Optically Stimulated Luminescence Dating of Quartz extracts from Red River sediments

OSL-SAR analyses were completed by an Automated Risø TL/OSL–DA–15 system, with excitation from an array of 30 blue (470 ± 20 nm) light-emitting diodes (LEDs) focused onto the quartz aliquot. Optical stimulation of grains was at 125° C with a heating rate of 5 °C/s. All SAR emissions were integrated for the first 0.8 s out of 40 s of stimulation, with background emissions at 30-40 s. The quartz luminescence emissions showed a dominance of a fast component with a > 90% reduction in signal after 3 s of blue-LED excitation. The fast ratio was calculated for each natural and the equivalent emission from a regenerative dose, with aliquots of <15 excluded from *De* analysis (Durcan and Duller, 2011).

Experiments were done to evaluate the effect of preheat temperatures (160, 180, 200, 220, and 240 °C) on isolating a robust, time-sensitive and thermal-transfer emission of the regenerative signal, prior to *De* assessment with SAR protocols. These experiments gave a known dose (5-30 Gy) and then evaluated which preheat resulted in recovery of this dose. There was concordance with the known dose for preheat temperatures above 200 °C for 10 s. A second “cut heat” at 200 °C for 10 s was applied prior to the measurement of the test dose. A final heating at 260 °C for 40 s was given to minimize carryover of luminescence to the succession of regenerative doses. A test for the reproducibility of the radiation-induced SAR ratio (Lx/Tx) was performed routinely by giving the same beta dose for the initial and the final regenerative dose, and evaluating the concordance of the SAR ratios, which should be within 10% (Murray and Wintle, 2003; Wintle and Murray, 2006).

The SAR protocols were used to resolve *De* for sixty-five quartz extracts for eolian and fluvial sediment associated with the Red River (Table A1; Fig. A1). Aliquots were removed from analysis if the fast ratio was < 15 (Durcan and Duller, 2011), the recycling ratio was outside of 0.90 to 1.10, the zero dose was > 5% of the natural signal, if the infrared depletion ratio was > 5%, or the error in *De* value was >10% (Wintle and Murray, 2006). Thus, the statistical significance of an *De* population was determined for 27 to 72 quartz aliquots (Table 1). Error analysis for *De* calculations for a quartz aliquot assumed a measurement error of 1% with 2000 Monte Carlo simulation repeats.

The *De* distributions were log normal and over 90% samples showed De values with overdispersion values between 9 and 23%. Overdispersion values < 20% are routinely assessed for quartz grains that are well solar reset, like aeolian sands (e.g., Wright et al., 2011) and this value is considered a threshold metric for calculation of a De value using the central age model (Galbraith and Roberts, 2012). Overdispersion values >20% may indicate mixing or grains of various ages or partial solar resetting of grains; the minimum age model (MAM; four parameters) may be an appropriate statistical treatment for such data (Fig. A1). However, overdispersion values between 20 and 32% may reflect a single De population, particularly if the De distribution is symmetrical, with the dispersion related to variability associated with micro-dosimetry and/or sedimentary processes (e.g., Arnold & Roberts, 2009). The MAM can effectively model the De components that are time dependent and inherited (Galbraith and Roberts, 2012), particularly using slice sampling statistics (Peng and Forman, 2019). Note all OSL ages are in respect to corrections in beta source collaborations (Autzen et al., 2022).

An assessment of the environmental dose rate (Dr) for each sample is a required metric for calculating a luminescence age. The Dr is an estimate of exposure of quartz grains to ionizing radiation (α, β, and γ) from the decay of the U and Th series, 40K, Rb, and cosmic sources during the burial period. The concentrations of these elements were determined on the bulk sediment by inductively coupled plasma mass spectrometry (ALS Laboratories, Reno, NV). A cosmic ray component, considering location, elevation and depth of strata sampled was calculated which includes the soft component (Prescott and Hutton, 1994; Liang and Forman, 2019). Moisture content (by weight) for the burial period was estimated from present values, sedimentology and in reference to field indicators on the height of the water table.

the Luminescence Dose and Age Calculation (LDAC) platform which unites De and Dr calculations, resolves a final OSL age with appropriate statistical and error analyses (Liang and Forman, 2019). The datum year for all OSL ages is AD 2010.

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| Table A1. Single Aliquot Regeneration protocols for OSL dating (SAR-OSL) | | |
|  | Steps | Time (seconds) |
| 1 | Dose (β) | - |
| 2 | Preheat (160-300) °C | 10 |
| 3 | OSL 125°C | 40 |
| 4 | Test Dose (β) | - |
| 5 | Cut Heat (160-220) °C | 10 |
| 6 | OSL 125°C | 40 |
| 7 | Annealing 260°C | 40 |

Diagram, engineering drawing

Description automatically generatedFig. A1. (a) OSL regenerative growth curves for quartz grains for samples BG4795, BG4982, BG4797 and BG4804 with inset figure showing representative shine-down curve for a natural emission. (b) Radial plots of equivalent dose (D*e*) values for aliquots. Shown is the two sigma standard error for D*e* from Peng and Forman computation (2019).

References

Arnold, L.J., Roberts, R.G., 2009. Stochastic modelling of multi-grain equivalent dose (D-e) distributions: Implications for OSL dating of sediment mixtures. *Quaternary Geochronology* 4, 204-230.

Autzen, M., Andersen, C.E., Bailey, M. Murray, A.S., 2022. Calibration quartz: An update on dose calculations for luminescence dating. *Radiation Measurements* 157, 106828.

Durcan, J.A., Duller, G.A.T., 2011. The fast ratio: A rapid measure for testing the dominance of the fast component in the initial OSL signal from quartz. *Radiation Measurements* 46, 1065-1072.

Fain, J., Soumana, S., Montret, M., Miallier, D., Pilleyre, T., Sanzelle, S., 1999. Luminescence and ESR dating-Beta-dose attenuation for various grain shapes calculated by a Monte-Carlo method. *Quat. Sci. Rev.* 18, 231-234.

Galbraith, R.F., Roberts, R.G., 2012. Statistical aspects of equivalent dose and error calculation and display in OSL dating: An overview and some recommendations. *Quaternary Geochronology* 11, 1-27.

Jain, M., Botter-Jensen, L., Singhvi, A. K., 2003. Dose evaluation using multiple-aliquot quartz OSL: test of methods and a new protocol for improved accuracy and precision. *Radiation Measurements* 37**,** 67-80.

Mejdahl, V. & Christiansen, H.H., 1994. Procedures used for luminescence dating of sediments. *Boreas* 13, 403-406.

Murray, A.S., Wintle, A.G., 2003. The single aliquot regenerative dose protocol: potential for improvements in reliability. *Radiation Measurements* 37, 377-381.

Peng, L, Forman, S. L., 2019. LDAC: An Excel-based program for luminescence equivalent dose and burial age calculations. *Ancient TL* 37 (2), 21-40; DOI: ancienttl.org/ATL\_37.htm#December.

Prescott, J.R. Hutton, J.T., 1994. Cosmic ray contributions to dose rates for luminescence and ESR dating: large depths and long-term time variations. *Radiation Measurements* 23, 497-500.

Wintle, A.G., Murray, A.S., 2006. A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating protocols. *Radiation Measurements* 41, 369-391.