

Optically stimulated luminescence cross-dating from quartz of different grain size

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ABSTRACT

Optically stimulated luminescence (OSL) signals from quartz can be used to determine when a sample was last exposed to sunlight. The energy absorbed since the last resetting (total dose) is measured using OSL and divided by the rate of storage (dose rate) to give the time elapsed from the last heating or daylight exposure. The general equation used to determine the age in luminescence dating is given by the ratio between the Equivalent Dose (ED) and the Annual Dose rate (AD). The ED, measured using luminescence signals, is the total absorbed dose starting from the last "zero event" while the AD, evaluated by in situ and extra situ radioactivity measurements, represents the rate at which energy is absorbed from natural radioactivity present in the sample and in the environment. Evaluation of the environmental dose rate component becomes a crucial point in dating since particular conditions of the site (heterogeneity, lack of structure...) could make age measurements inaccurate. In this work, the conditions to apply the subtraction dating are tested on a sediment of known age; it allows an age determination that is independent on the knowledge of the environmental dose-rate.

Section: RESEARCH PAPER

Keywords: Subtraction dating; dosimetric accuracy tests; partial bleaching test; age equations

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1. INTRODUCTION

Absolute dating by stimulated luminescence techniques is based on the ratio of the absorbed dose (Equivalent dose) since the last zeroing event and the absorbed dose rate (Annual Dose). The Equivalent Dose (ED) is obtained in laboratory from stimulated luminescence measurements with calibrated beta source, while the Annual Dose (AD) is derived from radioactivity measurements related to the U, Th, K content present in the sample and in the surrounding environment [1]. Evaluation of the environmental dose rate component becomes a crucial point in dating since particular conditions of the site (e.g., heterogeneity, lack of structure) could make age measurement complex and sometimes inaccurate [2], [3], [4], [5], [6].

In sediment, gamma attenuation is in a sphere with radius approximately up to ~50 cm and the dosimetric concept of infinite matrix can be used. In an infinite matrix, all of the specific energy emitted in the medium must also be absorbed there, provided that the medium is homogeneous over dimensions

significantly larger than the distance travelled by the relevant radiation. Thus, it is only necessary to find the energy emission rate to know the dose rate. For γ -rays, the infinite matrix is a body of sediment > 0.75 m in diameter.

In cases where the environment is heterogeneous on the scale of the attenuation distance of γ -rays (~30 – 50 cm), the infinite matrix may not be representative of the γ -dose rate. As an alternative, the γ -dose rate may be measured in situ at the sample location using, for instance, Thermoluminescence Dosimeters (TLDs). In practice, TLDs are left buried in the sampling points for 6–12 months, after which the absorbed dose is sufficient to induce a measurable signal [5]. The other common way of determining in situ γ -dose rates involves the use of a field γ -spectrometer, typically with a NaI(Tl) or LaBr probe [7].

Another possible approach, in the case of a heterogeneous environment is the combination of different luminescence dating techniques on the same sample: Fine and Coarse Grain (respectively FG and CG) methods. This approach allows an age

determination that is independent on the knowledge of the environmental dose-rate. The first authors that tested this technique, called subtraction dating, were Fleming and Stoneham (1973) [8] who, applying the thermoluminescence technique, carried out an experimentation on several terracotta stored in museums and on some pottery collected from archaeological sites without associated burial media, with accuracy higher than 12% under favourable conditions. Besides the aforementioned study, there are no available information in the current literature. A condition necessary to apply this technique is the high degree of accuracy of the absorbed dose measurements. This level of precision has been reached in the recent years by using dose quality assurances introduced in the field of dating and based on optically stimulated luminescence [9], [10], [11].

This work aimed to test the conditions for applicability of the subtracting dating method on a sediment of known age [12]. Absorbed dose measurements were obtained by optically stimulated luminescence on pure fine grain and coarse grain quartz; contributions to the annual dose were instead determined from natural radioactive isotopes concentrations through high resolution gamma spectrometry (HPGe) measurements [13].

2. AGE EQUATIONS

The use of different granulometric phases in luminescence dating involves different age equations. The first one describes the fine grain quartz fraction and is defined as:

$$Age(a) = \frac{ED_{FG}(Gy)}{(k \cdot \dot{D}_\alpha + \dot{D}_\beta + \dot{D}_{\gamma+cosm}) \frac{Gy}{a}}, \quad (1)$$

where ED_{FG} is the equivalent dose, k is the alpha efficiency, \dot{D}_α and \dot{D}_β are the annual dose contributions derived from alpha and beta decay of the radioactive contents present in the sample respectively. All these parameters provide the annual dose value of the sample. $\dot{D}_{\gamma+cosm}$ is the annual dose contribution resulting from gamma emissions of the radioactive material present in the environment and the cosmic dose value mainly dependent on the latitude and the depth from the Earth's surface [12].

In the case of Coarse Grain quartz, we have:

$$Age(a) = \frac{ED_{CG}(Gy)}{(f \cdot \dot{D}_\beta + \dot{D}_{\gamma+cosm}) \frac{Gy}{a}}, \quad (2)$$

where f is the attenuation factor which depends on the grain size of the measured fraction [13].

The two equations can also be written in the following way:

$$\left\{ \begin{aligned} Age \cdot (k \cdot \dot{D}_\alpha + \dot{D}_\beta + \dot{D}_{\gamma+cosm}) &= ED_{FG} \\ Age \cdot (f \cdot \dot{D}_\beta + \dot{D}_{\gamma+cosm}) &= ED_{CG} \end{aligned} \right. \quad (3)$$

$$\left\{ \begin{aligned} Age \cdot (k \cdot \dot{D}_\alpha + \dot{D}_\beta + \dot{D}_{\gamma+cosm}) &= ED_{FG} \\ Age \cdot (f \cdot \dot{D}_\beta + \dot{D}_{\gamma+cosm}) &= ED_{CG} \end{aligned} \right. \quad (4)$$

Subtracting member to member we get:

$$Age \cdot [k \cdot \dot{D}_\alpha + (1 - f) \cdot \dot{D}_\beta] = ED_{FG} - ED_{CG} \quad (5)$$

or

$$Age = \frac{ED_{FG} - ED_{CG}}{k \cdot \dot{D}_\alpha + (1 - f) \cdot \dot{D}_\beta}. \quad (6)$$

Equation (6) allows us to obtain the age even without knowing the environmental dose contribution.

All the contributions to the annual dose should be corrected by factors that consider the sample porosity and the average moisture level of the sample during its life.

3. MATERIALS AND METHODS

3.1. Sample preparation

The sample SD was prepared using the Coarse Grain (CG) and Fine Grain (FG) technique to obtain quartz with grain size respectively of $180 \mu\text{m} < \text{Ø} < 212 \mu\text{m}$ and $4 \text{mm} < \text{Ø} < 11 \mu\text{m}$. Organic and carbonate components have been removed by H_2O_2 (10 %, 72 hours) and by HCl (20 %, 2 hours). Quartz, feldspar and other mineral fractions were separated using a high-density liquid solution of sodium polytungstate. To eliminate the contribution of alpha radiation in the CG fraction, the outer layer of the grains was removed by etching with 40 % HF (50 min) followed by etching with 10 % HCl (15 min) to eliminate any fluoride formation.

3.2. Equivalent dose determination

Before the measurements of the equivalent dose on quartz, we carried out some tests to choose the optimal dose range for the regeneration measurements and the suitable pre-heating temperature. The purity of the extracted quartz was evaluated through a feldspar contamination test. The evaluation consists in determining the following parameter:

$$R = \frac{Post_{IRSL}/T_2}{L_1/T_1}, \quad (7)$$

where the numerator is the ratio between the OSL intensity measured after an irradiation and an InfraRed stimulation ($Post_{IRSL}$) and the intensity obtained after the application of a test dose T_2 , while the denominator is the ratio between the OSL intensity stimulated with blue light (L_1) after irradiation and the test dose T_1 . The value of R should be approximately 1 with tolerance of ± 0.10 .

OSL and IRSL signals were obtained by a Risø TL-DA-15 reader equipped with 41 blue LEDs with (470 ± 30) nm and a laser diode with (830 ± 10) nm, respectively. The stimulation units delivered 30mW cm^{-2} for OSL and 240mW cm^{-2} for IRSL at 90 % power, circa. Artificial luminescence signals were induced by ^{90}Sr - ^{90}Y calibrated beta sources integrated in the Risø systems, delivering 4.14Gy/min .

Equivalent dose measurements were carried out by applying Single Aliquot Regeneration (SAR) method on small aliquots [9]. The steps followed to carry out the SAR procedure are shown in Table 1, where the steps 4-8 are repeated for increasing doses: 1 Gy, 3 Gy, 6 Gy, 9 Gy, 12 Gy with a test dose of 1 Gy.

In this procedure, the OSL measurement is preceded by a pre-heating (Ph) in order to eliminate the contribution of shallow traps not useful for dating 27 small aliquots for FG and 20 small aliquots for CG.

3.3. Dosimetric accuracy tests

The applicability of SAR procedure was tested on each aliquot through measurements of the recycling ratio and recuperation [7]. A recycling ratio is derived from a repeated measurement of at least one dose point, to test the accuracy of the sensitivity correction. It is the ratio of the later regenerated value of L/T to the first (L_1/T_1) for $D = D_1$ and should be close to one.

Recuperation is measured by determining L/T for $D = 0 \text{Gy}$. This measurement determines the carry-over of any residual

Table 1. SAR procedure.

Step	Measurement	Observed
1	Dose, 0 Gy	
2	Ph@240°C, 10 sec	
3	OSL@125°C, 40 sec.	L_n
4	Dose, Y Gy	
5	Ph@240°C, 10 sec	
6	OSL@125°C, 40 sec.	T_n
7	Dose, X_i Gy	
8	Ph@240°C, 10 sec	
9	OSL@125°C, 40 sec.	L_i
10	Dose, Y Gy	
11	Ph@240°C, 10 sec	
12	OSL@125°C, 40 sec.	T_i

signal from the previous measurement cycle and should be small compared with L_n/T_n .

From measurements of the recycling ratio and recuperation tests it is possible to evaluate the reproducibility of the sensitivity-corrected luminescence response to doses given in the laboratory.

Although all the tests carried out are of great importance, the dose recovery ratio is the most complete test to evaluate the performance of the SAR procedure for any given sample. This test is carried out to assess if a specific SAR protocol can accurately determine a known dose administered when the sample is in a condition as close as possible to the initial reset before burial. To achieve this goal, any natural trapped charge population in an aliquot is first removed by artificial or daylight exposure without any thermal treatment, and then the aliquot is exposed to a known laboratory dose. Although the given laboratory dose is typically similar to the Equivalent Dose value, young samples often require a larger dose to achieve the needed precision. Thus, the given dose is measured using the chosen SAR protocol through the dose recovery ratio: it is the ratio of the measured dose to the given dose, and it should have a value close to one.

In the present paper, an evaluation of the accuracy of quartz in fine grain and coarse grain granulometries was carried out. The measurement sequence is a SAR applied to all the aliquots that have been irradiated with a known laboratory dose. Details of the used sequence are contained in Table 2. The integral signal of the first 1 s was used as OSL intensity, after subtraction of the background evaluated from the last 4 s of the total stimulation time.

Once the determination of the integration extremes of the signal, the ED value and the corresponding error for each aliquot was derived using the Analyst software. The values obtained, expressed in β s, were converted to Gy. The percentage accuracy was derived by means of the following expression:

$$A = \frac{\text{given_dose} - \text{measured_dose}}{\text{given_dose}} \cdot 100 \% . \quad (8)$$

The corresponding accuracy uncertainties were quantified applying the propagation of the uncertainty formula, considering a 2 % error on the calibration of the radiation source and thus on the given dose.

3.4. Partial bleaching test

One of the criteria needed for successful dating using Optically Stimulated Luminescence (OSL) is a complete resetting

Table 2. Measurement sequence for the dosimetric accuracy.

Step	Measurement
1	Dose, 2 Gy
2	Ph@240°C, 10 sec
3	OSL@125°C, 40 sec.
4	Dose, 0.69 Gy
5	Ph@240°C, 10 sec
6	OSL@125°C, 40 sec.
7	Dose, X_i Gy
8	Ph@240°C, 10 sec
9	OSL@125°C, 40 sec.
10	Dose, 0.69 Gy
11	Ph@240°C, 10 sec
12	OSL@125°C, 40 sec.

to zero, or to a known level, of the latent OSL signal at the time of deposition through exposure of the sample to zero event (sunlight). Incomplete resetting (partial bleaching) at deposition time leaves the material with an unknown residual signal leading to an apparent higher accumulated dose during the burial period and this in turn results in an overestimation of the age [14].

The literature suggests different methods to extract the information on incomplete resetting based on statistical and experimental approaches [15]. The evaluation of the bleaching degree allows the choice of the optimal model for the ED determination: Central Age Model (CAM) or Minimum Age Model (MAM) [16]. The CAM model, is based on the hypothesis that the logs of the individual estimated EDs are described by a normal distribution with a standard deviation that represents the over-dispersion of the data. In the MAM model, it is assumed that the sample is composed of two or more populations of different bleached grains, veering towards a multimodal distribution of ED. ED values result from a truncated log-normal distribution, where the lower truncation point corresponds to the mean logarithmic value of the grains with total bleaching (Minimum Age).

The Quantile-Quantile (Q-Q) plot was used to evaluate the ED distribution [17], i.e., a graphical tool for evaluating whether a set of data plausibly comes from a “normal” distribution. It is a scatter plot obtained by plotting two series of quantiles as a function of the other. Quantiles from a theoretical normal distribution are plotted on the horizontal axis and compared with a set of experimental data on the y axis. If both sets of quantiles come from the same distribution, then “normal”, one should see the experimental points overlapping with the theoretical line.

An experimental test to evaluate the bleaching time of an aliquot consists of exposing it to a light source for different illumination times before carrying out OSL measurements. The test was carried out on both FG and CG aliquots, using the sequence of measurements listed in Table 3. The procedures from the step 5 to the step 8 are repeated for the following illumination time, Δt_i : 10 s, 100 s, 200 s, 300 s, 400 s, 500 s.

The sequence involves irradiation with a β source, a preheating phase (200 °C, 10 s) with the aim of emptying the shallow traps, a blue diode stimulation using 3 % power source to simulate a natural light exposure. A different illumination time was simulated, with values of Δt_i = 10 s, 100 s, 200 s, 300 s, 400 s, 500 s. OSL intensity curves were measured for each Δt illuminance value.

For a quantitative evaluation of the change in signal intensity at different bleaching times, the integral signal of the first 1 s was

Table 3. Measurement sequence for the partial bleaching test.

Step	Measurement
1	OSL@125°C, 200 sec.
2	Dose, 21 Gy
3	Ph@200°C, 10 sec
4	OSL@125°C, 40 sec.
5	Dose, 21 Gy
6	Ph@240°C, 10 sec
7	Blue diode illumination@160°C, Δt_i , 3% power
8	OSL@125°C, 40 sec.

used as OSL intensity, after subtraction of the background calculated from the last 4 s of the total stimulation time. The normalization of the signals allowed the determination of the trend of the intensity variation. In this case, the error was the standard deviation of the normalized for I_{osl} measured for each aliquot.

3.5. Annual dose determination

The contributions to the annual dose from the radioactive chains natural of U and Th, and from the radioactive isotope of K were determined from the concentrations of these elements in the sample obtained through high resolution gamma spectrometry (HPGe) measurements. For conversion from concentration (% or ppm) to dose rate (Gy/ka), the correction factors listed in Table 4 [18] were used.

The contributions to the annual dose were corrected considering the sample humidity degree by means of the W and F factors using following equations:

$$\dot{D}_{\alpha corr} = \frac{\dot{D}_{\alpha}}{1 + 1.50 W F} \quad (9)$$

$$\dot{D}_{\beta corr} = \frac{\dot{D}_{\beta}}{1 + 1.25 W F} \quad (10)$$

$$\dot{D}_{\gamma corr} = \frac{\dot{D}_{\gamma}}{1 + 1.16 W F} \quad (11)$$

where:

$$W = \frac{\text{saturation_wet_weight} - \text{dry_weight}}{\text{dry_weight}} \quad (12)$$

and

$$F = \frac{\text{average_water_content_in_site}}{\text{saturation_water_content}} \quad (13)$$

The W factor was measured in laboratory. An average reference value of 0.5 was considered for the F factor since in the absence of data related to the weight of the extraction, the saturation fraction can only be estimated.

In the case of FG quartz, ^{241}Am calibrated alpha source delivering 2.7 Gy/min was used to determine the luminescence efficiency coefficient k necessary to correct the alpha dose contribution to the annual dose [19].

Table 4. Concentration-dose rate conversion factors [18].

	alpha	beta	gamma
K (1%)		0.7982	0.2491
Th (1 ppm)	0.7375	0.0277	0.0479
U (1ppm)	2.6760	0.1419	0.1096

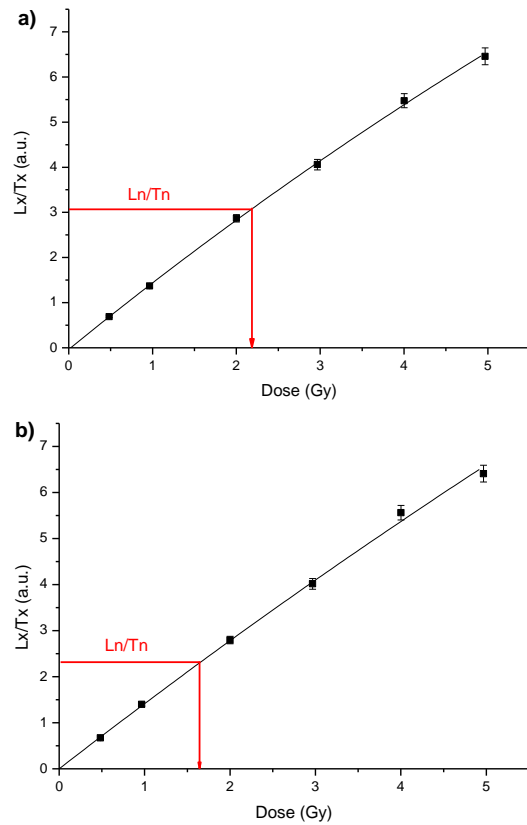


Figure 1. SAR examples obtained by FG technique (a) e CG technique (b).

4. RESULTS

4.1. ED determination

For FG and CG sizes, values of feldspar contamination $R = 0.97 \pm 0.03$ and $R = 0.99 \pm 0.02$ were respectively obtained. Results show an adequate purity of the extracted quartz. An example of SAR for FG and CG is shown in Figure 1. Extrapolation of the L_n/T_n ratio, allows the estimation the equivalent dose (Gy).

In Figure 2, the results in terms of histogram and Kernel Density Estimate (KDE) for the obtained ED values are shown. KDE is a non-parametric way to estimate the probability density function of a random variable. Kernel density estimation is a fundamental data smoothing problem where inferences about the population are made, based on a finite data sample.

The Quantile-Quantile (Q-Q) plot was used to evaluate the ED distribution for each group (CG and FG), Figure 3.

For both FG and CG samples, the experimental points follow the theoretical line; the behaviour suggests that the data are normally distributed. For ED calculation useful for dating, the use of the CAM model is therefore suggested. The results obtained by radial plot with CAM are reported in Figure 4.

ED results, for both fractions, are shown in Table 5.

Table 5. ED values and relative uncertainties obtained for the two measured fractions.

ED_{FG} (Gy)	ED_{CG} (Gy)
2.31 ± 0.03 (1σ)	1.61 ± 0.09 (1σ)

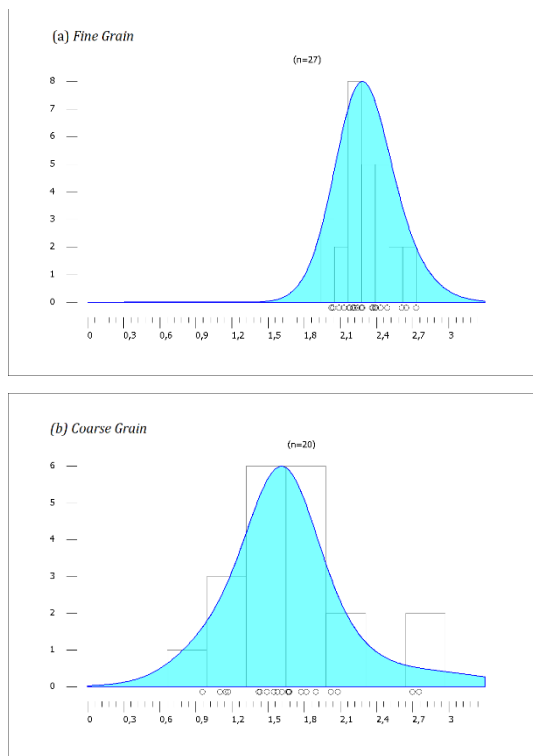


Figure 2. Histogram and KDE for ED values obtained by FG (a) and CG (b) techniques.

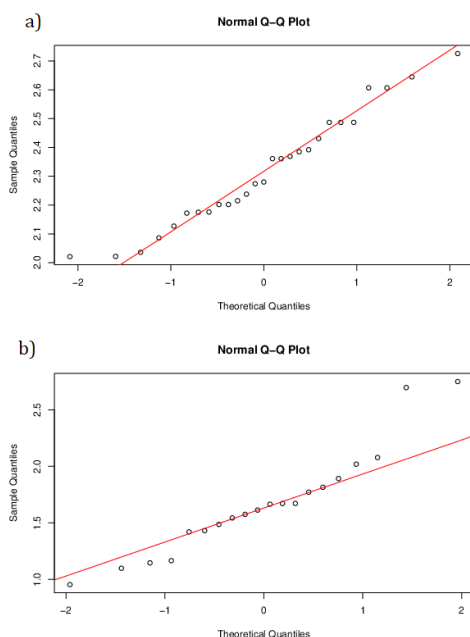


Figure 3. Q-Q plot of FG sample (a) and CG sample (b).

4.2. Dosimetric accuracy test results

As already discussed in the section 3.3 the tests for verifying the appropriate accuracy of the dosimetry data are: Recycling, Recuperation and Recovery tests.

In the case of both fine grain and coarse grain, values of $|\text{Recycling}| \leq 5\%$ and $|\text{Recuperation}| \leq 1\%$ were obtained. The percentage of discrepancy of the dose values obtained for each grain size, 27 aliquots for fine grain and 20 aliquots for coarse grain, against to the known dose value delivered was

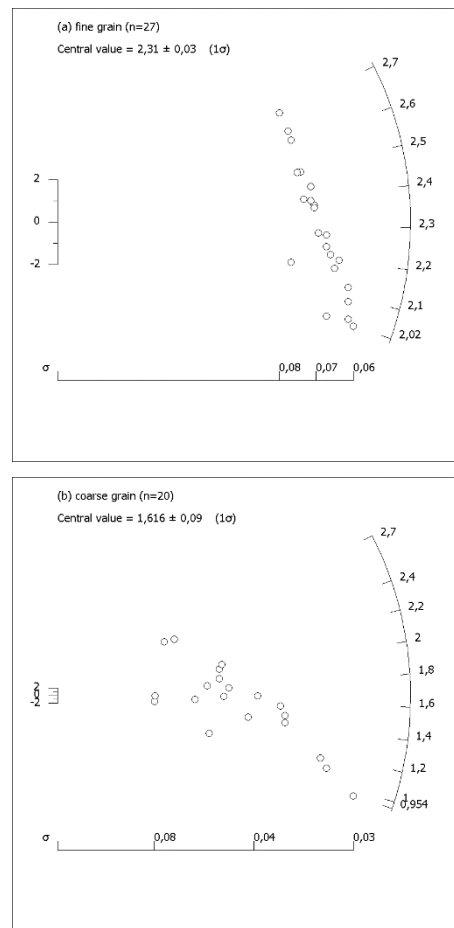


Figure 4. Radial plot with CAM for FG (a) and CG (b) samples.

evaluated. Figure 5 shows the results obtained through the box plot method.

The plots contain two outliers that fall outside the InterQuartile Range (IQR). These points lie outside a value of $\text{IQR} \times 1,5$, so they are outside the expected variation spectrum of the accuracy data.

As can be seen from the graph, the median of the FG data is not in the middle of the box, whereas that of the CG data is in the middle; this analysis suggests that, in the case of the FG, the distribution of accuracy is not symmetrical, whereas it is symmetrical in the case of the CG aliquots. Further evidence of the asymmetry of the data lies in the fact that in the FG the mean is higher than the median, whereas in the case of the CG the median and the mean coincide.

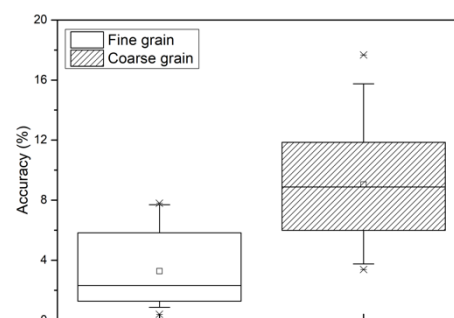


Figure 5. Box plot for FG and CG percentage accuracy values of the aliquots used for measurements.

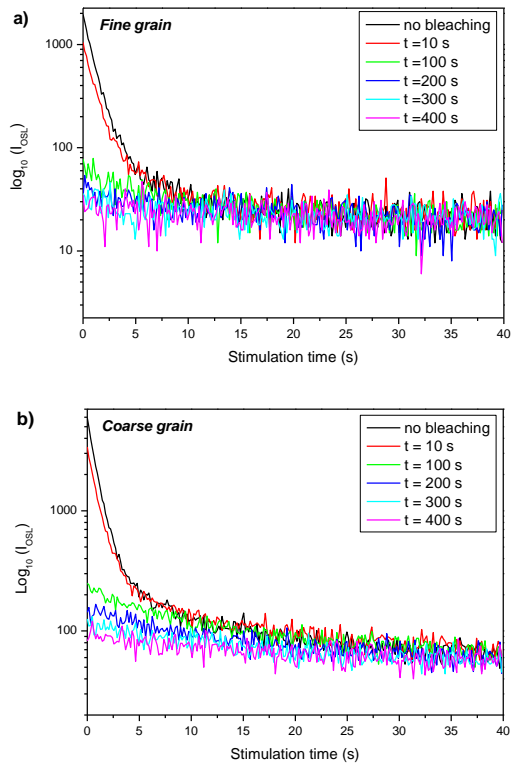


Figure 6. OSL curves for a CG aliquot (a) and an FG aliquot (b) obtained for different illumination time.

4.3. Partial bleaching test results

Figure 6a and Figure 6b contain an example of OSL luminescence curves obtained after different illumination times for a FG aliquot and a CG aliquot.

A time of 400 s is enough to bleach OSL emissions in the case of the CG granulometry. The result obtained in the case of coarse grain is non-trivial, as the total bleaching of the signal only occurs when simulating a 400 s exposure to natural light. It is therefore expected that the grains will be well bleached and negligible charge should remain in the easy-to-bleach OSL traps at the time of laying with a similar solar exposure time.

Figure 7 shows the different OSL intensity trend as a function of different bleaching time for a single aliquot of FG and CG fractions.

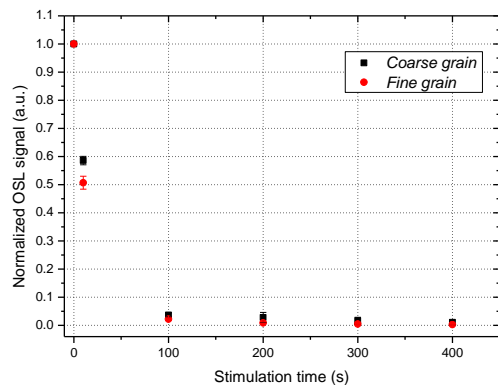


Figure 7. Normalized OSL curves for a CG aliquot and an FG aliquot obtained for different illumination time.

Table 6. Concentration of U, Th and K.

Sample	U (ppm)	Th (ppm)	K (%)
SD	0.23 ± 0.01	1.25 ± 0.04	0.10 ± 0.01

Table 7. Annual dose contributions corrected for humidity.

Sample	$D_{\alpha\text{corr}}$ (Gy/ka)	$D_{\beta\text{corr}}$ (Gy/ka)	$D_{\gamma\text{corr}}$ (Gy/ka)
SD	1.40 ± 0.11	0.13 ± 0.01	0.10 ± 0.01

As it can be seen from Figure 8, OSL intensity decreases in an exponentially decreasing trend with respect to bleaching time for the CG aliquots.

4.4. Dose rate determination

The contributions to the annual dose from the radioactive chains natural U and Th, and from the radioactive isotope of K were determined from the concentrations of these elements in the sample obtained by gamma spectrometry measurements at high resolution (HPGe), Table 6.

The contributions to the annual dose were corrected considering the sample humidity degree by means of the W and F factors. For SD sample $W = 0.13 \pm 0.01$ was obtained. An average reference value equal to 0.5 was used for the F factor.

Table 7 shows the annual dose contributions corrected for humidity.

For FG, from comparison of optically stimulated luminescence response between alpha and beta irradiation, the k value of 0.07 ± 0.01 was obtained. For CG, a mean attenuation factor (f) related to a grain size of 0.90 was used.

4.5. Age determination

Table 8 shows, for each technique, ED value, Annual Dose (AD), and age through the equation (1) for FG, (2) for CG, and (7) for Subtraction method (Sub).

The ages obtained through the three techniques, within the range of experimental errors, are in good accordance with each other, Figure 8.

Table 8. Equivalent Dose (ED), Annual Dose (AD) and Age for each technique.

	ED (Gy)	AD (Gy/ka)	Age (ka)
FG	2.31 ± 0.03	0.33 ± 0.02	6.91 ± 0.42
CG	1.61 ± 0.09	0.22 ± 0.01	7.22 ± 0.58
Sub	0.70 ± 0.09	0.11 ± 0.01	6.31 ± 0.44

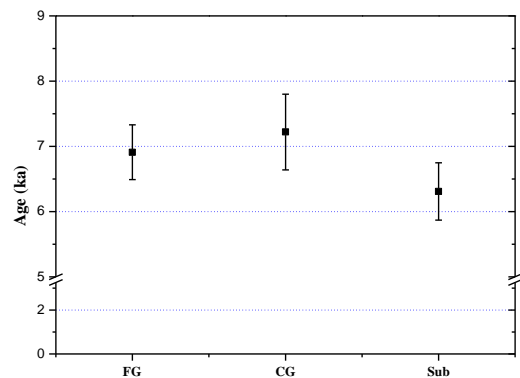


Figure 8. Obtained Age for SD sample by the three different techniques: FG, CG and Subtraction.

5. DISCUSSION AND CONCLUSION

The purpose of this work was to show a method for dating samples from highly heterogeneous environmental contexts and out-of-context samples. The results obtained show that this is possible if equivalent dose measurements are accompanied by appropriate dosimetric control tests. Notably, the purity of the quartz was first tested with a feldspathic contamination test, then the dose test was used to monitor changes in the sensitivity of the quartz during the readout and irradiation phases and the recycle test for the detection of any systematic errors in the ED determination. Finally, the recovery test allows the dosimetric accuracy of the equivalent dose measurement to evaluate. The possibility of applying subtraction dating methods could effectively eliminate the need to know the environmental dose rate, an important factor also in dating objects excavated many years earlier and now in museum collections.

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