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**Thermoluminescence and dosimetric characteristics study
of quartz samples from Seyhan Dam Lake Terraces**

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Abstract

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1. Introduction

The phenomenon of luminescence is related to the interaction of radiation with mineral grains, by the activation of and subsequent trapping of electrons at defects within the mineral lattice[1]. Thermoluminescence (TL), a type of luminescence, is defined as the emission of light from a semiconductor or an insulator when it is heated, because of the previous absorption of energy from irradiation[2]. Many of the natural minerals, especially different quartz minerals, have TL properties and quartz based minerals are popular to study their TL characteristics due to radiation sensitivity, basic structure of glow curve, and simple thermal treatment procedure. TL from quartz is commonly used in retrospective dosimetry, in particular for the dating of archaeological materials and sediments from the Quaternary period[1]. Different quartz minerals may show different TL properties and when the irradiated quartz minerals are heated from room temperature (RT) to a higher temperature, glow

curves from various forms of quartz exhibit great differences in shape[3-5]. Many investigations on quartz have been performed about its different properties [1, 6-11]. Some of the different studies about TL and OSL methods by using quartz samples are presented below:

A number of optical properties of a natural quartz that are related to the presence of electron traps having an optical depth of about 3 eV below the conduction band were described by Huntley and et al. [12], the effects of annealing, heating rate and fading (after annealing at 800°C) on the TL glow curves of natural quartz were investigated by Yüksel et al.[6], the use of TL method to distinguish between burned and unburned soil samples were investigated using quartz samples obtained from soil by Rengers et al. [13], the study of OSL dating of coarse-grain fluvial quartz using single-aliquot protocols on sediments was investigated by Fuchs and Lang [14], and in order to refine the OSL age of the last earthquake on the Dhesir fault in Central Iran, quartz samples were used by Fattahi et al. [15]. The aim of the present study is to characterize the quartz samples obtained from the soils of Seyhan Dam Lake Terraces using different methods. For this purpose, the method of separating quartz from soil, results of XRD, SEM-EDX, luminescence analyses, linear dose response range, minimum detectable dose (MDD) value and kinetic parameters (activation energy, frequency factor and order of kinetic) were presented.

2. Material and Methods

2.1. Sample Preparation

The soil samples were collected from Seyhan Dam Lake terraces in Adana and the sampling point location is 37°04'26.4"N 35°20'43.0"E. The collected soil sample from these terraces is seen in Fig. 1. Collected samples contain a mixture of various materials such as quartz, feldspar, fluorite, organic and carbonate. Mixed soil samples were gently grinded in an agate mortar to obtain grain sizes in the range 90–140 µm. In order to obtain pure quartz from soil samples, the following subsequent chemical processes were applied, respectively.

- i) The grinded mixture was sieved as wet. (It enables simple separation of fractions)

ii) The mixture was etched with hydrochloric acid (HCl, 10%) to eliminate carbonates that emit spurious signal in luminescence (for 1 hour) and then it was washed with distilled water three times.

iii) The mixture was etched with hydrogen peroxide (H_2O_2 , 30%) to eliminate organics (for 24 hours) and then it was washed with distilled water three times.

iv) The mixture was etched with hydrofluoric acid (HF, 38%) to eliminate feldspars and to remove effect of alpha particles contribution to do total dose (for 1 hour) and then it was washed with distilled water three times.

v) The mixture was again etched with HCl (10%) to eliminate fluorites for 1 hour and then it was washed with distilled water three times.

vi) A magnet was used to remove the iron from samples and then the samples were washed with distilled water three times.

vii) The obtained powder samples were dried in the oven at 40°C for 24 hours.

viii) The powder samples were weighed ($m_1 = 10.7$, $m_2 = 10.4$, $m_3 = 10.2$ mg) and three aliquots of each samples were mounted on stainless steel sample cups using silicon oil.

3. Experimental Procedure

The IRSL/OSL ratio test, X-ray diffraction (XRD), scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDX) and thermoluminescence (TL) methods were used for characterization of the obtained quartz samples. The XRD patterns of as-prepared samples were recorded on Rigaku SmartLab X-ray powder diffractometer with $\text{Cu-K}\alpha = 1.5406 \text{ \AA}$ over the range $2\theta = 10\text{--}90^\circ$. The morphological studies of the samples were done by the field-emission scanning electron microscope (Zeiss Supra-55) images. Thermoluminescence (TL) measurements were carried out using an automatic lexsys smart TL/OSL reader system. It has a standard bi-alkaline cathode UV-VIS Hamamatsu photomultiplier tube (PMT) unit (280–650 nm) and attached to filter pack consisting of a Schott-BG 39-glass-3 mm; AHF-BrightLine HC 575/25-Interference-5 mm filter (TL 565 nm). Beta (β)-irradiation was performed using a 1.85 GBq (50 mCi) $^{90}\text{Sr}/^{90}\text{Y}$ β -source (dose rate: 115 mGy/sec) with a maximum energy of 2.2 MeV. TL glow curve readouts were performed on a stainless

steel disc at a linear heating rate of 5°C/s up to 450°C . The background was subtracted from the TL data belonging to each one.

4. Results and Discussion

4.1. IRSL/OSL Ratio Test, XRD and SEM-EDX Analysis

The absence of feldspars in the obtained quartz samples was proven by an IRSL/OSL ratio test. The samples were irradiated beta dose of 1 Gy using $^{90}\text{Sr}/^{90}\text{Y}$ β -source and then first, blue light stimulation was carried out to achieve the measurements for 200 s at room temperature (RT). Secondly, infrared (IR) stimulation also was carried out in a similar way and decay curves of the samples were recorded (Fig. 2a). The IRSL/OSL ratio was calculated as 0.08. Therefore, the samples do not contain feldspars. If quartz stimulates with IR light, it has either a weak or no signal. Fig. 2b represents the XRD patterns of the samples and the XRD patterns of the obtained samples exhibit a set of sharp and well-defined diffraction peaks which are in good agreement with the pure quartz (The International Centre for Diffraction Data, ICDD, PDF Number: 01-085-1054).

Surface morphology, a plot of energy (keV) of the emitted X-ray photons as a function of intensity provides energy dispersive X-ray spectroscopy (EDX) spectra and the elemental composition of the sample are seen in the Fig. 3. The elemental analysis results represent the amount of silicon (Si) and oxygen (O) present in the sample. As seen in Fig. 3, obtained samples consist of 53.21% Si and 46.79% O elements.

4.2. TL Analyses

For TL analyses, quartz samples were irradiated with a β -source ~ 2 Gy. Irradiated quartz samples were heated from RT up to 450°C with a heating rate 1°C/s and then the TL glow curves were plotted (Fig. 4a). As seen in Fig. 4a, quartz samples have three TL glow peaks at 101, 135 and 355°C . Trap depths of the samples were calculated using TAnal software[16] for each peak and the three electron traps at 1.01, 1.04 and 1.62 eV shown in Fig. 5b were confirmed from TL studies.

TL dose response of the quartz samples were measured over the beta (β) dose range from 1 Gy to 10 Gy and TL glow curves were recorded for TL glow peak at 101°C. In order to determine the linear dose range, the heights of the TL glow peaks were used and the linear dose curve was plotted in a log-log scale (Fig. 5). As seen in Fig. 5, TL glow peak at 101°C has a linear dose range between 1 Gy and 5 Gy, and then it has sublinear dose range.

In the TL studies, determining the kinetic parameters, activation energy (E), order of kinetics (b), frequency factor (s), are important to explain the luminescence characteristics of the materials. For this reason, TL kinetic parameters of the quartz samples were determined using computerized glow curve deconvolution (CGCD), peak shape (PS) and isothermal decay (ID) methods for the first peak at 101°C. The samples were irradiated with beta dose of 2 Gy and TL glow curves were recorded from RT to 150°C to determine the TL kinetic parameters. However, TL glow curves were recorded after pre-heating process at 85, 95 and 105°C to determine the TL kinetic parameters for ID method.

TL glow curve of the samples were decomposed using TAnal software[16]. The accuracy of the presented value is supportable with figure of merit (FOM) value and the FOM value is 1.65% for this calculation. The obtained trap parameters were given in Table 1. TL peak at 101°C has a first order kinetic and the determined activation energies and the frequency factors of quartz samples using CGCD, PS and ID methods, are 1.01 ± 0.04 eV and $1.71 \times 10^{13} \text{ s}^{-1}$, 0.98 ± 0.05 eV and $2.50 \times 10^{12} \text{ s}^{-1}$ and 1.11 ± 0.06 eV and $3.90 \times 10^{12} \text{ s}^{-1}$, respectively.

Furthermore, in this study, minimum detectable dose (MDD) value of the quartz samples were also determined for the first TL peak using following equation. In order to calculate the minimum detectable dose three aliquots of the quartz samples were used for background variation measurements and the TL reader was calibrated using the samples irradiated with 2, 3, and 5 Gy beta doses which were the linear dose response range (Fig. 5 and Fig. 6).

$$D_0 = (B^* + 2\sigma_B)F$$

Where B^* is the mean TL background signal obtained from the samples, σ_B is the standard deviation of the mean background and F is the TL system calibration factor[17]. The MDD was found to be as 1.419 ± 0.071 mGy.

5. Conclusion

In this study, the quartz samples were obtained from the soil samples of Seyhan Dam Lake Terraces using different chemical processes. First, the obtained quartz samples were characterized to understand whether they were quartz or not. The IRSL/OSL ratio test, the XRD patterns and SEM-EDX analyses showed that the samples were quartz and it consists of Si (53.21%) and O (46.79%) elements. XRD patterns of the samples are compatible with the ICDD PDF Number: 01-085-1054 (SiO_2). The TL characteristics of the quartz samples have been investigated under β -irradiation. The TL peaks of the samples were observed at around 101, 135, and 355°C and the energy-level diagram of the quartz samples were drawn for the three electron traps at 1.01, 1.04 and 1.62 eV. TL peak at 101°C were studied in details. Linear and sublinear dose response range of the samples were determined as 1-5 Gy and 6-10 Gy for TL peak at 101°C, respectively. CGCD, PS and ID methods were used to determine E, b, and s parameters of the samples. The MDD value of the quartz samples for TL peak at 101°C was calculated and it was found as 1.419 ± 0.071 mGy.

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Figure 1. The collected soil sample of Seyhan Dam Lake terraces.

535x171mm (72 x 72 DPI)

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Figure 2. (a) The IRSL/OSL ratio test and (b) XRD pattern results for the obtained samples.

519x203mm (96 x 96 DPI)

Figure 3. The SEM/EDX analysis results for the obtained samples.

110x84mm (220 x 220 DPI)

Figure 4. (a) TL glow curve and (b) energy-level diagram of the obtained quartz samples.

558x182mm (96 x 96 DPI)

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Figure 5. TL dose response of the obtained quartz samples for TL glow peak at 101°C.

110x77mm (220 x 220 DPI)

Figure 6. TL glow peaks of the quartz samples irradiated with test doses.

283x222mm (96 x 96 DPI)

Tables**Table 1.** TL kinetic parameters obtained by CGCD, PS and ID methods for 2 Gy.

Methods	E (eV)	s (s ⁻¹)	b
CGCD	1.01±0.04	1.71×10 ¹³	1
PS	0.98±0.05	2.50×10 ¹²	1
ID	1.11±0.06	3.90×10 ¹²	1

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