



Non-contact photopyroelectric method for thermal diffusivity and effusivity investigation of porous solids. Theoretical approach, mathematical simulations and experiment.



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ABSTRACT. The suitability of two photopyroelectric (PPE) methods for measuring the thermal diffusivity and effusivity of porous solids is described. The main characteristic of both methods, associated with the possibility of investigating porous solids, is the absence of coupling fluid between the pyroelectric sensor and sample. The first method (proposed initially by Salazar et al. [1]) relies on the front detection configuration with opaque pyroelectric sensor and thermally thick sample. Based on the frequency scan of the phase of the PPE signal, this method leads to the direct measurement of the sample's thermal effusivity. The second method (proposed initially by Zammit et al. [2]) is based on two successive measurements (frequency scans), one in back and one in front PPE configuration, followed by a self-normalization of the phase vs. modulation frequency dependence. This method uses transparent pyroelectric sensor and backing material and allows the direct measurement of sample's thermal diffusivity. In the paper, both methods were adapted and improved for investigating some porous building materials of interest: old bricks with unknown values of thermal parameters.

1. INTRODUCTION. Among the PT techniques, the photopyroelectric (PPE) method proved to be one of the most performant. With the PPE calorimetry the heat, generated in the sample by the absorption of radiation, is measured with a pyroelectric sensor in good thermal contact with the sample. Various detection configurations (back and front), sources of information (amplitude and phase of the signal) and scanning parameters (chopping frequency of radiation and/or thickness of one liquid layer of the detection cell) have been used, depending on the purpose of the investigation. The best samples for PPE investigations are the liquids because the thermal contact between a liquid sample and a solid sensor is perfect and consequently, accurate results are expected. When investigating solids, a coupling fluid (liquid or paste) is used between sensor and sample, in order to assure a good thermal contact. For porous solids, any type of liquid or pasty coupling fluid penetrates into the sample and the obtained results are not accurate. The only alternative to investigate from thermal point of view such porous samples, by PPE method, is to replace the liquid/pasty coupling fluid between sensor and sample by a layer of air. Performing a search of the recent literature in the field, we found two methods that can be adapted for thermal effusivity and diffusivity investigations of porous building materials. The first one, proposed by Salazar et al. [1], is based on the frequency scan of the phase of the FPPE signal. The second method, proposed by Zammit et al., [2] is based on two successive measurements (frequency scans), one in back and one in front PPE configuration, followed by a self-normalization of the phase vs. modulation frequency behaviors. The paper presents the way in which these two above mentioned techniques have been adapted for the investigations of thermal diffusivity and effusivity of porous building materials; three old bricks have been selected as samples. For support, some materials with known values of thermal parameters (tefal, rubber) have been also investigated with the proposed methods.

2. THEORY

The PPE detection cells used in the two detection configurations are schematically described in Fig. 1.

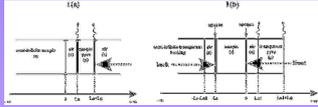


Fig. 1. Schematic view of the detection cell: 1(a) for Method 1, 1 (b) for Method 2

2.1 Method 1 [1]

Concerning the first method (Fig. 1a), the theory was developed in Ref. [1] and we will present here only the final result: The normalized FPPE signal (normalization performed with sensor without sample) is described by the relationship (the coupling fluid and the backing material were already replaced by air):

$$V_n = \frac{2A_0(A_1 + A_2)}{B_1 \cosh(q_a L_a) + B_2 \sinh(q_a L_a)} \quad (1)$$

where standard notation have been used [1].

Eq. (1) can be used to fit an experimental scan V_n vs. modulation frequency ω , with sample's thermal effusivity, e_p , heat losses through convection and radiation, h , and air gap thickness between sensor and sample, L_a , as fitting parameters.

Method 2 [2]

As it was previously mentioned, this second method (Fig. 1b) is based on two successive frequency scans, one in back and one in front PPE configuration, followed by a self-normalization of the phase vs. modulation frequency behaviors (phase BPPE/phase FPPE). The self-normalized phase of the PPE signal is given by the relationship [2]:

$$\varphi = -a_s L_s + \tan^{-1} \left(\frac{(1 - b_{BS}) \sin(2a_s L_s) e^{-2a_s L_s}}{(1 + b_{BS}) + (1 - b_{BS}) \cos(2a_s L_s) e^{-2a_s L_s}} \right) \quad (2)$$

where standard notations have been used [2].

Eq. (2) indicates that for high enough modulation frequencies the second term from the right side vanishes and one can easily obtain sample's thermal diffusivity. An analysis of Eq. (2) shows that if the backing material is air, we can approximate $b_{BS}=0$ and Eq. (2) takes the simplified form:

$$\varphi = -a_s L_s + \tan^{-1} \left(\frac{\sin(2a_s L_s)}{e^{2a_s L_s} + \cos(2a_s L_s)} \right) \quad (3)$$

Eq. (3) shows that using air as backing material one can measure in this configuration only the sample's thermal diffusivity, both in low and high frequency range.

However, if the backing material is air, when calculating the PPE signal in the back configuration, as pointed out in ref [1], we have to take into account the heat losses by convection and radiation at $x = -L_s$ interface (see Fig. 1b). Such a calculation leads to a more complicated relationship (see eq. 4) in which the phase of the self-normalized signal (back/front) depends on three fitting parameters: sample's thermal diffusivity, the thickness of the air gap between sensor and sample, and heat losses by convection and radiation.

$$U_N = \frac{H' \cdot e^{-a_s L_s} - \frac{2k_s \sigma_s}{k_s \sigma_s - h} \frac{2k_s \sigma_s}{H(1 + e^{-2a_s L_s})} \frac{-e^{-2a_s L_s} \left[1 - \frac{1}{2} (1 + e^{-2a_s L_s}) \right] - \left[\frac{1}{2} (1 + e^{-2a_s L_s}) - 1 \right]}{-e^{-2a_s L_s} \left[1 - \frac{1}{2} M \right] + 1 - \frac{1}{2} M} \quad (4)$$

$$M = 1 + e^{-2a_s L_s}$$

In order to avoid this complication, we prefer to use a transparent backing material and air gap between this backing and sample. This transparent backing will absorb the heat developed at the absorbent interface, and the whole layered cell (sensor/air/sample/air/backing) can be studied classically, assuming only the heat propagation by conduction.

CONCLUSIONS. The main adaptations and improvements performed for the two PPE methods/configurations are summarized in the following. Concerning Method 1, used for thermal effusivity investigations, the limitation imposed in the variation range of two fitting parameters (h and L_a) certifies the unicity of the solution found for the value of the thermal effusivity (eliminates the degeneracy). Concerning Method 2, the insertion of two layers of air at the interfaces sensor/sample and sample/backing respectively, constraints the applicability degree of the configuration to the measurement of thermal diffusivity only. However, the combination of these two techniques allows the complete thermal characterization of the investigated building materials. From technical point of view, if Method 1 requires samples with only one flat surface in contact with the sensor, Method 2 requires a more complicated processing of the samples: two flat surfaces and a sample thickness generally lower than 1mm. Consequently, this second method will introduce some limitations connected with samples porosity: samples with large pores become transparent for the laser and cannot be investigated. Another limitation of the methods is connected with the layer of air at the two interfaces. If the rugosity of the sample is too high, the air layer is too thick and consequently, (i) air acts as a low-pass filter in frequency dependent investigations and (ii) the signal/noise ratio in Method 2 is lower than in classical PPE measurements.

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References

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- [2] U. Zammit, F. Mercuri, S. Paoloni, M. Marinelli, R. Pizzoferrato, Simultaneous absolute measurements of the thermal diffusivity and the thermal effusivity in solids and liquids using photopyroelectric calorimetry, J. Appl. Phys. 117, (2015) 105104, 1-8.

3. RESULTS

Fig. 2 displays the frequency behavior of the normalized FPPE phase for each of the three investigated samples, together with the best fit performed with Eq. (1)

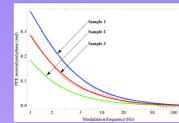


Fig. 2. The frequency behavior of the normalized FPPE phase for the investigated samples. The best fit performed with Eq. (2) is also presented.

Table 1 contains the results obtained for the thermal effusivity of the investigated samples, together with the values obtained for the other two fitting parameters, h and L_a .

sample	Thickness (μm)	Thermal effusivity ($\text{Ws}^{-1}\text{m}^2\text{K}^{-1}$)	h (Wm^2K^{-1})	L_a (μm)
1	320	1174 ± 117	42	17
2	770	888 ± 43	39	46
3	750	942 ± 75	40	26

Table 1. The values of the fitting parameters for the investigated samples.

A typical result obtained for the phase of the PPE signal for the same sample 2, as obtained with Method 2, is displayed in Fig. 3.

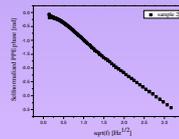


Fig. 3. The frequency dependence of the self-normalized PPE phase (back/front) for sample 2.

Fig. 4 display the behavior of the self-normalized PPE phase as a function of \sqrt{f} for all investigated samples (in the samples thermally thick regime) and Table 2 contains the values obtained for the thermal diffusivity, conductivity and volume specific heat (the thermal conductivity and volume specific heat have been obtained by combining the results from both Methods 1 and 2)

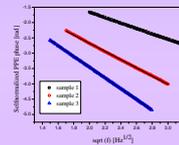


Fig. 4. Self-normalized PPE phase as a function of \sqrt{f} for all investigated samples, in the samples thermally thick regime.

sample	Thermal diffusivity ($10^6 \text{m}^2\text{s}^{-1}$)	Thermal conductivity ($\text{Wm}^{-1}\text{K}^{-1}$)	Volume specific heat ($10^6 \text{Jm}^{-3}\text{K}^{-1}$)
1	0.41 ± 0.02	0.75 ± 0.07	1.82 ± 0.12
2	1.04 ± 0.03	0.91 ± 0.04	0.87 ± 0.04
3	0.56 ± 0.02	0.70 ± 0.05	1.55 ± 0.08

Table 2. The values of thermal diffusivity e , thermal conductivity k and volume specific heat C measured for all three samples of old bricks.