with a well-known

reference sample [8]. Recently a method was proposed [9] in which the pyroelectric sensor itself was used as a reference sample. It is based on the fact that, for a simplest configuration (air-PE sensor-sample-air), at high modulation frequencies the FPPE signal does not depend any more on the sample's thermal properties. The main drawback of this method is that a very thick PE sensor must be used to avoid the frequency region in which piezoelectric effects become important. These effects could be caused by thermal deformation of the detector or as a consequence of thermoelastically generated acoustic waves generated in the sample and reaching the sensor [10], and their contribution to the pyroelectric signal have been studied by several authors [11–19]. As a consequence of using a very thick sensor, the modulation frequencies used in the mentioned method [8–12] are very low, so that the integration times of the lock-in amplifier and the measurement time become large and the use of a preamplifier with very high input impedance is mandatory. Optimizing the experimental conditions it is possible to minimize the piezoelectric contribution. One way to do this is diminishing the PE dimensions, maintaining the sensor flat and supporting it under tension so that the bending resonance frequencies become very high [20]. But the reduction of the PE dimensions enhances the oscillatory heat flux from the sample and sensor towards the body of the measurement cell so that experimental results can be

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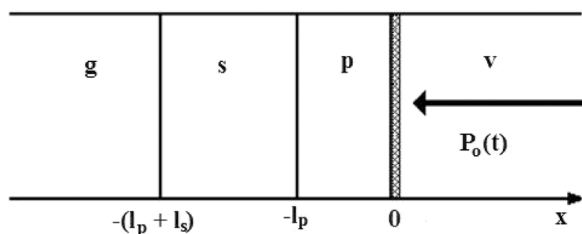


Fig. 1. Schema of the experimental i-PPE configuration. The colored layer at the  $x = 0$  coordinate corresponds to black paint used to increase the absorption of light.

distorted [21]. This problem can be avoided using a special design for the metallic coatings of the PE sensor [22] or by putting the PE sensor in contact with a material that dissipates the acoustic waves [11,19,23], e.g. using a viscous liquid window, as will be done in the present work.

## 2. Methodology

The Fig. 1 shows schematically the experimental configuration used in the i-PPE technique [9], where  $v$  represents a viscous transparent liquid,  $g$  is air,  $p$  is the PE sensor,  $s$  is the sample and  $P_0(t)$  is the power of the amplitude modulates laser beam.

The voltage generated between both sides of the PE sensor due to the periodical heating is given by [24]:

$$V_p(t) = \frac{pP_0(1-R)}{4C_p} \Gamma \exp(i\omega t) = A\Gamma \exp(i\omega t), \quad \omega = 2\pi f \quad (1)$$

where  $P_0$  is the laser power amplitude,  $p$  is the pyroelectric coefficient,  $C_p$  is the pyroelectric sensor electrical capacity and  $R$  is its optical reflection coefficient. For a thermally thick sample (the thermal diffusion length of the thermal wave being much smaller than the sample's thickness), the coefficient  $\Gamma$  is given by [9]

$$\Gamma = \frac{(1 - e^{-\sigma_p l_p})(1 + R_{vp})}{k_p \sigma_p^2 l_p} \left[ \frac{R_{sp} e^{-\sigma_p l_p} + 1}{R_{vp} R_{sp} e^{-2\sigma_p l_p} - 1} \right] \quad (2)$$

In Eq. (2) the following notation has been used:

$$R_{ij} = \frac{1 - b_{ij}}{1 + b_{ij}}$$

$$b_{ij} = \frac{e_i}{e_j}$$

$$\sigma_j = (1 + i)a_j$$

$$a_j^{-1} = \mu_j = \sqrt{\frac{\alpha_j}{\pi f}}$$

where  $\omega = 2\pi f$ ,  $\alpha_i$ ,  $e_i$  and  $k_i$  are the thermal diffusivity, effusivity and conductivity of the  $i$ -th region of Fig. 1 ( $i = s, p, v$ ),  $\mu_i$  is the thermal diffusion length and the parameter  $R_{ij}$  represents the thermal wave reflection coefficient at the  $i$ - $j$  interface. Note that the only thermal parameter of the sample from which the parameter  $\Gamma$  depends is the thermal effusivity,  $e_s$ .

The photopyroelectric cell used in our experiments is shown schematically in Fig. 2.

The front window is an optical transparent viscous liquid (VL) and must be thermally thick, so that the thermal wave generated at the PE sensor becomes strongly attenuated near the glass – viscous liquid interface. Therefore, the chosen thickness of the VL layer was 4 mm, i.e. ten times greater than the value of thermal diffusion length at the lowest used modulation frequency. The VL is located in a sort of container, formed by a ring of plexiglass glued to a transparent glass. The seals do not allow leakage of the VL. The PE sensor chosen for the experiments was a PVDF foil ( $\alpha_p = 0.58 \times 10^{-7} \text{ m}^2/\text{s}$  and  $e_p = 559.4 \text{ W s}^{1/2} \text{ m}^{-2} \text{ K}^{-1}$  [25]) with a thickness of 28  $\mu\text{m}$ .

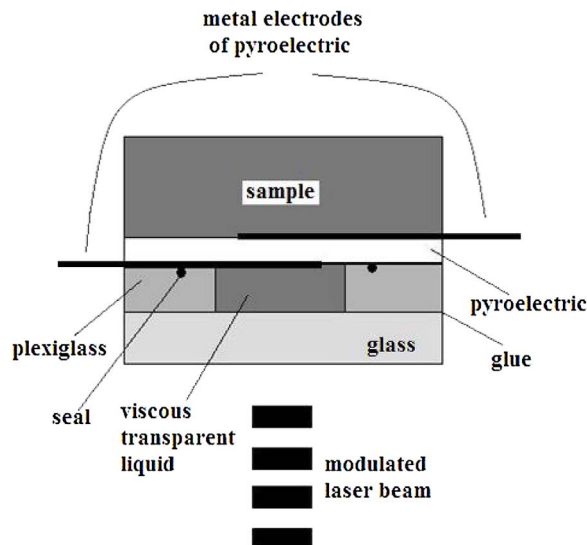


Fig. 2. The photopyroelectric cell (schematically). The PE voltage is generated only in the area in which there are metallic layers at both sides of the PE sensor [22].

After several experiments using different transparent liquids and PE sensor dimensions, we resorted to use a corn syrup as a VL (trade mark Karo-Bebé, [26]). Using a pyroelectric sensor diameter of 8 mm and a laser beam spot on it of 6 mm, it was seen that the thermoelastic effect doesn't appear at frequencies lower than 400 Hz for the samples of interest (e.g. common liquids). Note that the thermoelastic effect manifests itself with a suddenly increase of the PE signal phase instead of the awaited decrease with frequency when this effect is absent [15].

This corn syrup is a mixture of various saccharides and water with unknown proportion, so we could not find data on their thermal, optical and rheological properties. So we measure the parameters of interest. Its viscosity, measured with a LVT (Brookfield) viscometer was found as  $\eta = 3.4 \text{ Pa s}$ , and its thermal effusivity and diffusivity, measured by photopyroelectric methods, were  $e_v = 1116.0 \text{ W s}^{1/2} \text{ m}^{-2} \text{ K}^{-1}$  and  $\alpha_v = 1.0 \times 10^{-7} \text{ m}^2/\text{s}$ , respectively. The optical absorption spectrum of a typical syrup sample measured with a UV-vis spectrometer (DR6000 Hach) is shown in Fig. 3. It can be seen that for the used laser (LDM-3 LASEREX Technologies) wavelength,  $\lambda = 650 \text{ nm}$ , the syrup can be considered almost transparent (the heat generated by the small optical absorption for a nominal laser power of 100 mW is negligible).

Due to the small diameter of the PE sensor, the electrical capacity is also very small ( $C_p \sim 120 \text{ pF}$ ). In order to work near the ideal voltage

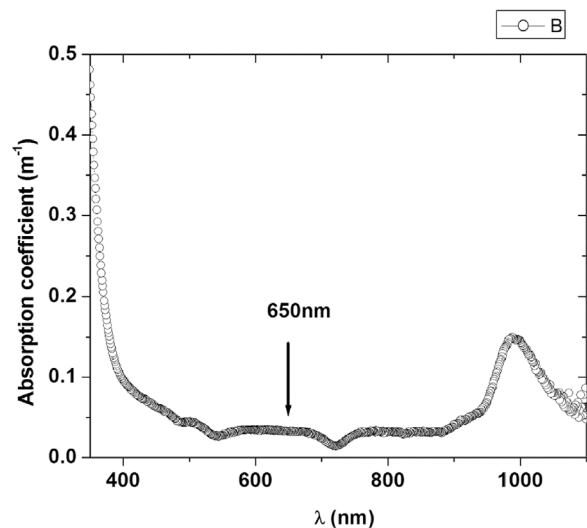


Fig. 3. The optical absorption spectrum of Karo Bebé corn syrup.

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