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Analysis of pulsed photoacoustic signal: theoretical study of the diffusion of chromophores in the human skin

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Abstract. Pulsed photoacoustic spectroscopy (PPAS) provides a convenient means for in vivo and in situ monitoring of human skin properties and surface concentrations of locally applied substances, such as drugs and cosmetics. In this article, we applied the one-dimensional theoretical model of Mandelis and Royce (J. Appl. Phys. 50, 6 (1979)) to simulate the photoacoustic response of human skin and diffusion phenomena through it, for various optical absorption coefficients and sample thicknesses. This approach is analyzed through the temperature and pressure variations in the photoacoustic cell.

PACS. 82.80.Kq Energy-conversion spectro-analytical methods (e.g., photoacoustic, photothermal, and optogalvanic spectroscopic methods) – 87.64.-t Spectroscopic and microscopic techniques in biophysics and medical physics – 87.15.Aa Theory and modeling; computer simulation

1 Introduction

Photoacoustic spectroscopy has been applied to an impressive variety of fields ranging from semiconductor materials to medicine. This non-destructive technique allows for a spatial scan inside the medium and can be applied even to opaque media. Therefore, this spectroscopic technique presents a particular interest for complex and biological systems. The re-discovery of photoacoustic spectroscopy in the early 1970s [1], nearly a century after the pioneering work of Arthur Graham Bell [2], has led to rapidly advancing research into a plethora of related spectroscopic and analytical techniques.

The conventional methods of analysis, used to investigate the properties of molecules inside a medium, involve the measurement of the transmission and reflection coefficients of the material, to determine the absorption of a given chromophore, to an average value of the property over the thickness of the material being investigated (thin film, solution, crystal). It does not provide any depth-related information of the presence of the substances in the material, *i.e.* the local concentration.

In contrast photoacoustic spectroscopy (PAS) allows non-transparent materials, either homogeneous or heterogeneous, liquids or solids, biological or synthetic, to be characterized.

Pulsed photoacoustic spectroscopy (PPAS) has several features that make it an appropriate technique for *in-vivo* skin condition measurement. It probes only a small area, so it can be used on all parts of the body and it uses laser probing of low irradiance, so it is non-destructive.

Typical relative increases are between 0.01 °C and 0.001 °C and can be detected through a miniature microphone in contrast with other techniques like opto-thermal transient emission radiometry (OTTER) which induce several degrees of temperature increase [3–6].

This technique performs a spatial scan of the heat emission inside the material. The depth of this scan depends on several factors such as the thickness, the optical absorption coefficient and the thermal diffusivity of the material. In the case of classical (modulated) photoacoustic spectroscopy (MPAS), the light beam is modulated in intensity by a mechanical chopper placed on the light path of a high power lamp. Pulsed photoacoustic spectroscopy (PPAS) remains similar, in principles, to MPAS except that the excitation light is a pulse of short duration.

The basic principles of photoacoustic spectroscopy consist in irradiating the sample with light and measuring the heat response induced by this light. The acoustic oscillation is generated by the expansion and contraction of a boundary layer created by thermal diffusion waves from inside the solid, following the absorption and optical-to-thermal energy conversion of (harmonically chopped or pulsed) radiation. MPAS is a narrow- band measurement of amplitude and phase of a signal at the same frequency as the excitation, whereas PPAS is a wideband measurement and a convolution of the impulse response signal

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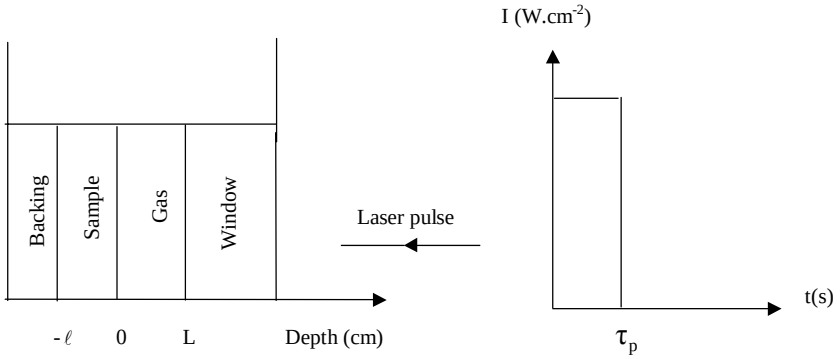

Fig. 1. Representation of the different photoacoustic cell regions.

$$\frac{\partial^2 T_{\rm s}(x,t)}{\partial x^2} - \frac{1}{\alpha_{\rm s}}\frac{\partial}{\partial t}T_{\rm s}(x,t) = \frac{-\eta\beta I_0}{K_{\rm s}}\exp(\beta x) \quad 0 < t \le \tau_{\rm p} \quad \text{and} \qquad -\ell \le x \le 0$$
$$= 0 \quad t > \tau_{\rm p} \quad -\ell \le x \le 0 \tag{1}$$

with the temporal excitation waveform. However, there is a relationship between the two, *via* the Fourier transformation, if the response of the system is linear.

The heat emission from the sample can be detected, directly, by an IR sensor as in photothermal spectroscopy, a technique called infrared photothermal radiometry (PTR) [7,8], by a piezoelectric sensor in contact with the sample [9,10], or by a pyroelectric sensor made from a material such as polyvinylidene fluoride in photopyroelectric spectroscopy (PPES) [11,12] and indirectly, by measurement of the pressure variation in a gas buffer enclosed with the sample in the detection cell [1,13–17]. Lahjomri *et al.* [17] have developed a pulsed photoacoustic spectrometer which offers the possibility of *in-vivo* measurements and the characterization of the phenomenon of the diffusion of chromophores inside human skin.

On the theoretical level, the photoacoustic response signal of a sample excited by a pulse light of very short duration was determined by Mandelis and Royce [14].

An analysis of this signal, would make it possible to define the effect of the physical parameters of the medium, and consequently, to proceed starting from this signal, to the measurement of characteristic parameters of the diffusion kinetics.

In this article we propose the study of the influence of the physical parameters through the analysis of the variation of the surface temperature and pressure of a human skin sample, excited by a light pulse, according to the Mandelis and Royce theory [14].

2 Device physics

In the Mandelis and Royce theory of pulsed photoacoustic spectroscopy, optical, acoustic and thermal processes occurring in a photoacoustic cell excited by a light pulse of short duration are treated in a one-dimensional approximation. The laser spot-size is supposed wide enough compared to the sample length l (cm) so that one-dimensional thermal wave propagation is satisfied [11]. A block diagram of a photoacoustic cell is reported in Figure 1. The sample having an optical absorption coefficient β (cm^{-1}) is supported on an optically transparent backing.

The photoacoustic cell, hermetically closed contains the backing, the sample, the gas layer of length L (cm), which contains an optically transparent gas (air) and the light pulse enters the cell through a non-absorbing window. The nonradiative deexcitation process, following the excitation of the sample by a light pulse in the form of Heaviside function of duration $\tau_{\rm p}$ (s) and an irradiance of I_0, and the absorption of this light in the bulk of the sample, are taken to be instantaneous.

One can note that this technique gives the possibility of adjusting the light pulse duration – by choosing different laser sources – (100 μs to about 100 fs) to the characteristic time of the phenomenon under investigation (several ms to about 100 fs).

The thermal diffusion equations for each of the regions of the cell can be written, with only that for the solid containing a distributed heat source for the duration of the pulse:

See equation (1) above

where $T_{\rm s}(x,t)$ is the temperature of the sample, η is the efficiency of the nonradiative process, $K_{\rm s}$ and $\alpha_{\rm s}$ are the thermal conductivity and the thermal diffusivity, respectively.

Three similar equations, but with no source term, hold for the other regions of the cell.

The analytical resolution of equation (1) includes different methods like separation of variables and the method of images for reducing the dimensionality and simplifying the equations, also Laplace and Fourier transformation, and more recently Green's functions methodologies [21,22].

Taking into account the conditions of temperature and heat flux continuity between the regions at all times,

$$\Delta\hat{p}(s) = \left(\frac{\eta\beta I_0 p_0[1-\exp(-s\tau_\mathrm{p})]}{sk_\mathrm{s}L\alpha_\mathrm{g}T_0(\beta^2-a_\mathrm{s}^2)}\right)\Big[\cosh(a_\mathrm{g}L)-1+D\sinh(a_\mathrm{g}L)\Big] \times\left(\frac{(r-1)(b+1)\exp(a_\mathrm{s}\ell)-(r+1)(b-1)\exp(-a_\mathrm{s}\ell)+2(b-r)\exp(-\beta\ell)}{(b+1)[(1+gD)s+(D+g)C]\exp(a_\mathrm{s}\ell)-(b-1)[(gD-1)s+(g-D)C]\exp(-a_\mathrm{s}\ell)}\right) \quad (2)$$

Mandelis and Royce [14] solve the time domain equations in Laplace space s by taking their Laplace transforms to reduce the partial differential equations in (x,t) to a set of ordinary differential equations in x.

The general expression for the Laplace transform of the photoacoustic signal is then [14]:

See equation (2) above

where $a_\mathrm{s} = (s/\alpha_i)^{1/2}$ with $i = s, g$; $q = a_\mathrm{s}\,l$; $r = \beta/a_\mathrm{s}$; $C = \cosh(a_\mathrm{g}\,\ell)$; $b = (k_\mathrm{b}\,a_\mathrm{b})\ /\ (k_\mathrm{s}\,a_\mathrm{s})$; p_0 and ℓ are respectively the initial pressure in the PAS cell and the sample thickness.

B and D are quantities much smaller than unity, typically on the order of 10^{-3}.

This expression is inverted to obtain $\Delta P(t)$, the PAS response, for several special cases.

Practically, the microphone is sensitive to the variations of the pressure inside the gas buffer of the cavity yielding a pressure variation response pulse $(\mathrm{d}P(t)/\mathrm{d}\,t)$ and the pressure inside the cavity can therefore be obtained upon time integration [17].

The PAS signal is governed by three parameters: the optical absorption length ($\mu_\beta = 1/\beta$), the thermal diffusion length μ_s which is given in MPAS by:

$$\mu_\mathrm{s} = \sqrt{\frac{2\alpha_\mathrm{s}}{\omega}} \quad (3)$$

where ω is the angular modulation frequency of the laser intensity and α_s is the thermal diffusivity of the sample, the last parameter being the thickness of the sample l.

In impulse and step excitation, there is no unique diffusion length, because the heat continues to spread as time proceeds. Instead, a time-dependent diffusion length for unidimensional problems can be defined by a relationship such as:

$$\mu_\mathrm{s} \approx \sqrt{2\alpha_\mathrm{s}t}. \quad (4)$$

This definition is not unique, but other definitions would deviate from it only by a numerical factor of the order of unity [20].

The comparison between these three parameters $(\ell, \mu_\beta, \mu_\mathrm{s})$ gives six special cases given in the theory of Rosencwaig and Gersho [1], and only four cases in the theory of Mandelis and Royce [14].

The time-domain model of the theory of Mandelis and Royce [14] in Laplace space is the equivalent of the Rosencwaig-Gersho model [1] in the frequency domain, but with each pulse displaying in the time-domain development of the photoacoustic signal, that information obtainable in the modulation mode by separate measurements at different modulation frequencies.

Early times (high modulation frequencies) contain surface information, later times provide weighted information from the interior of the sample. This is the basis of depth profiling, which gives PPAS the ability to sense changes in material properties with depth.

Therefore, on the practical level [15–17], one can thus deduce from the temporal signal generated by an impulse of light, the frequency response that one would obtain by the technique of the modulated photoacoustic spectroscopy defined by the theory of Rosencwaig and Gersho [1]. The exploitation of the signal in the frequency domain leads more easily to the determination of thermal diffusion depth. This last parameter is fundamental in profilometric investigations. It represents the layer of the medium, from which the signal emanates and thus yields the exact probed thickness.

3 Results and discussion

By using the expression for temperature and pressure variations (expression (15) and (17) of Ref. [14]) we evaluate numerically, both in frequency domain and time domain, the temperature and pressure variations, by using adequate expressions for the error function and the complementary error function [18].

For displaying certain aspects of the different curves, it is imperative to use logarithmic or otherwise non-linear scales for amplitude and/or time axes.

Figure 2 shows the temperature and pressure variations, $\theta(t)$ and $\Delta P(t)$, of *Stratum Corneum* ($\ell = 10\,\mu m$) following an excitation (*i.e.* absorption) by laser beam. For this case the backing of the sample is the epidermis. In Figure 3 the sample is a diffusing substance and the backing is the human *Stratum Corneum*. The following thermophysical parameters have been used in calculating the curves. Values of physical parameters of the human skin have been taken from reference [19]:
$K_\mathrm{Sc} = 0.21\ \mathrm{W\,m^{-1}\,K^{-1}}$, $K_\mathrm{E} = 0.42\ \mathrm{W\,m^{-1}\,K^{-1}}$, $C_\mathrm{Sc} = 1.2\ \mathrm{kJ\,kg^{-1}\,K^{-1}}$, $C_\mathrm{E} = 1.2\ \mathrm{kJ\,kg^{-1}\,K^{-1}}$, $\rho_\mathrm{Sc} = 3.6 \times 10^3\ \mathrm{kg\,m^{-3}}$, $\rho_\mathrm{E} = 3.6 \times 10^3\ \mathrm{kg\,m^{-3}}$, $\alpha_\mathrm{Sc} = 4.9 \times 10^{-4}\,\mathrm{cm^2\,s^{-1}}$, $p_0 = 1$ atm, $\alpha_\mathrm{g} = 0.2\ \mathrm{cm^2\,s^{-1}}$. The pulse power flux was $I_0 = 3 \times 10^3\ \mathrm{W\,cm^{-2}}$ of duration $\tau_p = 10^{-6}$ s, and the cell length was chosen close to 0.1 cm, a typical PAS cell dimension.

Figure 3 shows the temperature variations with time for various values of the optical absorption coefficient β of the diffusing substance. For optically transparent substances (the optical penetration depth is much larger than the thickness of the sample), the temperature varies linearly with β. For this example the thermal parameters of the substance were identical to those of the skin.

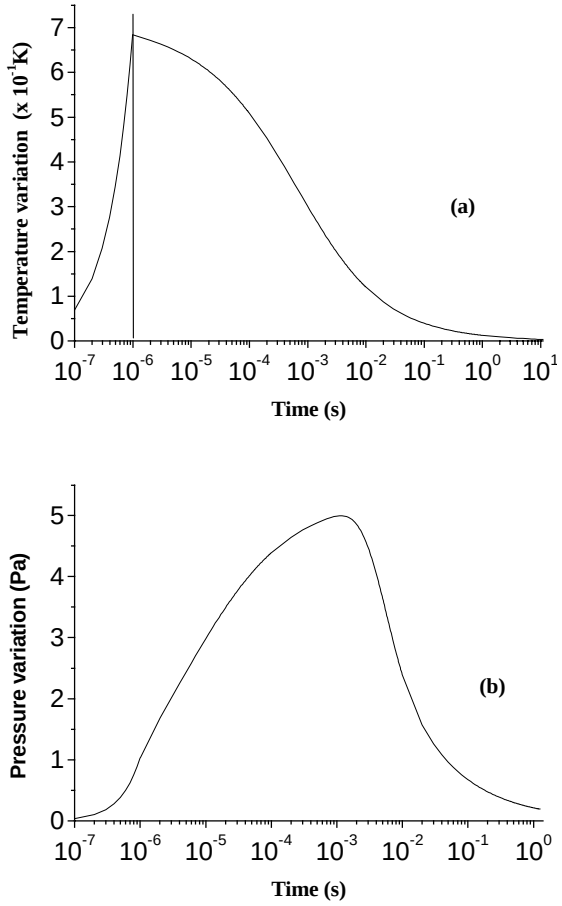

Fig. 2. Representation of temperature (a) and pressure (b) variations of a *Stratum Corneum* sample of thickness 10^{-3} cm, optical absorption coefficient $\beta = 10^3$ cm^{-1} and thermal diffusivity $\alpha_s = 4.9 \times 10^{-4}$ cm^2 s^{-1}.

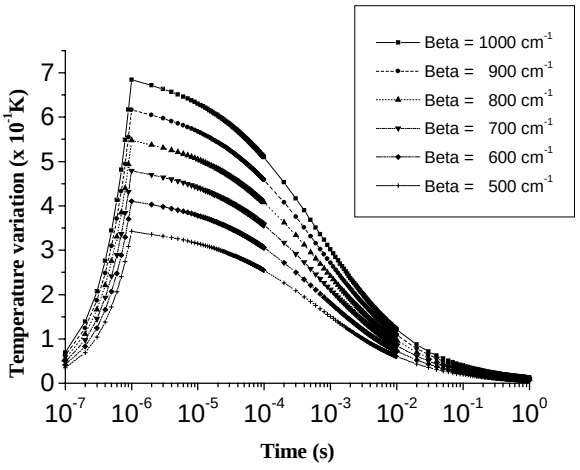

Fig. 3. Temperature variations $\theta(0,t)$ of the interface (sample-gas) of an optically thin diffusing substance of thickness $\ell = 10^{-3}$ cm and thermal diffusivity $\alpha_s = 4.9 \times 10^{-4}$ cm^2 s^{-1} for different values of the optical absorption coefficient β.

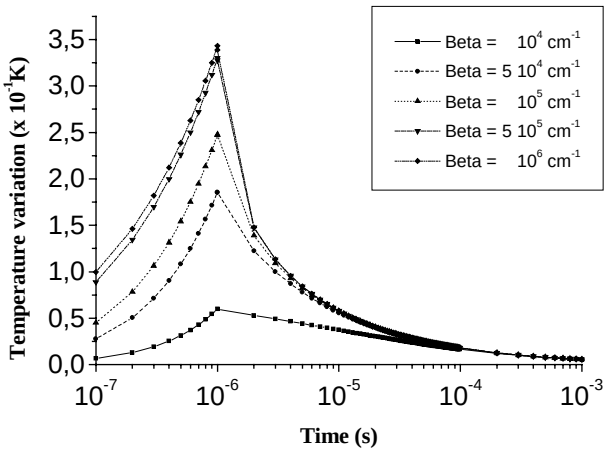

Fig. 4. Temperature variations $\theta(0,t)$ of the interface (sample-gas) of an optically opaque diffusing substance of thickness $\ell = 10^{-3}$ cm and thermal diffusivity $\alpha_s = 4.9 \times 10^{-4}$ cm^2 s^{-1} for different values of the optical absorption coefficient β.

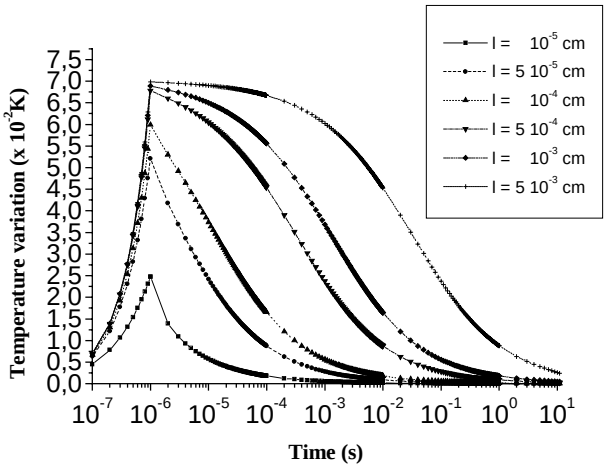

Fig. 5. Temperature variations $\theta(0,t)$ of the interface (sample-gas) of an optically thin diffusing substance of optical absorption $\beta = 10^3$ cm^{-1} and thermal diffusivity $\alpha_s = 4.9 \times 10^{-4}$ cm^2 s^{-1} for different thicknesses.

The temperature variation of the substance is seen to increase during the pulse and to reach its maximum value at the end of the pulse after which it decays to the background value *via* heat conduction into the backing (*Stratum Corneum*) and into the gas.

Figure 4 shows temperature variations for an optically opaque sample of diffusing substance. It can be seen that the temperature amplitude is relatively insensitive to large variations in β at experimentally attainable observation times. For small times, the temperature variation profile exhibits a β dependence, whereas for later times, $\theta(0,t)$ exhibits no dependence on β, in contrast to the optically thin sample case, where the amplitude varies linearly with β at all times. One can note that for the case of optically opaque sample, since the optical absorption depth is much smaller than the thickness of the sample, the temperature profile is independent of the thickness of the sample.

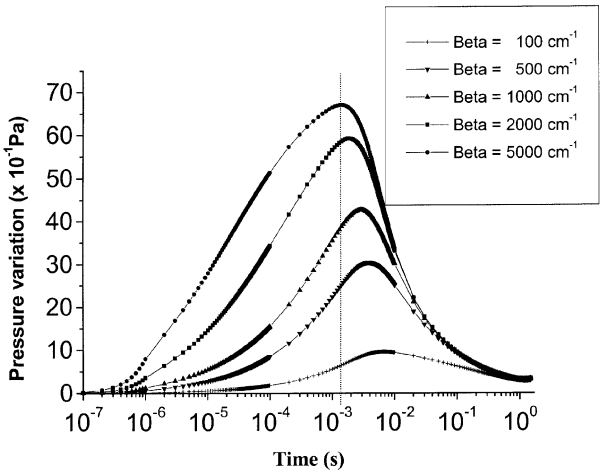

Fig. 6. Pressure variations $\Delta P(t)$ in the gas buffer of an optically opaque diffusing substance of thickness $\ell = 10^{-3}$ cm and thermal diffusivity $\alpha_{\rm s} = 4.9 \times 10^{-4}$ cm^2 s^{-1} for different values of the optical absorption coefficient β.

Figure 5 shows temperature variations of an optically thin sample (diffusing substance) *versus* time for different thicknesses of the sample. The thickness parameter (ℓ) simulates the effect of the thickness of solutions deposited on *Stratum Corneum*. The variation in temperature of the surface of the solution $\theta(0,t)$ is always maximum at the end of the pulse whatever the thickness of the sample, however the results show that the greater thickness corresponds to the slower decrease of its temperature to the baseline value.

The pressure in the gas (Fig. 6), for a diffusing substance sample, does not have its maximum value at the end of the pulse but continues to increase as a function of time. The position of the maximum in the $\Delta P(t)$ curves is very important in the study of the diffusion of drugs into the *Stratum Corneum*. Indeed the value $\Delta P_{\max}$ holds for the amount of chromophores contributing to the signal response.

The diffusion of chromophores into skin is monitored, as in MPAS, through the amplitude of the response signal, $\Delta P_{\max}$. In addition, the PPAS gives access to the time parameter $t_{\max}$ of the response pulse [15–17]. This time factor gives two types of additional information on the medium investigated: a global time delay of heat propagation, and a time profile of heat emission as detected by the microphone. It's important to remember that $\Delta P_{\max}$ gives information about the amount of drugs (absorbing chromophores) and $t_{\max}$ about the depth in the sample from which the major emission occurs. Because acoustic waves propagate very rapidly in air (at a speed of 343 m/s at room temperature), the $t_{\max}$ value, typically between 7 and 12 ms [17], represents the time needed for the main heat emission to diffuse from the inside of the sample to the surface in contact with the air buffer. This assumes that pressure-wave transfer from the surface/boundary layer of heat to the microphone occurs adiabatically and instantaneously.

In previous experimental studies, realized in Leblanc's group [15–17], the photoacoustic response in the frequency domain has been obtained by applying the Fast Fourier Transform (FFT) to the time-domain photoacoustic response. Such a task requires a good choice of the time base and an adequate number of data points, to be able to obtain the response both in low frequencies and high frequencies with a minimal error. In our case, the photoacoustic response in frequency domain has been evaluated using expression (2).

This expression is convenient and presents the major advantage of being able to simulate the photoacoustic response for any duration of the light pulse, even if the time-domain response is only limited to light pulse duration higher than or equal to, 10^{-6} s in the Mandelis and Royce model [14].

Figure 6 shows the pressure variation response of an optically opaque diffusing substance for different values of the optical absorption coefficient β. The $\Delta P_{\max}$ value decreases with β and it is always shifted in time, in contrast to the pressure variation response for different values of the thickness or the thermal diffusivity where the $\Delta P_{\max}$ always occurs at the same $t_{\max}$ for either an optically thin or optically thick sample. This is a direct consequence of the relaxation time τ_β in the expression of the pressure variation, which is given in the case of an optically opaque sample by [14]:

$$\tau_\beta = 1/\left(\beta^2 \alpha_{\rm s}\right). \tag{5}$$

Since the optical absorption coefficient is a square value in the expression (5), the shifted values of $\Delta P_{\max}$ are essentially due to the variation of β more so than the thermal diffusivity $\alpha_{\rm s}$. Also, the optical absorption coefficient is an order of 10^3 while the thermal diffusivity coefficient is only an order of 10^{-4}.

This result is very important in that sense, that it gives for the first time an explanation of the decrease of $\Delta P_{\max}$ observed in previous studies [15–17].

The results of Leblanc's group [15–17] show that the decrease of $\Delta P_{\max}$ is Erfc-like *versus* the kinetic time T. However this behavior was not attributed to any physical parameter.

Figure 7 shows the photoacoustic response in Laplace space (frequency domain), plotted from 10 Hz to 10^3 Hz, for the same sample and for different values of the optical absorption coefficient β.

As mentioned above, all materials can be classified in PAS among six cases depending on three parameters: the sample thickness (ℓ), the thermal diffusion length ($\mu_{\rm s}$) and the optical penetration length (μ_β). Each of these cases follows a specific power law of their $\Delta P(f)$ dependence *versus* the frequency: $\Delta P(f) \propto f^n$. In a Log-Log representation of their spectrum, the exponent can be obtained from the slope of the curve. Any modification in the slope can be due to the change in the frequency which induces a variation in the thermal penetration length $\mu_{\rm s}$. Such variations in the slope can be related to the six cases of the Rosencwaig and Gersho theory [1]. Perturbations of the spectrum can also occur from a modification inside

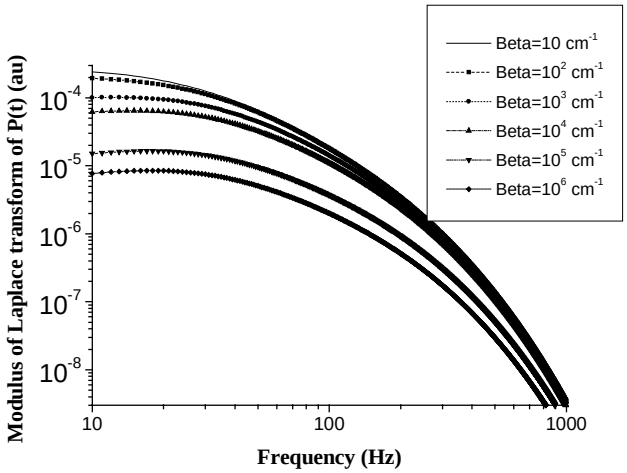

Fig. 7. Representation of the photoacoustic response in frequency domain of a diffusing substance of thickness $\ell = 10^{-3}$ cm and thermal diffusivity $\alpha_s = 4.9 \times 10^{-4}$ cm^2 s^{-1} for different values of the optical absorption coefficient β.

a medium caused by chemical or photochemical reaction. Such perturbation can be caused by a modification of the thermal and optical characteristics of the sample. Another source of perturbation of a spectrum can arise from a multilayered structure of a sample like skin. The variation of the slope is then due to discrete values of μ_s and μ_β, each corresponding to one layer of the stratified medium.

Figure 7 shows the variation in the slope of the frequency response for different values of the optical absorption coefficient β. For this task we have used the modulus of expression (2). Similar curves were plotted for different thickness and thermal diffusivity of the sample but did not exhibit any difference compared to Figure 7.

One can note that PPAS has the advantage of being more revealing of optical, thermal and geometrical nature of the studied medium than other spectroscopic techniques, especially for stratified media such human skin.

4 Conclusion

The study has demonstrated the validity of the Mandelis and Royce theory of pulsed photoacoustic spectroscopy for the characterization of human skin and monitoring the diffusion of topically applied drugs in human skin.

The photoacoustic response of an excited sample of *Stratum Corneum* and another one of diffusing substance was monitored through the plot of both the temperature and pressure variations for different values of the thickness and the optical absorption coefficient.

For the case of optically transparent samples and for a given thickness, the peak of PPAS response will be the same for all values of β (provided $\mu_\beta > \ell$) but the amplitude of the signal will vary linearly with β. For samples of the same β but different thickness, the PPAS response will develop more slowly as the sample thickness increases, as was indicated in Figure 5.

In the case of optically thick sample the PPAS response is independent of the sample thickness and does not vary linearly with β.

The shifted values of $\Delta P_{\max}$ on the time axis (Fig. 6), are in agreement with previous results [15,17] and were attributed, for the first time, to the variation of the optical absorption coefficient β. The representation of the photoacoustic response in the frequency domain demonstrates the potential use of PPAS for new insights into penetration processes because depth profiling can be performed. This profiling is of particular interest for penetration studies in human skin, which is known as a heterogeneous medium.

This study has several applications in cosmetic and pharmaceutical industries.

Work is now in progress to refine the theoretical model of PPAS, especially in the frequency domain, to allow the characterization of human skin, particularly the *Stratum Corneum*, and the quantification of the diffusion of topically applied substances into human skin.

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