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The Investigation of Thermoluminescence Properties of Tooth Enamel

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Abstract: In this study; Thermoluminescence(TL) properties of tooth enamel which was obtained from health teeth in the Faculty of Dentistry Ege University was investigated . The tooth enamel was exposed to X-ray and TL spectra were taken with different heating rates (β) (2-10^{o}C/s). It was observed that there is a broad TL peak approximately at 210 ^{o}C for tooth enamel in the range of 50-400 ^{o}C. The dependence of heating rate and exposed X-ray dose (dose response) of the 210 ^{o}C peak were investigated, as well as kinetic parameters were calculated using peak shape and heating rate methods for tooth enamel.

Keywords: Tooth enamel, Thermoluminescence, Kinetic parameters

Diş Minesinin Termolüninesans Özelliklerinin İncelenmesi

Özet: Bu çalışmada, Ege Üniversitesi Diş Hekimliği Fakültesi'nden alınan sağlıklı diş minelerinin termolüminesans (TL) özellikleri incelenmiştir. Diş minesi X ışınları ile uyarılarak farklı ısıtma hızlarında (β) (2-10^{o}C/s) TL spektrumları alınmıştır. 50-400 ^{o}C aralığında diş minesi için yaklaşık 210 ^{o}C' de geniş bir TL piki olduğu gözlenmiştir. 210 ^{o}C' deki pikin ısıtma hızı ve X ışını dozuna bağlılığı (doz yanıt) incelenmiş, aynı zamanda pik şekli ve ısıtma hızı metodları kullanılarak diş minesi için kinetik parametreler hesaplanmıştır.

Anahtar kelimeler: Diş minesi, Termolüminesans, Kinetik parametreler

1. Introduction

According to studies tooth enamel is the hardest part of tooth and protects the dentine. In terms of composition, it consists of 95% inorganic minerals (fluorapatite and hydroxyapatite), 1% organic minerals and 3% water [1-4]. As mention above, the mineral components of enamel are fluorapatite [$Ca_5F(PO_4)_3$] and hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$]. Apatites are a family of compounds characterized by a similar structure, albeit with different compositions. The hydroxyapatite content of mature enamel is 96% by weight and 85% by volume. The rest is protein 1% and water 3%. In the process of enamel development, the protein content decreases from 20% (by weight) at the secretory level down to 1% in the end of the maturation stage [5-6]. For over thirty years tooth enamel has been known as dedector for in vitro dosimetry [7]. Because of this feature tooth enamel is used for age and emergence dose estimation [8-14]. In previous studies, Toktamış et al in study to investigate thermoluminescence properties of fluorapatite mineral in tooth enamel under the

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different annealing temperatures with various annealing times and lanse fluorapatite mineral in tooth enamel exhibits TL properties. Annealing of the sample affects extremely the glow curve and causes a huge enhancement in the sensitization of TL peak[4]. Secu et al in other research the thermoluminescence response to gamma-ray irradiation of tooth enamel is reported. They showed that the TL was attributed to the recombination of CO^{-}_2 radicals incorporated into or attached to the surface of hydroxyapatite crystals. In addition, in this study kinetic parameters obtained after the deconvolution into single peaks of the TL curve recorded following gamma-ray irradiation (200 mGy) and after subtraction of the second TL curve[10].

In this study; we focused on TL dose response, fading and calculation of kinetic parameters for tooth enamel.

2. Experimental

Human health teeth were obtained through Faculty of Dentistry Ege University. Teeth excerpted for medical reasons were used in this study and were provided with no individual indentifying information. Because of requirements of ethic, no information available regarding treat histories of the donors. The health situations of teeth were determinate based on eye inspection. Tooth samples were disinfected in a 5% sodium hydrochloride water solution for 4 hours followed by drying before use. Teeth crowns were separated by cutting 2-2.5 mm sizes with a diamond saw (Dates, HTS-4SM). This saw was replaced with iced distilled water to prevent possible chemical contamination of the sample and to prevent resetting of the luminescence by localized heating [15]. Finally, cleaning of the sample with acetone for 3 minute in a per removed metal restudies from drilling and slicing.

A X-ray source (single phase and water-cooled Matchlett OEG-50A) which delivers about 30 $Gy.min^{-1}$ doses was used to irradiate to sample in a environment that dark and room temperature. All TL measurements were performed with a TL reader system (TLD RA'94) at different heating rates. The sample that exposed to X-ray was read out in an N_2 atmosphere with regard to abstain from undesirable signals.

3. Experimental Results and Discussion

3.1. XRD experiment

The X-ray diffraction (XRD) pattern and peak ID report of tooth enamel were performed by a PANanlytical Empyrean X-ray diffraction equipment and X'Pert High Score Plus program, are shown in figure 1 and table 1. The PANanlytical Empyrean X-ray diffraction equipment is designed to multiple purpose configurations. The X-ray tube is a part of equipment and operates at 10-60 kV rated voltage, 8-60 mA rated current and 6 kW maximum rated output phase control. Fixed divergence slit size in X-ray generator is 0.4785 (^{o}). The result of phase analysis for tooth enamel with X'Pert High Score Plus program was showed that planes of the *Fluorapatite* [$Ca_5F(PO_4)_3$] such as (002), (121), (112) and (310).

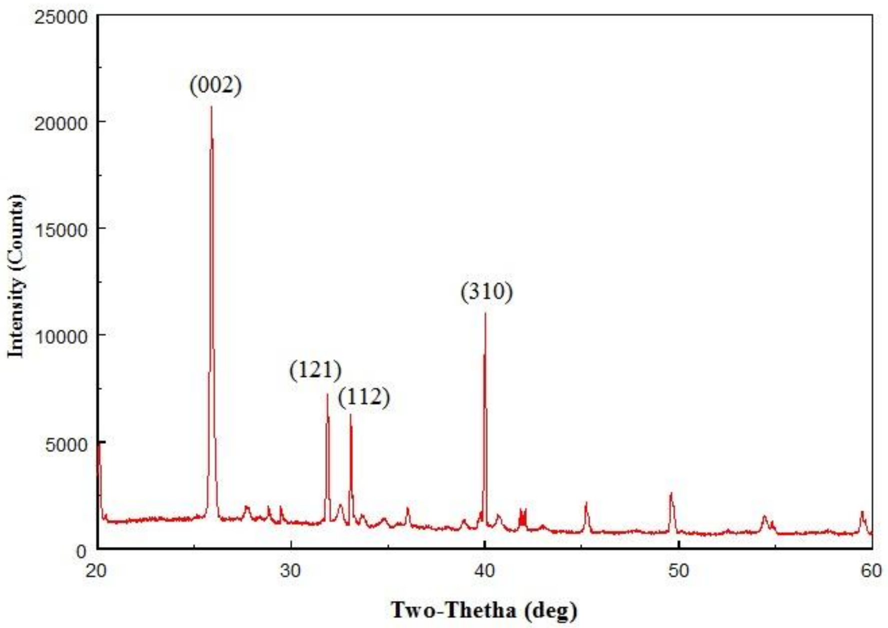

Figure 1. X-ray diffraction (XRD) pattern of tooth enamel.

Table 1. The peak ID report of tooth enamel [16].

2θ	Phase ID	Height (%)	d(Å)	*hkl*
25.864	*Fluorapatite*	56.1	3.4420	(002)
31.927	*Fluorapatite*	100.0	2.8008	(121)
32.250	*Fluorapatite*	59.5	2.7735	(112)
40.043	*Fluorapatite*	16.7	2.2498	(310)

3.2. Dose Response

The aim of this part of study is to investigate the effects of radiation dose on the TL glow curves. Dose response is a specific experiment in the dosimetric studies for getting knowledge about connection between TL intensity and applied dose. Figure 2 (a) shows the change of TL glow curve as a function of irradiated doses 600- 1800 Gy and the variation of area under TL peak as a function doses in Figure 2 (b).

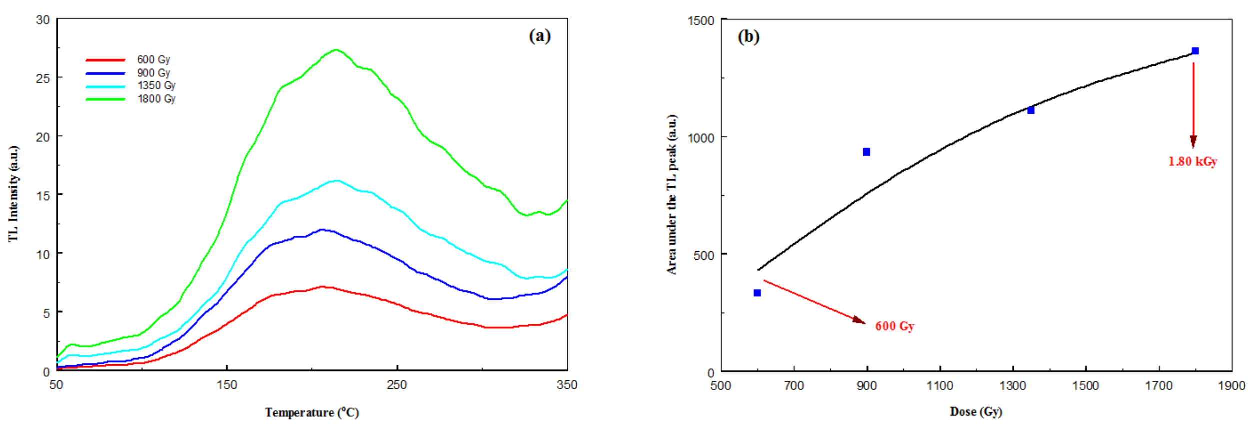

Figure 2. The dose response of the sample for different higher doses (600 Gy to 1.80 kGy)

It is clear that the area under TL peak increases whit increasing irradiated doses; however acceptable linearity in dose response is not observed for the sample. At the same time it was not observed saturation point because of not reached higher doses and x-ray filament heating.

3.3. Fading experiments

Other important point is fading experiment for tooth enamel. In figure 3 the variations of glow curve and normalized area under the glow curve are given. It seen that there is no important change in TL peak temperature and shape of glow curve. Normalized area under the curve and maximum TL intensity fall to 30% of initial values at 32 h (1920 min.) of storage time in dark room. Following 32 h of storage time about same is seen in area under the curve and maximum TL intensity.

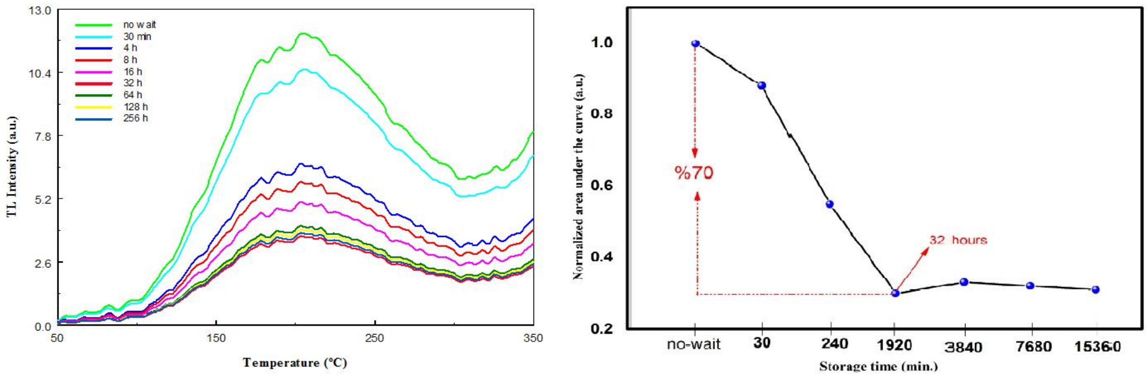

Figure 3. Fading of tooth enamel irradiated with 900 Gy and normalized area under the glow curve as a function of storage time in dark room.

3.4. Determination of kinetic parameters

In order to completely characterize the TL properties, one should have an idea of the kinetic parameters of the glow peak like activation energy (E), the order of kinetics (b) and frequency factor (s) of the traps responsible for TL. These parameters determine the stability of traps. In the general for a better stability of the traps, the activation energy (E) should be high which results in the higher glow peak temperature and hence less fading. It was used peak shape and heating rate methods for determined this parameters for TL peak at 210 ^{o}C of the sample of tooth enamel.

3.4.1. Peak shape method

The shape or geometrical properties of a glow peak depends on the order of kinetics involved in TL process. From the shape of glow peak, one can get information regarding the trap depth, frequency factor and order of kinetics utilizing peak temperature (T_m) and two temperature corresponding to the half the peak intensity on the rising side (T_1) and falling side (T_2) of the glow curve. The total peak width at half the maximum intensity ($\omega = T_2 - T_1$), the lower temperature half with ($\tau = T_m - T_1$) and upper temperature half with ($\delta = T_2 - T_m$) were calculated from which the symmetry factor $\mu = \delta/\omega$ was determined. Using the symmetry factor, the order of kinetics is obtained from Chen's plot [17-18]. The activation energy and frequency factor were determined using the relations formulated by Chen [19] and are applicable for any order of kinetics given by,

$$E_\alpha = c_\alpha\left(\frac{kT_m^2}{\alpha}\right) - b_\alpha(2kT_m) \tag{1}$$

$$s = \frac{\beta E}{kT_M^2} exp\left(\frac{E}{kT_M}\right) \tag{2}$$

where, α stands for δ, τ and ω. Here,

$$c_\tau = 1.510 + 3.0(\mu - 0.42) \quad b_\tau = 1.58 + 4.2(\mu - 0.42) \tag{3}$$

$$c_\delta = 0.976 + 7.3(\mu - 0.42) \quad b_\delta = 0 \tag{4}$$

$$c_\omega = 2.52 + 10.2(\mu - 0.42) \quad b_\omega = 1 \tag{5}$$

Using the above equations the trap depth and frequency factor were determined for different heating rates from 2°C/s to 10 °C/s and the mean values of E_ω, E_δ, E_τ and s_ω, s_δ, s_τ obtained. The average value the trap depth and frequency factor is obtained by this method is 0.496 eV and $1.09\text{x}10^5 s^{-1}$for irradiated 600 Gy tooth sample. The symmetry factor obtained by this method is 0.64 eV corresponding to the order kinetic of 1.75.

3.4.2. Heating rate method

In this method TL glow curves of X-ray irradiated 600 Gy tooth enamel are recorded at different heating rates (2 °C/s, 4 °C/s, 5 °C/s, 6 °C/s, 8 °C/s and 10 °C/s). It has been observed that no important change in the glow curve structure at various heating rates. In spite of the fact that there is no important change in the glow curve structure, glow peaks showed a systematic shift in peak positions with heating rate (Fig. 4). The glow peak position showed a shifting from 135°C to 265°C for the change of heating rate of 2 °C/s to 10 °C/s. The whole glow peak is shifted to higher temperatures as heating rate increases in a manner depending on the half life and time spent at each temperature. Temperature lag is expected in thick samples at higher heating rates. Using condition for maximum TL intensity [20] a graph was plotted between $\ln(T^2{}_m/\beta)$ and $1/kT_m$ (Fig. 4) where β is linear heating rate in °C s^{-1} and T_m is glow peak temperature in °C. From the slope and intercept of the straight line, the activation energy and the frequency factor are determined. The value of the activation energy and the frequency factor determined by this method is 0.154 eV and $4.6\text{x}10^6$ s^{-1}, approximately.

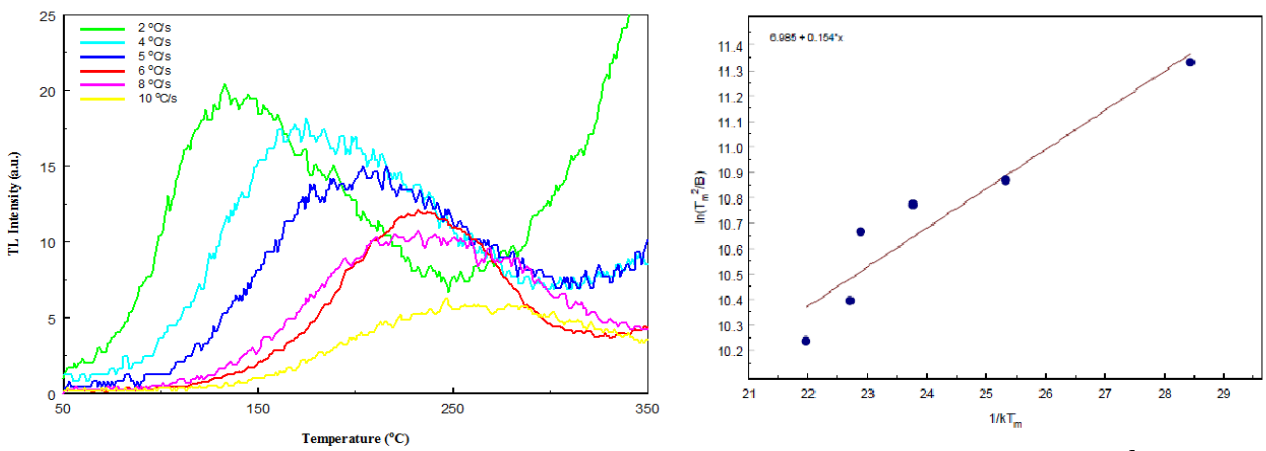

Figure 4. Variation of TL glow peak position for different heating rates and plot of $\ln(T^2{}_m/\beta)$ versus $1/kT_m$ for tooth enamel.

As a result in this study, the properties of TL were investigated and calculated kinetic parameters for tooth enamel. TL dose response of the sample shows that there is not good linearity behavior whereas the low fading rate (after 32 hours). Property of the TL signal shows that the sample is bent of producing high stability free radicals by radiation under room temperature. However, it is necessary to carry out additional research to investigate whether the deproteinization treatment with hydrazine preserves the radiation-induced TL signal [10,21]. In addition, annealing may be performed on the sample. If the important increase in sensitivity obtained with tooth enamel irradiated after deproteinization and annealing treatment is also obtained on tooth enamel irra-diated before deproteinization and annealing treatment, a very significant result has been obtained for the use of TL with tooth enamel in vitro dose restoration studies.

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