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Single-grain TT-OSL bleaching characteristics: Insights from modern analogues and OSL dating comparisons

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ABSTRACT

Previous assessments of thermally transferred optically stimulated luminescence (TT-OSL) signal resetting in natural sedimentary settings have been based on relatively limited numbers of observations, and have been conducted primarily at the multi-grain scale of equivalent dose (D_e) analysis. In this study, we undertake a series of single-grain TT-OSL bleaching assessments on nineteen modern and geological dating samples from different sedimentary environments. Daylight bleaching experiments performed over several weeks confirm that single-grain TT-OSL signals are optically reset at relatively slow, and potentially variable, rates. Single-grain TT-OSL residual doses range between 0 and 24 Gy for thirteen modern samples, with > 50% of these samples yielding weighted mean D_e values of 0 Gy at 2σ. Single-grain OSL and TT-OSL dating comparisons performed on well-bleached and heterogeneously bleached late Pleistocene samples from Kangaroo Island, South Australia, yield consistent replicate age estimates. Our results reveal that (i) single-grain TT-OSL residuals can potentially be reduced down to insignificant levels when compared with the natural dose range of interest for most TT-OSL dating applications; (ii) the slow bleaching properties of TT-OSL signals may not necessarily limit their dating applicability to certain depositional environments; and (iii) non-trivial differences may be observed between single-grain and multi-grain TT-OSL bleaching residuals in some modern samples. Collectively, these findings suggest that single-grain TT-OSL dating may offer advantages over multi-grain TT-OSL dating in certain complex depositional environments.

1. Introduction

The favourable dose saturation properties of thermally transferred optically stimulated luminescence (TT-OSL) signals offer potential for establishing extended-range luminescence chronologies that exceed the traditional upper age limits of quartz OSL dating (e.g., Wang et al., 2006; Duller and Wintle, 2012; Arnold et al., 2015). However, TT-OSL signals have been shown to be optically reset at a considerably slower rate than conventional OSL signals (e.g., Duval et al., 2017), meaning there is greater potential for insufficient signal resetting and associated TT-OSL age overestimation in any dating study. TT-OSL bleaching characteristics have been assessed using several approaches in the recent literature. Daylight bleaching experiments performed on a small number of samples have shown that several weeks or months of natural sunlight exposure are typically required to deplete TT-OSL signals to within 10% of background (e.g., Jacobs et al., 2011; Arnold et al., 2013; Demuro et al., 2015). However, similar sized TT-OSL signal reductions have been observed over much shorter (< 1 h) daylight exposure times for some samples (Athanassas and Zacharias, 2010). TT-OSL depletion rates on the order of multiple days have also been reported from several

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solar simulator bleaching studies (e.g., Tsukamoto et al., 2008; Hernandez et al., 2012; Brown and Forman, 2012; Duval et al., 2017), albeit using different experimental conditions and simulated daylight intensities. In spite of these generally slow optical bleaching rates, equivalent dose (D_e) assessments performed on modern and very young samples suggest that adequate TT-OSL signal resetting down to sufficiently low levels is possible in some sedimentary environments. Multi-grain residual D_e values of 5–19 Gy have been reported for several modern aeolian sediments from Eurasia and South Africa (see Duller and Wintle, 2012). Arnold et al. (2014) reported a similarly sized multi-grain D_e of 7.3 ± 0.8 Gy for a modern slopewash and aeolian deposit from north-central Spain, while multi-grain residual D_e values of several tens of Gy have been obtained for coastal and lacustrine shoreline deposits from South Africa and Australia (Jacobs et al., 2011; Fu et al., 2017). In contrast, very large multi-grain TT-OSL residual doses of 250–300 Gy have been reported for modern suspended sediments and overbank deposits from the Yellow River (Hu et al., 2010), potentially cautioning against the suitability of TT-OSL dating in turbid and UV-depleted fluvial settings.

While these various TT-OSL bleaching assessments have proved insightful, they are based on a relatively modest number of observations (n = < 20 samples) and further work is needed to better characterise TT-OSL signal resetting across a broader range of natural contexts using complementary types of experimental procedures. Additionally, all existing assessments of TT-OSL bleaching characteristics, with the exception of one study (Fu et al., 2017), have been performed at the multi-grain scale of D_e analysis. It remains unclear, therefore, whether TT-OSL residual doses reported in existing modern analogue studies partly reflect averaging effects arising from simultaneously measuring grains with different bleaching histories, signal compositions or TT-OSL source trap properties. For samples with inherently bright signal intensities, single-grain TT-OSL dating offers the potential to evaluate, or even circumvent, any potential averaging effects. Single-grain TT-OSL has recently been applied at several independently dated archaeological sites from Spain and Australia (e.g., Demuro et al., 2014; Arnold et al., 2015; Hamm et al., 2016). These single-grain studies have also revealed that multi-grain TT-OSL signals may be dominated by grains with unfavourable TT-OSL behaviours (e.g., Arnold and Demuro, 2015) and that apparent multi-grain TT-OSL residual doses of several tens of Gy may result from the inclusion of grain types that are routinely rejected by single-grain quality assurance criteria (Fu et al., 2017). Such complications require further examination, and additional single-grain bleaching assessments are needed to better characterise TT-OSL signal resetting at the most fundamental scale of D_e analysis.

The aims of the present study are threefold: (i) To examine the TT-OSL bleaching characteristics of quartz samples from a range of depositional environments using three complementary approaches; namely, daylight bleaching experiments, examination of modern sample D_e datasets, and comparisons of replicate TT-OSL and OSL ages for geological dating samples. The first two of these approaches permit examination of TT-OSL resetting properties under controlled bleaching conditions and in analogous natural depositional contexts, while the latter favours assessments of bleaching histories that are directly relatable to individual dating samples; (ii) To assess whether the bleaching properties of TT-OSL signals limit their dating applicability to certain depositional settings, environmental conditions or age ranges; (iii) To compare TT-OSL residual doses and bleaching trends at different scales of D_e analysis.

2. Sample details and experimental procedures

This study incorporates nineteen samples collected from a diverse range of depositional environments across Spain and Australia (Fig. S1). These two geographic regions have been targeted for their generally bright single-grain quartz TT-OSL signal characteristics (e.g., Arnold et al., 2015; Hamm et al., 2016), while individual sites within these regions have been selected to encompass a variety of natural bleaching conditions. Thirteen samples were collected from actively accumulating, or very recently accumulated, surface sediment deposits that were expected to yield burial doses close to, or consistent with, 0 Gy (assuming adequate signal bleaching during transportation). These samples represent modern analogues for associated archaeological, palaeontological and palaeoenvironmental dating samples being studied as part of recent or ongoing TT-OSL dating projects (e.g., Arnold et al., 2014; Demuro et al., 2014; Fu et al., 2017). Two shallow cave infill samples from the middle Pleistocene palaeoanthropological sites of Galería and Sima del Elefante, Atapuerca, (ATG10-3, ATE10-13) have been chosen for the daylight bleaching experiments, owing to their relatively high and comparable mean burial doses, and uniformly bleached single-grain TT-OSL D_e distributions (e.g., Demuro et al., 2014; Arnold et al., 2015). Single-grain TT-OSL and OSL dating comparisons were performed on four late Pleistocene samples from southern Kangaroo Island that exhibit different types of OSL D_e distributions, and that lie within typically routine OSL dating ranges (mean D_e values = 17–103 Gy). Two of these samples (KHC-KI5, KI14-5) were collected from relatively deep exogenous infill deposits preserved within Kelly Hill Cave (McDowell et al., 2013), and located ~25 m from the nearest palaeoentrance (Arnold et al., in prep). A third sample (KI14-12) was collected from a proximal (shallow) exogenous infill deposit preserved immediately beneath a former external opening of the same cave system. The fourth sample (KI14-1) was derived from a well-bedded coastal aeolianite deposit (Bridgewater Formation) found at the Boar Beach trace fossil site (Camens et al., 2017).

To achieve the main study aims, we have chosen to focus on single-grain TT-OSL and OSL analyses, which enable in depth assessments of bleaching adequacy in the absence of potential grain averaging effects. The details of the TT-OSL and OSL dating procedures employed in this study, including the quality assurance criteria used to eliminate unreliable grains, are provided in Arnold and Demuro (2015), Arnold et al. (2016) and the Supporting Information (Fig. S2-3; Table S1-3). D_e values were determined for individual quartz grains using the single-aliquot regenerative-dose (SAR) procedures shown in Table S1. Table S3 summarises the environmental dose rates for the Kangaroo Island dating samples, calculated using a combination of *in situ* field gamma-ray spectrometry (Arnold et al., 2012) and low-level beta counting (Bøtter-Jensen and Mejdahl, 1988).

3. TT-OSL daylight bleaching tests

To investigate the effects of controlled daylight exposure on single-grain TT-OSL D_e datasets, we bleached subsets of prepared quartz grains from samples ATG10-3 and ATE10-13 for 42 days on a south-facing exterior window ledge in Burgos, Spain. The original (unbleached) D_e datasets for these two samples exhibit relatively low overdispersion of 23–27%, and the majority of individual D_e estimates are consistent with single dose populations centred on central age model (CAM) D_e values of 540–572 Gy (Fig. 1a–b). The unlogged D_e datasets exhibit multiplicative D_e uncertainty properties (Fig. S4), and are normally distributed (ATG10-3) or slightly positively skewed (ATE10-13) according to the criterion outlined by Bailey and Arnold (2006) (Table S4).

After 6 weeks of daylight exposure, the weighted mean (CAM) D_e values for both samples were reduced by ~90% (Fig. 1c–d; Table S4). These depletion rates are consistent with that obtained for a multi-grain TT-OSL sample by Demuro et al. (2015) under analogous experimental conditions. Though both samples retain weighted mean (unlogged CAM; CAM_{UL}) residual D_e values of 54–65 Gy, complete resetting of burial doses is possible for a significant proportion of the measured grains in each sample. Between 38 and 52% of the daylight-bleached grains have D_e values consistent with 0 Gy at 2σ after 42 days of daylight exposure (Table S4). The D_e distributions are also characterised by higher overdispersion values of 49–57% and significantly enhanced

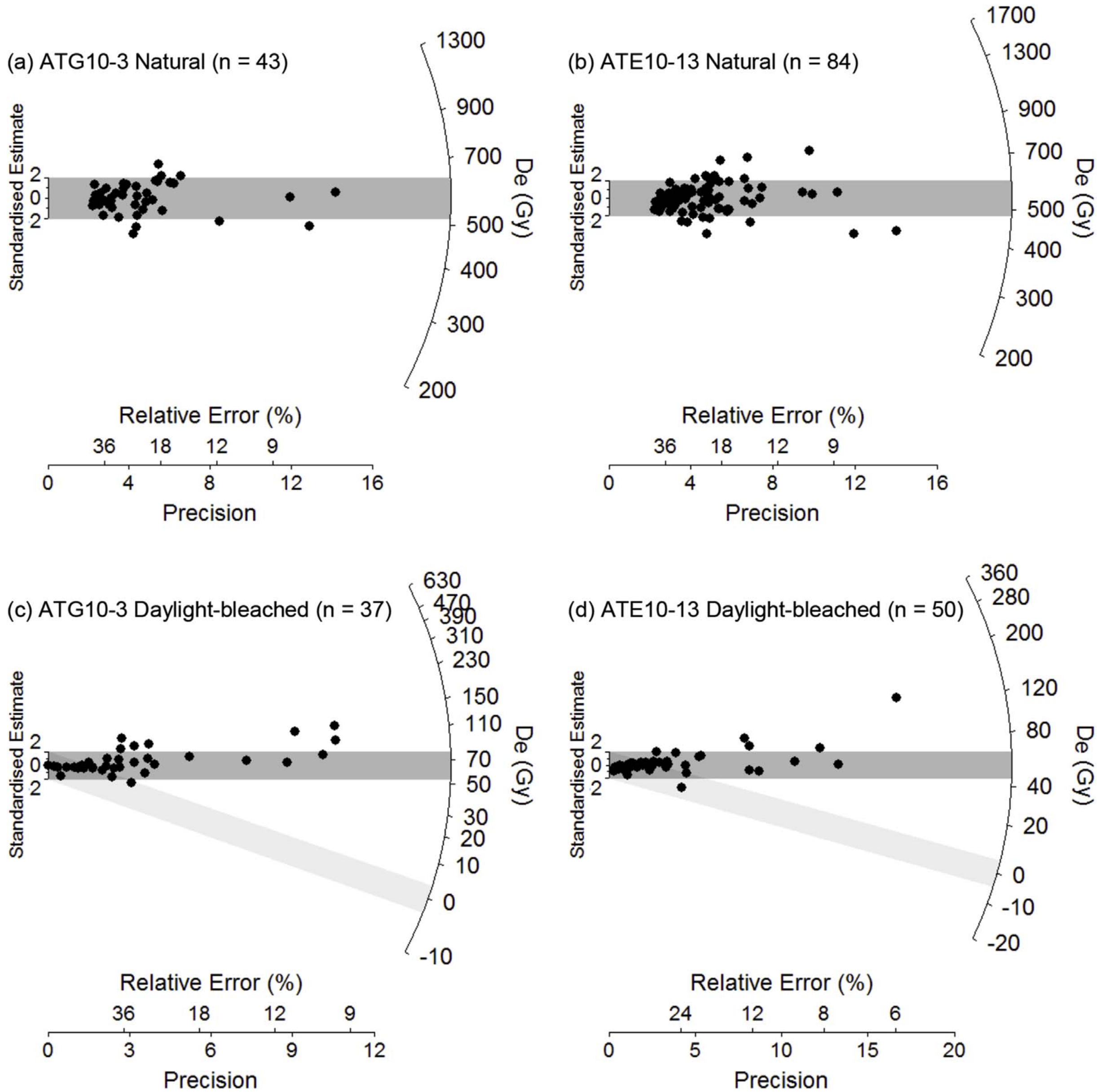

Fig. 1. Natural and daylight-bleached single-grain TT-OSL D_e distributions for samples ATG10-3 and ATE10-13 from Atapuerca, Spain. Daylight bleaching experiments were conducted on monolayers of prepared quartz grains during July–August in Burgos, Spain (N 42° 21′ 00″ W 03° 42′ 24″, 860 m.a.s.l.). The daylight-bleached grains were agitated every few days to ensure homogenous exposure of all grain surfaces during the 42 day bleaching period. The dark grey bands are centred on the weighted mean D_e values, which have been calculated using the CAM for the natural D_e datasets and the CAM_{UL} for the daylight-bleached D_e datasets. The light grey bands in plots (c) and (d) are centred on the target residual dose of 0 Gy. Radial plots (c) and (d) have been plotted using a modified log transformation of $z = \log(D_e + a)$ (Galbraith and Roberts, 2012), to more easily accommodate both the large and small (negative and near zero Gy) D_e values observed in these datasets. The standard errors of these modified log transformed datasets are given relative to $D_e + a$, where $a = 20$ Gy for the daylight-bleached datasets of ATG10-3 and $a = 30$ Gy for the daylight-bleached dataset of ATE10-13.

positive skewness, and therefore appear to resemble heterogeneously bleached single-grain D_e datasets (e.g., Olley et al., 2004; Arnold et al., 2009).

It is difficult to determine whether these heterogeneous D_e distribution characteristics reflect genuine inter-grain differences in TT-OSL signal depletion rates or whether they are a reflection of pre-existing inter-grain differences in natural D_e values prior to bleaching. The former interpretation may be supported by published evidence suggesting that (i) TT-OSL signals are composites of multiple signal components with different detrapping probabilities (e.g., Tsukamoto et al., 2008; Brown and Forman, 2012; Demuro et al., 2015), and that (ii) inter-grain differences in TT-OSL behaviours (e.g., source traps and signal stabilities) are common in at least some samples (e.g., Arnold and Demuro, 2015; Duval et al., 2017; Bartz et al., this volume). Further support comes from Table S4, which shows that the higher overdispersion and enhanced skewness of the daylight-bleached datasets cannot be recreated by simply scaling the original D_e datasets by the average depletion rates measured in the bleaching experiments (0.11 ± 0.01 for ATG10-3 and 0.10 ± 0.01 for ATE10-13). It is also possible, however, that some of the enhanced overdispersion in the daylight-bleached D_e datasets may be caused by the increasing influence of intrinsic sources of D_e scatter over low dose ranges (e.g., different responses of individual grains to the SAR conditions). Fig. S4 shows that the daylight-bleached D_e datasets exhibit distinctly different D_e uncertainty properties in comparison to the natural D_e datasets (additive rather than multiplicative D_e uncertainty relationships), reflecting the dominance of different types of experimental D_e scatter over low dose ranges.

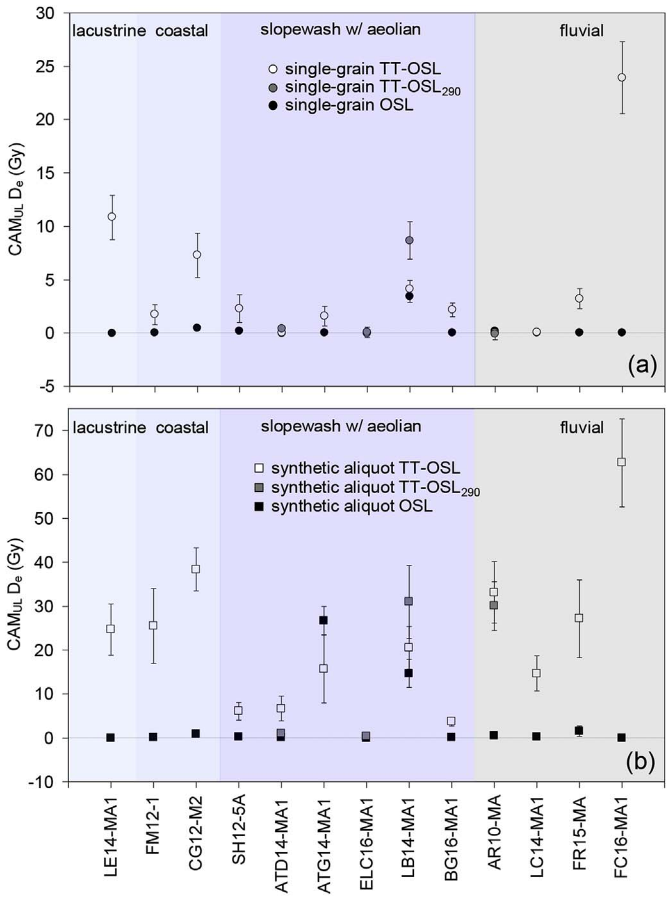

Fig. 2. (a) Single-grain TT-OSL, TT-OSL_{290} and OSL CAM_{UL} D_e values obtained for the modern analogue samples. (b) Synthetic aliquot TT-OSL, TT-OSL_{290} and OSL CAM_{UL} D_e values obtained for the modern analogue samples. Synthetic aliquot D_e values were obtained by summing the signals of all accepted and rejected grains types on each single-grain disc (equivalent to multi-grain aliquots containing 100-grains each). The horizontal lines mark the expected D_e value (0 Gy) for these samples.

4. Modern analogue D_e datasets

Ten of the thirteen modern samples yielded weighted mean (CAM_{UL}) single-grain OSL D_e values equivalent to 0 Gy at 2σ. Twelve of these samples also have OSL CAM_{UL} D_e values of < 0.5 Gy, and > 80% of their measured grain populations yielded modern D_e values at 2σ (Figs. 2–3, S5, Table S5). These OSL datasets confirm that the collected samples are genuinely modern and have experienced at least several minutes of relatively homogenous daylight exposure prior to their recent deposition. The single-grain TT-OSL results for the modern samples are similarly encouraging, especially given the slower bleaching rates and non-zero Gy mean residual doses observed in the daylight bleaching experiments. Seven of the thirteen modern samples yield TT-OSL CAM_{UL} D_e values equal to 0 Gy at 2σ (Table S5). The majority of samples have TT-OSL CAM_{UL} D_e values < 5 Gy; only three samples (LE14-MA1, CG12-M2 and FC15-MA1) have higher CAM_{UL} D_e values of 5–25 Gy (Fig. 2). The weighted mean residual D_e for all thirteen samples is 3.8 ± 1.4 Gy for the TT-OSL datasets, compared to 0.01 ± 0.01 Gy for the OSL datasets. The SG TT-OSL_{290} protocol, which is designed to maximise TT-OSL contributions from higher temperature source traps (Arnold and Demuro, 2015), yields CAM_{UL} D_e residuals that are statistically indistinguishable from their corresponding OSL and TT-OSL D_e values at 2σ (Table S5, Fig. 2). Although only applied to four samples, the TT-OSL_{290} signal therefore appears to be bleachable down to relatively low residual doses in some natural depositional contexts.

The TT-OSL D_e distribution characteristics and weighted mean

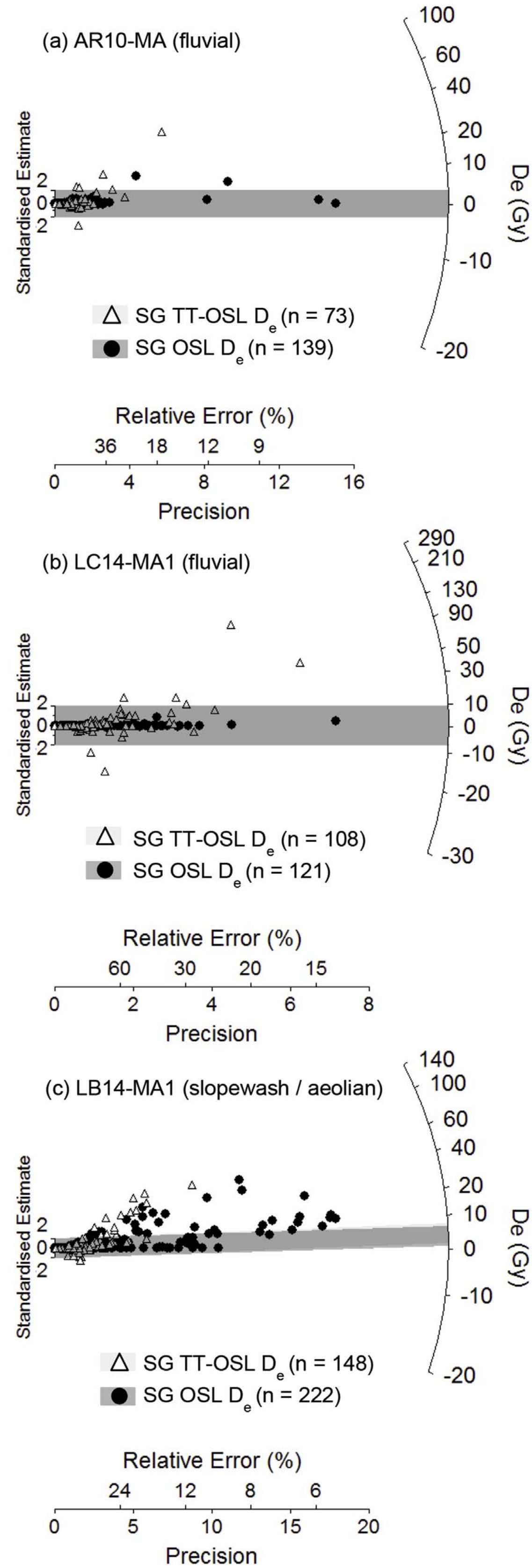

Fig. 3. Representative modified log transformed radial plots showing single-grain TT-OSL and OSL D_e distributions for the modern analogue samples. See Fig. 1 caption for details of the plotting procedure. An *a* offset value of 30 Gy was used to create plots (a) and (c). An *a* offset value of 40 Gy was used to create plot (b). The radial plots are centred on the expected D_e value of 0 Gy for each sample, while the light grey and dark grey bands are centred on the TT-OSL and OSL CAM_{UL} D_e values of each sample, respectively.

residual D_e values vary significantly between sites from the same depositional setting (Figs. 2–3), highlighting that it may not be appropriate to generalise about TT-OSL bleaching adequacy on the basis of depositional context alone. The single-grain TT-OSL D_e distributions of all samples contain minor populations of high D_e values when compared with their OSL counterparts (Fig. 3, Fig. S5). In some cases, the TT-OSL datasets exhibit more pronounced asymmetric tails of high D_e values (Fig. 3c) and CAM_{UL} overdispersion values of several Gy. However, all of the single-grain TT-OSL datasets contain significant populations (61–95%) of 'modern' grains that yield 0 Gy D_e values at 2σ (Table S5, Fig. S5). For ten of the samples, the proportion of modern grains observed in the TT-OSL datasets are similar to (i.e., within 10% of) the proportions of modern grains recorded in the corresponding OSL datasets.

The modern analogue 'synthetic aliquot' D_e datasets (equivalent to multi-grain aliquots containing 100-grains) reveal several interesting trends (Fig. 2, Table S6). The sample-averaged synthetic aliquot OSL residual D_e is 0.67 ± 0.35 Gy for the thirteen samples, which is consistent with the sample-averaged single-grain OSL residual of 0.01 ± 0.01 Gy at 2σ. By contrast, the sample-averaged synthetic aliquot TT-OSL residual D_e (19.9 ± 4.3 Gy) exceeds its single-grain counterpart by a factor of five to six. Additionally, only one of the modern samples (ELC16-MA1) has a synthetic aliquot TT-OSL D_e value equal to 0 Gy at 2σ. The synthetic aliquot TT-OSL D_e values obtained in this study (0.3–63 Gy) overlap with multi-grain TT-OSL residual values reported elsewhere for modern analogues (e.g., Jacobs et al., 2011; Duller and Wintle, 2012; Arnold et al., 2014). For our datasets, comparisons undertaken at different scales of D_e analysis suggests that the systematically larger multi-grain TT-OSL residuals primarily arise from the inclusion of grain types that are rejected by the single-grain quality assurance criteria. There appears to be noticeable inter-sample variability in the types of rejected grains that exert strong multi-grain averaging effects, as might be expected for such a geographically diverse sample dataset. For instance, the presence of rejected grains with very slowly decaying TT-OSL signals appears to chiefly influence the multi-grain D_e results of samples CG12-M2 and LE14-MA1 (see also Tsukamoto et al., 2008; Demuro et al., 2015). For many of the other samples (e.g., FC16-MA1, ATD14-MA1, FM12-1), grains displaying anomalous dose-response properties or unsuitable recycling ratios appear to exert non-neutral effects on the final multi-grain D_e values.

5. Single-grain TT-OSL and OSL dating comparisons

The four late Pleistocene dating samples from Kangaroo Island display different types of single-grain OSL D_e distributions (Fig. 4, Table S3), and therefore provide useful datasets for evaluating TT-OSL bleaching suitability across a range of dating contexts. Sample KI14-12, collected close to a cave palaeoentrance, yielded homogenous OSL and TT-OSL D_e datasets (Fig. 4a) with low overdispersion values of 17–19%, and indistinguishable CAM OSL and TT-OSL ages of 54.2–55.0 ka (Table S3). The consistency of these results supports the applicability of TT-OSL at this locality, and suggest that the Kelly Hill Cave infill deposits were exposed to prolonged daylight prior to entering the karst system.

Samples KHC-KI5 and KI14-5, collected from a deeper chamber within the same cave system, exhibit more heterogeneous OSL D_e distributions, higher overdispersion values of 30–37%, and their D_e datasets are better represented by the minimum age model (MAM) according to the maximum log likelihood criterion of Arnold et al. (2009) (Fig. 4b–c, Table S3). These complex D_e characteristics are interpreted as reflecting the entrainment of grains from pre-existing cave sediments during the transportation of predominantly well-bleached, externally derived sediments through the closed cave system (Arnold et al., in prep). The TT-OSL D_e datasets of these heterogeneously bleached samples exhibit pronounced residual D_e populations and higher overdispersion values than their OSL counterparts. In spite of their seemingly complicated depositional history, consistent TT-OSL and OSL ages of 16.1–18.2 ka and 67.3–67.7 ka were obtained for samples KHC-KI5 and KI14-5, respectively, using the MAM.

The OSL D_e dataset of sample KI14-1, collected from the Boar Beach fossil dune sequence, is characterised by low-to-moderate overdispersion and is well represented by the CAM according to its maximum log likelihood score (Fig. 4d, Table S3). The corresponding TT-OSL D_e dataset of KI14-1 exhibits moderate overdispersion of 42% and a more noticeable tail of high D_e values, which could suggest that daylight exposure was not long enough to completely reset the TT-OSL signal of all grains prior to deposition. Though the MAM-4 is statistically favoured over the CAM for this dataset, the TT-OSL ages obtained using both age models (115.2 ± 7.9 ka and 138.2 ± 9.3 ka, respectively) are consistent with the corresponding OSL age of 137.4 ± 8.5 ka at 2σ (CAM data not shown in Table S3).

6. Discussion and conclusions

The results of this study provide several useful insights into TT-OSL dating bleaching characteristics at different scales of D_e analysis. Daylight bleaching tests confirm that ~6 weeks of exposure may be needed to reduce sample-averaged single-grain TT-OSL residuals to within 10% of background; though complete signal resetting is possible for up to 50% of individually measured grains over the same time period. The CAM_{UL} residual doses (0.1–23.9 Gy) obtained across a range of modern environments are noteworthy given these relatively slow daylight bleaching rates. The favourable modern analogue bleaching results imply prolonged surface residence times at the sites considered here. Alternatively, the sediment samples may have experienced progressive attenuation of residual signals prior to final deposition via multiple cycles of erosion, transportation and re-deposition (see Stokes, 1992).

Importantly, the modern analogue residual doses observed in this study are relatively low in comparison to the natural dose range of interest for typical TT-OSL dating applications. Residual D_e values on the order of 10^{-1}–10^{1} Gy are unlikely to compromise single-grain TT-OSL applicability beyond existing uncertainties in most middle or early Pleistocene dating studies. These unbleached TT-OSL residuals may give rise to more significant systematic age offsets when dating Holocene or late Pleistocene samples, particularly at the multi-grain scale of analysis. However, the low single-grain residuals obtained for many of the modern samples, and the consistent OSL and TT-OSL ages observed for the Kangaroo Island samples, suggest potential for reliable TT-OSL dating over shorter timescales at some sites.

Our various bleaching assessments suggest that single-grain TT-OSL dating suitability is not necessarily limited to certain depositional environments, as is sometimes assumed. Significant variation exists in the magnitudes of modern residual doses recorded both within and between different sedimentary settings (Fig. 2). The consistency of comparative OSL and TT-OSL ages from Kangaroo Island also supports the applicability of single-grain TT-OSL dating in some relatively complex sedimentary contexts, as long as appropriate statistical age models are considered. Though these findings are promising, our empirical datasets are relatively limited, and there remains a need to undertake site-specific bleaching assessments in any TT-OSL dating study; especially those conducted in high-latitude settings and depositional environments not covered by our modern analogue dataset. A potentially useful approach for assessing bleaching adequacy might involve comparisons of ages or D_e values obtained with multiple luminescence signals that bleach at different rates. Such assessments have been widely used in post-IR IRSL studies (e.g., Murray et al., 2012), with parity in ages or D_e values being used to support adequate resetting of the slower bleaching signal, all things being equal. The results of our comparative TT-OSL and OSL dating study support those of Demuro et al. (2015) and Demuro et al. (this volume), and suggest that such differential bleaching assessments could provide useful insights into single-grain TT-OSL suitability in

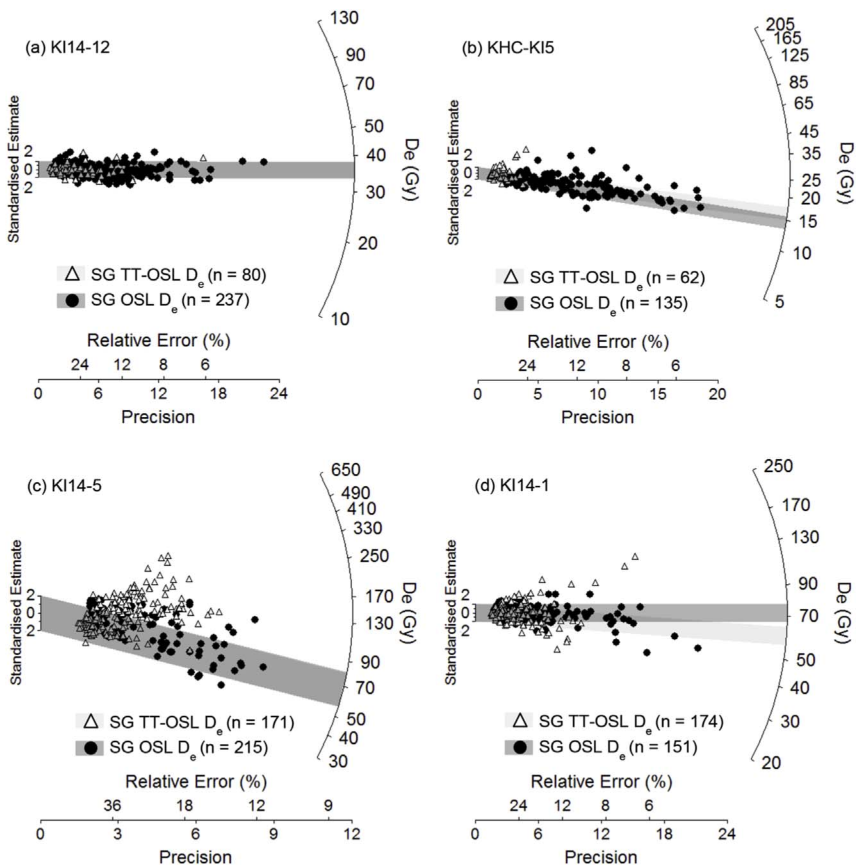

Fig. 4. Paired single-grain TT-OSL and OSL D_e distributions for the Kangaroo Island dating samples, shown as radial plots. Each radial plot is centred on the TT-OSL CAM D_e value. The light grey and dark grey bands are centred on the TT-OSL and OSL D_e values used to calculate the final ages of each sample (see Table S3 for details).

routine dating applications.

Our modern analogue D_e datasets, together with those reported by Gliganic et al. (2017), provide useful constraints on the amount of overdispersion observed in well-bleached modern or very young samples from a diverse range of settings. Well-bleached modern samples, with CAM_{UL} D_e values of 0 Gy at 2σ, yield unlogged overdispersion values of 0.12 ± 0.05 Gy for single-grain OSL datasets and 1.4 ± 0.5 Gy for single-grain TT-OSL datasets (Fig. S6a-b, Table S6). In the absence of site-specific constraints on underlying overdispersion, these average values might provide useful first order approximations for the σ_b parameter of the unlogged minimum age model (MAM_{UL}) and unlogged finite mixture model (FMM_{UL}); which should be specified in Gy when analysing heterogeneously bleached or mixed single-grain datasets containing 0 Gy or negative D_e values. When applying the conventional (logged) MAM and FMM, it may also be worthwhile considering the typical single-grain TT-OSL overdispersion values reported so far for well-bleached and unmixed geological (non-modern) samples. These published D_e datasets yield a mean overdispersion value of 21 ± 2% (Table S7, Fig. S6c), which is consistent with that reported for 'ideal' single-grain OSL samples (20 ± 1%; Arnold and Roberts, 2009).

Finally, our results show that significant differences may be observed between single-grain and multi-grain TT-OSL bleaching residuals for some modern samples. Assessment of multi-grain TT-OSL bleaching characteristics may be complicated by averaging effects of unsuitable grain types that are routinely rejected in single-grain analysis, paralleling observations reported in some OSL dating studies (e.g., Demuro et al., 2013; Arnold et al., 2013). These results also reinforce the findings of Arnold and Demuro (2015), which showed that the summed (multi-grain) TT-OSL characteristics of samples may not necessarily be representative of TT-OSL-producing grains that are individually considered suitable for dating.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.quageo.2018.01.004.

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