Optically stimulated luminescence dating of heat retainer hearths from the Sahara: Insights into signal accumulation and measurement.

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Abstract

Heat retainer hearths are a prominent component of the Holocene archaeological record of a number of drylands. Rocks within these hearths were fired in antiquity, emptying the optically stimulated luminescence (OSL) source traps of mineral grains within the rock. Since partial bleaching and mixing of grains within a lithified heat retainer is impossible, these rocks offer the opportunity to test our understanding of OSL signal accumulation and measurement processes. First, we show that OSL ages calculated using grains size fractions from 4-11 µm up to 180-210 µm are indistinguishable for a single heat retainer, indicating that the environmental and instrumental dose rate correction factors routinely used in luminescence dating are accurate. Second, we used single-grain dose recovery and equivalent dose measurements to determine the overdispersion due to beta microdosimetry. For the heat retainers measured in this study, overdiseprsion due to beta microdosimetry ranges from 8.9 ± 1.8 to 20.3 ± 1.6 %. Third, we investigate the impact of mechanical crushing on the measured equivalent dose from quartz, to test the potential of using this technique to liberate dateable material from heat retainers which are not acid soluble. A small (<1 Gy) but significant increase in equivalent dose is observed in crushed zero-age samples, but the equivalent doses of crushed and uncrushed Holocene quartzes are indistinguishable. We conclude that crushing is a viable method for extracting dateable material from a heat retainer, but that some knowledge of the dosimeter's mean grain size is required for calculation of an accurate environmental dose rate.

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1. Introduction

Heat retainer hearths are a prominent component of the Holocene archaeological record, and are particularly prevalent in the Sahara and Arabian Peninsula, where they are frequently associated with surface scatters which attest to the presence of ancient nomadic pastoralists. Rocks, which acted as heat retainers while the hearth was in use, are heated to sufficiently high temperatures to completely empty the OSL source traps. Consequently, mineral grains from within these heat retainer rocks are dateable using optically stimulated luminescence (OSL) techniques, providing an estimate of the time elapsed since last firing (Armitage and King, 2013; Rhodes et al., 2010; Rhodes et al., 2009). These fired heat retainers are often the only dateable and unequivocally anthropogenic component of the surface archaeological record. OSL dating of hearths is therefore potentially an important tool for understanding the nature of the surface archaeological record in drylands. Furthermore, fired hearth stones offer a number of advantages when studying the accumulation of the OSL signal. First, since the heat retainers are fired in a lithified state, and since post-firing mixing of mineral grains within a heat retainer is impossible, OSL dating of multiple grain-size fractions from a single rock allows the accuracy of grain-size specific corrections to environmental and instrumental dose rates to be tested. Second, since post-firing mixing of mineral grains is impossible, and since Armitage and King (2013, their Figure 5) have demonstrated that sunlight does not deplete the OSL signal in the inner portion of hearth rocks, sand sized quartz extracted from hearth rocks may be used to investigate single-grain equivalent dose (D_e) overdispersion due to beta microdosimetry. In the present study we: 1) Conduct paired single-grain dose recovery and D_e measurement experiments to investigate the degree of overdispersion caused by beta microdosimetry; 2) Calculate ages for a range of grain-size fractions extracted from a single heat retainer to test the internal consistency of the OSL method, and 3) Investigate the possibility of extending the OSL dating technique to non-carbonate lithologies where mineral grains cannot be extracted from a heat retainer by acid dissolution.

2 Materials and methods

A total of thirteen heat retainers were collected from five hearths at the Al Wafa archaeological site, in the Libyan Sahara. A full site description is found in Armitage and King (2013). Briefly, Al Wafa is a gently sloping basin characterised by shallow, ephemeral drainage channels cut into an extensively deflated surface composed of unconsolidated sand protected by gravel-pebble sized carbonate clasts. The landscape is dominated by ~100 discrete hearths, which are usually seen as mounds rising a few tens of centimetres above the surrounding land surface (Figure 1), and date from ~7-9 ka (Armitage and King, 2013). These mounds are believed to result from topographic inversion,

with the hearths originally consisting of cobble lined pits dug into soil, which was subsequently deflated when the Sahara dried ~5 ka (Armitage et al., 2015). The cobbles, which served as heat retainers when the hearth was in use, are clasts of sandy limestone, which outcrops at the margin of the Al Wafa basin. Approximately cuboid heat retainers with no visible fracturing were sampled from discrete hearths.

The outer light exposed portions of the heat retainers was removed by repeated immersion in 1.16 M HCl, until the length of each axis of the clast had been reduced by a minimum of 25 mm. The dating sample was obtained by dissolution of the remaining material. Quartz separates were produced using the methods summarised in Supplemental Material Section A. For samples FZ39-48, only the 210-180 µm fraction was prepared for measurement. For samples FZ84, 85, 95 and 96, dating samples were prepared from the 4-11, 11-20, 20-40, 40-60, 60-90, 90-125, 125-150, 150-180 and 180-210 µm fractions. All size fractions were mounted on 10 mm aluminium discs. Additional single-grain analysis (212-180 µm grains) was performed using standard Risø single-grain sample holders.

All OSL measurements presented in this study were carried out using a Risø TL/OSL-DA-15 dating system (Bøtter-Jensen et al., 2003), fitted with a single-grain OSL attachment (Duller et al., 1999). Optical stimulation of single aliquots was carried out using a blue (470 ± 30 nm) light emitting diode (LED) array with a power density of 33 mW/cm^2, while single-grains were stimulated using a 10 mW Nd : YVO_4 solid-state diode-pumped green laser (532 nm) focussed to yield a nominal power density of 50 W/cm^2. Infra-red (IR) stimulation was carried out using an IR (870 nm) laser diode array. OSL was measured using an Electron Tubes Ltd 9235QB15 photomultiplier tube with 7.5 mm of Hoya U-340 filter interposed between the sample and photomultiplier. Irradiation was carried out using a 40 mCi ^{90}Sr/^{90}Y beta source, calibrated relative to the National Physical Laboratory, Teddington ^{60}Co γ-source (Hotspot 800) (Armitage and Bailey, 2005). Due to the spatial heterogeneity of beta emitters across the active face of the ^{90}Sr/^{90}Y beta source, it was necessary to apply a grain position correction to single-grain D_e values, using the method of (Armitage et al., 2011).

Equivalent doses were determined using the single-aliquot regenerative-dose (SAR) method (Murray and Wintle, 2000). Following Armitage and King (2013), who worked on heat retainers from the Al Wafa site, we adopted a preheating regime of 260 °C, 10 s for PH1 (the pre-heat prior to measurement of natural or regenerated luminescence) and 220 °C, 10 s for PH2 (the pre-heat prior to measurement of the test dose luminescence response) for all measurements. OSL measurements were made at 125 °C, using blue diodes (40 s) for aliquot measurements or a green laser (2 s per grain) for single-grain measurements. For blue diode stimulation, the OSL signal was that recorded during the first 0.36 s of

stimulation, and a background signal from the last 4 s of stimulation was subtracted. For green laser stimulation, the OSL signal was that recorded during the first 0.3 s of stimulation, and a background signal from the last 0.3 s of stimulation was subtracted (Thomsen et al., 2005). Curve fitting, and D_e determination, were performed using version 3.24 of the Luminescence Analyst software (Duller, 2007). We adopted the single-grain rejection criteria of Armitage et al. (2011), Supplementary Material Section B.

3 Age estimates generated using different grain-size fractions

In principle, any grain-size fraction from fine silt to fine sand can be used to measure the equivalent dose of a sample. For any given sample, the grain size used may be dictated by availability or the post-depositional processes which a sample has experienced e.g. silt may be translocated by percolating water in a sandy sediment, but be the only clast size available in a loess deposit. Consequently, sedimentary characteristics and geomorphic setting often necessitates the measurement of different grain size fractions for samples within the same dating study. However, numerous factors determining the environmental or laboratory dose rate experienced by the luminescence dosimeter vary with grain size. These variations may be progressive, e.g. the ~13% increase in beta dose rate from a Risø irradiation unit as grain size increases from 4-11 µm to >63 µm (Armitage and Bailey, 2005) or abrupt e.g. at the transition between sand-sized material, which are routinely etched to remove the alpha dosed rind, and silt-sized material. If a single dating project uses more than one grain size fraction, then the minimum requirement for a successful study is that different grain sizes yield the same luminescence age for sediments which were deposited simultaneously. In practice, the types of sedimentary deposit which are normally targeted for OSL dating tend to be well sorted. Conversely, the heat retainers from Al Wafa contain quartz grains from <4 to >210 µm in diameter, all of which must have a common OSL age, making these samples ideal for testing the internal consistency of OSL ages produced using different grain size fractions.

Twelve aliquots of quartz from each size fraction from each of four samples were measured using a single luminescence reader. Across the range of grain sizes measured, progressive variations in environmental alpha (Brennan et al., 1991) and beta (Guérin and Mercier, 2012) attenuation factors, and laboratory beta dose rate (Armitage and Bailey, 2005) occurred. An abrupt alteration in dose rate occurred between the 60-90 and 90-125 µm fractions due to HF etching of >90 µm grains, resulting in the loss of 100% of the environmental alpha dose (making the common assumption of isotropic etching), and 1% of the environmental beta dose (Brennan, 2003). An abrupt change in laboratory dose rate is also present between 20-40 and 40-60 µm, since the <40 µm grains were deposited from suspension, coating the entire face of the disc, whereas the >40 µm grains were attached using

Silkospray applied via a 5 mm diameter mask. However, since the instrument was calibrated using samples mounted by the same methods, no correction for the known variation in instrumental dose rate between the core and periphery of the disc was made (Ballarini et al., 2006).

Equivalent dose, dose rate and calculated age are presented for nine different grain size fractions in the range 4-210 μm (Tables S2-S4). For sample FZ84 (Figure 2), dose rate falls steadily as grain size increases, from a value of 0.98 ± 0.07 Gy/ka (4-11 μm) to 0.83 ± 0.05 Gy/ka (180-212 μm), a decrease of ~18 %. For all four of the samples for which data are available, equivalent dose also falls with increasing grain size, though for each sample there are outliers from this general trend (e.g. 11-20 μm in Figure 2). Nonetheless, ages calculated for each grain size fraction from a single sample are consistent with each other. This result indicates that for these samples at least, the dose rate correction factors used in this study produce consistent ages across the full range of grain sizes commonly adopted for luminescence dating work.

4 Single-grain overdispersion due to beta microdosimetry

Overdispersion (OD), the relative spread of equivalent doses after measurement uncertainties are excluded, is a key input for several of the statistical models used in single-grain dating e.g. the Minimum Age Model (MAM) (Galbraith et al., 1999) and the Finite Mixture Model (FMM) . However, successful application of these models requires an estimate of the OD of a well-bleached sample, which is otherwise identical to the sample being measured. Incorrect estimation of the well-bleached OD may lead to the adoption of the incorrect statistical model, and the production of an incorrect age. For example, an erroneously young age would be calculated if an underestimate of the well-bleached OD caused the MAM to be applied to a sample that was not partially bleached. The heterogeneous distribution of beta activity within a sediment, e.g. where occasional potassium feldspars are present within a quartz rich sand (Mayya et al., 2006), has the potential to cause such an underestimation of the well-bleached OD. Although some success has been achieved in modelling OD due to beta microdosimetry (Cunningham et al., 2012; Guérin et al., 2015; Martin et al., 2015), few studies have measured OD in ancient sediments where other causes may be excluded or isolated. Heat retainers which were fired in antiquity offer the opportunity to isolate OD due to beta microdosimetry, since lithified samples cannot experience post-depositional mixing, and firing completely empties the OSL source traps (Armitage and King, 2013, section 8), eliminating the possibility of heterogeneous bleaching. Consequently, OD observed in a D_e dataset (OD_{De}) from a heat retainer consists of contributions from the measurement process (e.g. instrumental variability and intra-sample variation in luminescence characteristics) and from beta microdosimetry. Since the OD observed in a dose recovery dataset (OD_{Rec}) is caused by the measurement process alone, it is

possible to isolate OD due to beta microdosimetry (OD_{β}) by subtracting quadratically OD_{Rec} from OD_{De}.

Dose recovery measurements were made on six samples from Al Wafa. Following Armitage and King (2013), single-grain dose recovery experiments were performed by heating samples to 350 °C for 120 s (to empty the OSL source traps) after which a beta dose similar to D_e (the "known dose") was applied. The "recovered dose" was then measured, using an identical measurement sequence to that used for measuring the equivalent dose. Single-grain equivalent dose, dose recovery and OD values are presented in Table S6. The "dose recovery ratios" (recovered dose/known dose) ranged from 0.96 ± 0.01 to 1.00 ± 0.01, with a mean value of 0.99 ± 0.01, indicating that the measurement parameters used in this study are appropriate for the Al Wafa samples. OD_{rec} values ranged from 6.3 ± 0.6 % to 9.7 ± 1.1 %, with a mean value of 7.6 ± 1.2 %. OD_{β} values ranged from 15.8 ± 1.8 to 27.1 ± 1.6 %, with a mean value of 22.6 ± 4.1, while OD_{De} values ranged from 18.5 ± 1.5 % to 28.2 ± 1.5 %, with a mean value of 23.9 ± 3.6 %.

The wide range of OD_{β} values from heat retainers from a single location, and derived from a single geological unit, suggests that the effects of beta microdosimetry is highly sample dependant. This implies that, even for multiple samples from a single location, the well-bleached OD required for the application of several statistical models may need to be determined for each individual sample. In addition, only one of the six samples measured yielded an OD_{De} value below 20%. Several studies (e.g. Armitage et al., 2011) have used an OD_{De} value of 20% to distinguish between well-bleached, undisturbed samples (OD_{De}<20%) and samples which have been subject to partial bleaching or post depositional mixing (OD_{De}>20%). Our results contribute to a growing body of literature (Guérin et al., 2015; Thomsen et al., 2016) which does not support the use of a 20% OD_{De} threshold. Instead a site-specific, or possibly even a sample specific, OD_{De} estimate for a well-bleached sample is required. In the absence of such an estimate, partial bleaching or post depositional mixing should be diagnosed based upon a knowledge of the sample's depositional context and burial history. This approach risks confirmation bias (the tendency to observe effects which are expected to be present), and highlights the need for a robust method for estimating the OD expected from a well-bleached sample.

5 Extending the OSL dating technique to non-carbonate lithologies

Where heat retainers are composed of carbonate rock, extraction of dateable material is easily achieved using progressive HCl dissolution to remove the outer light-exposed portions of the rock (e.g. Armitage and King, 2013). Conversely non-carbonate heat retainers have been subject to more

complicated preparation procedures. For example, Rhodes et al. (2010) removed the light exposed portion of the rock using a geological hammer, followed by two cycles of mechanical crushing (one producing pebble sized fragments, and the other yielding sand sized fragments) and a short HF etch to remove surface crushing effects and feldspar. This latter approach has the disadvantage of being time-consuming and, depending upon the mineralogical composition of the parent rock, potentially yielding a polymineral sample with unpredictable luminescence characteristics. An alternative approach is to remove the outer portions of the rock with a geological hammer, and crush the remaining dating fraction in a mill, preparing the resulting powder using the standard method for 4-11 μm silt. The advantages of this approach is that it is less labour intensive, is likely to yield a more predictably pure quartz separate, and presents the sample to the luminescence reader in a predictable geometry. As with the method of Rhodes et al, (2010), this method requires a realistic approximation of the mean grain size of the dosimeter before crushing to calculate an accurate dose rate. More significantly, a crushing induced increase in signal has been observed for burnt flint samples (Aitken, 1985), section 7.1), while recent evidence suggests that crushing or shearing may either reduce or reset the natural luminescence signal (Bateman et al., 2012; Swift et al., 2011).

To test the possibility that milling induces a luminescence signal, a selection of seven zero age quartz separates were measured in their crushed and uncrushed states. Similarly, to test the possibility that milling reduces the luminescence signal, a selection of five quartz separates from Holocene dune samples (D_e ranging from 2-20 Gy) were measured in their crushed and uncrushed states. Finally, to test the utility of crushing as a method for extracting dateable quartz from non-carbonate heat retainers, small chips from the core of the four heat retainers reported in Section 3 were crushed, and the De compared to that for material extracted via sequential dissolution in HCl. Crushed samples were prepared by loading ~2 g of material into a light-tight stainless steel grinding jar, and grinding at 20 Hz for 120 s using a Retsch Mixer Mill MM400. The 4-11 μm fraction was subsequently extracted from the milled material using the method described in Supplemental Material Section A. Results are presented in Supplementary Material Tables 3-5.

Of the seven zero age samples measured (Table S7), four showed a significant increase in equivalent dose upon crushing, while three yielded indistinguishable values. The mean increase in equivalent dose due to crushing was 0.25 ± 0.14 Gy, ranging from 0.08 ± 0.05 Gy for a Libyan desert dune sand (Armitage et al., 2007) to 0.93 ± 0.33 Gy for a South African coastal dune sand (this study). From these data it is apparent that crushing can induce a significant increase in D_e, but that the effect is neither universal nor uniform. None of the five Holocene samples yielded crushed/uncrushed ratios distinguishable from unity (Table S8), suggesting that any signal increase due to crushing is either

negligible or masked by other sources of D_e scatter. Crushed material from the cores of the four heat retainers yielded equivalent doses consistent with the range of values obtained from uncrushed material (Tables S2-S5). However, to determine an age, the mean grain size of the uncrushed quartz must be known to determine the applicable dose rate. Without this information i.e. assuming mean grain size of the dosimeter to be somewhere between 4 and 210 μm, a large uncertainty term needs to be used to account for the ~15 % variation in environmental dose rate between these grain sizes. In the case of the four heat retainers measured here, the effective grain size of the quartz prior to crushing may be determined by calculating the dose rate required for the equivalent dose of the crushed material to yield the mean age determined using the various grain size fractions presented in Section 3. This analysis indicates that heat retainers contained grains with a mean effective size of 40-60 μm (n=2), 90-150 μm (n=1) and >210 μm (n=1). For the samples in question, environmental dose rates decrease by 7.7 ± 0.8 % from 40-60 to 180-210 μm. Consequently, estimation of the mean grain size either by visual inspection or petrography may yield acceptably precise estimates of the environmental dose rate. These data suggest that crushing is a viable technique for extracting dateable mineral separates from heat retainers with non-carbonate lithologies.

6 Conclusions

Heat retainers from ancient hearths offer the opportunity to test our understanding of OSL signal accumulation and measurement processes. We measure equivalent doses from a range of grain sizes commonly adopted in luminescence dating studies. For each hearth stone, different size fractions yield similar ages, indicating that for our samples at least, the instrumental and environmental dose rate correction factors used in luminescence dating yield internally consistent (accurate?) results. In addition, since mineral grains within heat retainers cannot be subject to partial bleaching or post firing mixing, overdispersion in single-grain data from these samples is due to measurement processes and beta microdosimetry. The latter may be isolated by subtracting quadratically the single-grain overdispersion obtained from a dose recovery experiment on the same sample. This approach yielded values for overdispersion resulting from beta microdosimetry ranging from 15.8 ± 1.8 to 27.1 ± 1.6 %. Our results suggest that the application of a standard overdispersion threshold (e.g. 20%), above which processes such as mixing or partial bleaching may be diagnosed, may not yield accurate results. Lastly, the possibility of extracting dateable material from heat retainers via mechanical crushing was explored. A small but significant increase in equivalent dose was observed for a number of quartz separates from zero-age samples after crushing. However, it was not possible to distinguish the equivalent dose from crushed and uncrushed fractions of five Holocene samples. These results suggest that crushing is a viable method for extracting dateable material from a rock, but we note that

some knowledge of the dosimeter's mean grain size is required for calculation of an accurate environmental dose rate.

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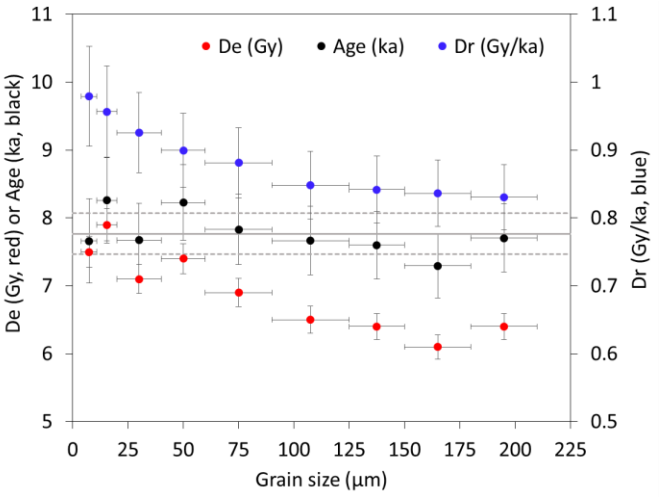
Figures

Figure 1: A small hearth at Al Wafa. The hearth mound is ~1 m in diameter.



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291 Figure 2: Equivalent dose (D_e), environmental dose rate (D_r) and calculated age for different grain
292 size fractions of quartz from heat retainer sample FZ84. The thick grey line represents the mean
293 age, calculated using data from all grain sizes, while the dashed lines represent ± 1 standard
294 deviation.

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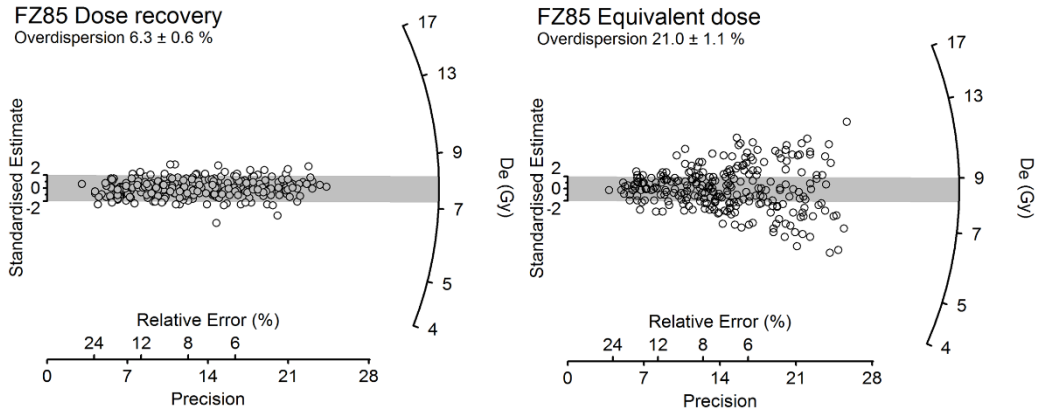

Figure 3: Radial plots of equivalent doses for individual quartz grains from heat retainer sample FZ85. The grey bars are centred at the equivalent dose calculated using the central age model (Galbraith et al., 1999). The left radial plot represents data from a dose recovery experiment, whereas the right panel shows the natural equivalent dose distribution.

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