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Dose estimation and dating of pottery from Turkey

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A B S T R A C T

The luminescence method is a widely used technique for environmental dosimetry and dating archaeological, geological materials. In this study, equivalent dose (ED) and annual dose rate (AD) of an archaeological sample were measured. The age of the material was calculated by means of equivalent dose divided by the annual dose rate. The archaeological sample was taken from Antalya, Turkey. Samples were prepared by the fine grain technique and equivalent dose was found using multiple-aliquot-additive-dose (MAAD) and single aliquot regeneration (SAR) techniques. Also the short shine normalization-MAAD and long shine normalization-MAAD were applied and the results of the methods were compared with each other. The optimal preheat temperature was found to be 200 °C for 10 min. The annual doses of concentrations of the major radioactive isotopes were determined using a high-purity germanium detector and a low-level alpha counter. The age of the sample was found to be 510 ± 40 years.

1. Introduction

Luminescence phenomenon can be used to date deposition sediments and archaeological materials such as pottery, brick and ceramic. The luminescence method is a widely used technique for environmental dosimetry (Engin, 2007; Filho et al., 2005; Yoshimura and Yukihara, 2006; Yusoff et al., 2005; Yüce et al., 2010). As far as dating is concerned, the phenomenon of luminescence can be subdivided according to the kind of energy supply during stimulation into thermoluminescence (TL, stimulated by heat) and optical stimulated luminescence (OSL, stimulated by light) (Aitken, 1990; Atlıhan and Meriç, 2008; Huntley et al.,1985; Lang and Wagner, 1996; Martini and Sibilia, 2001; Meriç et al., 2009). Depending on the wavelength of light used for stimulation, luminescence is termed as blue light stimulated luminescence (BLSL), green light stimulated luminescence (GLSL) or infrared stimulated luminescence (IRSL).

Sediments and some archaeological artifacts contain polyminerals composed of crystal structure. When these minerals extracted from a buried material that are subjected to ionizing radiation (alpha, beta and gamma) from radionuclei (i.e. U, Th and K) in the surrounding soil, this natural irradiation causes ionization of valance electrons and creates electron/hole pairs. Then these free electrons and holes are trapped at pre-existing lattice defects within the crystal structure of the mineral. Since these electrons accumulate with time, their amount and thus the intensity of the luminescence signal can be used for dating. When this crystal is subsequently excited by heating or by exposure to light, electrons can be released from the traps and recombine with the holes. The recombination energy is emitted as luminescence (Aitken, 1985).

Luminescence dating requires the measurements of two quantities. These are equivalent dose and annual dose rate. Absorbed dose by the buried material is proportional to luminescence quantity that is proportional to trapped charge concentration; this can be related to the time in which the crystal is subjected to ionizing radiation and called equivalent dose. ED is measured using luminescence techniques. Annual dose rate is due to the natural radioisotope content of the sample, its surronding environment and cosmic rays. The age of the material can be calculated by the equivalent dose divided by the annual dose rate.

Two basic procedures have been developed in evaluation of equivalent dose (paleodose). These are MAAD and SAR procedures (Aitken, 1985; Aitken, 1998). In order to perform MAAD procedure several discs (aliquots) are used while the SAR procedure requires a single disc. In MAAD, each aliquot is different from the others with respect to the amount of the sample it contains. Normalization is necessary to find each disc's average sensitivity. For this, the OSL signal resulting from a short exposure to the stimulating light source is measured for each; to avoid significant age underestimation the exposure must be short enough for the depletion of the signal to

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be not more than 1 or 2%; hence the term short-shine normalization is also used (Aitken, 1998). However statistical errors can occur due to the short time (0.1 s, 0.2 s) of shine in normalization. Therefore, in this study, a different MAAD procedure (long shine normalization) was applied. Also the short shine normalization-MAAD was applied and the results of the short shine normalization-MAAD were compared with the results of the long shine normalization-MAAD method.

In the present work, a pottery sherd that was approximately 20 cm of the depth below the surface was taken from the archaeological site Çanaklık-Side (Antalya/Turkey). Side, which is one of the oldest settlements in Anatolia, was established before 7th century BC. Archaeologists are interested in the age of collected samples.

2. Experimental procedures

2.1. Apparatus

The apparatus used in this study, an Optical Dating System 9010 Reader, was developed by Spooner et al. (1990). Because the technique uses infrared photons (wavelength 880 ± 80 nm) as a stimulation source, the measured signal is called infrared stimulated luminescence (IRSL).

The basic luminescence measurement system incorporates an infrared light-emitting diode (IR-LED) module based on the design described by Spooner et al. (1990). All data were collected using an IRSL add-on unit for the 9010 automated reader, using TEMT 484 IR diodes that run at 40 mA giving a power of about 30 mW/cm^2. Luminescence was detected using a Thorn EMI 9235 QA photomultiplier tube.

Concentration of potassium, uranium and thorium were determined by a high-purity germanium detector (coaxial-type HPGe detector, ORTEC Ltd.). Additionally, uranium and thorium were determined by 7286 low-level alpha counter as they have alpha activity. The type of photomultiplier tube used in the 7286 is an EMI 6097 B.

2.2. Sample preparation for equivalent dose determinations

All operations are carried out in the subdued red light to avoid bleaching effects. The outer surface (3 mm) of the pottery was removed. The outer layer was discarded for following reasons:

– There may be a reduced level of luminescence in the outer surface because of the effect of sunlight.
– Soil contamination must be avoided because of its high level of geological luminescence.

The sample was crushed gently until it was powder. It was washed in 10% HCl and 35% H_2O_2 to remove carbonates and organic materials. Afterwards it was washed in distilled water and then was dried and sieved to obtain the size of fine grain (< 20 μm). Then, the grains were deposited in a thin layer with silicon spray on the aluminium discs (aliquots) of 10 mm diameter and 0.5 mm thickness. These discs were then placed on sample trays of the OSL dating system.

3. Applied methodology

3.1. Preheat procedure

For OSL dating, it is important to select a preheating procedure, which is necessary to eliminate the thermally unstable IRSL components. The preheat procedure of 5 min at 220 °C on the basis of a natural dose/(natural+additive) dose 'plateau' test has been used for the multiple-aliquot dating, as originally developed by Rhodes (1988). The preheat region is determined as the flat region in the preheat temperature zone versus the ratio of natural dose to natural+additive dose count graph.

We then focused on researching whether a flat region as in the curve obtained by the natural dose/(natural+additive) dose technique is also obtainable in the regenerate dose technique.

Fifty-one discs were prepared and divided into 17 groups, i.e., 3 discs for each group.

Nine of these 17 groups were used for creating count–temperature (at constant time) curves and the remaining 8 groups were used for creating count–time (at constant temperature) curves. The steps of the procedure were as follows:

(1) All discs were bleached with sunlight and irradiated to 10 Gy of beta rays and then left for 24 h. These discs were then normalized using a shine of diode light with the power 30 mW/cm^2 for 5 s.
(2) Each of the nine groups (count–temperature at constant time) had been kept in the furnace at different temperatures between 0 and 280 °C at a constant time of 5 min.
(3) Luminescence counts were measured against temperature and plotted. The preheating temperature had been chosen as 200 °C.
(4) To find the duration time of preheating, a procedure similar to the one explained above was followed. The furnace temperature was kept at 200 °C, but each of the 8 groups (count–time at constant temperature) were heated over different time intervals.
(5) Luminescence counts were measured against time and plotted as a function of time.

3.2. Equivalent dose measurements

3.2.1. MAAD calculations

3.2.1.1. MAAD with a short shine (0.2 s) normalization

(1) Thirty discs were prepared and divided into 6 groups, i.e., 5 discs for each group.
(2) The aliquots were normalized (using a short shine of 0.2 s). The time was chosen such that the decrease in luminescence during illumination was very small and could be neglected.

Table 1
Dose rates dating result of the archaeological sample.

	Dose rates (mGy/a)			ACD* (mGy/a)	W**	Annual dose (mGy/a)
	Alpha	Beta	Gamma			
Alpha counting	3.218	2.803	1.863	0.160	0.5 ± 0.032	8.044
Gamma spectrometry	1.785	1.984	1.150			5.079

* Annual cosmic dose.
** Saturation water content.