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CALIBRATION OF THERMOLUMINESCENCE OPTICALLY STIMULATED LUMINESCENCE READER USING QUARTZ IN DATING LABORATORY, MALAYSIAN NUCLEAR AGENCY

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ABSTRACT

The TL OSL reader is a tool for dating applications in a variety of fields. One of the main components of an TL OSL reader is the irradiation source. Calibration of irradiation sources for readers can be carried out using quartz. It is important to calibrate the reader before performing the dating method to ensure equivalent dose (De) within the acceptance range. Quartz was measured using the Single Aliquot Regeneration (SAR) protocol. Measurements obtained an average dose rate of 28.87 ± 0.43 Gy (n = 10) with a ratio in the range <5%. In addition, the measurement also found that the value of 2θ is equivalent to 70%. The central point for dose distribution was at 28.30 Gy, with only 3 quartz samples getting dose readings outside the dispersion bar. The overdispersion measured doses were 26.62, 27.68 and 31.56 Gy. The ratio of received dose/measured dose is in the range of 0.887-1.052, which showed that there was no significant signal loss. Therefore, we conclude that this reader is acceptable for carrying out the dating method and it is recommended to use different doses as well as more detailed SAR protocols for further study.

Keywords: thermoluminescence, optically stimulated luminescence, quartz, calibration

INTRODUCTION

A Thermoluminescence Optically Stimulated Luminescence (TL OSL) reader is a tool to carry out the dating method based on the luminescence signal in sample minerals such as quartz or feldspar (Preusser et al., 2008). Studies using this reader are concentrated in many fields such as archaeology (Afouxenidis et al., 2007), geochronology (Sanderson & Murphy, 2010) and geomorphology (Muñoz-Salinas et al., 2011). One of the important components in the reader is the irradiation source. It is used to provide homogeneous irradiation in ensuring detection and stimulation on a wide range for dating methods (Richter et al., 2013). Calibration of the irradiation source for the reader was performed using quartz that had been exposed to known radiation.

Quartz is one of the minerals found in nature that can be used in the dating method which aims to identify its properties on radiation and annealing (Koul et al., 2016). The quartz is an aliquot that contains natural paleodoses which can receive laboratory-made dose radiation that has a known dose strength (Kadereit & Kreutzer, 2013). Calibration using quartz is an important step to ensure that the equivalent dose (De) values obtained are reliable, accurate and unbiased (Guérin & Valladas, 2014). Through the use of this quartz, the reader provides information regarding the equivalent dose of this reference material. This equivalent dose is obtained by comparing the luminescence signal that responds to the radiation dose and the luminescence signal that responds to different irradiation times by using the radiation source to be calibrated (Richter et al., 2020). Therefore, this study aims to calibrate the radiation source of the TL OSL reader by using quartz that has been validated to ensure that the equivalent dose is within the accepted range before performing the dating method

MATERIALS, METHODS AND EXPERIMENTAL SETTING

We have received approximately 5 grams of quartz (a verified reference material) that has been prepared according to a study by (Richter et al., 2020) for which sediment samples were taken from the Holocene desert in Schletau, Germany.

Coarse grains (100–300 µm) of the sediment were extracted and heated at 500ºC for 5 hours to zeroing the luminescence signal on the sample. Thereafter, the samples were treated with 30% HCl to remove carbonates. Then, the samples were treated using 30% H_2O_2 to remove organic matter. For the purpose of quartz enrichment, the samples undergo a process using a heavy liquid mineral separation with sodium polytungstate at densities of 2.62 and 2.67 gcm^{-3}. Etching was performed using 40% HF and washing in 30% HCl and then washed in a dispersion material to dissolve the fine-grain particles attached. The dried samples were then sieved at grain sizes between 90–160 µm. Thereafter, the sample was exposed to a daylight lamp (OSRAM Ultra Vitalux 300W with cooling) for 8 hours.

The quartz received had an equivalent dose of 30 Gy, similar to the quartz in study (Richter et al., 2020). The next procedure is carried out in a dark room aimed at preventing the quartz from being exposed to daylight which could cause zeroing of the luminescence signal on the sample (Osunkwor & DeWitt, 2021). The grains of the quartz were placed in 10 stainless steel discs measuring 10 mm in diameter that had been put with silicon oil. After that, all the discs are placed on sample wheels to be loaded into the reader.

Measurements were carried out using the LEXSYG SMART System from Freiberg Instruments (Richter et al., 2015). The reader was equipped with a Sr-90 beta radiation with a radioactivity of 1.85 GBq. Use of blue LED (458 nm) for the purpose of optical stimulation with a maximum rate of 100 mW/cm^{-2}. The detection window used is a combination of BSL/TL, which includes 2.5 mm thick Hoya U340, 5 mm thick BP 365/50 and Schott NG4. Thermal stimulation is carried out through a metal ceramic plate capable of reaching a maximum of 700 ºC to allow the sample to be heated accurately and can be repeated in a homogeneous (Lomax et al., 2014).

Calibration using quartz in this study is based on Single Aliquot Regeneration (SAR) protocol (Murray & Wintle, 2000), whose setting can be seen in TABLE 1. All aliquots (n = 10) undergo a complete SAR protocol cycle for each quartz. The beta irradiation time is framed based on the irradiation time capable of inducing the OSL signal and is carried out in a dark room to ensure that all aliquots have the same bleaching history (Guérin & Valladas, 2014). In this study, 10 s was the test dose. Therefore, each increase in the irradiation time of 50, 100 and 150 s should be followed by 10 s of irradiation. The main purpose of each aliquot obtaining the test dose was to ensure the consistency of the measurements at the beginning and end of the study. In this protocol, preheat is carried out after irradiation and before OSL measurements to allow the transfer of electrons from the trapped (Frouin

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et al., 2017). An overview of the SAR cycle for this study is shown in FIGURE 1. Each aliquot was preheated at 200 ºC for 10 s while heating at 160ºC. The OSL signal for the blue signal stimulus was stimulated for 50 s at a temperature of 125 ºC.

TABLE 1 shows the SAR protocol cycle settings for each quartz sample. The experimental dose is 10 s, where each increase in irradiation dose 50, 100 and 150 s will be followed with 10 s to ensure consistency of irradiation.

Setting	Temperature (ºC)	Time (s)
Irradiation	-	10, 50, 100, 150
Test	-	10
Preheat	200	10
Preheat	160	0
OSL	125	50

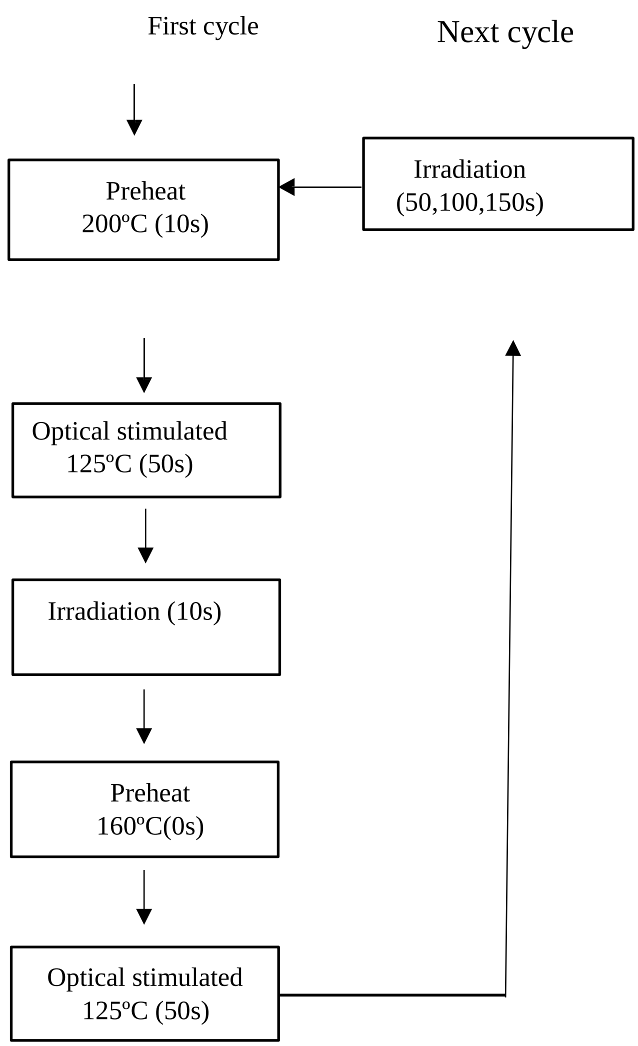

FIG 1 shows an overview of the SAR cycle for each sample. The cycle includes preheating, optic stimulation and irradiation.

RESULTS AND DISCUSSION

The results of the luminescence signal for quartz samples are shown in TABLE 2. According to studies conducted by (Richter et al., 2020), the most accurate and simple calibration using OSL can be obtained by interpolating the OLS-Beta normalisation response from gamma dose quartz to a single dose growth curve beta arising from the OSL-Beta normalisation response at several beta doses. The interpolation of the normalised signal OSL is conducted with three or more points using square linear fit (Guérin & Valladas, 2014). Therefore, we applied this interpolation to each quartz. An example of interpolation can be seen as the curve in FIGURE 2.

The mean of the interpolated dose was 28.87 ± 0.43 Gy (n = 10), as in FIGURE 3, using the SAR procedure. The ratio of dose is consistent and unity in the range of 5%. This leads to an assumption that the SAR protocol and the stability of the reader guarantee the signal obtained during this study (Tribolo et al., 2019). In addition, the standard deviation for all quartz samples (n=10) was approximately 4%. This gives the impression that there is no significant change in the optically stimulated luminescence signal produced by the reader experiencing uncertainty (fluctuation) either in the short or long term (Hansen et al., 2015). Furthermore, the use of the SAR protocol should reach a unity value, but a range within ± 10% is acceptable in the application of the dating method (Richter et al., 2020).

The central point for the dose distribution was at 28.30 Gy, as in FIGURE 4 obtained via the abanico plot (Dietze et al., 2016). The value of 2θ is equivalent to 70%, which indicates only 3 quartz samples got dose readings located outside the dispersion bar as marked in a grey area in FIGURE 4. The three overdispersion equivalent doses were 26.62, 27.68 and 31.56 Gy. These three doses are acceptable because they are still in the range of ± 10%. Meanwhile, 10 samples were insufficient to produce a useful abanico plot curve for the study (Dietze et al., 2016). Thus, the curve is only to give a conclusion in general without considering the polarity as similar to the study conducted by (Douglass et al., 2006).

TABLE 2 shows the results of luminescence signals for 10 quartz samples.

Quartz Sample, n	Given dose, Gy	Measured dose, Gy	Measured dose/Given dose	Error
1	30	27.97±0.57	0.932	0.33
2	30	31.56±0.63	1.052	0.42
3	30	28.85±0.75	0.962	0.6
4	30	28.69±0.63	0.956	0.6
5	30	28.6±1.02	0.953	0.75
6	30	29.24±0.81	0.975	0.8
7	30	29.85±0.64	0.995	0.72
8	30	29.67±0.58	0.989	0.66
9	30	27.68±0.41	0.923	0.73
10	30	26.62±0.40	0.887	0.53

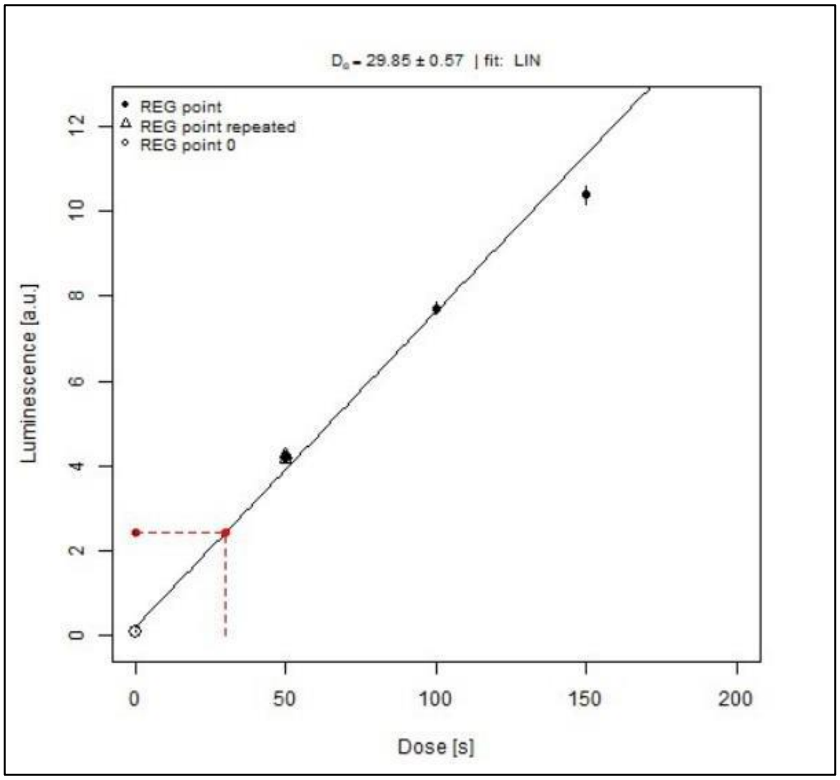

FIG 2 shows an example of interpolation of the dose growth curve that obtains an equivalent dose of 29.85 Gy. The interpolation of the normalised signal OSL is conducted using square linear fit at points 50,100,150 Gy.

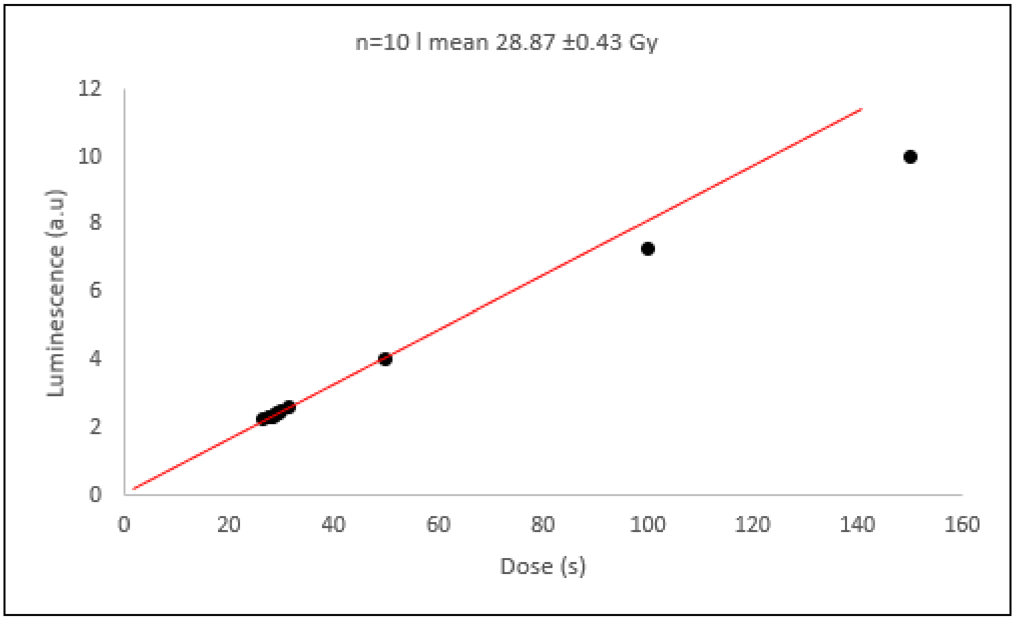

FIG 3 shows the average interpolation dose for all 10 quartz samples was 28.87 ± 0.43 Gy using the SAR protocol.

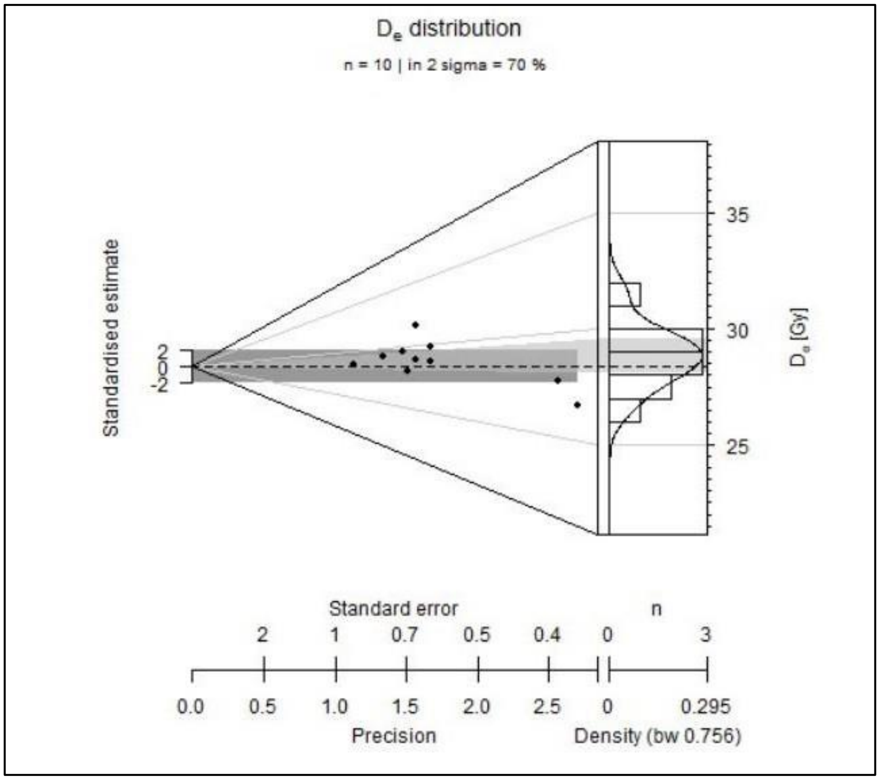

FIG 4 is an abanico plot showing the central point of the dose distribution at 28.30 Gy and value of 2θ equivalent to 70%. The overdispersion doses are 26.62, 27.68 and 31.56 Gy.

CONCLUSION

The TL OSL readers is a tool for dating applications in various fields. The approach of using quartz for the calibration of radiation sources for TL OSL readers is an alternative approach. It is an important step to understand all forms of variability and accuracy for equivalent dose readings. The mean measured dose in this study was average 28.87 ± 0.43 Gy with a ratio in the range <5% using SAR protocol. Abanico plot shows that the central point of dose distribution is 28.30 Gy, with a 2 θ value equal to 70%. It is indicating that 7 quartz sample readings lie on under dispersion region. Quartz samples were measured and produced a ratio measured dose/given dose between 0.887-1.052, which showed that there was no significant signal loss and was almost in line with the results of the study conducted by (Richter et al., 2020) whose ratio between 0.992 - 1.017. Therefore, we concluded the equivalent dose reading produced by the reader in this study was acceptable because it was within range. Indeed, the use of quartz for the calibration of TL OSL readers in the Dating Laboratory, Malaysian Nuclear Agency is to ensure the reliability and accuracy of the dose equivalent reading before using it for the dating method of any sample. Thus, further studies can be performed using a variety of quartz and more detailed SAR protocols such as the study (Hansen et al., 2015).

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