To,

The Editor, EarthArXiv,

Dear Editor,

I am herewith submitting a manuscript entitled "*How Robust are Single Aliquot Regeneration paleo-doses using single grains of Quartz: The role of change in luminescence sensitivity during the measurement of natural luminescence*" by Naveen Chauhana, H. M. Rajapara, , J. Feathers, S. T. Garnett, R. J. Wasson, M.K. Jaiswal and A. K. Singhvi for publication in *EarthArXiv*. The manuscript contains original ideas and new data which are not previously published or under consideration for publication elsewhere.

The work investigates the inter-grain and inter-sample variations in luminescence responses of natural Quartz (SiO_2) and its effect on luminescence doses. The study provides rigorous single grain data analysis which tries to investigate role of luminescence sensitivity for radiation dosimetry in natural samples. The work will be useful for improving the existing methods for dating.

Thanking you,

With warm regards,

Naveen Chauhan

How Robust are Single Aliquot Regeneration paleo-doses using single grains of Quartz: The role of change in luminescence sensitivity during the measurement of natural luminescence

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ABSTRACT

Luminescence dating using quartz is generally carried out using the single aliquot regeneration (SAR) protocol (Murray et al., 2021; Murray and Wintle, 2000). Singhvi et al. (2011) suggested a methodological improvement in the SAR protocol to account for the changes in the luminescence sensitivity during the readout of natural OSL signal. It was suggested that such changes are common and if not corrected for, can lead to systematic offsets in the SAR based luminescence ages. Singhvi et al. (2011) proposed a Natural Correction Factor (NCF) to correct for such changes in the luminescence sensitivity.

This suggestion implied that, in the absence of any experimental procedure to routinely measure changes in the luminescence sensitivity of individual grains, currently used automated single grain measurements and age determination based on them could be erroneous (Chauhan and Singhvi, 2019). This contribution examines a possible way such that automated single grain measurements can be used through segregation of luminescence signals from grains whose signal potentially have a NCF closer to unity.

Examination of SAR measurements on single grain data provided three important observations viz;

a) dispersion in single grain paleo- doses decreased with the signal to background ratio(R),
b) the dose recovery tests on young samples (with low natural doses), additionally irradiated with a larger laboratory dose, indicated that, majorly the samples with high signal / background ratio (R), typically > 100, returned paleodoses closer to the expected dose. Grains with lower R gave lower paleo-doses with a higher dispersion;
c) Optical decay curves of samples with R > 3 and R > 100 differed and only grains, with similar and near ideal decay shapes comprising only the fast component, yielded concordant paleo-doses.

These led to a suggestion that segregation of paleo-doses based on their R values and optical decay shapes could possibly provide a diagnostic filter for grains that do not suffer sensitivity changes and, ages based on such grains would be more reliable. At this stage this study is presented as an idea with reasonable premise and promise, some encouraging results on the proof of the concept and we suggest for a community wide effort towards a reevaluation of reported single grain paleo-doses based on two criteria viz. the R value and optical decay shapes, so that more realistic single grain luminescence ages are obtained.

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1. Introduction

Development of automated single grain optically stimulated luminescence (OSL) measurements offered the promise of improved precision due to capabilities of obtaining paleo-doses on large number of grains. These also opened up the prospects of isolating grains based on their bleaching histories. Various analysis protocols were developed for analysis of single grain paleodoses (Bailey and Arnold, 2006; Chauhan and Morthekai, 2017; Chauhan et al., 2021; Chauhan and Singhvi, 2011; Galbraith and Roberts, 2012; Galbraith et al., 1999; Guérin et al., 2015). Studies on variabilities in single grain luminescence and their possible import on dating application were also reported by e.g. Adamiec (2000); Chauhan and Singhvi (2011); Duller (2012); and Yoshida et al. (2000). Use of single grain luminescence also led to identification of complicating factors such as,

a) the realization that natural beta dose at the single grain level is heterogeneous (Chauhan et al., 2021; Guérin et al., 2015; Jacobs et al., 2006; Mayya et al., 2006; Thomsen et al., 2005). This required a revision of the age equation for the computation of single grain luminescence ages, (Chauhan et al., 2021; Mayya et al., 2006; Morthekai, 2007), and
b) identification of undocumented and unaccounted for changes in OSL sensitivity of single grains during the preheat and readout of their natural OSL (Chauhan et al., 2009; Chauhan et al., 2015; Chauhan and Morthekai, 2017; Chauhan and Singhvi, 2019; Singhvi et al., 2011). This led to the present study.

Singhvi et al. (2011) pointed out that the SAR protocol (Murray et al., 2021; Murray and Wintle, 2000), used for the estimation of paleo-doses, does not take into account changes in the luminescence sensitivity of aliquots during the measurement of their natural signal. Thus, in the conventional SAR protocol, measurement of natural signal and the following regenerated signals are carried out with differing luminescence sensitivities. Such differences, compromise both the precision and the accuracy of luminescence ages. Using the proportionality of the 110°C TL glow peak of quartz with its OSL, an aliquot -based correction factor was proposed. This correction factor was the ratio of the luminescence sensitivity of the 110°C TL glow peak of a sample as received, and that after the preheat and the measurement of natural OSL. This factor was termed by Singhvi et al. (2011) as the natural correction factor (NCF) and its applicability was demonstrated by Chauhan et al. (2015); and Chauhan and Morthekai (2017) besides others.

Table-1 SAR measurement protocol

1	Dose D_i^*
2	Preheat (240 °C, 10 sec)
3	OSL (125 °C, 1 sec) (L_x)
4	Test Dose
5	Preheat (240 °C, 10 sec)
6	OSL (125 °C, 1 sec) (T_x)
7	Return to step-1

$^*D_i$ is zero for burial dose measurement. D_i given in step of R_1, 2R1, $4R_1$, 0, R_1 following saturating exponential behavior of dose build-up during sediment burial.

The use of the NCF with the SAR protocol on single aliquots generally gave lower paleo-doses (D_e's), with lower dispersion and permitted dealing with those samples where the intensity of natural OSL plotted way above the regenerated growth curves,(Yoshida et al., 2000). Such observations clearly indicated the presence of unaccounted changes in sensitivity in the SAR protocol. Chauhan and Singhvi (2019) measured sensitivity change during the measurement of natural OSL of single grains and suggested that the sensitivity variations in single grains during measurement of natural signal can be significant and should be considered while using single aliquot SAR measurements. Despite a considerable scatter, the data showed a general trend for NCF approaching unity with the increasing luminescence sensitivity of individual grains. The authors also suggested that dull grains might have an adverse role to play during single aliquots dose determination.

Taking this observation forward, this study explored the possibility of reducing / eliminating the need to measure the change in sensitivity of individual grains so that the automated measurement could still be carried out with additional data processing to collate paleodoses of only those grains which had likelihood of their NCF being closer to unity. Thus, the premise that brighter grains had NCF values closer to unity was explored and the dependence of single grain paleo-doses on their luminescence sensitivity was examined. This premise was further tested using suitably designed dose recovery tests. The results also provided a physical basis for various empirical approaches like the fast ratio and super-grains aimed to improve the dating results (Duller, 2012; Durcan and Duller, 2011).

2. Samples and sample preparation

The proof of concept was tested using samples from: a) magnetic termite mounds of *Amitermes Meridionalis* from Northern Territory, Australia (12.5392°S and 131.1014°E); b) aeolian dune sands from Thar Desert India (27.4952°N and 70.3003°W) and, c) fluvial sands from R. Kaveri, India, (10.9058°N and 79.5906°E) and Tamilnadu (13.2140N, 79.8328E). These sites were selected due to the diversities of the provenance of sediments and their varied depositional environments. Inferences from these studies were further tested by reanalyzing single grain data on fluvio-aeolian samples from archaeological sites each in South Carolina, USA; Sao Paulo, Brazil and Mwulu's cave in South Africa (Feathers, 2003; Feathers et al., 2020 and unpublished work). Overall the choice of samples provided data on quartz from diverse provenance and geological histories.

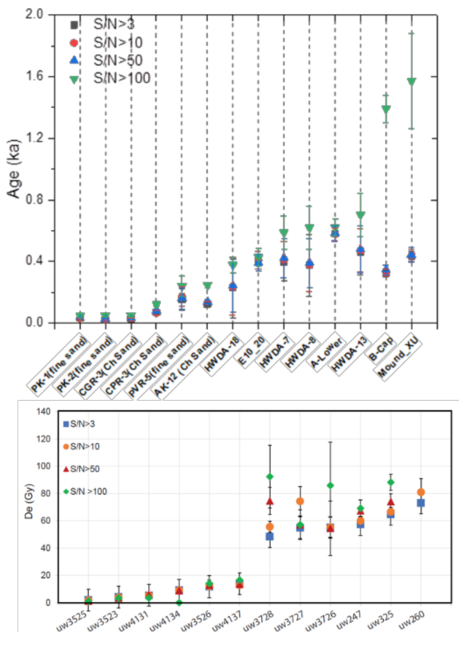


Figure-1 Variation of estimated Age and dose with respect to different single grain luminescence S/N ratio. X-axis is sample code with increasing age. Notice that the ages agree in some cases and diverge in others. Similar trend is to be expected for paleo-doses given that the dose rate in each case is assumed to be same.

The samples were collected in metal pipes (Chandel et al., 2006) and quartz grains were extracted using standard procedures, i.e. sequential steps of pretreatments with 3.7 % (1N) hydrochloric acid (HCl) and 30 % hydrogen peroxide (H_2O_2), drying and sieving for 150 – 210 µm diameter grains, and isolation of quartz using a Frantz Magnetic separator (Porat, 2006). Quartz rich fractions were then treated with 40 % hydrofluoric acid (HF) for 80 min to remove the α-irradiated surface and dissolve residual feldspar grains. After HF, an immediate treatment with 37 % HCl was carried out to convert insoluble fluorides to soluble chlorides. This fraction was re-sieved to obtain quartz grains > 150 µm. Purity of quartz OSL signal due to contamination by feldspar OSL, was tested using Infra-Red Stimulated Luminescence (IRSL).

3. Measurements

The measurement protocol including sample preparation procedures for the samples from the USA, Brazil and South Africa, have been described in Feathers (2003); and Feathers et al. (2020). Measurements in India used a standard Risø TL/OSL single grain reader TL/OSL DA-20 (Bøtter-Jensen et al., 2000; Bøtter-Jensen et al., 2010), with a Nd : YAG green laser as the stimulation source and detection optics comprising Hoya U-340 and BG-39 filters in the front of an EMI 9635QA Photomultiplier tube (Aitken, 1998). Thermal treatments and optical stimulations were carried under ultrapure nitrogen atmosphere with a flow rate of 1 l/min. A 10 sec pause after each thermal cycle allowed the grains to reach a thermal equilibrium. A five-point single-aliquot regenerative-dose (SAR) protocol was used for the dose response curve (Table-1; Wintle and Murray (2006)) with a preheat of 240 °C for 10s and optical stimulations at 125°C. SAR paleo-doses for samples were computed using integrated photon counts for an initial 0.1 sec as the signal and the final 0.2 seconds as the background.

The selection criterion was: <5 % recuperation ratio, recycling ratio within ±10 % of 1, <10 % test dose error and < 2.5 % measurement error. Typically, about 1000 grains were measured for each sample and of these 221-760 grains satisfied the measurement criteria.

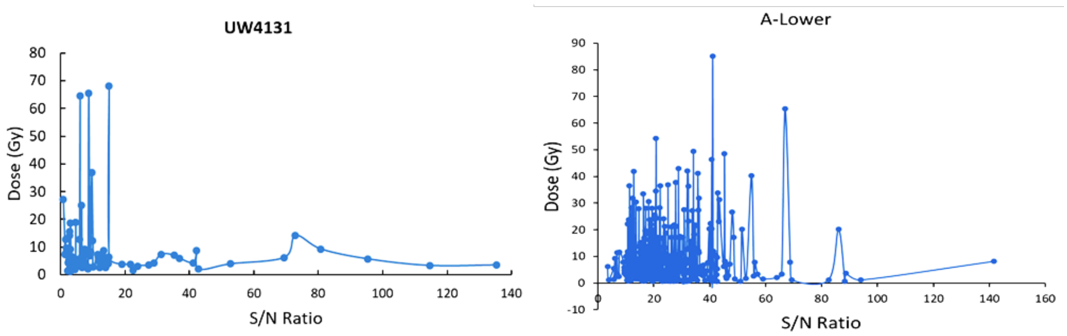


Figure 2 Variation in De as a function of R. Notice the large fluctuations which reduce as R approaches >100.

The data for each sample was then segregated based on the response of grains to a fixed test dose of 0.5 Gy. The ratio of signal (S) to background (B), or S/B, was called the R value. Groups of grains with R > 3, > 10, > 50 and >100 were examined as separate subsets, noting that data classified as R > 3 included grains in the classes R > 10, >50 and >100 and likewise the data for R > 10 included all those grains with R > 50 and R > 100. Light sum of grains with R > 100 suggested that about 14% of the signal was contributed by these < 5 % grains.

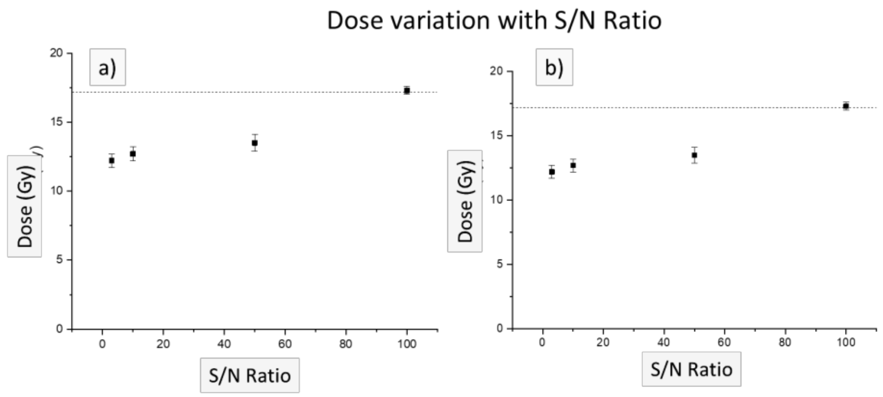
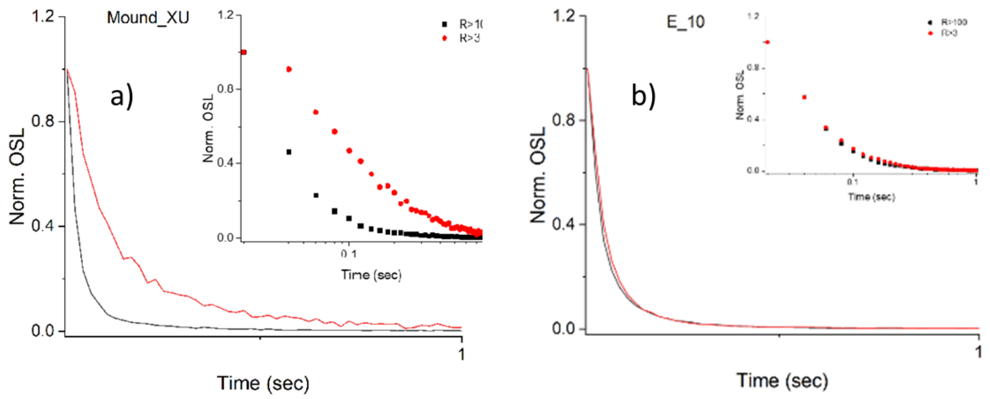


Figure 3 a) Dose recovered from Mound XU (Natural + Beta). Natural dose considered as 4.6±0.8 Gy. b) Dose recovered from the sample B-Cap having Natural and known laboratory Beta doses). Natural dose as computed

based on SAR is 2.1±0.1 Gy. In both the cases the, added laboratory beta dose is 15.1±0.1 Gy. Dashed line indicates the expected total dose of the grains after administering laboratory dose.



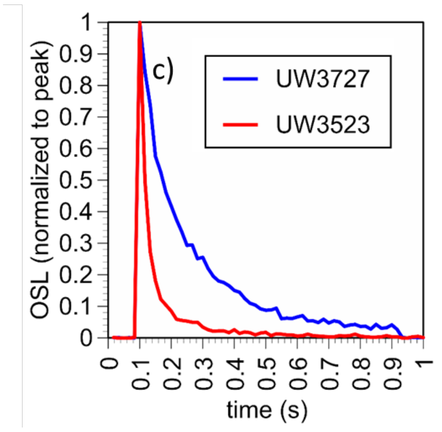


Figure 4 a) Optical decay curve for Mound XU, R>100 (Natural dose 4.6±0.8 Gy) black squares and Mound XU, R>3 (Natural dose 1.26±0.1 Gy) red circles. (Logarithmic scale, inset) b) Optical decay curve for E_10, R>100 (Natural dose 1.15±0.02 Gy) Black squares and B-Cap, R>3 (Natural dose 1.10±0.02 Gy) red circles. (Logarithmic scale, inset).c) Optical decay curves of grains from UW3727 (South Africa), which has a S/N of 0.63. The other is from UW3523 (Brazil), which has a S/N of 40. Noteworthy is that UW3727 has a significant medium component, lacking in UW3523.

The second experiment comprised a dose recovery test using the SAR protocol. A beta dose of 15.1±0.1 Gy was added to young samples on top of low natural dose (few Gy) and the total dose (natural + added dose) was recovered. A test dose 2.2±0.1 Gy was used. As the expected dose was known (being largely the added dose), this experiment helped test the effect of sensitivity change on paleo-doses. A sensitivity change would lead to a recovered dose different from expected dose.

4. Results

Table 2 provides the paleo-dose data for the original samples. Figure 1, provide the same in a graphical form for ease of visualization. Figure 2 provides the results of the dose recovery test. Figure 3 provides the optical decay curves for R > 3 and > 100, indicating the presence of a medium component.

Several noticeable features were observed,

1. The difference in paleo-doses for R > 3 to R > 100 (plotted and tabulated in Table 2. Fig 1), was sample dependent and ranged no change to up to >100%.
2. The plots for the D_e as a function of R fluctuated to up to R < 50 after which it tended to stabilize around a mean value (Figure 2).
3. Results of the modified dose recovery test (Figure 3) comprising recovery of anticipated dose from a young sample along with an added 15.1 Gy beta dose. Typically, 300 grains were measured for each sample and, their D_e estimated. Grains with R > 100, provided results closer to the expected value and the grains with R <

50 resulted in values lower than 15 Gy. Based on Chawla et al. (1998), we assumed that a small dose of 15 Gy would not induce and sensitivity change.

4. OSL decay curves for R>3 and R>100 (Figure 4) of individual grains, suggests that paleo-dose for R >3 and R >100 were similar whenever the decay curve shapes of R>3 and R>100 were similar. The data suggest that a medium component of the OSL decay curve, may have caused sensitivity changes.
5. Despite limitations of statistics, the data from the UW-series, , (Table 2) showed a similar trend with most samples showing a difference in paleo-doses by over 30-50% and a few showed no change. The decay curve shapes indicated the presence of medium component for samples where the paleodose depended on the R value.

5. Discussion:

The results suggest:

1) Change in luminescence sensitivity during the read out of natural signal at single grain level is common and usage of SAR paleo-doses, in the manner carried out at present, may lead to erroneous ages.
2) Such a sensitivity change could occur across the components of OSL decay curves and therefore the use of fast ratio, may also lead to difficulties. Stated simply, a higher fast ratio may not be a guarantee against sensitivity change.
3) Analysis of paleo-doses with individual grain S/N ratio can help. It is suggested that variation of De along with their R value can be useful in isolating single grain paelodoses where the change in luminescence sensitivity is minimal.
4) Grain dependent changes in luminescence sensitivity leads to dispersion in paleodoses and these add to the dispersion caused by heterogeneity both due to incomplete bleaching and in the spatial heterogeneity of the natural beta dose.
5) Given the rapid bleachability of the fast component of Quartz OSL significant sensitivity variation during natural measurements, it is reasonable to suggest that sensitivity change and heterogeneity in the natural beta dose are perhaps more serious causes of dispersion in single grain paleodoses than heterogeneous bleaching. In fact perhaps heterogeneous bleaching has been erroneously implicated as the cause for dispersion in paleo-doses.
6) The observed decrease in the difference between the recovered and expected paleo-doses with increasing R accords with the suggestion of Chauhan and Singhvi (2019). These studies also provide a physical basis of earlier suggestion of the use of super (bright) grains and fast ratio for robust De results (Duller, 2012; Durcan and Duller, 2011). It is hoped that with more data and analysis a robust protocol for single grain based ages can be developed.

6. Conclusion

This study suggests:

1. Sensitivity change during the SAR measurement of single grain paleo-doses are likely to cause systematic errors in the estimation of paleo-doses. This occurs due to change in the OSL sensitivity of a grain during the read out of natural OSL.
2. Thus, a grain specific NCF correction is necessary implying that automated single grain luminescence measurements, may not lead to proper results.

Table-2 a) Samples measured in India. b) Samples measured at Seattle, USA (UW series) and India (SEN-06, DTL-1)

Table-2a	R >3			R >10			R >50			R >100			% change in dose for Grains with R>3 and R>100
Sample Code	De Gy	Number of Grains	% Number of Grains of R>3	De Gy	Number of Grains	% Number of Grains of R>3	De Gy	Number of Grains	% Number of Grains of R>3	De Gy	Number of Grains	% Number of Grains of R>3	
PK-1(fine sand)	0.07±0.02	278	100	0.07±0.01	256	92.09	0.08±0.01	112	40.29	0.11±0.02	19	6.83	57.14
PK-2(fine sand)	0.06±0.01	267	100	0.07±0.01	257	96.25	0.08±0.01	104	38.95	0.13±0.03	18	6.74	116.67
CPR-3(Ch Sand)	0.22±0.04	339	100	0.21±0.05	329	97.05	0.25±0.04	125	36.87	0.39±0.05	22	6.49	77.27
CGR-3(Ch Sand)	0.09±0.01	428	100	0.11±0.01	404	94.39	0.11±0.02	108	25.23	0.17±0.03	17	3.97	88.89
AK-12 (Ch Sand)	0.47±0.04	549	100	0.51±0.03	522	95.08	0.52±0.04	123	22.40	0.94±0.05	14	2.55	100.00
PVR-5(fine sand)	0.23±0.09	376	100	0.26±0.08	361	96.01	0.24±0.09	139	36.97	0.36±0.07	18	4.79	56.52
HWDA-7	1.68±0.53	678	100	1.73±0.49	641	94.54	1.78±0.53	154	22.71	2.48±0.43	9	1.33	47.62
HWDA-8	1.47±0.78	581	100	1.48±0.67	543	93.46	1.53±0.61	178	30.64	2.43±0.52	11	1.89	65.31
HWDA-13	1.92±0.61	586	100	1.96±0.57	545	93.00	1.99±0.61	143	24.40	2.93±0.54	15	2.56	52.60
HWDA-18	1.03±0.87	445	100	1.05±0.81	433	97.30	1.08±0.77	134	30.11	1.68±0.17	22	4.94	63.10
E10_20	1.10±0.02	441	100	1.10±0.02	407	92.29	1.05±0.02	162	36.73	1.15±0.02	50	11.34	4.55
Mound XU	1.26±0.04	221	100	1.28±0.05	186	84.16	1.29±0.11	23	10.41	4.58±0.84	2	0.90	263.49
B-Cap	0.48±0.02	320	100	0.49±0.02	268	83.75	0.51±0.04	34	10.63	2.06±0.07	8	2.50	329.17
A-Lower	1.48±0.03	760	100	1.48±0.03	726	95.53	1.49±0.09	36	4.74	1.59±0.08	6	0.79	7.43

Table-2b **Sample Codes**	**Estimates**	**R <3**	**R >3**	**R >10**	**R >50**	**R >100**
UW3812	N	-	89	63	25	8
	CAM De (Gy)	-	6.8 ± 0.6	6.4 ± 0.6	5.9 ± 0.8	5.8 ± 1.1
	OD (%)	97.0	76.0	76.3	66.5	54.5
UW4066	N	12	73	41	14	6
	CAM De (Gy)	0.8 ± 0.4	0.4 ± 0.1	0.22 ± 0.04	0.12 ± 0.02	0.11 ± 0.02
	OD (%)	119.0	114.0	105.9	38.1	45.6
UW4067	N	7	73	50	16	5
	CAM De (Gy)	2.8 ± 1	0.3 ± 0.1	0.19 ± 0.03	0.13 ± 0.02	0.12 ± 0.02
	OD (%)	69.0	143.0	109.0	45.0	34.5
UW4068	N	5	60	39	13	9
	CAM De (Gy)	1.4 ± 0.6	0.3 ± 0.1	0.2 ± 0.02	0.21 ± 0.02	0.2 ± 0.02
	OD (%)	41.5	118.5	34.5	16.0	19.7
UW4131	N	14	81	37	7	2
	CAM De (Gy)	8.6 ± 1.5	5.9 ± 0.5	5.5 ± 0.6	5.8 ± 1.1	
	OD (%)	28.5	63.5	59.5	49.0	>100
UW4134	N	15	100	49	9	
	CAM De (Gy)	11.4 ± 2.1	9.1 ± 0.6	9.1 ± 0.7	8.8 ± 1.9	No Data
	OD (%)	47.5	60.0	54.5	65.0	
UW4137	N	16	56	35	4	
	CAM De (Gy)	14.9 ± 2.5	13.9 ± 0.9	12.8 ± 1.1	13.5 ± 1.6	No Data
	OD (%)	45.0	44.1	45.2	22.5	
UW3525	N	1	101	71	19	9
	CAM De (Gy)	1.5 ± 3.1	1.8 ± 0.2	1.7 ± 0.2	1.1 ± 0.3	1.1 ± 0.2
	OD (%)	-	79.0	78.0	90.0	35.0
UW3526	N	8	129	87	16	3
	CAM De (Gy)	16.5 ± 3.8	11.8 ± 0.3	12 ± 0.3	13 ± 0.7	14.1 ± 2.5
	OD (%)	36.0	20.7	20.5	21.1	29.5
UW3727	N	76	42	16	2	
	CAM De (Gy)	34.7 ± 2.9	55.7 ± 6.5	74.3 ± 10.7	-	No Data
	OD (%)	56.0	61.5	45.5	NA	
UW3728	N	18	68	27	6	
	CAM De (Gy)	46.4 ± 6.7	48.6 ± 3.2	55.6 ± 4.1	69.3 ± 57.6	No Data
	OD (%)	33.0	37.0	19.5	>200	
SEN-06	N			112	64	23
	CAM De (Gy)	No Data	No Data	63.2 ± 2.4	62.6 ± 3.1	62.6 ± 5.3
	OD (%)			40.0	39.4	39.6
DTL-1	N					
	CAM De (Gy)	No Data				
	OD (%)					

3. A practical solution for a continued use of automated measurement is to use those grains with larger R values. Sensitivity change provides a basis for previously reported approaches (Duller, 2012; Durcan and Duller, 2011).
4. The optical decay curve shape depends on the R value, both due to presence of medium and slow components (Murari, 2008). Optical decay curve of grains with R>3 shows a medium component and those with R >100 approach a near perfect optical decay curve for a robust SAR analysis (Murray et al., 2021).

In conclusion we suggest a community wide assessment of the impact of sensitivity change through a reanalysis of existing data for additional insights. This would include recalculation of ages using the R value and the comparison of optical decay parameters for R > 3 and R > 100.

Such a community effort will ensure that OSL- SAR based single grain ages are robust. It is conceivable that in future studies, the choice of proper R value to isolate those grains that do not suffer from sensitivity change would be determined by, 1) measuring and arranging grains by their R value, 2) recomputing the paleo-dose by successively removing grains with lower R value, and 3) finding a plateau region where removal of grains does not change the dose value. It should be easy to integrate this in age determination protocols.

Acknowledgements

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Contributions

AKS conceived the study. HR carried out the measurements and this work was a part of his doctoral thesis. NC supervised the experimental work, carried out deconvolution calculations and sensitivity segregated analysis. MJ provided the samples from Kaveri basin and shared SAR data. SG and RJW collected samples from termite mounds HR, RJW and AKS collected the Thar Samples. JF measured the data on samples from the USA, Brazil and South Africa. AKS and NC developed the manuscript with all the authors contributing towards the final version.

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