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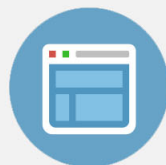
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In vitro thermal diffusivity measurements as aging process study in human tooth hard tissues

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In the present work, the Open Photoacoustic Cell Technique was used to find effective thermal diffusivity of human tooth hard tissues, a thermal variable of great interest in the biological science, and inorganic materials. The aging process of the tooth enamel and dentin was analyzed through its effective thermal diffusivity. The study *in vitro* of these samples showed an increase and posterior decrease with aging of the samples. The values found for the enamel and dentin samples, varied from $(36 - 55) \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$ and $(20 - 32) \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$, respectively. © 2013 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4832481>]

I. INTRODUCTION

Tooth enamel is the hardest and most highly mineralized substance of the body and with dentin, cementum, and dental pulp is one of the four main tissues which make up the tooth in vertebrates. Dentin essentially forms the complete tooth root except for a thin overcovering of cementum. The dentin is about 15 times more organic than enamel¹ and it has sensitivity to stimuli physical and/or chemical unlike enamel, considered an inert tissue. The pulp region of the tooth is much like vital tissue elsewhere in the body, consisting of many blood vessels, nerves, and cells. Enamel and dentin present a thermodynamic reaction when heat is applied. Both tissues contract, and this process is faster in dentin than in enamel.²

Concern about heat transfer effects^{1,3,4} in teeth began after the development of higher and higher speed dental drills that aided quick removal of carious formations. As drill speeds increased and bur-cooling options become more numerous, specific evaluations of the resulting effects on teeth were made. Nowadays in dental bleaching, using heat² should take into account that thermal loads are major risk factor for destruction of tooth structures.

In order to understand the processes of heat transfer through the dental tissues, it is important to find the thermal diffusivity of human dental samples, such as tooth enamel and dentin. The thermal diffusivity determines the rate at which heat diffuses through the material. A study of the literature reveals data on thermal diffusivity; however, the reported values were different between them.³⁻⁶ Braden⁴ studied the conduction in teeth and the effect of lining materials and calculated the thermal diffusivity for teeth from temperature changes measurements occurring at the enamel/dentin interfaces. Brown *et al.*⁵ reported measurements of the density and specific heat of teeth, as well as values of

thermal diffusivity based on thermal conductivity measurements. Panas *et al.*⁶ discussed the difficulties in identify the thermophysical properties of human tooth hard tissues and how some of the obtained results differ from the data presented in the literature, mainly about thermal diffusivity investigations.^{3,7} According to them, the reasons for that are both difficulties in obtaining the adequate specimens and complicated structure of the investigated material, which makes the measurements more difficult. The differences could be related to personal differences of the hard tooth specimens. On the other hand, many modern medical therapy methods are based on thermal treatment in which the thermophysical properties are essential to mathematical modeling involved in the therapy planning.

Photothermal techniques already have been used as diagnostic technique of several defects and features of human teeth.⁸⁻¹⁰ Therapy with topical application of fluoride or new drugs can be monitored since the thermal diffusivity is known.¹¹ The Open Photoacoustic Cell Technique (OPC)¹²⁻¹⁴ is a non-destructive and low-cost technique used to find the thermal diffusivity *in vitro* with minimal amount of sample, specially dental materials.¹⁵⁻¹⁷

In this context, the OPC technique became apt to find the effective thermal diffusivity of human dental samples. Furthermore, due to thermal diffusivity sensitivity about morphological changes of the materials,¹⁸ this variable in the aging process of human samples was investigated.

II. EXPERIMENTAL SETUP

The Open Photoacoustic (PA) Cell consists in a commercial electret's microphone that receives directly on top a flat slab of a solid sample. The resulting pressure fluctuation in the photoacoustic gas chamber can be calculated using the Rosencwaig and Gersho (RG) thermal diffusion model.¹⁹ For an opaque sample, pressure variation $P(\omega)$ in the electret's foil is given by¹²

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$$P(\omega) = \frac{\gamma P_0 I_0 (\alpha_g \alpha_s)^{1/2}}{\ell_g T_0 k_s \omega} \times \frac{\exp \left[j \left(\omega t - \frac{\pi}{2} \right) \right]}{\sinh(\sigma_s \ell_s)}, \quad (1)$$

and it is a function of modulation of light with $\omega = 2\pi f$ and f is the modulation frequency. In Eq. (1), $\gamma = c_p/c_v$ is the specific heat ratio; P_0 and T_0 are the ambient pressure and temperature; I_0 flux of monochromatic light incident; ℓ_i , k_i , and α_i are the thickness, thermal conductivity, and diffusivity, respectively, $i = "g"$ (gas) and $i = "s"$ (sample); the complex diffusion coefficient $\sigma_i = (1+j)\alpha_i$ is frequency dependent; here, α_i is thermal diffusion coefficient. Depending on the linear thermal expansion coefficient and thickness of the sample, a thermoelastic contribution may appear in thermally thick regime ($\ell_s \alpha_s \gg 1$).¹² If this contribution can be separated out at higher frequencies, as is the case with our photoacoustic experiments, it may be theoretically ignored. Then, the OPC amplitude, Eq. (1), is simplified substantially in the thermally thick regime.¹⁷ The resulting expression is

$$P = \left(\frac{A}{f} \right) \times \exp(-b\sqrt{f}). \quad (2)$$

The coefficient A contains the instrumental transfer function and is independent of the samples thickness. So the thermal diffusivity can be calculated from the exponential dependence of the signal that allows one to fit the parameter $b = \sqrt{\pi \ell^2 / \alpha_s}$; and so forth, the thermal diffusivity since thickness is known.

The experimental OPC system for performing the frequency scans of the dental samples is shown in Fig. 1. It consisted of a 12 mW HeNe laser at 632 nm (Coherent) mechanically chopped (SR540, Stanford Research System) and uniformly focused directly the sample. The sample was fixed using a small amount of vacuum grease on top of an electret microphone as shown in Fig. 1. A lockin amplifier (SR530, Stanford Research System) was used to analyze the amplitude of the microphone signal. The electret microphone has a non-flat frequency response from 4–150 Hz. Its frequency response was obtained by running a frequency scan of a 102 μm -thick aluminum (Al) sample. This sample is thermally thin ($\ell_s \alpha_s \ll 1$) up to 10 kHz, and one would expect the dependence of the PA signal on the modulation

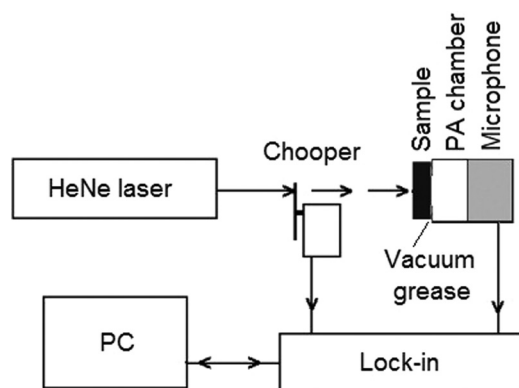


FIG. 1. OPC experimental setup.

frequency to be $f^{-1.5}$.¹² Therefore, the frequency scan allows to get the microphone response function (χ).²⁰

III. MATERIALS

A. Dental enamel

In general, the tooth is composed of three parts: enamel, dentin, and pulp. Dental enamel density decreases from its surface to the area close to the enamel-dentin junction (EDJ). This mature structure is basically composed by inorganic substances, 95% of mineral components, 4% of organic components, and 1% water. The basic structure of enamel is formed by prisms of hydroxyapatite (HAp) crystals that are generally at right angles to the surface. Unlike dentin and bone, enamel does not contain collagen in its composition. Enamel is a nonvital tissue that is incapable of regeneration and it has acquired a complex structural organization and a high degree of mineralization rendered possible by the almost total absence of organic matrix in its mature state. Enamel is translucent, and varies in color from light yellow to gray-white.²¹

B. Dentin

The main structure of the tooth is formed by dentin, which lies just under the enamel. Dentin is a bonelike matrix characterized by multiple closely packed dentinal tubules that traverse its entire thickness. It has an elastic quality that is important to the functioning of the tooth; the elasticity provides flexibility and prevents fracture of the overlying more brittle enamel. Mature dentin has yellowish color and is made up of about 70% inorganic material, 20% organic material, and 10% of water. The inorganic part of dentin consists of substituted hydroxyapatite in the form of small plates. The organic part is about 90% collagen.²¹ An approximate representation of human teeth is given in Fig. 2, where a molar is shown.

C. Sample preparation

Thermal diffusivity measurements of tooth structures were performed on molar and premolar human teeth and aged between 17–61 years. Healthy teeth had been extracted for aesthetic and therapeutic reasons and were sliced to get facets, containing enamel-dentin junction using a low-speed diamond saw. Then, the same saw wore away the concave part of enamel to produce a flat surface. Posterior, the EDJ

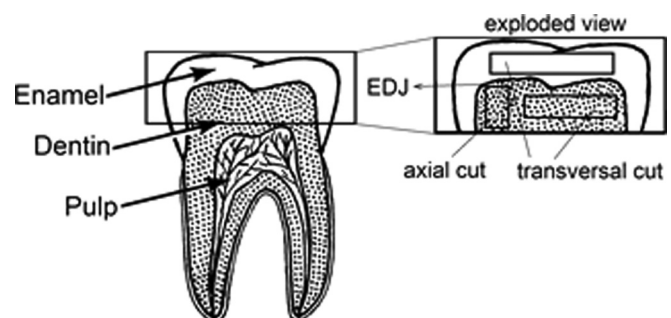


FIG. 2. Diagram of typical teeth showing enamel, dentin, and pulp regions.

was separated by use of a low-speed carborundum saw, to produce small discs about 10.0 mm diameter. To get about 200 μm thickness, a manual polishing of small facets was carried out with sandpaper (ultra-fine 500 grit). The dental enamel was cut in the transversal direction (sketched in Fig. 2). Dentin was cut in the transversal and axial direction (sketched in Fig. 2). Apparently, the dentinal tubules direction does not influence the thermal diffusivity.^{3,22} Finally, the thickness of samples was measured with a digital micrometer (Digimatic Mitutoyo) having an accuracy of 1 μm .

The implicit condition for optical opaqueness required in arriving at Eq. (1) was met by using a circular 20- μm -thick Al-foil (2 mm in diameter), attached to the front side of the sample, also using a thin layer of oil. The thermal thickness of the foil was thus negligible compared to the thicknesses sample.¹⁷ Furthermore, as before described, the aluminum foil is thermally thin for modulation frequencies up to some few kHz ($\alpha_{\text{Al}} = 0.92 \text{ cm}^2 \text{ s}^{-1}$), so that it introduces neither significant attenuation in the PA signal amplitude nor significant delay in its phase.¹³ On the other hand, Balderas-López *et al.*¹⁶ demonstrated that to fulfill the earlier condition, the value range of frequency should be restricted ($f \ll 25 \text{ Hz}$) when a dielectric sample has a metal overlayer of 20 μm thick. Thus, we measured the effective thermal diffusivity of the system 20- μm -thick Al- dental sample.

IV. RESULTS AND DISCUSSION

Open Photoacoustic Cell experiments were performed three times in several runs of 4–60 Hz modulation-frequency range. The photoacoustic signal amplitude was normalized by χ . Figure 3(a) shows typical normalized OPC signal amplitude for the dental enamel sample (age: 17 years), on a logarithmic scale. The log plot shows that the thermal diffusion model is valid in the range of frequencies 4–15 Hz, behaving as a thermally thick sample¹⁹ ($f^{-1.5}$). However, for frequencies greater than 49 Hz, the signal amplitude behaves as f^{-1} , what is expected for high modulation frequencies when the contribution from thermoelastic bending is dominant.^{13,16,17,23} Figure 3(b) is semilogarithmic graph of the corresponding normalized amplitude for the dental enamel sample [same sample of Fig. 3(a)] as function of the square root of modulation frequency. In the 4–15 Hz frequency range, the signal amplitude is dominated by an exponential behavior, $\sim \exp(-bf^{-1/2})$, as predicted by the thermal diffusion model, for a thermally thick sample. Furthermore, in this frequency range, we can consider the behavior of the aluminum foil as thermally thin and the sample as thermally thick.¹⁶ After finding the variable b through fitting the photoacoustic signal, the thermal diffusivity was calculated. Initially, we find the average thermal diffusivity on dental enamel sample (age: 17 years) as being $\bar{\alpha}_{\text{enamel}} \approx (47 \pm 3) \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$. This value is 0.2% and 12% higher than the value reported by Brown⁵ ($46.9 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$) and Braden^{4,24} ($42.0 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$), respectively, using another technique. Although the value found was the effective diffusivity of the system 20- μm -thick Al-dental sample, preliminary simulations, using the two-layer model,¹³ showed that our conclusions can be extended to own dental sample.

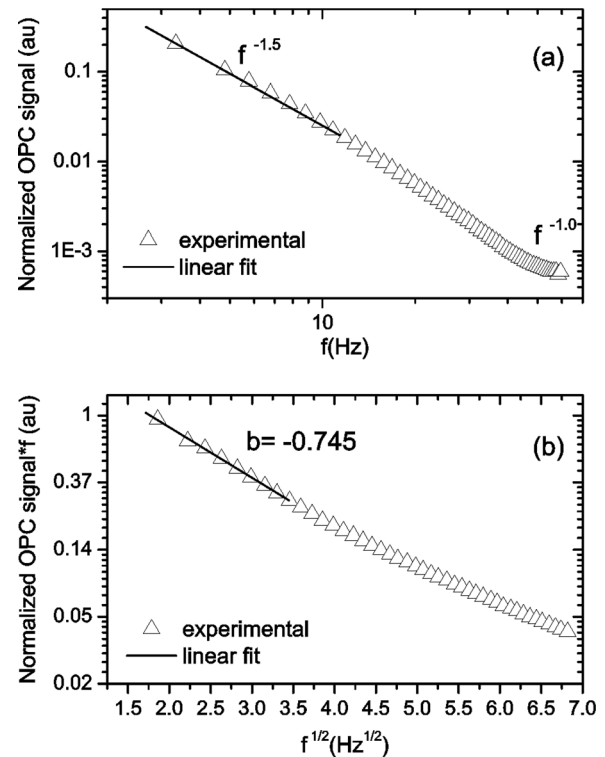


FIG. 3. Frequency scan for the dental enamel (age: 17 years): (a) Typical normalized photoacoustic signal amplitude (S/χ) as a function of the modulation frequency, and (b) Typical linear plot of normalized OPC signal multiplied by f as function of the square root of f .

Then, the values for average thermal diffusivity as age function were determined to enamel and dentin. The signal dependence with frequency presents a peculiar slope for each sample but all were like that shown in Fig. 3. The thickness (l_s), the average slope (b_{av}), and the average thermal diffusivity (α_{av}) as age function of the samples are presented in Table I. The reported standard deviation was calculated as the experimental error on the b value in Eq. (2) by using the standard formula for error propagation.

TABLE I. Effective thermal diffusivity of dental enamel and dentin. b_{av} and α_{av} represent the average value on all the slopes and thermal diffusivities, respectively, measured for each sample.

Age (years)	Thickness (μm)	b_{av} (slope)	α_{av} ($10^{-4} \text{ cm}^2 \text{ s}^{-1}$)
Dental enamel			
17	296	0.76 ± 0.02	47 ± 3
32	284	0.68 ± 0.02	55 ± 2
48	260	0.64 ± 0.02	52 ± 3
55	340	0.90 ± 0.02	45 ± 3
61	238	0.70 ± 0.02	36 ± 3
Dentin			
17	180	0.65 ± 0.02	23 ± 2
17	183	0.69 ± 0.02	22 ± 2
17	191	0.71 ± 0.02	23 ± 2
19	184	0.73 ± 0.02	20 ± 1
32	188	0.73 ± 0.02	21 ± 1
47	217	0.70 ± 0.02	32 ± 2
48	187	0.61 ± 0.02	29 ± 2
55	220	0.74 ± 0.02	29 ± 1
61	209	0.73 ± 0.02	26 ± 1

Fig. 4 shows the thermal diffusivity behavior as age function for the dental samples; enamel, see Fig. 4(a) and dentin, see Fig. 4(b). The thermal diffusivity evolution reveals a non-linear increase and posterior decrease with aging of the samples (61 years measured limit). The values variation found for the enamel and dentin samples were $(36 - 55) \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$ and $(20 - 32) \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$, respectively. Braden^{4,24} found for same occlusal cavity $\alpha_{enamel} \approx 42.0 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$ and $\alpha_{dentin} \approx 26.0 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$, which could suggest the age of 58 years for his sample. On the other hand, due to curve concavity in Figs. 4(a) and 4(b), enamel and dentin should not be used as age determination.²⁵ The literature review about human dentin thermal diffusivity measurement revealed a great variation of the values obtained by several authors.^{3-5,7,26} Panas *et al.*⁷ and Borovskiĭ *et al.*²⁶ found for dentin thermal diffusivity the values 19.2×10^{-4} and $26.5 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$, respectively, using the flash laser method. Magalhães *et al.*³ found for five dentin samples, whose ages ranged from 18 to 26 years, values between 19.9×10^{-4} and $26.5 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$ using the same methodology. These authors³ also measured density, open porosity, and specific heat, and they concluded that the properties range appreciably according to donor and the tooth sampled. They suggested that other factors will also have an influence, such as the individual's age and tooth anatomy. In addition, there is thermal data obtained by other in vitro techniques on natural nanostructured hydroxyapatite extracted from fish bone waste,²⁷ with $\alpha_{HAp} \approx 20 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$, and on bovine bone,²⁸ with $\alpha_{bone} \approx (31 - 54) \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$. The difference noticed here can be attributed to differences in the total mineral content and the compositional features of the coatings and natural bone.

The results in Fig. 4 show that the transient heat transfer occurs much more rapidly in the enamel ($\approx 87\%$) than in the

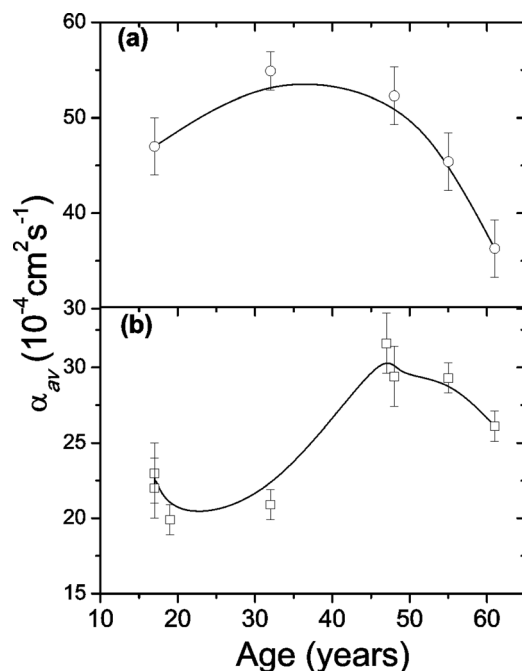


FIG. 4. Average thermal diffusivity as a function of the age: (a) dental enamel and (b) dentin.

dentin. The enamel and dentin are mineralized tissues and contain the same inorganic components, but differ in morphology and organic composition. Perhaps, the thermal diffusivity difference can be explained by the collagen and dentinal tubules presence in the dentin.²¹

Figure 4(a) shows that the thermal diffusivity for the tooth enamel increases smoothly between 17–30 years and then decrease about 23% to 61 years. Dentin presents in Fig. 4(b), a slight decrease of the thermal diffusivity 17 to 32, a sudden increase (33%) to 50 years, and consequent reduction of 19% to 61 years.

The tooth surface has a thin layer of aprismatic enamel whose hydroxyapatite crystals are randomly arranged. This layer is positioned above prismatic enamel, whose HAp crystals are directionally arranged. However, in the elderly tooth, the aprismatic enamel no exists and the prismatic enamel is in constant contact with the fluids of the mouth. Young enamel also received little masticatory load and have no change in the crystal. With age, enamel becomes progressively worn in regions of masticatory attrition. Teeth darken with age. Although darkening could be caused by the organic material addition to enamel from the environment, darkening also may be caused by a deepening of dentin color (the layer becomes thicker with age) seen through the progressively thinning layer of translucent enamel.²¹ The enamel/dentin thickness increases gradually with age, the rate of this increase is large in the younger age groups, decreasing in intensity gradually in younger adults with tendency to stabilization.²⁹ With aging, the enamel permeability decreases. Young enamel behaves as a semipermeable membrane, permitting the slow passage of water and substances of small molecular size through pores between the crystals. With age, the pores diminish as the crystals acquire more ions and as the surface increases in size.²¹ In the recent researches,³⁰ it is found that the mineral density, as well, calcium, and phosphorus weight percent decreases from the outer to the inner enamel layers in the young or the old age groups. Moreover, the differences these properties between the two age groups are significantly different only in the outer enamel layer.

The dentin, like all body tissues, undergoes change with time. During the natural course of aging, there is a gradual reduction of the tubules diameter due to mineral deposition within the lumen interior. When this occurs in several tubules in the same area, the dentin assumes a glassy appearance and becomes translucent (or sclerotic). Sclerotic dentin is found often near the root apex in teeth from middle-aged people. Associated with sclerotic dentin are an increased brittleness and a decreased permeability of the dentin. The continued dentin deposition results in an increase in the degree of mineralization of dentin with age,^{31,32} as well as changes in the mineralized collagen matrix.^{33,34} Due to the consequent changes in the mineral to the collagen ratio, this process may influence the degree of hydrogen bonding that occurs with the loss of water and the shrinkage extent as a result of dehydration. Wang *et al.*³⁵ studied the aging importance to dehydration shrinkage of human dentin and found that the degree of anisotropy in the shrinkage increases from the pulp to the EDJ and is the largest in the young dentin ($23 \leq \text{age} \leq 34$). Another age change was an increase in dead tracts within dentin.

There is no doubt that the age microstructural changes in the dental samples affect the thermal diffusivity. Recalling that the samples in this *in vitro* study were only dental enamel prismatic and all samples were dehydrated.

Sugawara and Yoshizawa³⁶ reported that for porous materials when the thermal conductivity of the solid constituent is larger than of the fluid in the pores, the thermal conductivity of a porous material is decreased with porosity increment. On the other hand, if the thermal conductivity of the solid constituent is smaller than of the fluid, the thermal conductivity of a porous material is increased with porosity increment. In this context, the decrease of the thermal diffusivity with increasing age can be explained by the air presence ($\alpha_{air} \approx 1,900 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$)³⁷ and resulting decrease in pore and/or dentinal tubules. Mainly for dentin, the variation of the results for young people can have to the process of formation and growth (of the proper aging). This is probably due to the real stage of protein consolidation in the collagen. El-Brololossy *et al.*³⁸ observed an increase of the values of thermal diffusivity of remineralized artificially carious enamel and dentin. The largest values were for the samples treated with Zeolite followed by bioactive glass in the form of sealing of enamel pores and plugging of dentinal tubules. In this case, probably the thermal conductivity of the fluid is smaller than the thermal conductivity of the dental samples. So in the present work, it is important to note that the measurements of thermal diffusivity were made *in vitro* and for the measures *in vivo*, the dentinal pores would be filled with dentinal fluid. Therefore, there would be a higher presence of water ($\alpha_{water} \approx 14 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$),³⁷ and consequently greater heat conduction.

The variation in results depending on the age groups show that you should not use samples from different people as single sample measurement as well as average values of the thermophysical properties of human tooth samples. In dental, procedures should be checked the close relationship between the amount of heat used and preserving tooth structure.

V. CONCLUSION

Considering the importance of Biological Studies of human parts including teeth, through the Open Photoacoustic Cell technique, the value for the effective thermal diffusivity of the human tooth hard tissues as age function was measured. The results showed a possible decrease in thermal diffusivity with the age. The values found for the enamel and dentin samples varied from $(36 - 55) \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$ and $(20 - 32) \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$ respectively, which agrees with values found in literature. We believe that the values found here can be very worthy when tabulated and may be not only used for any drugs delivery studies that need to take into account the heat propagation but also to know or calculate the depth profiling of therapeutical substances applied to dental tissues.

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