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Optically Stimulated Luminescence (OSL) Dosemetry Characteristics of Quartz Grains Extracted from Some Sediments from Chad Basin, N.E. Nigeria

[1, 2]J.D. Abafoni, [2]A.S. Murray, [2]R. Sohbati and [3]S.P. Mallam
[1]Department of Physics, University of Maiduguri, Nigeria
[2]Nordic Laboratory for Luminescence Dating, Riso National Laboratory, DK 4000, Roskilde, Danmark
[3]Centre for Energy Research and Training, A.B.U., Zaria, Nigeria

Abstract: The Single Aliquot Regenerative dose (SAR) protocol has received popular application in Optically Stimulated Luminescence (OSL) dosemetry due to its ability to correct for numerous sensitivity changes that result during equivalent dose measurements. The basic luminescence dosemetry characteristics and SAR performance evaluation tests have been applied on quartz grains collected from the Chad basin using Risoe TL/OSL reader. The outcome shows that the measured equivalent Doses (D_e) are independent of thermal pre-treatment and charge recuperation, thermal transfer of charges and repeatability of measurements are all negligible within operational conditions. The dose recovery quality of the grains are within acceptable recommendations. Heterogeneous exposure of grains has been observed to have prevailed throughout transport and deposition of sediments and the mean of <5% of measured D_e have been used as dose estimates from the grains. None of the two deposition environments studied (aeolian and fluvial) showed any evidence of homogeneous resetting of luminescence signal in quartz grains during transport and deposition.

Key words: Equivalent dose, dose recovery, regenerative dose, OSL, heterogeneous bleaching, Nigeria

INTRODUCTION

Quartz is among the most readily available mineral grains used in luminescence dosemetry, especially for the dating of geologic and archaeologic materials. Luminescence dating principle employs the fact that Optically Stimulated Luminescence (OSL) intensity observed when already irradiated quartz grains are optically stimulated is used to estimate the radiation dose accrued since deposition. This accrued dose comes from the action of naturally occurring radioactive elements in the matrix surrounding the mineral. A detailed account of the luminescence dating method has been given by several researchers (Aitken, 1985, 1998; Botter-Jensen and Murray, 1999; Murray and Olley, 1999; Murray and Wintle, 2000).

The accurate estimation of the radiation dose responsible for the natural OSL signal in the laboratory can only be possible if the following conditions hold (Murray and Wintle, 2000):

- Charge competition to fill empty traps must be the same for natural and laboratory irradiation
- Stability of filled traps should equally be attainable in both natural and laboratory conditions
- Luminescence signal per unit trapped charge must be the same during stimulation of both natural and laboratory induced signals

Through the use of dose recovery test, the first condition can be confirmed by Murray and Wintle (2003, 2006). The OSL signal emitted within the first 0.45 sec of illumination is dominated by signal whose lifetime is approximately 10^8 years at temperature not >20°C. The same condition is observable when laboratory irradiation is used, only that the signal is accompanied by some that have lifetime of 400 years at the same temperature (Murray and Wintle, 1999). These observations however, prove the stability of the luminescence signal in both circumstances at least for the dateable time range for the quartz OSL dating technique (<10^6 years).

The third condition is a property of the specific quartz grains which is controlled by factors that influence sensitivity changes such as thermal treatment, transport and depositional conditions and mineralization, etc. (Murray and Wintle, 2000). It is the aim of this study to use the newly modified SAR protocol to check the performance of quartz grains extracted from the Holocene environment of Chad basin, N.E. Nigeria (Fig. 1).

Corresponding Author: J.D. Abafoni, Department of Physics, University of Maiduguri, Nigeria

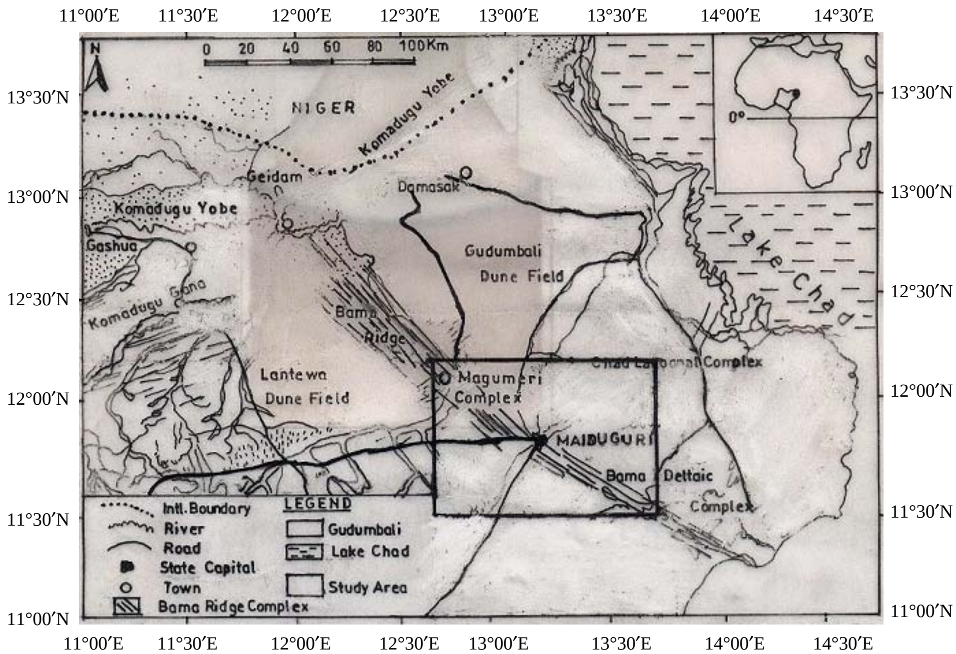

Fig. 1: Map of North Eastern Nigeria showing the ridge and sampling site modified by Thiemeyer (1995)

MATERIALS AND METHODS

Sample collection: Samples for OSL measurement are normally collected in such a way that no part of it is exposed to day light or any other form of brightness that may result in the stimulation of the quartz grains. For this reason, samples were collected by forcing a length (≈50 cm) of metallic or PVC pipe into the deposit. A black cloth was used for cover against any possible exposure and immediate capping of the ends of the pipes were done to ensure no part of the collected rudiment was in any way exposed to day light.

In the laboratory, the samples were recovered under subdued red light (>6000 nm). About 10 cm from each end of the pipes were rejected and separated from the main OSL sample. Samples for this study were deliberately collected from two deposition environments (aeolian and fluvial) for simple comparison.

Sample preparation: All samples were water washed and treated with 10% HCl and 30% H_2O_2 to remove carbonates, organic and clayey materials, respectively. Samples were again put into 40% HF to dissolve and remove feldspar and to etch 10 μm outer layer on quartz grains most accessible to the α-particle. Further treatment with 10% HCl was done to remove acid soluble fluorides resulting from HF treatment. This method of sample preparation was adopted because only quartz crystals were required for the study, otherwise heavy-liquid separation (sodium polytungstate 2.62-2.70 g cm^{-3}) can be used to separate quartz from feldspars (Aitken, 1985, 1998; Hilgers *et al*., 2001).

Analytical method: Optically stimulated luminescence measurement was done using the automated Risoe TL/OSL Reader model DA-20 with all its accessories in place including an internal $^{90}Sr/^{90}Y$ β-source for sample irradiation. All samples were mounted onto 9.8 mm diameter stainless steel discs using silicone oil spray (silkospray). For equivalent dose estimation, each disc contained about 4 mg of grains in the size range of 90-212 microns. Samples were all tested for feldspar contamination using Infrared Stimulation Luminescence (IRSL) before carrying out OSL measurements using the blue Light Emitting Diodes (LEDs), wavelength 470±30 nm and U340 detection filters. Details of luminescence measurement instrumentation are provided by Botter-Jensen (2000). The modified Single Aliquot Regenerative dose (SAR) protocol is used for measurements, Table 1 shows the structure of the measurement sequence. A growth curve (dose vs. response) was generated for each sample, from which the dose corresponding to the natural signal marked on the response axis was obtained via interpolation (Fig. 2).

The recuperation study was done by measuring the OSL signal without administering any dose to the sample after the largest regenerative dose signal was measured. The signal was used to assess the level of thermal transfer

Table 1: Measurement sequence for SAR (OSL)

Steps	Sequence of procedures	Observation
1	Give dose, D_i	-
2	Preheat at 260°C for 10 sec	-
3	Measure OSL for 40 sec at 125°C	L_i
4	Give test dose D_T (sample specific)	-
5	Cut heat at 220°C	-
6	Measure OSL for 40 sec at 125°C	-
7	Measure OSL for 40 sec at 290 (clean out)	T_i
8	Return to step 1 (for i = 1, 2, 3)	-

For natural dose, i = 0 and D_0 is the natural dose. Regeneration doses are D_1-D_3 with corresponding signals L_1-L_3

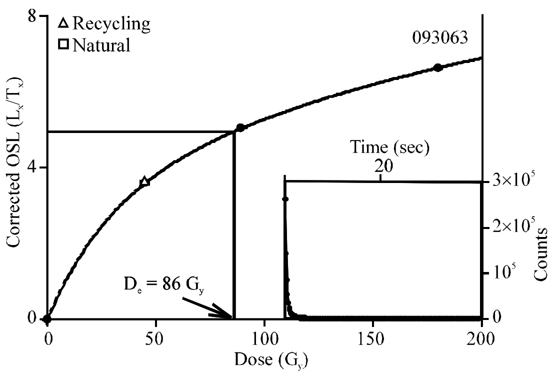

Fig. 2: A typical sensitivity corrected growth and decay curves of a quartz sample. The quartz sample used here was extracted from sediments collected from a Palaeo-beach ridge in the basin (The Bama Beach Ridge BBR)

during measurement cycles (Murray and Wintle, 2003, 2006). Repeat of one of the regenerative doses was done to reveal the variation in sensitivity of the sample. The recycling ratio fell within the acceptable range 0.9≤x≤1.1 (Murray and Olley, 1999) and anything outside this was rejected (Table 2). The ability for a sample to accurately recover a given dose is a necessary step in luminescence dosemetry and dating (Murray and Wintle, 2003). The idea used here was to create a natural scenario in the laboratory where a sample was bleached of its latent luminescence signal and was given a known dose to be recovered. In this research, each sample was bleached with OSL blue LED for 100 sec and was kept for 10 k sec (10000 sec) so that charges transferred to shallow light sensitive traps during bleaching may decay back to the OSL main trap after which the bleaching was repeated. The regeneration doses given in this test were 65, 100 and 135% of the equivalent dose of the sample under consideration, a test dose of 10-20% was adopted throughout the measurement.

The plot of measured dose against given dose is shown in Fig. 3a. The effect of preheat temperature on the dose recovery property of a representative of the samples is also shown in Fig. 3b.

Table 2: Summary for the D_e measurement using OSL

Sample ID	Riso code	Depth (m)	Grain size (μm)	(n)	D_e (G_y)
BR 1	*093056	18.5	90-212	15	131.00±8.2
BR 5A	*093057	18.0	90-212	12	85.30±5.7
BR 5B	*093058	16.5	90-212	12	87.10±6.0
BR 5C	*093059	15.3	90-212	12	79.40±8.0
BR 5D	*093060	14.0	90-212	18	76.34±5.1
BR 5E	*093061	13.4	90-212	16	119.70±7.0
BR 5F	*093062	12.1	90-212	29	80.46±5.0
BR 5G	*093063	10.4	90-212	42	51.52±1.0
BR 5H	*093064	8.0	90-212	18	21.50±0.5
BR 5I	*093065	6.0	90-212	20	27.77±2.0
BR 5J	*093066	5.4	90-212	30	19.55±1.0
BR 5	*093067	5.0	90-180	30	20.82±1.0
BR 3	*093068	4.0	90-180	30	11.46±0.5
1 BR K	*093069	4.6	90-212	15	21.20±1.0
2 BR K	*093070	9.5	90-180	12	26.00±0.6
1 BR M	*093071	2.0	90-180	26	9.26±0.4
2 BR M	*093072	4.4	90-180	12	15.60±0.4
CFRM 2	*093074	-	90-180	12	22.00±2.1
CFRM 4	*093075	1.3	90-180	18	4.40±0.4
CFRM 5	*093076	2.6	90-212	18	5.90±0.2
CFRM 7	*093077	4.4	90-180	15	48.12±4.0
CFRM 8	*093078	1.2	90-180	15	11.36±0.8
CFRM 11	*093080	4.9	90-212	12	95.53±9.0
CFRM 10A	*093081	5.0	90-212	19	95.00±4.8
CFRM 10B	*093082	4.1	90-212	12	71.70±7.0

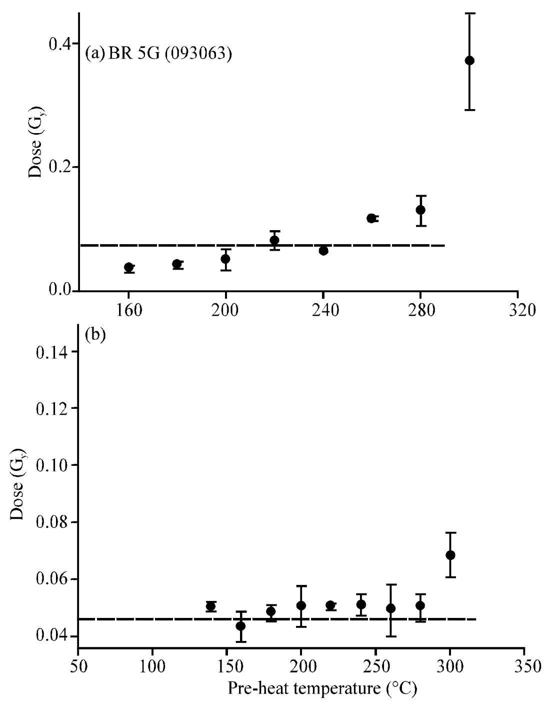

Fig. 3: Thermal transfer check on some selected sample representatives; a) Alluvial and b) Aeolian. There is slight recuperation of charges at higher temperatures in both categories

RESULTS AND DISCUSSION

The independence of the equivalent dose on temperature pre-treatment of the sample are shown

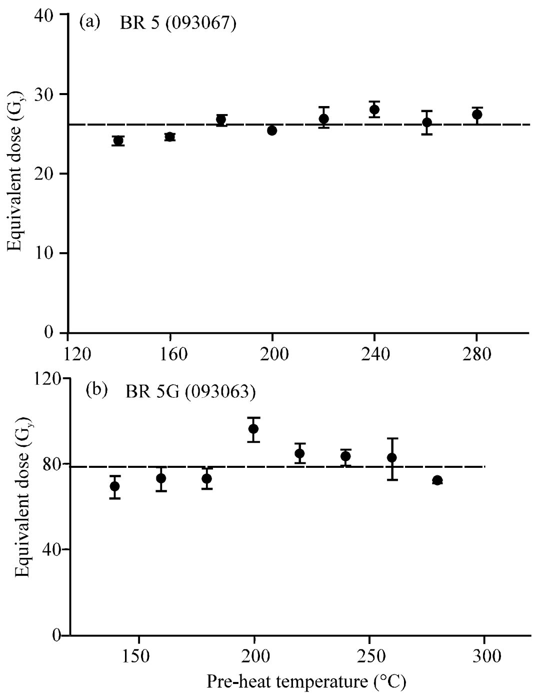

Fig. 4: Dependence of D_e on pre-heat temperature for some selected sample representatives

graphically in Fig. 4a and b. Temperature range, 140-300°C have been used for this test. The temperature that correspond to the plateau in the plot (260°C for most samples) was used as the preheat temperature in routine OSL measurements.

This clearly indicates that the sensitivity change correction adopted in the SAR protocol works well for these samples. This was further confirmed by the outcome of the repeatability test shown in Fig. 5 and 6 for both aeolian and fluvial deposits. The dependence of the recycling ratio on temperature was tested and the result show that the acceptable limit (0.9-1.1) remain unchanged for most samples at measurement temperatures 240-260°C. The ratio changes at higher temperatures (Fig. 5a and b). Signal recuperation during cycles of measurement was checked and signal intensity at zero dose was <0.1% of the equivalent dose for all samples. This value is much less than the acceptable level of 5% (Murray and Wintle, 2000). The dependence of this on temperature shows that at high temperature (>280°C), significant recuperation occurs (Fig. 5a and b).

The thermal transfer of charges tested for these samples also reveal the effects of high temperature treatments, this is shown in Fig. 3a and b. Dose recovery in these quartz samples was successful. This is shown in Fig. 7a where the measured to given dose ratio of the

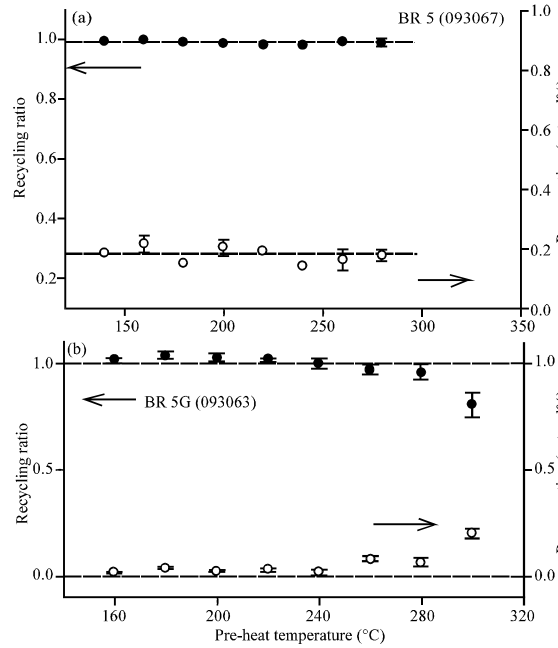

Fig. 5: Recycling ratio and charge recuperation plots. There is consistency with the recommended acceptable limits. In general, recuperation is <0.5% for: a) aeolian and b) fluvial samples. Likewise OSL recycling ratio fall within $0.9 \le x \le 1.1$ for both sedime

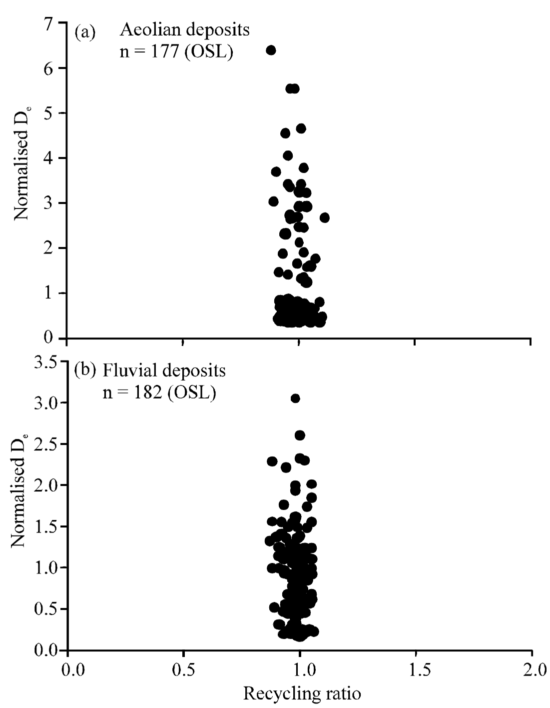

Fig. 6: Dependence of recycling ratio on equivalent dose. The consistency even at high dose levels; a) point B deposits' b) point A deposits

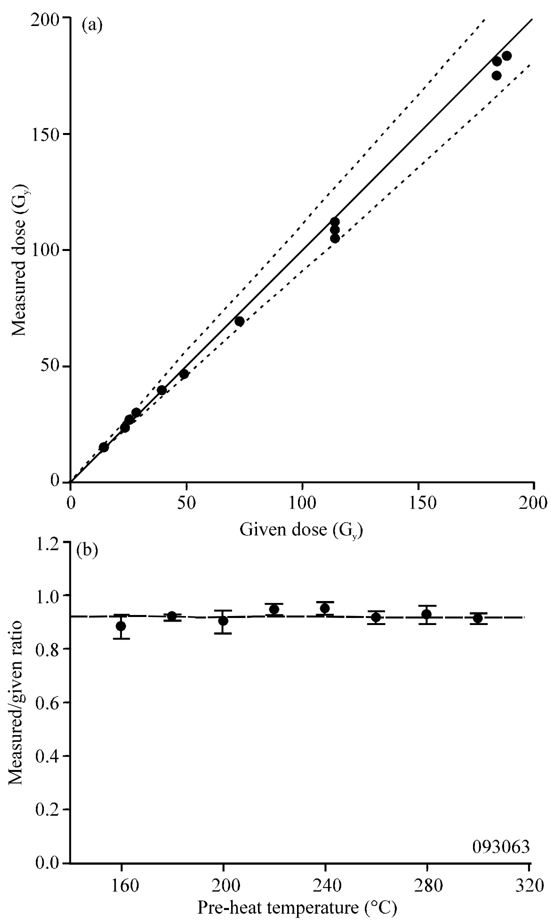

Fig. 7: a) Measured dose vs. given dose plot for 20 samples. There is insignificant deviation from 1:1 ratio and b) measured dose/given dose vs. pre-heat temperature for a sample representative

tested samples fall within the acceptable 10% limit. With equivalent doses from these grains, it would not be necessary to rely age constraint when dating archaeologic or geologic materials. The consistency of the ratio with increase in preheat temperature proves the effectiveness of the sensitivity correction in SAR protocol. The heterogeneous resetting of latent luminescence signal during transport and deposition of these grains was considered. The equivalent dose distributions shown in Fig. 8 are asymmetric indicating that heterogeneous partial resetting of signal prevailed throughout transport and deposition periods. The results obtained here agrees with other researches in the sub-region (Thiemeyer, 1992; Gumnior and Thiemeyer, 2003; Gumnior and Preusser, 2007). Luminescence signal per unit trapped charge can be adversely affected by degree of change in sensitivity of quartz crystals. Variation in sensitivity during routine OSL measurements arises from past and present thermal treatments. A very important assumption of the SAR procedure is that the sensitivity measured using a test dose is proportional to the sensitivity associated with the preceding natural or regenerative dose (Murray and Roberts, 1998; Murray and Wintle, 2000). Confirmation of this assumption has been revealed in the independence of D_e on preheat temperature. Other characteristics such as point recycling, charge recuperation and thermal transfer of charges show stability in sensitivity within the operational temperatures. The performance of these quartz samples in OSL dosemetry was culminated in the dose recovery test. This test was used to assess the reliability of a measured dose from a sample whose history is not known. Such a test can be trusted in luminescence dating applications where age constraint is lacking.

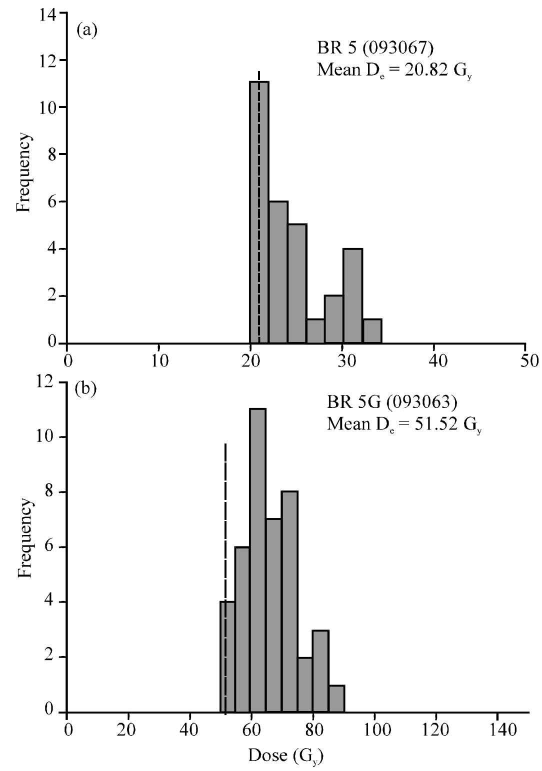

Fig. 8: Equivalent a) dose distribution plots and b) Aeolian deposits and c) fluvial deposit. The dashed lines show the mean value the <5% of the individual aliquot measurements

Even though, low number of aliquots (maximum of 42) was used in this study due to sample quantity, the coarse grains of quartz have performed fairly well under the SAR protocol. Reliable ages can be obtained when these grains are used in dating applications.

CONCLUSION

It is however, necessary to point out that quartz OSL signal saturates at high level of doses. This can be seen in the dose vs. response growth curve. The young samples investigated in this were not affected by this saturation problem hence, dose recovery and eventual equivalent dose estimation are commendable.

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