



## LUMINESCENCE DATING STUDIES OF YEŞİLOVA HOYUK

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**Abstract:** Ceramic findings collected from Yeşilova Hoyuk located in Izmir were dated using the thermoluminescence dating technique. The area is of significant archaeological importance since it is the first prehistoric settlement in Izmir. Recent archeological observations suggest that human occupation of the region took place about 8500 years ago comparing to previously determined dates of 5000 years.

Three samples collected from the same archaeological layer (Neolithic period) in Yeşilova Hoyuk were dated using the thermoluminescence method. Archaeological doses (AD) were obtained by single aliquot regenerative dose method (SAR) for thermoluminescence (TL) using coarse grain quartz minerals extracted from samples. Thick and thin  $Al_2O_3:C$  thermoluminescence dosimeters (TLD) were used to determine the annual dose rate.

The archaeological doses were found to vary from  $25.91 \pm 0.78$  to  $26.82 \pm 0.68$  Gy, and the annual doses were found to be between  $3.34 \pm 0.24$  and  $3.47 \pm 0.24$  mGy/a. The ages obtained for the samples were determined to be  $6000 \pm 830$  BC,  $5740 \pm 670$  BC and  $5460 \pm 740$  years for samples ND1, ND2 and ND3, respectively, which supports the prediction of archeologist that the sampling layer dates from the Neolithic period.

**Keywords:** TL dating, archaeology, Neolithic period, quartz,  $Al_2O_3:C$ .

### 1. INTRODUCTION

Izmir has been a meeting point of Aegean, Mediterranean and Balkan civilizations, as well as domestic cultures of the Southeast Anatolia. Given its strategic location, inter-regional migration, invasion and trade have played an important role in the development of the area during the earliest times. Izmir should not only be seen as a cultural transition zone, but also as a region influenced by very large geographical areas in cultural history. The city is one of the oldest settlements of the Mediterranean basin. The recent discovery (in 2003) of Yesilova Hoyuk,

situated in the plain of Bornova, reset the starting date of the city's history further back than was previously known.

The settlement in Yesilova Hoyuk includes three cultural layers. Starting from the surface soil, these layers were put into groups as follows; the first layer: Early Bronze Age-Roman period, second layer: Chalcolithic period (2 levels) and the third layer: (8 levels) Neolithic Period, from which samples were collected. The Neolithic settlement, at a depth of 4 meters under the surface level, had started in the Neolithic period and reached its zenith towards the end of the Neolithic. The same settlement existed partially through the Chalcolithic period. Accordingly, Yesilova Hoyuk experienced settlement

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spanning at least fifteen hundred years. It was the longest and thickest cultural level in which there were traces of mud and clay deposits due to the flooding from nearby streams (Derin, 2007).

Findings obtained from the settlement area indicate a community which regularly consumed sea-foods such as mussels and snails, and that their activities were, at least partially, also directed towards agricultural production. Numerous bone fragments provide evidence for extensive hunting of animals in the neighborhood, principally wild cattle, pigs and deer, which were consumed as food. The Yesilova Hoyuk had rich water resources and a generous flora, which were highly important conditions for the subsistence of Neolithic people (Derin *et al.*, 2010).

The thermoluminescence dating method has been used to determine the age of Neolithic ceramic samples collected from the Yesilova Hoyuk. The method is the most preferred dating method for fired or heated materials such as ceramic, pottery and bricks. The archeological age is determined from the ratio of the archaeological dose, which is the total accumulated dose following the last heating of the sample, to annual dose originating from the cosmic rays and naturally occurring radioisotopes (K, U and Th) in the surrounding soil and the material itself.

Quartz is the most common mineral in the earth's crust after the feldspar. Nearly 100% of its composition consists of SiO<sub>2</sub> (>97.8 wt% SiO<sub>2</sub>). It is the main component of nearly all volcanic rocks and secondary material in sediments. Thermoluminescence dating of quartz has been extensively used since the early 1970s (Sampson and Fleming, 1972; Aitken, 1985; Hütt *et al.*, 2001; Göksu *et al.*, 2001; Benea *et al.*, 2007; Veronese *et al.*, 2008).

The mineral particle size is of great importance in studies of luminescence dating. There are two different particle sizes used in luminescence dating, namely coarse grains (>90 µm) and fine grains (4-11 µm). The use of coarse grained minerals has some advantages such as the elimination of the effects of alpha particles. In addition coarse grains contain single mineral, which are well understood in terms of their luminescence characteristics. However, many ceramic samples do not have sufficient amount of coarse grained minerals (Barnett, 2000; Feathers, 2003).

Single aliquot regenerative dose method for thermally stimulated luminescence dating was first proposed by Wintle and Huntley (1979) in order to determine the archaeological doses. The application of this method for TL (SAR TL) was proposed by Hong *et al.* (2006). In this method, first, the natural TL signal of the sample is measured. Then the growth curve is obtained by exposing the sample to known regeneration doses with an appropriate radiation source in the laboratory (Hong *et al.*, 2008; Song *et al.*, 2009). Archeological dose is then taken as the value of the dose given to a sample so as to restore the natural TL signal. The advantage of this method is that the natural TL signal can easily be assessed

even for the older samples, in which the natural TL signal is on the corresponding portion of the non-linear growth curve (Liritzis, 2000). The method is reliable since no extrapolation in the growth curve is required to determine the archaeological dose (Duller, 1994; Lepper *et al.*, 2003). Multiple aliquots of the sample may also be used in TL dating; however, many sample discs considered to be identical to each other have to be used, while only one disc is sufficient to determine archaeological dose in single aliquot method (Murray and Wintle, 2000; Murray and Wintle, 2003). In addition, the SAR method does not require any normalization in order to correct mass and sensitivity difference between multiple discs.

In this study, the archeological age of samples collected from the Yesilova Hoyuk in Izmir were determined using coarse grains of quartz mineral extracted from the fired ceramics after applying the SAR TL protocol. This is the first attempt to determine the archeological age of the site using the luminescence dating technique.

## 2. MATERIAL AND METHOD

### Sampling site

Environmental variables were key factors determining the siting and development of settlements in historical times. The favorable climatic and geographical conditions at Yesilova Hoyuk made it a convenient and advantageous location for human habitation compared to other cities. The remains belong to the oldest settlements at the center of Izmir are found in Yesilova Hoyuk (38°26'29''N-27°12'51''E) (Fig. 1).

The plain has rich water sources fed from many streams. Thick alluvial layers have accumulated over time at the site, which are the product of flooding by the streams running over the plain. In a similar way, Yesilova Hoyuk, which is buried by a thick layer of alluvial deposits, is a mound-type settlement. The layer corresponding to the Neolithic period in which the samples were taken is about 3 meters in depth. It is the thickest and longest cultural times in Yesilova.

### Sampling and sample preparation

In thermoluminescence dating, the natural TL signal of the sample should be reset by heating at high temperatures (Aitken, 1985). The ceramic samples were collected from the layer considered to be from the Neolithic period of human occupation as estimated by archaeologist at Yesilova Hoyuk (III. Layer shown in Fig. 2). They are handmade, good quality pottery. The colors of these samples are reddish, revealing that they were well fired. Therefore, the samples used in TL dating were collected from this layer.

Ceramic samples include different kind of crystalline inclusions such as quartz, feldspar and zircon. These minerals behave like a dosimeter for naturally occurring radiation of internal and environmental radioactivity. In

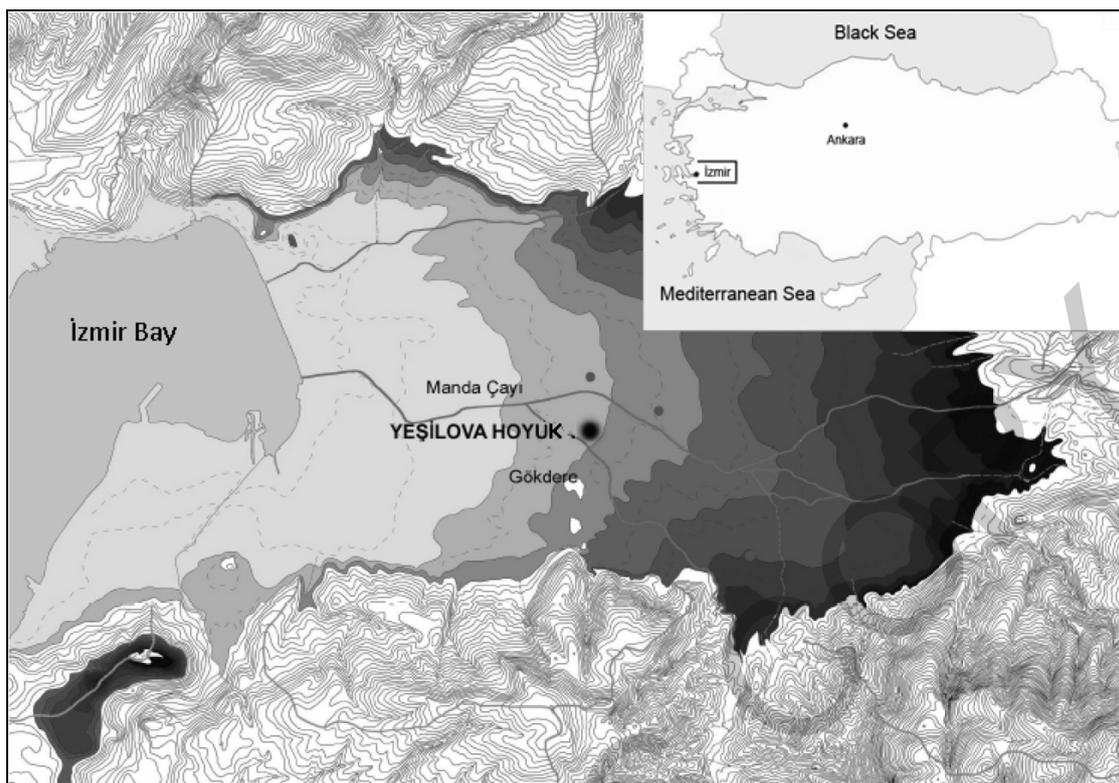


Fig. 1. Location of the Yeşilova Hoyuk, Izmir, Turkey. The map shows the geographic surface pattern of the area.

this study coarse grain quartz minerals extracted from samples, ranging from 140 to 250  $\mu\text{m}$  in size, were used, and all preparation procedure was done under dim red light. During sample preparation, the outer surface of the samples ( $\sim 2$  mm) was firstly removed mechanically to eliminate beta radiation dose contribution from surrounding soil to the sample. The remaining inner part of the samples were gently crushed and sieved. The coarse grains obtained were washed in 10% HCl acid and treated with  $\text{H}_2\text{O}_2$  to remove carbonates and organic materials, respectively. Quartz grains were extracted by density separation using sodium poly-tungstate solutions of 2.60 and 2.75  $\text{g}\cdot\text{cm}^{-3}$ . Then they were etched with 40% HF acid for 40 minutes in order to remove the alpha particles dose contribution, and washed with dilute HCl acid to remove any precipitated fluorides. After each chemical procedure, the extracts were washed with pure water and dried. Finally, samples were wet sieved and dried again. These treatments were applied to eliminate some undesirable materials and to obtain pure quartz minerals. Finally, extracted quartz minerals were placed on stainless steel discs of 10 mm in diameter (Aitken, 1985; Benea *et al.*, 2007; Veronese *et al.*, 2008).

#### Determination of Archaeological Dose

Archaeological dose was determined by single aliquot regeneration (SAR) dose method, which decreases the error arising from sample inhomogeneity among the discs

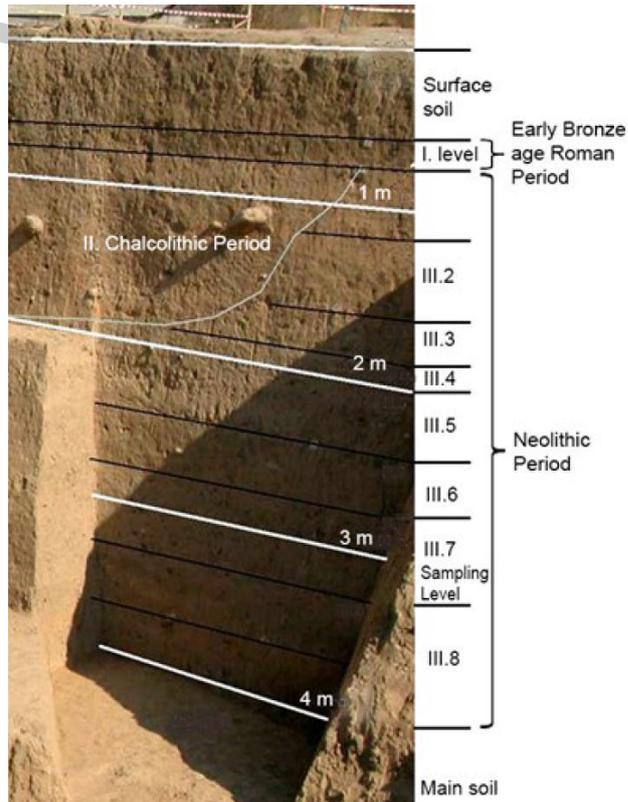


Fig. 2. Horizontal profile of the site in Yeşilova Hoyuk, showing the sampling level.

(Murray and Wintle, 2003; Hong *et al.*, 2006). The dose can be determined with improved precision as each aliquot is self-normalizing. Another important advantage of the method is that the mass of sample required is reduced to a minimum, which may be critical in certain situations. On the other hand, the method has a drawback due to a probable change of the sample sensitivity during irradiation and heating cycles (Hong *et al.*, 2008; Wallinga *et al.*, 2000).

In order to be able to choose a stable temperature interval for the glow curve of the samples the plateau test was performed (Aitken, 1985). For this, the natural TL glow curve is compared with the glow curve after laboratory irradiation. The region in which a plateau is observed is thermally stable since the natural TL signal and signal from laboratory irradiated sample have a constant ratio. The archaeological dose was determined by integrating the plateau region of the corresponding glow curves, and the background signals recorded after each measurement subtracted.

### Determination of Annual Dose Rate

One of the most important processes in luminescence dating is to determine the radiation dose to which the sample has been exposed per year, namely annual dose rate. In coarse grain method, this is the product of three components; i) beta dose contribution from the ceramic samples itself, ii) gamma dose contribution from the associate soil, and iii) some contribution from the cosmic radiation. TLD measurements were used to determine annual dose rate.

In TLD measurements, thin  $\text{Al}_2\text{O}_3:\text{C}$  and thick  $\text{Al}_2\text{O}_3:\text{C}$  TL dosimeters were used to assess beta and gamma – cosmic radiation contribution to annual dose, respectively.

### TLD measurements

The gamma and cosmic radiation dose contribution to annual dose rate was determined using thick  $\text{Al}_2\text{O}_3:\text{C}$  dosimeter. In order to remove any existing charge from traps, dosimeters were first annealed at  $900^\circ\text{C}$  for 15 minutes. Then, they were embedded into a 30 cm deep hole at the sampling location within a plastic tube for 12 months to take into account seasonal variations.

The contribution of beta particles present in quartz crystals to the annual dose was determined using thin  $\text{Al}_2\text{O}_3:\text{C}$  dosimeters (Göksu *et al.*, 1999). The dosimeters were annealed at  $400^\circ\text{C}$  for 30 minutes to remove any existing trapped charges. Two grams of dried archaeological samples were powdered and placed in plastic sample holders with quartz to ensure electronic equilibrium. Since the holders are fully filled by the sample, it can be accepted as beta thick. In order to block the alpha particles from uranium and thorium series the holder was covered with a thin Mylar sheet (density  $1.0\text{ mg/cm}^2$ ) and then the dosimeters were placed on top of the holders.

They were kept in a lead shielded chamber for 2 months in order to protect from background gamma rays. To evaluate the background radiation in the chamber, pure quartz powder sample was also used under the same conditions (Göksu and Schewenk, 2000; Göksu *et al.*, 2001). After two months of storage time, accumulated dose of both thin  $\text{Al}_2\text{O}_3:\text{C}$  dosimeters and pure quartz powder were determined by recording their TL glow curves. Since the TL signals from pure quartz is due to the background beta and gamma radiations in the lead chamber they were subtracted from the accumulated dose of  $\text{Al}_2\text{O}_3:\text{C}$  dosimeters in order to assess the beta dose contribution ( $D_\beta$ ) from the sample. All of these measurements were conducted in  $2\pi$  geometry; therefore, the results were multiplied by a factor of 2 in order to simulate the beta radiation field of the sample (Veronese *et al.*, 2008).

Approximately 6-10  $\mu\text{m}$  of the outer surface of the coarse grain quartz minerals was etched during the chemical procedure (Bell and Zimmermann, 1978; Aitken, 1985). Because of the elimination of the outer surface being exposed to external alpha particles, alpha radiation dose contribution to the annual dose was neglected.

### Water content in the samples

The water content of a sample is an important parameter because of water dose absorption. Water in archaeological samples absorbs some of the radiation that prevents part of the radiation energy reaching to quartz grains. For this reason dose rate in dry sample may be higher than the humid sample. This may cause underestimation of age. Therefore, the percentage of water in the samples ( $W\%$ ) was determined in the laboratory. Eventually, in order to obtain the exact dose rate of beta and gamma radiation to the sample, the measured values were corrected by multiplying with convenient correction factors (Aitken, 1985). Therefore the measured value of  $D_\beta$  and  $D_\gamma$  dose must be multiplied by appropriate correction factor so that the correct beta dose rate absorbed by the quartz grains is obtained. On the other hand, measured value of  $D_{\gamma+c}$  doses do not require any correction since the dosimeters were buried to sampling point in which they were exposed to the all seasonal variations.

### TL Measurements

All TL curves were recorded using a Harshaw TLD 3500 reader in nitrogen atmosphere at a heating rate of  $2^\circ\text{C}\cdot\text{s}^{-1}$ . Background measurements were conducted after each measurement and subtracted from individual TL measurements. In order to protect the photomultiplier tube from blackbody radiation, a 3 mm heat absorbent filter (Schott KG-1) has been placed in front of the tube. Irradiations were carried out using  $^{90}\text{Sr}/^{90}\text{Y}$  beta source. The equivalent dose rates being exposed to samples were calibrated using  $\text{Al}_2\text{O}_3:\text{C}$  dosimeters and pure quartz samples irradiated at Çekmece Nuclear Research and

Education Center, Secondary Standard Dosimetry Laboratory. Thus, it was determined that the beta source at the laboratory delivered an equivalent dose rate of  $3.10 \pm 0.15$ ,  $5.10 \pm 0.16$  and  $5.30 \pm 0.60$   $\text{mGy} \cdot \text{s}^{-1}$  for thick, thin  $\text{Al}_2\text{O}_3:\text{C}$  dosimeters and quartz, respectively.

### 3. RESULTS AND DISCUSSION

The archaeological dose was determined by using the single aliquot regeneration dose procedure for TL measurements (Hong *et al.* 2006). In this study three samples collected from the same archaeological layer of the site were used, and five aliquots were prepared for each sample in order to reduce the statistical error.

Firstly, the natural TL glow curves were recorded and compared with the glow curves obtained after laboratory irradiations of the samples. The temperature region in which a plateau is observed is thermally stable since the natural TL signal and signal from laboratory irradiated sample have a constant ratio. Therefore, the temperature region from 275 to 300°C was considered as a plateau region in this study (Fig. 3).

Later, dose response experiments were conducted for each sample, obtaining a linear response from 2.4 to 38.4 Gy. For a double check, we have also conducted given to measured dose experiments, and observed a unity, revealing that there were no sensitivity changes due to preheating and irradiation cycle. In order to determine the archeological dose, TL experiments were conducted on five aliquots prepared from each sample. A 220°C preheat temperature was applied to the samples in order to suppress the unstable low temperature TL signals. The natural TL signals of quartz minerals extracted from the samples were recorded. In order to determine the equivalent

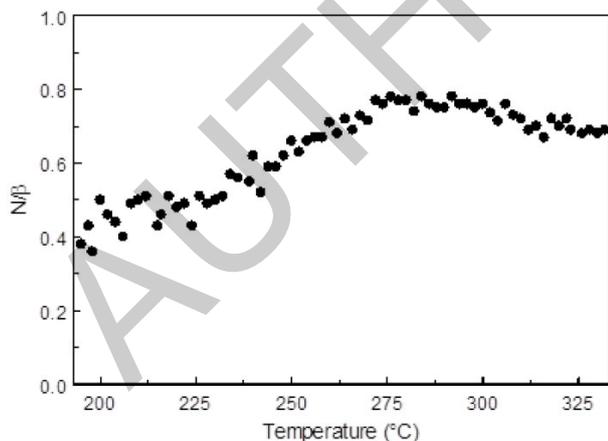


Fig. 3. A plot of the plateau curve as obtained by comparing the shape of the natural glow curve with the shape of the regenerated one.

lent dose of these signals, artificially induced TL signals were recorded after the samples were irradiated with different laboratory doses. Fig. 4 represents the glow curves recorded for natural and regenerated TL. Later, the TL signals between 275 and 300°C, corresponding to the plateau region, were integrated after subtracting the background signals, collected after each experiment under the same conditions. The integrated TL signals were plotted against regenerative doses in order to produce growth curves for each sample, in which archaeological doses were determined (Fig. 5). The archaeological doses were found to be  $27.70 \pm 1.17$ ,  $27.55 \pm 1.15$  and  $26.50 \pm 1.08$  Gy for ND1, ND2 and ND3 samples, respectively.

Gamma ( $D_\gamma$ ) and cosmic ( $D_c$ ) radiation dose contribution to annual dose were determined to be  $1.67 \pm 0.06$   $\text{mGy/a}$  by using thick  $\text{Al}_2\text{O}_3:\text{C}$  TL dosimeters. Beta dose contribution ( $D_\beta$ ) to annual dose from the samples itself were also determined as  $2.50 \pm 0.54$ ,  $2.62 \pm 0.49$  and  $2.54 \pm 0.49$   $\text{mGy/a}$  by using thin  $\text{Al}_2\text{O}_3:\text{C}$  TL dosimeters for dried ND1, ND2 and ND3 samples, respectively.

The saturation water contents of the samples (W%) were determined to be  $35 \pm 0.42$  for ND1,  $32 \pm 0.38$  for ND2 and  $28 \pm 0.37$  for ND3 samples. The sampling site was exposed to flooding several times from nearby rivers through history (Derin *et al.*, 2010); therefore, the fractional uptake F was considered to be  $0.8 \pm 0.2$  according to these climatic factors of the site (Aitken, 1985). The moisture corrected beta dose rate was calculated as  $1.85 \pm 0.28$ ,  $1.99 \pm 0.24$  and  $2.00 \pm 0.28$   $\text{mG/a}$  using equations given by Adamiec and Aitken (1998) for ND1, ND2 and ND3 samples, respectively.

The annual dose for coarse grain samples in the current study was determined from the following equation (Aitken 1998):

$$\text{Annual Dose} = 0.90D_\beta + D_\gamma + D_c \quad (3.1)$$

and they were determined to be  $3.34 \pm 0.24$  for ND1,  $3.46 \pm 0.21$  for ND2 and  $3.47 \pm 0.24$   $\text{mGy/a}$ . Using the age equation;

$$\text{Age} = \text{Archaeological Dose} / \text{Annual Dose} \quad (3.2)$$

the ages of the samples were calculated to be  $6000 \pm 830$ ,  $5740 \pm 670$  and  $5460 \pm 740$  yr BC for ND1, ND2 and ND3, respectively. Uncertainties obtained are in good agreement with literature (Hütt *et al.*, 2001; Benea *et al.* 2007; Veronese *et al.* 2008, Meriç *et al.* 2009). The obtained results were summarized in Table 1.

The burnt wooden samples obtained from the same Neolithic level of Yeşilova were dated using radiocarbon dating method by Dr. Bernhard Weninger at the University of Köln. The date of the sample was determined to be  $7505 \pm 37$   $^{14}\text{C}$  BP (6440-6360 (63.9%) and 6290-6270 (4.3%) cal BC). (Derin *et al.*, 2010).

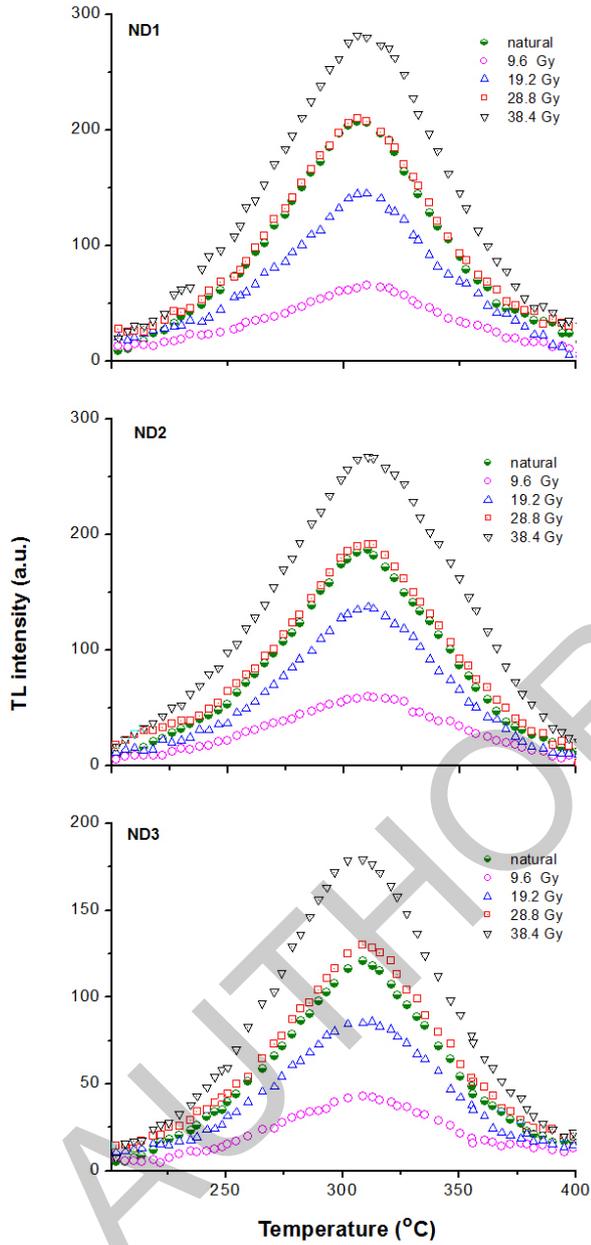


Fig. 4. Natural and regenerated TL glow curves of quartz grains ex-

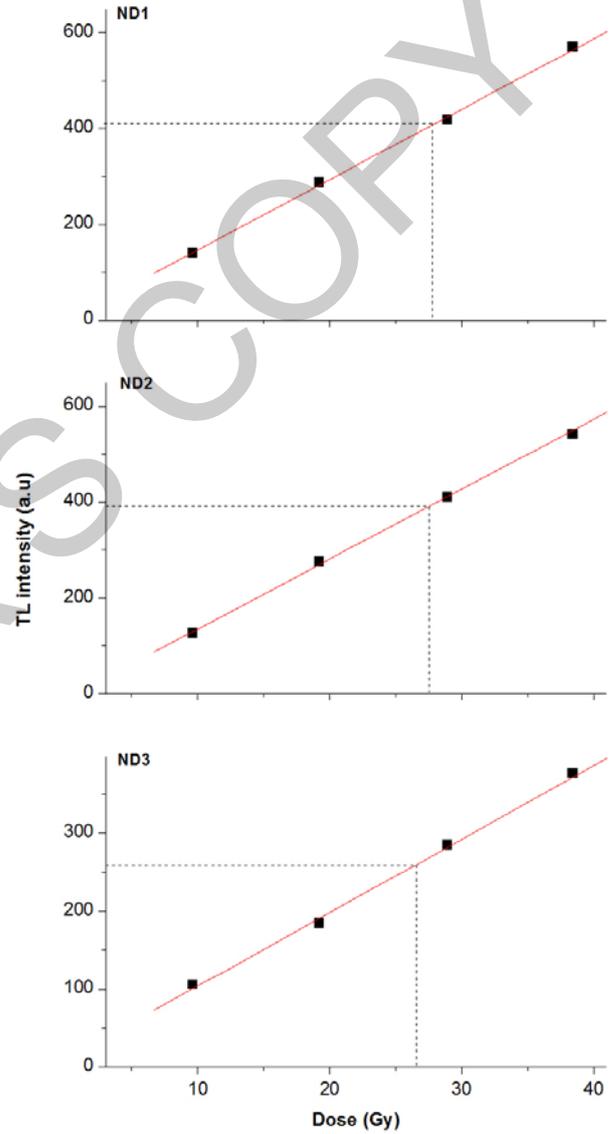


Fig. 5. Growth curves used in determining the archaeological dose of the quartz samples using SAR TL technique.

Table 1. Results of calculated archaeological dose, annual dose rates and ages.

| Sample ID | $D_{\beta}$<br>(mGy/a) | $D_{\gamma+c}$<br>(mGy/a) | Total Annual Dose<br>(mGy/a) | Archaeological<br>Dose (Gy) | Age<br>(yr BC) |
|-----------|------------------------|---------------------------|------------------------------|-----------------------------|----------------|
| ND1       | $1.85 \pm 0.28$        | $1.67 \pm 0.06$           | $3.34 \pm 0.24$              | $26.77 \pm 0.84$            | $6000 \pm 830$ |
| ND2       | $1.99 \pm 0.24$        | $1.67 \pm 0.06$           | $3.46 \pm 0.21$              | $26.82 \pm 0.68$            | $5740 \pm 670$ |
| ND3       | $2.00 \pm 0.28$        | $1.67 \pm 0.06$           | $3.47 \pm 0.24$              | $25.91 \pm 0.78$            | $5460 \pm 740$ |

#### 4. CONCLUSION

The historical development of Izmir city has been clarified through the Yeşilova Hoyuk excavations and dating studies. The discovery of the Yeşilova Hoyuk changed the date of first human occupation of the city, which was previously known to be about 5000 BP.

In this study, the archeological samples collected from the Yeşilova Hoyuk, which belong to the Neolithic period, were dated using the thermoluminescence dating technique. Coarse grained quartz minerals extracted from archeological ceramics and the single aliquot regenerative dose method were used to determine archaeological dose. In addition, the annual dose was determined using thin and thick  $Al_2O_3:C$  TL dosimeters.

The main sources of uncertainty, which were calculated using the error propagation rules and the suggestions of Aitken (1985), are the results of the manual irradiator and manual TLD reader. On the other hand, confidence in the TL dates is given by their close correspondence with radiocarbon dates obtained for the same occupation levels. Therefore, by comparing the radiocarbon and TL ages in the study region, it is reasonable to express that there is a good agreement between the obtained ages and also that of the archaeologists' prediction.

To the best of our knowledge, no other luminescence dating study has been reported in this region thus far, although there exist some luminescence dating studies conducted on archeological samples from different archeological sites (Meriç *et al.* 2009; Kıyak *et al.*, 2010). Therefore, the present work was the first application of the thermoluminescence dating technique for the site. It is believed that the results obtained in this study may contribute to the promotion of the luminescence dating technique and supply valuable information for further studies in Yeşilova Hoyuk.

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