Deviations In The Accumulated Dose For Archaeological Dating

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Abstract

The archaeological dating by thermoluminescence (TL) method is obtained through the determination of the accumulative dose and the annual. In this work, an evaluation of two factors which can cause deviations in the accumulative dose in archaeological dating of sediments by TL method was carried out.

Keywords: TL, Deviations, Archaeological Dating

1. Introduction

The thermoluminescence (TL) dating is done measuring the light emitted after heating the material and it will be released only if another exposition to an exciting agent occurs again. The TL intensity of the emitted light depends on the radiation dose and this is the basis of the TL dating [1] [2]. The TL light intensity can be changed due to previous exposure to sunlight or to thermal heating, leading to an accumulative dose lower than expected. [3] [4].

The materials dating is very important in several areas [1],[2]. However, the results sometimes cause doubts about the reliability of TL dating technique. Due to this, some dating tests with different techniques have been performed and therefore, it can be seen with deviations of ages [5].

Some recent techniques such as single aliquot regenerative dose (SAR) minimize the effect of inaccurate dose determination, [6], but the effects on treatment of statistical data are not minimized. In the SAR protocol proposed by Murray & Wintle, it is demonstrated that using techniques of dating single grains with successive replicates on the same sample can diminish the effect of interference of the environment - as an undue exposure to light; for instance- and then, increase the reproducibility of the broadcast signal optically stimulated light. The effects of ultraviolet light on a sample of dating are a well known process. However, the study of the replicates of the samples for dating by thermoluminescence technique has not yet been fully explained.

It is important to remember that the results provided by any person should be acceptable to all potential users, requiring a quality indicator to ensure the confidence of this result; this indicator is played by statistical tools. Therefore, to verify the accuracy of the effect of the TL signal between replicates, the Student t test and Cochran test were used.

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COCHRAN TEST (Q test): is performed to verify if the treatments have identical effects. The Cochran test is a non-parametric test for analyzing randomized complete block designs where the response variable is a binary variable (i.e., there are only two possible outcomes, which are coded as 0 and 1). The Cochran test assumes that there are c experimental treatments (c >= 2). The Cochran test is based on the following assumptions: a) the blocks were randomly selected from the population of all possible blocks; b) the outcomes of the treatments can be coded as binary responses (i.e., a "0" or "1") in a way that is common to all treatments within each block. Assuming that it only has value suspected in the extremities of the distribution; the Cochran test is equal to the Dixon test. After organizing the experimental data in the crescent order; it must be applied the Q test on the extreme values at left and right. [7]

The test is very simple and it is applied as follows:

1. The N values comprising the set of observations under examination are arranged in ascending order: 
   \( x_1 < x_2 < \ldots < x_N \)

2. The statistic experimental Q-value (\( Q_{exp} \)) is calculated. This is a ratio defined as the difference of the suspect value from its nearest one divided by the range of the values (\( Q \): rejection quotient). Thus, for testing \( x_1 \) or \( x_N \) (as possible outliers) we use the following \( Q_{exp} \) values:

   For minimum value:
   \[
   \frac{x_2 - x_1}{x_{n-1} - x_1}
   \]  
   (Eq. 1a)

   For maximum value:
   \[
   \frac{Z_n - Z_{n-1}}{Z_n - Z_2}
   \]  
   (Eq.1b)

   While: \( x \) is anyone value from data set.

3. The obtained \( Q_{exp} \) value is compared to a critical \( Q \)-value (\( Q_{crit} \)) found in tables. This critical value should correspond to the confidence level (cl) we have decided to run the test (usually: cl=95%).

4. If \( Q_{exp} > Q_{crit} \), then the suspect value can be characterized as an outlier and it can be rejected, if not, the suspect value must be retained and used in all subsequent calculations.

   The null hypothesis associated to Q-test is as follows: "There is no a significant difference between the suspect value and the rest of them, any differences must be exclusively attributed to random errors"[8].

   A table containing the critical Q values for cl 90%, 95% and 99% and N=3-10 is given in RORABACHER (1991).

T STUDENT TEST: is applying on two dating sets to verify similarities between them. In this work the T test has been applied on the all values (10 values) of each peak of the energetic emission, for each replicate (replicates: five for nature, 10 Gy, 40 Gy and 50 Gy; seven for 30 Gy and eight for 20 Gy). In T test a grouping standard deviation (SG) is used, as showed in Equation 2, and the end calculation is given to Equation 3.

\[
S_G = \sqrt{\frac{\sum_{i=1}^{N_1} (x_i - \bar{x}_1)^2 + \sum_{j=1}^{N_2} (x_j - \bar{x}_2)^2 + \sum_{k=1}^{N_3} (x_k - \bar{x}_3)^2 + \ldots}{N_1 + N_2 + N_3 + \ldots - N_f}}
\]  
   (Eq.2)

\[
t = \frac{\bar{x}_1 - \bar{x}_2}{S_G}
\]  
   (Eq.3)

2. Experimental

In a dark room, sediment samples collected from Lajeado, Rio Grande do Sul state, were initially pulverized, and sieved to have a particle size diameter between 0.080 and 0.180 mm. Around 700mg of each sample was leached with 5ml of HCl 37.7% (v/v) solution for 40 min, then the resulting solution was discharged and the residue was
washed out with purified water. Then the residue was leached again with 5 ml of HF 10% (v/v) during additional 40 minutes. After discharging the solution and washing the residue out, it was kept for about 24 hours for complete dryness. The samples were separated into four groups and irradiated with a dose rate of 43Gy/h at Center of Radiation Technology, Institute of Nuclear Research and Energy (IPEN-CTR). After the samples irradiation, they were exposed to ultraviolet light, the samples were also thermally treated at 200°C in order to eliminate peaks in the region of 150°C that might interfere with measures commonly used in dating peaks (regions of 220 -325°C).

3. Results

Initially there were the effects of ultraviolet light on a sample of dating [1]. This phenomenon already studied [1],[2],[8],[9],[10], can lead to dating with large discrepancies in determined age. Figure 1 shows the effect of UV light on the intensity of TL light emission, it is easily observed after the sample is exposed to UV light TL signal decreases sharply. The Figure 2 represents the curve of the TL intensity of light emission as a function of the radiation dose.

Figure 1- TL Glow curves

Figure 2. TL plot for method additive

Figure 3 shows the natural sample and samples with additional doses. It's also shown the behavior of the TL signal for each replicate with a difference in signal strength between them.

Figure 3. Glow curve for natural sample and additional doses.
In Figure 4, data from 1 a 50Gy are shown. It is noted large variations of the data set for the peaks. If these curves are used in linear regressions, some overlaps will occur. So there is a need for knowing the effect of reproducibility in each replicate in order to be used the adequate points in the regression. To check whether the extreme points of a replicate may be excluded from the composition of sets of dating, the Cochran test was applied in five points that compose the average dose in a plot of TL intensity as a function of dose. Thus, different regressions were obtained when higher values of TL intensity (high) or lower values of intensity (low) are used in the regression.

The sample used in the study was separated into three subsamples, the first was not exposed to any treatment, the second was treated thermally and the third was treated with UV light. Subsequently, these three subsamples were dated by the additive method. The results are shown in Table 1.

<table>
<thead>
<tr>
<th>Accumulated Dose D_{ac} (Gy)</th>
<th>TL Additive Method (182.05)</th>
<th>TL with Thermal treatment (175.00)</th>
<th>TL with UV exposure (169.04)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Age (years)</td>
<td>9.10X10^4</td>
<td>8.75X10^4</td>
<td>8.45X10^4</td>
</tr>
<tr>
<td>Deviation (Gy)</td>
<td>14.5</td>
<td>11.3</td>
<td>14.5</td>
</tr>
<tr>
<td>Dose variation over the tuning curve (Gy)</td>
<td>13-18</td>
<td>9-19</td>
<td>8-22</td>
</tr>
</tbody>
</table>

By applying the test of Cochran on the values of each replicate, the results show that it is not possible to discard values at 95% of confidence level. The Student's t test applied in two sets of data at the exactly point of temperature peak used for dating, shows no significant differences between all experimental values with 95% of confidence level. The calculated value of Student t test was 1.6, which is lower than the tabled Student's t test value (95%). So, we applied the Cochran test in the average value of TL intensities of the replicates at each point, and it was found that there are no significant differences to eliminate any data with a confidence level less than 95%. Thus, Figure 4 can be obtained by the dataset that are the confidence limits for the method.
The curves of the reproducibility and repeatability are showed in Figure 4, they are limits in which the measured values will meet.

CONCLUSION

The success of this archaeological research is evidenced by the good results obtained in statistical tests. The results showed that routine experiences such as heat treatment and UV exposure can significantly affect the determination of cumulative dose. So, it is clear that a careful evaluation of uncertainty is of fundamental importance since there is no standard procedure for determining the age of archaeological materials, and also there are no certified reference materials applied to this determination. By applying statistical tests there was little variation between the points of the temperature range at 225-270 °C, each point corresponding exactly to at least 05 replicates. The maximum and minimum emission of TL should be disregarded to replicate, since they are suspected for the calculation of the average of TL intensities for dating.

REFERENCES


