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Comparison of OSL and ITL Measurements on Quartz Grains Extracted from Sediments of the Chad Basin, N.E. Nigeria

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Abstract: Optically Stimulated Luminescence (OSL) dating using quartz has now been confirmed to be limited to sediment ages <150 Ka due to the saturation levels of the dose-response curve. Thermoluminescence (TL) of unheated quartz has been used in the past to date sedimentary sequences beyond 200 Ka but became very unpopular among users because of the poor reproducibility observed in the measurement procedure (multiple aliquot) and inadequate resetting of the TL signal at 325°C. The possibility of using Isothermal Thermoluminescence (ITL) on quartz to extend the dating range has been suggested by many workers. Using the Single Aliquot Regenerative dose protocol (SAR), the OSL and ITL (at 270°C for 600 sec) signals have been compared using quartz grains extracted from sediments collected from the Nigeria sector of the Chad basin. The preliminary finding of this research shows that the equivalent doses obtained using ITL signals from the younger samples (<30 Ka) agrees with OSL doses. On the other hand, ITL underestimates the OSL counterpart in the older samples (>100 Ka).

Key words: Isothermal TL, OSL, thermoluminescence, regenerative dose, aliquot, equivalent dose

INTRODUCTION

Luminescence technique in dating applications have developed tremendously over the past few decades. The limit of this dating technique is determined by a variety of factors such as luminescent material type, prevailing depositional circumstances (in environmental materials) and luminescence signal measurement styles and techniques employed. It is generally accepted that quartz OSL using the Single Aliquot Regenerative dose protocol (SAR), developed by Murray and Roberts (1998) gives reliable Equivalent Dose (De) measurements by correcting for emerging sensitivity changes during measurements. However, the quartz OSL often saturates when large doses are involved e.g., >200 Gy (Huot *et al*., 2006).

This saturation problems in quartz OSL have restricted its success to young samples (<50 Ka) only. Thermolumminescence (TL) signal from quartz have shown slow saturation levels that can enable estimation of doses upto 500 Gy, Aitken (1985), Jain *et al*. (2005). Unfortunately, TL signal have proved difficult to be bleached optically (Aitken, 1985; Jain *et al*., 2005; Buylaert *et al*., 2006) and its use can easily present dose over estimation, especially in samples with short burial times. The potential of isothermal Thermoluminescence (ITL) signal in luminescence dosemetry have been investigated by researchers Jain *et al*. (2005), Buylaert *et al*. (2006), Choi *et al*. (2006) and Huot *et al*. (2006) with the singular intention to extend the dating range using quartz. This initial investigations targeted the ITL signal at 310°C originating from the famous thermally stable 325°C TL peak. This approach has successfully extended the age range using SAR protocol in sediments from indo-gangetic plains (Jain *et al*., 2005, 2007) and Korean Peninsula (Choi *et al*., 2006).

Further investigations on samples from many other sedimentary environments and locations shows that the increase in measured doses result in over estimation of the expected doses, Buylaert *et al*. (2006). The over estimation resulting from the use of ITL at 310°C has been blamed on an irreversible sensitivity change that occurs during the course of measurement, Vandenberghe *et al*. (2009). Sequel to this development, Vandenbeaghe *et al*. (2009), reports the adoption of ITL at 270°C following a preheat at 300°C for 10 sec as the alternative approach to achieve minimal sensitivity changes in ITL measurements.

In this study, ITL at 270°C was applied to quartz grains extracted from Aeolian and fluvial sediments with the intention to investigate the potentiality of using ITL at 270°C in dating applications comparing with the already established quartz OSL dating technique.

MATERIALS AND METHODS

Fourteen samples were isolated for this investigation. They comprise of aeolian and fluvial samples collected

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Table 1: Measurement sequence for SAR-OSL

Steps	Sequence of procedures	Observation
1	Give dose (Di)	-
2	Preheat at (240-260°C) for 10 sec	-
3	Measure OSL for 40 sec at 125°C	Li
4	Give test dose D_T (sample specific)	-
5	Cut heat at (220-240°C)	-
6	Measure OSL for 40 sec at 125°C	-
7	Measure OSL for 40 sec at 290 (clean out)	Ti
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

Table 2: Measurement sequence for SAR-ITL

Steps	Sequence of procedures	Observation
1	Give dose (Di)	-
2	Preheat at 300°C for 10 sec	-
3	Heat to 270°C and hold for 600 sec	ITL-Li
4	Give test dose D_T (sample specific)	-
5	Preheat at 300°C for 10 sec	-
6	Heat to 270°C and hold for 600 sec	ITL-Ti
7	Measure OSL for 40 sec at 290 (clean out)	-
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

from Chad basin, N.E. Nigeria. Bulk of the samples came from a palaeo beach ridge. The quartz fractions fall between the diameter range of 90-212 μm and were prepared through sieving and chemical treatment using standard procedures given by Aitken (1985) 10% H_2O_2, 10% HCl and 40% HF under subdued red or orange light (>600 nm). Feldspar contamination was checked by exposing the grains to infrared (IRSL) stimulation (830 nm). Samples showing IRSL signals >10% of OSL counterparts were rejected in this investigation.

The automated Riso TL/OSL Reader Model DA-20 fitted with $^{90}Sr/^{90}Y$ beta source was employed in the measurement of both OSL and ITL signals. Quartz OSL stimulations were done at 125°C for 40 sec using blue LED (470 nm) delivering about 50 mW cm^{-2} at the sample position. ITL was observed at 270°C for 600 sec at 2°C sec^{-1} rate. Luminescence signal filtration was through 7.5 mm of UV-filter (Hoya U-340).

All the samples were first subjected to OSL analysis using the newly modified SAR protocol (Table 1), Murray and Wintle (2003). The test dose administered was sample specific (<20% of D_e). Standard luminescence characteristics were observed in all the samples which indicates that the SAR OSL protocol is very suitable for equivalent dose estimation for these samples (Table 1). The correction of sensitivity changes embedded in the SAR procedure was satisfactory as evidenced by perfect recycling ration (0.9<x<1.1) and negligible charge recuperation (<5% of the natural value). A more reliable and highly rated performance test of the SAR procedure, the dose recovery test was carried out for each sample. This tests the overall accuracy of the SAR protocol and can be trusted where age control on dated materials is absent. In dose recovery measurements, samples were bleached of natural doses and same amount were administered artificially to be recovered using SAR procedure. In order to achieve this, regenerative doses in steps of 65, 100 and 135% of the given dose were administered and equivalent doses (D_e) were calculated via interpolation on the dose versus response curves (Fig. 1). In perfect dose recovery, the ratio of measured dose to given dose is unity (1:1), Fig. 2. Isothermal Thermoluminiscence (ITL) measurements were made at

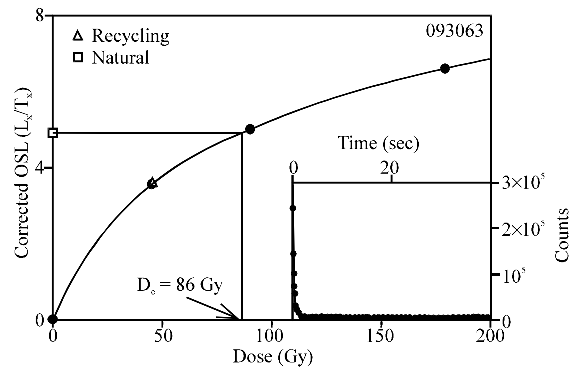

Fig. 1: A typical growth and decay curves of a quartz sample. This curve comes from a fine to medium well sorted fluvial sediment at the ridge center

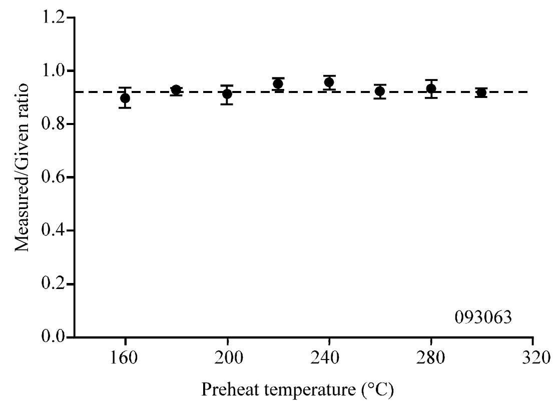

Fig. 2: Measured dose/given dose vs. preheat temperature. Note the relationship of the points

270°C for 600 sec after a preheat at 300°C for 10 sec on each of the samples. Table 2 shows the SAR-ITL measurement protocol adopted in this research. A blue LED stimulation at 280°C for 40 sec was applied at the end of each cycle to rid the traps of any possible charge transfers during measurements. The dose versus response curves generated shows that ITL curves saturates earlier than the OSL curves in the older samples. Though the calculated equivalent doses in the younger samples are in agreement, the ITL growth curves are in saturation (Fig. 3 and 4).

RESULTS AND DISCUSSION

The dose versus response growth curves obtained through the SAR-ITL have shown saturation earlier than those of SAR-OSL for both young and old samples (Fig. 3a). The equivalent doses calculated (Table 3) show close agreement among the range of younger samples and large differences in the older ones. The ITL D_e values are lower than the OSL D_e as shown in Fig. 3b. The dose recovery tests conducted on selected samples using the subtraction technique were successful since the doses were recovered (triangles in Fig. 4). Two samples 1 BRM and BR.5J were used in this test in which 100 and 200 Gy were added and recovered. Both recovered doses fall close to the 1:1 line in Fig. 4. This success largely confirms the potentiality of SAR-ITL in dosemetric applications. The signal investigated here is weak in intensity, Vandenberghe *et al.* (2009) this may be responsible for the behaviour observed in this investigation.

A key observation noted during this study as the difficulty in bleaching the ITL signal at 270°C in the laboratory. Exposing the samples to solar simulator for 4 h in the laboratory could not remove the signal. This contradicts assertions that the signal is readily bleachable.

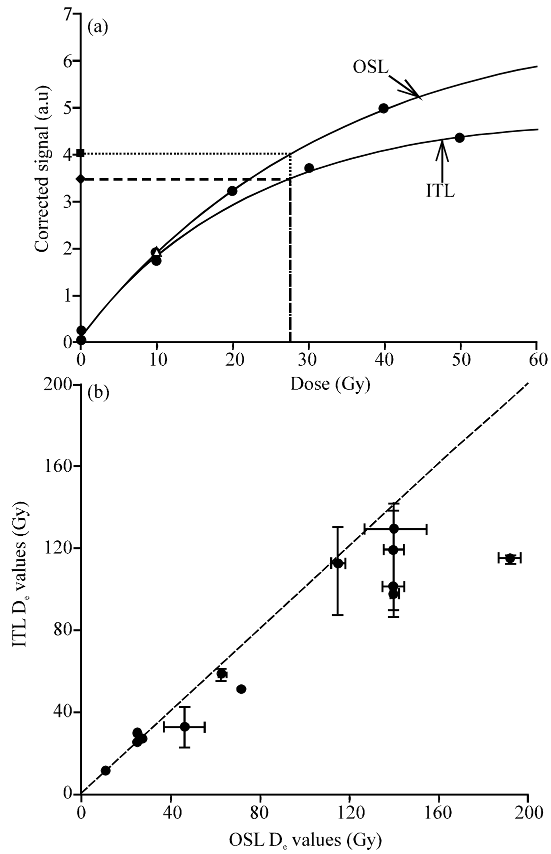

Fig. 3: a) Dose-response curve comparison for a young sample, ITL D_e = 22.95±2.0 Gy and OSL D_e = 23.16±0.64 Gy; the latent ITL and OSL signals are not the same but almost same D_e is responsible for the signals and b) the ratio of ITL D_e to OSL D_e falls below 1:1 line for most of the measured samples

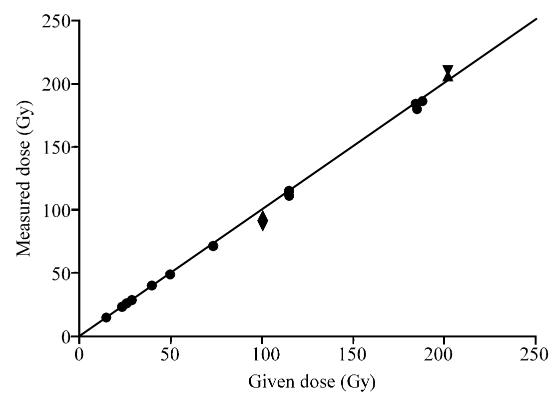

Fig. 4: Measured dose vs. given dose. Shaded triangles represent the ITL doses using the subtraction technique

Table 3: Summary of the samples' ITL and OSL ages as calculated using arithmetic means and compared

Sample ID	Riso code	Depth (m)	WC (%)	Grain size (μm)	Dose rate (Gy/Ka)	ITL D_e (Gy)	n	ITL age (Ka)	D_e OSL	n	OSL (age)
BR5A	93057	18.0	3.0	90-212	3.1±0.18	109.5±21.5	6	35.0±7.0	113.0±7.00	12	36.0±3.0
BR5B	93058	16.5	3.0	90-212	3.3±0.17	128.6±13.8	6	39.0±4.7	141.0±14.0	12	43.0±5.0
BR5C	93059	15.3	1.2	90-212	3.2±0.17	124.7±15.0	6	39.0±5.1	144.0±9.00	12	41.0±3.4
BR5D	93060	14.0	2.0	90-212	3.5±0.19	119.0±20.0	6	34.0±6.0	143.0±6.00	18	42.0±3.0
BR5E	93061	13.4	5.6	90-212	3.4±0.16	124.0±5.20	6	36.5±2.1	116.0±2.00	18	44.0±3.0
BR5F	93062	12.1	2.4	90-212	2.7±0.21	95.4±8.60	12	35.3±3.7	60.0±1.00	18	22.0±1.0
BR5G	93063	10.4	2.1	90-212	2.8±0.18	50.2±4.50	6	17.9±1.9	26.0±0.50	18	9.0±0.5
BR5H	93064	8.0	1.8	90-212	3.0±0.17	25.7±1.00	6	8.6±0.5	32.0±1.00	18	11.0±0.6
BR5I	93065	6.0	2.0	90-212	2.9±0.20	29.2±3.50	6	10.1±1.3	22.0±0.30	18	7.0±0.4
BR5J	93066	5.4	2.0	90-212	3.2±0.16	27.6±4.20	6	8.6±1.9	24.0±1.00	30	7.5±0.8
BR5	93067	5.0	2.0	90-180	3.3±0.17	35.4±3.50	6	10.7±1.2	23.0±0.80	18	7.0±0.4
IBRM	93071	2.0	2.0	90-180	2.8±0.17	12.3±1.50	6	4.4±0.4	11.8±0.40	18	4.0±0.2
CFRM7	93077	4.4	2.2	90-180	2.7±0.19	56.3±5.70	6	20.9±2.0	64.3±5.00	12	24.0±2.0
CFRM10A	93081	5.0	6.0	90-212	2.5±0.18	101.3±4.00	6	40.5±3.3	140.0±8.00	18	72.0±5.4

CONCLUSION

The result emerging from this preliminary investigation shows clearly that SAR-ITL at 270°C for 600 sec after a pre-heat at 300°C for 10 sec is not working for this particular group of quartz grains. It is obvious in this research that gross de-sensitization of the quartz grains has occurred resulting in the under estimation of doses. Investigations are still going on in respect of ITL techniques in dosemetry and it is hoped that further light can be shed on the application of this technique.

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