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ESR/OSL Dating and Firing Temperature Determination of Archeological Pottery from Kumyer Location in Turkey

Ulku Sayin[1,2] · Shin Toyoda[3] · Ilker Isik[4] · Gamze Ekici[5] · Ulku Rabia Yuce Semiz[6] · Recep Biyik[7] · Ayhan Ozmen[1,2]

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Abstract

Archeological pottery is a good material that carries important data about ancient human life. Electron spin resonance (ESR) and optically stimulated luminescence (OSL) techniques are used for dating of a pottery found at Kumyer archeological site by rescue excavation from Muğla city in Turkey located at the western part of Anatolia. The ages estimated by ESR and by OSL methods are 4750 ± 400 a and 4100 ± 400 a, respectively, beinag consistent with an archeological analogical estimate of 3000–2000 B.C. In case of a broad signal overlapped with Al center, to take the first peak is recommended as the signal intensity of the Al center for ESR dating experiment. Firing temperature of sample is found to be more than 600 °C using the thermal properties of the E_1' center. ESR has an advantage in dating of ancient potteries as the method can confirm that the heating temperature has been high enough to erase the dating signals.

✉ Ulku Sayin
ulkusayin@gmail.com

1 Department of Physics, Selçuk University, Konya, Turkey

2 Selçuk University Advanced Technology Research and Application Center, Konya, Turkey

3 Institute of Paleontology and Geochronology, Okayama University of Science, Ridai, Okayama, Japan

4 Department of Protection and Restoration of Cultural Assets, Selçuk University, Konya, Turkey

5 Vocational School of Health Services Opticianry Programme-KTO Karatay University, Konya, Turkey

6 Turkish Energy, Nuclear and Mineral Research Agency, Sarayköy Nuclear Energy Research Institute, Ankara, Turkey

7 Turkish Energy, Nuclear and Mineral Research Agency, Nuclear Energy Research Institute, Istanbul, Turkey

1 Introduction

Clay is a widely available, easily worked raw material that can turn into potteries by baking at a high temperature. The potteries are important archeological items that provide information about the life styles of ancient people, their culture and their history [1]. Mostly archeologists determine the age of pottery from its morphological characteristics and specificities, but in some cases, due to changing ancient traditional habits or interfering the excavation levels, such relatively determined age may not be sufficient and reliable. Electron spin resonance (ESR) and optically stimulated luminescence (OSL) are those of the techniques for "*absolute*" dating [2–6]. In applying these methods, the dating signals are assumed to be zeroed at the time that the object was manufactured by firing.

Thermoluminescence (TL) technique has been a most widely used dating method of heated objects as pottery [7]. The principles of TL and OSL dating methods are similar [6] and it has been shown in many studies that the use of OSL method in pottery dating gives reliable results [8–11].

As for ESR dating of pottery, Maurer et al. in 1981 [12] first showed that ESR dating of pottery should, in principle, be possible from the results of a simulating experiment. Since then, until a trial study by Bartoll and Ikeya in 1997 [13], there were no dating research activities on applications to archeological pottery; in fact, their study was the first and successful result using the Al center in quartz extracted from a pottery. In the following studies [14, 15], however, a signal called the "E′ center" was used for ESR dating of pottery. Although these studies look successful where the ESR ages are consistent with the OSL ages, the g factor for the "E′ center", g = 2.00035 is not consistent with that of the E_1' center, g = 2.001 [16] in the ESR measurement condition with the relatively larger scan range and with the larger modulation amplitude. Even if they have observed the E_1' center, it is quite probable that the increase of the signal intensity with dose was due to the formation of "counterfeit" signal, as indicated by Toyoda and Schwarcz in 1997 [17].

Actually, the ESR signals used for dating potteries are those in quartz. There has been a long history of dating quartz starting an application to fault gouge [18]. While some studies showed successful results [19, 20] while in some of them, the results are problematic [21]. Recently, several basic studies on dating quartz were performed [22, 23] as well as a new dating protocol is proposed [24]. In such circumstances of researches in ESR dating of quartz, pottery would be another case to check the validity of ESR dating of quartz using artificially heated material.

In the present study, archeological pottery taken from Kumyer Location at the western part of Turkey in Muğla city was dated by ESR and OSL techniques. There are several studies that concluded different cultures lived in Western Anatolia [25–27]. However, the best of our knowledge there is not any investigation about the Kumyer Location. Archaeometric studies on ceramic artifacts belonging to Kumyer Location Necropolis area can solve cultural synchronization and chronological problem which still exist on both sides of Aegean.

The main purpose of this study is how ESR dating of pottery works to obtain the ages for archeological purposes. This work is the second ESR dating study

of ancient pottery using Al center after Bartoll and Ikeya in 1997 [13], and first compared with OSL dates, after confirming using the E_1' center that the firing temperature has been high enough for zeroing the signals.

2 The Sampling Location

Kumyer Location is a Necropolis (Cemetery) area located within the Turkish Coal Enterprises decoupage area near the province of Muğla, Yatağan District, Yeşilbağcılar Resort. In 2009, rescue excavations were carried out at this decoupage area with permission of Turkish Excavations Department of the Ministry. At that year, the location was registered as a protected area and 99 graves with the potteries as gifts and 7 pottery kilns were revealed by rescue excavations [28].

Kumyer Location is an arid area. Latitude, longitude, and altitude of the location are 37°21′18.5" N, 28°03′14.6" E and 450 m, respectively. The investigated pottery shown in Fig. 1 was taken from the 1 m burial depth of the surface.

3 Investigation by ESR

3.1 Sample Preparation for ESR

Pottery sample was carried to the laboratory by avoiding sunlight and humidity. First, collected samples were washed with distilled water and 2 mm surface of the samples was scraped to eliminate the part affected by alpha and beta particles, and one with possible sunlight effect [7]. After scraping, the samples were gently crushed in a mortar, and were sieved to 125–250 μm grain sizes, etched with 36% HCl (overnight) to remove carbonates, washed with distilled water and dried [2]. The samples were etched with 40% HF solution for 40 min to remove feldspars and

Fig. 1 A picture of Kumyer pottery, analyzed in the present study

diffused impurities [13, 29]. After rinsing and drying, magnetic separator was used to eliminate magnetic grains and get pure quartz. The chemical and physical treatments all quartz samples were sieved again to get 75–125 μm grain sizes.

Ten aliquots of 100 mg quartz subsamples were prepared and nine of them were irradiated to doses up to 280 Gy by gamma rays from a ^{60}Co source at Seconder Standard Dosimetry Lab. (SSDL), Turkish Energy, Nuclear and Mineral Research Agency, Nuclear Energy and Research Institute (Istanbul/Turkey). Using the ionization chamber (PTW TM3002, PTW Unidos-Webline T10021), the dose rate in the area where the sample was irradiated was measured as 387 ± 4.25 mGy/min. The ESR signal intensity of the Al hole center was measured for these aliquots.

To measure the intensities of the E_1' center, quartz grains were extracted with the same preparation protocol above from the raw clay material, most probably from which the pottery had been made. An aliquot of pottery quartz irradiated to 200 Gy was heated at 300 °C for 15 min before the measurement for the E_1' center after the measurement for the Al center.

3.2 ESR Measurements

A JEOL JESFa-300 X-band CW-ESR spectrometer at Selçuk University of Turkey was used for 123 K and 300 K temperature ESR measurements. This spectrometer has cylindrical TE_{011} cavity with unloaded Q value 18.000. The scan field can be set 0–1.4 T range. Low-temperature measurements (123–300 K) were done by JEOL ES-DVT4 variable temperature controller. JEOL JES-PX-2300 X-band CW-ESR spectrometer with nitrogen gas flow system, CT-470 at Okayama University of Science in Japan was used for the measurements at 81 K. For recording the spectra of Al center, the following ESR spectrometer parameters were set; microwave power 5 mW, width of the magnetic field ± 10 mT around the center field 322 mT, scanned in 1 min with accumulation of 7 scans, modulation width 0.1 mT, time constant 0.03 s, modulation frequency of 100 kHz. To minimize anisotropic effects, spectra were recorded for five different orientations at 35° intervals and the average value of intensity was used for calculations.

For the room temperature measurements for the E_1' center, ESR spectrometer conditions were center field; 333.23 mT, sweep width; ± 4 mT, modulation amplitude; 0.12 mT, microwave frequency; 9.339 GHz, time constant; 0.03 s, microwave powers 0.1 mW, 0.01 mW and scanned in 1 min with accumulation of 10 scans. The Mn^{2+}/MnO standard sample was used to calibrate the spectrometer sensitivity in both spectrometers.

3.3 ESR Results

3.3.1 The Al Center Signal

By considering the spectra recorded at 81 K; $[AlO_4]^o$ hole center was identified in the natural quartz [13]. ESR spectral pattern of $[AlO_4]^o$ center with the g values of

several peaks given in Fig. 2 shows a complex structure due to the hyperfine splitting, nuclear Zeeman and quadrupole terms [30].

Toyoda and Falgueres in 2003 [31] suggested that for dating quartz samples such as granitic or sedimentary origin, the signal intensity from the first peak (g = 2.0181) to the last peak (g = 1.9928) of Al center is to be used as first proposed by Yokoyama et al. in 1985 [32] while the fifth peak was recommended by Maurer et al. in 1981 [12], also fifth and seventh peak was by Bartoll and Ikeya in 1997 [13]. However, in the present sample, a broad isotropic signal overlapped with Al center, shown in Fig. 3a, was detected at room temperature and at 81 K. The origin of this signal at g = 2.0035 is not known but may possibly be comparable with the organic radical observed in dentin at g = 2.0046 [33] because of the similarity of the g factors. The signal may possibly have been formed by burning of some organic matter in the material of pottery. In the isochronal annealing experiment for the sample with a dose of 100 Gy (Fig. 3b), while the signals of Al center were observable at 180 °C, only a singlet signal was seen after the annealing at 270 °C and 360 °C. As the first peak (g = 2.0181) is not affected by this unknown signal, this first peak of the Al center was used in D_E determination by the additive dose method.

3.3.2 Dose Response and D_E

ESR signal intensity (I) of the first Al peak, used for dose response study, was determined using I = [PH/(Amp*m(g)] equation. Here, PH is the peak height of the considered signal (Fig. 2), Amp is the spectrometer amplitude, m (g) is the sample mass in gram. The dose response of the Al center is shown in Fig. 4a. The errors shown in the figure correspond to the standard deviations of the signal intensities for the five measurements recorded with 35° intervals. The dose response curve of Al center was fitted to a linear function; and the value of equivalent dose, D_E, was calculated using Y2Science ESR Dating Program to be $D_E = 22.5 \pm 1.2$ Gy. As can be seen in

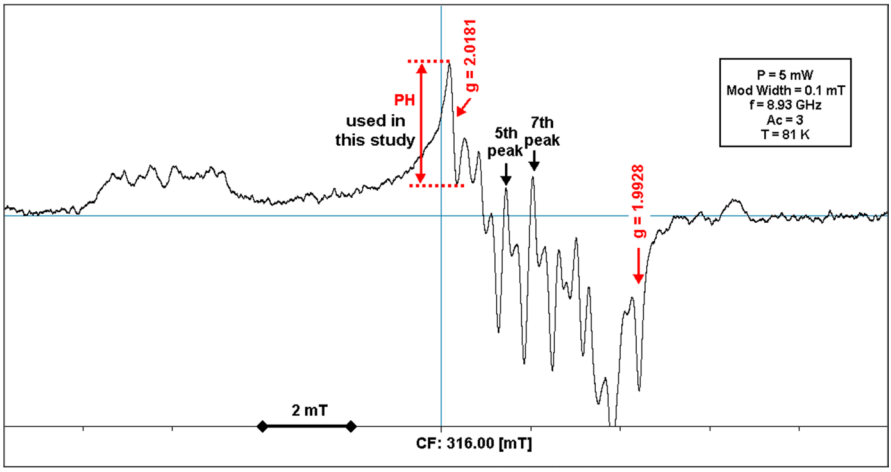

Fig. 2 An ESR spectrum of $[AlO_4]^o$ hole center observed in 280 Gy irradiated quartz

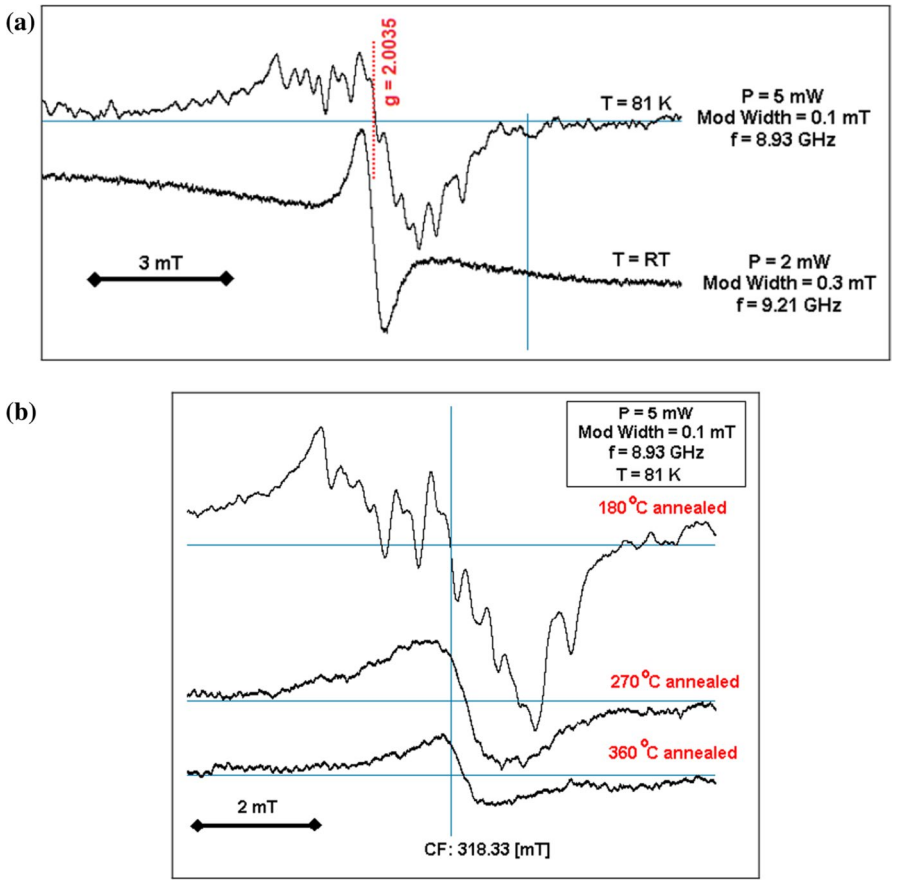

Fig. 3 **a** ESR spectra of non-irradiated quartz samples of pottery at 81 K and room temperatures, **b** ESR spectra of 100 Gy irradiated samples at the 180 °C, 270 °C and 360 °C annealing temperatures

Fig. 4b, the ESR signal intensity of the first peak used in the dose response curve increases significantly as the radiation dose increases.

3.3.3 Determination of Firing Temperature of the Pottery

The E_1' center is an electron trapped at an oxygen vacancy in crystalline quartz [34]. Jani et al. in 1983 [35] reported its precise principal g values as 2.00179, 2.00053 and 2.00030 and proposed that the increase of the signal intensity on heating is due to the transfer of the holes released from $[AlO_4]^o$ to neutral oxygen vacancies with two electrons (Si–Si bond) to form the E_1' center. Toyoda and Ikeya in 1991 [16] found that even after the annealing of the signal of the E_1' center, it recovers after gamma ray irradiation and following heating at 300 °C. Using this feature, they examined the thermal stability of the oxygen vacancies, the precursor of the E_1' center, to find that it decays above 450 °C and almost

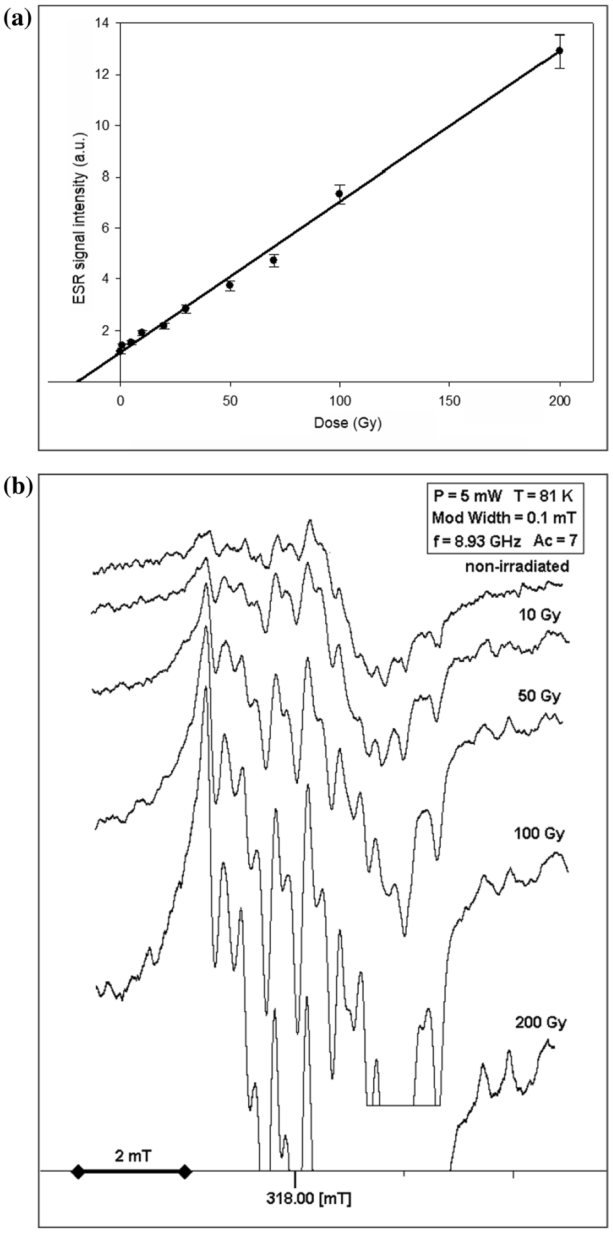

Fig. 4 **a** The dose response of the Al center **b** ESR spectra of non-irradiated and irradiated samples

completely decays at 600 °C. Based on this phenomenon, they lately proposed a protocol to estimate the temperature of lithic heat treatment [36]. This protocol was applied in the present study to investigated Kumyer pottery.

As shown in Fig. 5, an ESR signal of the E_1' center was observed in the raw clay material which most probably the pottery was made of, however, no signals were observed at room temperature in quartz extracted from the pottery. As the E_1' center did not recover by gamma irradiation and following heating, no observable number of oxygen vacancies are present in quartz extracted from pottery, indicating that the quartz in the pottery has been heated at a temperature above 600 °C (to be categorized to "Degree 2–2" according to the flow chart in Fig. 2 of Toyoda and Ikeya in 1993 [36]). As it is well known that any dating signals of ESR or OSL are erased at this temperature range, the result confirms that the ESR and OSL ages for the present pottery sample will not have any age offset.

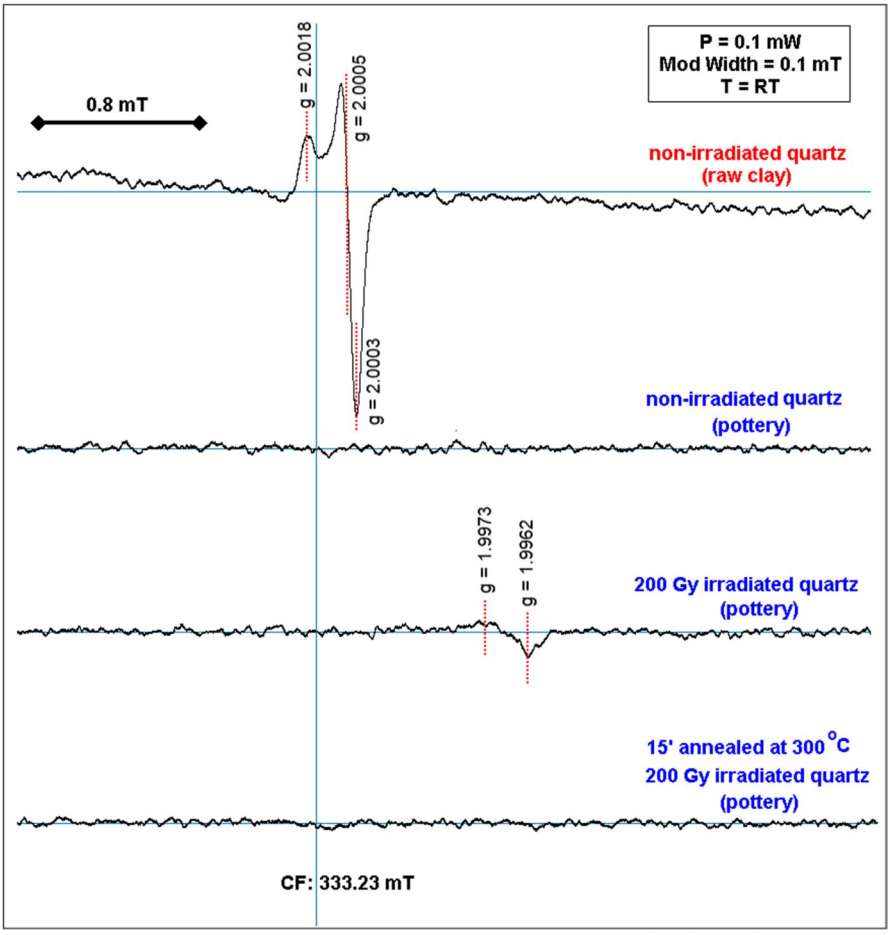

Fig. 5 ESR experimental results of firing temperature determination; the ESR spectra of non-irradiated raw clay quartz, non-irradiated pottery quartz, 200 Gy irradiated pottery quartz and 15' annealed at 300 °C of 200 Gy irradiated quartz

4 Investigation by Optically Stimulated Luminescence (OSL)

4.1 Sample Preparation for OSL

Under controlled light at a wavelength of 590 nm, approximately, 2–3 mm from all surfaces of the potsherd was removed using a utility knife. Samples were prepared by the coarse grains quartz inclusion technique. The sample was gently crushed by hand with an agate mortar and the sand size grains (90–180 μm) extracted by dry sieving. The coarse grains were extracted by routine treatment (10% HCl, 35% H_2O_2, 40% HF; 10% HCl and distilled water). The test sieving was made to select quartz grains 90–180 μm in size. All samples were mounted on stainless-steel discs using silicone-based spray.

The purity of the quartz on six aliquots was monitored by IR check measurement [37]. The ratio between the IR signal and the post-IR blue signal was smaller than 10% for all aliquots.

4.2 The Experimental Equipment

All continuous wave optically stimulated luminescence (CW-OSL) measurements were performed using the standard Riso TL/OSL system (Model TL/OSL-DA-20) installed at the TL/OSL dosimetry laboratory of Sarayköy Nuclear Research and Training Center. The samples were stimulated with blue LEDs (470 nm) at 90% power (36 mW/cm^2). The irradiations were performed using a ^{90}Sr/^{90}Y beta source (40 mCi) with a calibrated dose rate of 0.140 Gy/s. Luminescence was detected with a photomultiplier tube (PMT) bi alkali EMI 9235QA which has an extended UV response with maximum detection efficiency between 300 and 400 nm. Photon detection was measured using a 7.5 mm Hoya U-340 detection filter, which has a peak transmission around 340 nm.

4.3 OSL Results

Equivalent dose was estimated for the sample using an improved single-aliquot regenerative-dose (SAR) protocol (with 260 °C preheat for 10 s, and 220 °C cut heat) [38, 39]. All OSL curves were measured at a stimulation temperature of 125 °C for 5 s. The mean dose recovery ratio (measured/given dose) for six aliquots was 0.99 ± 0.02 giving confidence of the OSL dating test. The luminescence signal was calculated as the first 0.8 s of the resulting decay curve minus the background, which was averaged over the last 4 s.

Typical SAR–OSL growth curve (a) and decay curve (b) for one of the aliquots of the sample are shown in Fig. 6. Since the grow curve was nonlinear, the growth curve was fitted to a single saturation exponential function. Recycling ratio was 0.99 ± 0.01, recuperation point was nearly zero and the growth curves

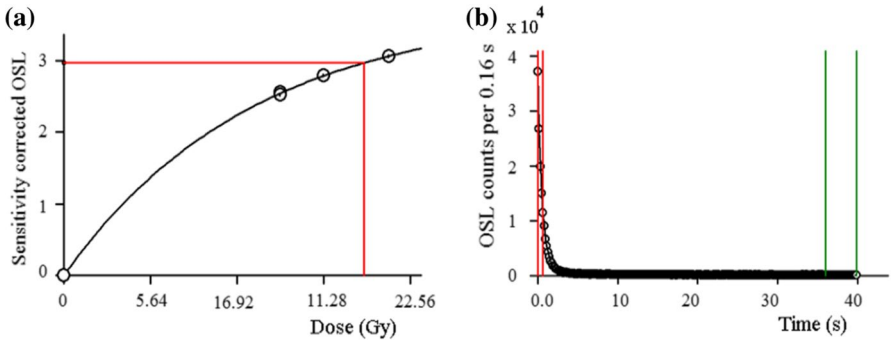

Fig. 6 **a** A typical OSL shine down curve **b** a dose response curve obtained by SAR-OSL of the pottery sample for the determination of equivalent dose

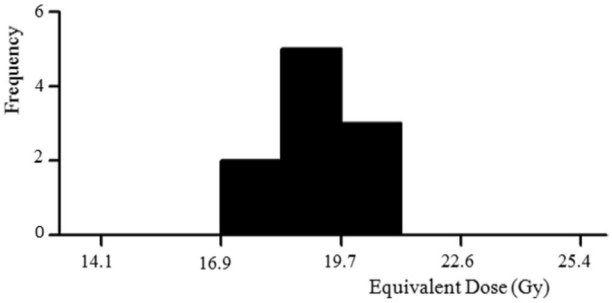

Fig. 7 D_E distribution of pottery sample obtained by OSL

pass remarkably close to the origin for almost all aliquots. These data show that SAR protocol worked correctly in the sample.

Figure 7 presents the representative D_E distribution for one of the aliquots of the sample as histogram plot. D_E distributions in the sample are same in generally and show non-scattering distribution. This indicates proper resetting of the luminescence signal at the time of pottery firing. Average equivalent doses for Kumyer potsherd, based on 11 aliquots, are determined $D_E = 19.2 \pm 1.5$ Gy by OSL technique.

5 Dose Rates

The dose rate was calculated from the concentrations of radioactive elements in the samples, determined by the gamma spectrometry. A Canberra High-Purity Germanium (HPGe) detector installed at the radioactivity measurement laboratory of Sarayköy Nuclear Research and Training Center was used for the measurements for the fired pottery and the surrounding sediment. The obtained elemental concentrations of potassium, uranium, and thorium (Table 1) were converted into dose rates using the conversion factors [40]. The alpha dose rates were neglected as the

Table 1 Radionuclide concentrations and dose rate data for the pottery and surrounding sediment

Sample	Radionuclide concentrations (Bq/kg)			Water content (%)		Grain size (μm)	Dose rate (Gy/ka)			
	U	Th	K				Beta	Gamma	Cosmic	Total
Pottery	72.4 ± 7.3	73.3 ± 7.2	708 ± 82	2.8	For OSL	140	2.88	0.41	0.20	4.68
					For ESR	90	2.95			4.75
Sediment	50.1 ± 5	50.3 ± 4.9	577 ± 78	2.1			–	1.19		

surface of the quartz grains were etched by the HF treatment. The beta ray dose rate was corrected with the attenuation factors for the grain size [41]. The saturation water content of the sample (W) was measured as 28.26% using PresicaXM60 gravimetric moisture analyzer. The fraction for the water uptake during burial (F) was considered as 0.1, thus moisture correction was done using the 2.8% water content value. The cosmic dose rate was calculated [42], considering the site altitude (0.45 km), geomagnetic latitude (37° N), longitude (28° E) and burial depth (1 m) of the sample. The beta dose rate within the pottery was considered as the dose rate given to the quartz grains extracted from the pottery while the contributions from the gamma dose rate from the surrounding sediment was assumed to be 80% and that from the pottery was to be 20% as the thickness of the pottery is 1.3 cm, referring to the contribution values in [43] which was originally given by Mejdahl [44]. The total dose rate values were calculated to be $D = 4.75 \pm 0.36$ (Gy/ka) for ESR and $D = 4.68 \pm 0.35$ (Gy/ka) for OSL. The radionuclide concentrations and dose rate data are shown in Table 1.

6 ESR, OSL Ages and Discussions

By dividing the D_E's by the dose rates, the ages were obtained to be $T_{ESR} = 4750 \pm 400$ a for ESR and 4100 ± 400 a for OSL, in agreement with T_{ESR} within the statistical error. For Kumyer pottery the D_E values, dose rates and ages obtained by ESR and OSL dating techniques were given in Table 2.

Due to the analogical interpretations [27, 45] by considering the form and clay properties of pottery, archeologists were concluded that the age of Kumyer pottery is related to Early Bronze Age (3000–2000 B.C.). Determined ESR and OSL ages are in good agreement with this assessment. Furthermore, other archeological samples revealed by same rescue excavations at 2009 point out the First (3000–2700 B.C.) and Second (2700–2400 B.C.) Period of Early Bronze Age [28]. That is why, although OSL and ESR ages represent the Early Bronze Age, ESR age is thought to be more compatible with archeological estimated age. In addition, dating results of Kumyer are in good agreement with the determined relative and absolute ages of regions close to this location, as an example the ancient pottery belonging to Yarbaşı Location, the nearest area to Kumyer, was investigated by analogical methods and it was determined that the ESR and OSL ages of sample are compatible with Early Bronze Age [46, 47].

Although ESR dating of quartz of tephra has problems [21] with using the Al center, it is well demonstrated that pottery is a good material for ESR dating in the

Table 2 Dating results of Kumyer pottery

Dating technique	D_E (Gy)	Dose rate (Gy/ka)	Age (a)
ESR	22.5 ± 1.2	4.75	4750 ± 400
OSL	19.2 ± 1.5	4.68	4100 ± 400

present paper, being consistent with the OSL age, where the ESR method has an advantage that the signal will also tell whether the pottery has been heated at a temperature high enough for bleaching the signal completely, hence, the offset of the ages.

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