Empirical insights into multi-grain averaging effects from ‘pseudo’ single-grain OSL measurements

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ABSTRACT

In this study we assess the signatures of multi-grain averaging effects for a series of sedimentary samples taken from the archaeological site of Hotel California, Atapuerca, Spain. We focus on the special case of equivalent dose (D\text{e}) measurements made on single-grain discs that contain more than one quartz grain in each of the individual grain-hole positions of unmixed standard single-grain aliquots of sedimentary quartz. In particular, the averaging effects of very small multi-grain aliquots of sedimentary quartz, and (ii) assessing the suitability of ‘pseudo’ single-grain D\text{e} measurements for this particular dating application. Pseudo single-grain OSL measurements made on standard discs loaded with 90–100 μm grains (equivalent to ~30 grains per hole) yield significantly different D\text{e} distribution characteristics and finite mixture model (FMM) burial dose estimates compared with single-grain OSL measurements. Grains with aberrant luminescence behaviours, which are routinely rejected during single-grain analysis, exert strong averaging effects on the pseudo single-grain and multi-grain aliquot D\text{e} distributions. Grain-hole averaging effects arising from pseudo single-grain measurements also give rise to ‘phantom’ dose components and are apt to provide bias assessments of quartz signal characteristics and grain type classifications. Though this is a site-specific study, it serves as a cautionary note for interpretations of other pseudo single-grain OSL and D\text{e} datasets – particularly those obtained from measurements of discs containing several tens of grains per hole that those derived from complex depositional environments. The use of custom single-grain discs drilled with smaller sized grain holes is recommended as a means of limiting grain-hole averaging effects when dealing with very fine (<180 μm) sediments.

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1. Introduction

Recent experiments undertaken by Arnold and Roberts (2009) on simulated D\text{e} datasets suggest that significant problems can be encountered when dating mixed sedimentary samples with traditional multi-grain aliquot equivalent dose (D\text{e}) estimation techniques. In particular, the averaging effects of multi-grain D\text{e} analysis can give rise to ‘phantom’ dose components – discrete dose populations that do not correspond to original mixing components – which prevent the correct number of ‘genuine’ dose components being identified with the finite mixture model (FMM) of Galbraith and Green (1990). Importantly, the modelling findings of Arnold and Roberts (2009) suggest that the FMM should only be applied to single-grain D\text{e} datasets and that multi-grain averaging effects can be severe enough to cause unreliable burial dose estimation when measuring even small aliquots that consist of a few tens of grains. A number of empirical single-grain studies have also shown that multi-grain averaging effects can produce systematic biases in D\text{e} distributions for unmixed samples that contain large populations of aberrant quartz grains (e.g. Thomsen et al., 2003; Jain et al., 2004; Demuro et al., 2008).

In this study we aim to assess the significance of such averaging effects for empirical multi-grain D\text{e} datasets from a complex archaeological setting. For this purpose we focus on the special case of D\text{e} measurements made with single-grain discs that contain more than one quartz grain in each of the individual grain-hole positions. This situation commonly arises when ‘standard’ single-grain discs (Fig. S1a) drilled with a 10 × 10 array of cylindrical holes, each 300 μm in depth and 300 μm in diameter (Bøtter-Jensen et al., 2003), are loaded with inappropriately sized quartz grains. Grain packing calculations (Table S1) reveal that the individual grain-hole positions of standard single-grain discs are expected to contain ~3 grains when loaded with 180–212 μm grains and as many as ~30 grains when loaded with 90–100 μm grains. These predicted quantities are supported by visual inspection of preloaded single-grain discs under a binocular microscope.
(Fig. S1b–d), though there are minor hole-to-hole variations in grain numbers related to non-spherical grains, incomplete filling of grain holes and uneven loading across the disc plane. ‘Pseudo’ single-grain $D_s$ measurements made in this manner clearly do not provide true single-grain resolution, but, instead represent very small multi-grain aliquots; thus providing ideal case studies for assessing the signatures and extent of averaging effects in complex sedimentary samples. It is also important to assess the broader implications of undertaking pseudo single-grain measurements in routine dating applications, since this can be a common practice when dealing with very fine sediments devoid of suitably coarse (212–250 μm) quartz.

2. Sample details and experimental procedures

The samples used in this study were taken from the open-air, Middle Palaeolithic archaeological site of Hotel California (Navazo, 2006; Navazo et al., 2008), located in the south-western foothills of the Sierra de Atapuerca, north-central Spain. Four samples were collected from the main archaeological horizons (Units 1–5; Table 1). These units consist of massive slits and clays with scattered boulders of flint and quartzite, which have accumulated by slopewash erosion and colluviation of nearby Lower Pleistocene fluvial terrace deposits (Benito-Calvo et al., 2008). Two replicate samples (HC10-1 and HC10-4) were taken from the same depth in the uppermost archaeological horizon (Unit 5), while the two remaining samples (HC10-2 and HC10-3) were obtained from the lower archaeological horizons (Units 2 and 1, respectively) overlying Neogene marls. The sedimentological properties of the Hotel California deposits indicate that these samples could have been affected by several potential sources of extrinsic $D_s$ scatter, namely insufficient bleaching at deposition (short-distance alluvial/coluvial transportation), post-depositional mixing (anthropogenr disturbance, bioturbation, desiccation crack formation, illuviation) and beta-dose heterogeneity (proximity to large boulders, micro-scale variations in clay formation). This complex burial history increases the likelihood that these samples will contain grains with distinctly different burial doses, which, according to the results of Arnold and Roberts (2009), suggests they may be well-suited for studying multi-grain averaging effects.

Though lacking site-specific independent age control, the presence of over 1800 Mousterian lithics from Units 1–5 indicates a Late Pleistocene (or earlier) age for these samples (equivalent to a $D_s$ estimate of at least 35 Gy). Additionally, $D_s$ estimates of less than ~70 Gy should not be expected for the in situ, fully bleached grain compositions of these samples since the ages derived from such low doses would underestimate the last known, reliably dated Neanderthal finds in Europe at ~28 ka (Finlayson et al., 2006).

Single-grain, pseudo single-grain and small (~80-grain) multi-grain aliquot $D_s$ estimates were obtained using the single-aliquot regenerative-dose (SAR) procedures (Murray and Wintle, 2000) shown in Table S2. Further details of the optically stimulated luminescence (OSL) dating procedures and equipment employed in this study, as well as the SAR rejection criteria used to eliminate unreliable aliquots/grains, are provided in the Supporting Information (SI). Single-grain $D_s$ measurements were made by loading standard single-grain discs with 212–250 μm grains (Fig. S1b). Pseudo single-grain $D_s$ measurements were performed on standard-sized discs containing ~30 grains per hole and ~3 grains per hole, which were produced by loading individual grain-hole positions with 90–100 μm and 180–212 μm grains, respectively (Fig. S1c and d). To test the suitability of our experimental conditions, we performed dose-recovery tests on quartz grains of sample HC10-1 after bleaching their natural OSL signals using blue LEDs at ambient temperature ($2 \times 1$ ks illuminations separated by a 10 ks pause) and administrating a known laboratory dose of $D_s$.

Table 1

OSL sample details, $D_s$ summary statistics and age model results.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Strat. unit</th>
<th>Disc type</th>
<th>Grain size (μm)</th>
<th>Aliquot size (no. of grains)</th>
<th>No. of measurements</th>
<th>CAM results</th>
<th>FMM results</th>
<th>Proportion of grains or aliquots (%)</th>
</tr>
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<tr>
<td>HC10-1</td>
<td>V</td>
<td>SG(s)</td>
<td>212–250</td>
<td>~1</td>
<td>118/1000</td>
<td>72 ± 6</td>
<td>137 ± 10</td>
<td>4 k₃ 128 ± 6 63 ± 6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SG(c)</td>
<td>90–100</td>
<td>~3</td>
<td>87/700</td>
<td>65 ± 6</td>
<td>142 ± 11</td>
<td>4 k₃ 118 ± 7 56 ± 7</td>
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<tr>
<td></td>
<td></td>
<td>SC(c)</td>
<td>73/2600</td>
<td>~3</td>
<td>70/600</td>
<td>62 ± 7</td>
<td>151 ± 13</td>
<td>4 k₃ 143 ± 11 56 ± 9</td>
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<td></td>
<td></td>
<td>SG(s)</td>
<td>90–100</td>
<td>122/700</td>
<td>64 ± 5</td>
<td>208 ± 13</td>
<td>4 k₃ 275 ± 9 83 ± 4</td>
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<td></td>
<td></td>
<td>MG</td>
<td>90–100</td>
<td>~80</td>
<td>11/20</td>
<td>15 ± 4</td>
<td>354 ± 18</td>
<td>2 k₃ 396 ± 36 53 ± 3</td>
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<td>HC10-4</td>
<td></td>
<td>SC(c)</td>
<td>90–100</td>
<td>~3</td>
<td>58/1800</td>
<td>38 ± 6</td>
<td>146 ± 12</td>
<td>3 k₃ 119 ± 9 84 ± 7</td>
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<td></td>
<td>V</td>
<td>SG(s)</td>
<td>90–100</td>
<td>~28</td>
<td>113/500</td>
<td>42 ± 9</td>
<td>218 ± 12</td>
<td>4 k₃ 290 ± 13 50 ± 9</td>
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<td></td>
<td>MG</td>
<td>90–100</td>
<td>11/20</td>
<td>6 ± 4</td>
<td>380 ± 11</td>
<td>1 k₃ 375 ± 25 100</td>
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<td>HC10-2</td>
<td>II</td>
<td>SG(s)</td>
<td>212–250</td>
<td>~1</td>
<td>92/900</td>
<td>40 ± 5</td>
<td>224 ± 12</td>
<td>2 k₃ 160 ± 14 56 ± 11</td>
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<td></td>
<td></td>
<td>SG(c)</td>
<td>90–100</td>
<td>~3</td>
<td>66/1400</td>
<td>47 ± 7</td>
<td>196 ± 15</td>
<td>2 k₃ 149 ± 11 73 ± 8</td>
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<td></td>
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<td>MG</td>
<td>90–100</td>
<td>~28</td>
<td>133/400</td>
<td>42 ± 4</td>
<td>284 ± 13</td>
<td>3 k₃ 358 ± 14 77 ± 6</td>
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<td></td>
<td>SC(c)</td>
<td>90–100</td>
<td>15/24</td>
<td>42 ± 5</td>
<td>242 ± 25</td>
<td>2 k₃ 354 ± 19 54 ± 15</td>
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<td></td>
<td></td>
<td>MG</td>
<td>90–100</td>
<td>~3</td>
<td>60/1500</td>
<td>43 ± 6</td>
<td>233 ± 17</td>
<td>3 k₃ 201 ± 14 70 ± 8</td>
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<td></td>
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<td>SC(s)</td>
<td>90–100</td>
<td>119/500</td>
<td>38 ± 4</td>
<td>289 ± 13</td>
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<td>14/20</td>
<td>20 ± 5</td>
<td>405 ± 25</td>
<td>2 k₃ 361 ± 18 70 ± 16</td>
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</tr>
</tbody>
</table>

a Disc type: SG(s) = standard single-grain discs with 300 × 300 μm holes, SG(c) = custom single-grain discs with 100 × 300 μm holes, MG = traditional multi-grain aliquot discs. CAM = central age model, FMM = finite mixture model (Galbraith et al., 1999; Galbraith and Green, 1990).

b Number of $D_s$ measurements that passed the SAR rejection criteria/total number of grains, pseudo single-grain aliquots or multi-grain aliquots analysed.

c CAM and FMM burial dose estimates have been derived using the standardised $D_s$ values ($D_s$) calculated using Eq. (S1), to enable direct comparisons between measurements made using different grain-size fractions.

d The burial dose obtained using the most statistically suitable age model (i.e., according to the log-likelihood score criterion outlined by Arnold et al., 2009) is shown in bold for each $D_s$ dataset.

e FMM was fitted by varying the common overdispersion value ($\sigma$) between 20% and incrementally increasing the specified number of $k_s$ components. The FMM parameter values shown here were obtained from the ‘optimum’ FMM fit (i.e., the fit with the lowest BIC score: Arnold and Roberts, 2009). The FMM dose component containing the highest proportion of individual $D_s$ values has been selected for the final FMM $D_s$ calculation.

f $D_s$ and overdispersion values have been selected for the $D_s$ calculation.
100 Gy. These tests yielded weighted mean ratios of measured-to-given dose that were consistent with unity for multi-grain aliquots (1.01 ± 0.02, n = 6), single-grain measurements (1.01 ± 0.02, n = 70) and pseudo single-grain measurements made on discs containing ~30 grains per hole (1.00 ± 0.01, n = 106). Overdispersion values of 1 ± 5% and 3 ± 3% were obtained for the single-grain and pseudo single-grain dose-recovery $D_e$ datasets, indicating that certain sources of intrinsic $D_e$ scatter (i.e., those originating from the beta-dose-recovery experimental conditions themselves, or from grain-to-grain variations in luminescence responses to the fixed SAR conditions) are relatively minor for these samples.

3. Assessments of multi-grain averaging effects

3.1. Analysis of $D_e$ distributions

Individual $D_e$ values presented in this study have been standardised to a reference grain-size of 100 μm ($\bar{D}_e$; see SI for derivation) to enable direct comparisons between measurements made with different grain fractions. Fig. 1a shows that the single-grain $\bar{D}_e$ distribution of HC10-1 displays a distinctly heterogeneous pattern of dose estimates. Individual $\bar{D}_e$ values cover a very broad range (3–370 Gy) and have a high overdispersion value of 72% (Table 1). The $\bar{D}_e$ distribution is characterised by an elongated, asymmetric tail of higher $\bar{D}_e$ values, a continuum of low $\bar{D}_e$ estimates, and a dominant clustering of values in the lower-middle portion of the distribution. Application of the FMM to this $\bar{D}_e$ dataset (see SI for further details) reveals the presence of 4 discrete dose populations, with the dominant dose component (i.e., that containing the highest proportion of individual $D_e$ values) aligning with the large clustering of $\bar{D}_e$ estimates at ~130 Gy (Table 1). The other samples share very similar complex single-grain $\bar{D}_e$ distribution characteristics to HC10-1 (Fig. S2). Each of these single-grain $\bar{D}_e$ distributions are characterised by more than one FMM dose component and each displays an overdispersion of ~40% (Table 1), which is significantly higher than the ~20% value commonly reported for ‘ideal’ (i.e., well-bleached and undisturbed) sedimentary quartz samples (Olley et al., 2004; Arnold and Roberts, 2009; Arnold et al., 2011). The absence of a similar amount of $\bar{D}_e$ scatter and overdispersion in the single-grain dose-recovery datasets for HC10-1 could imply that additional sources of extrinsic scatter contribute to the spread of these
single-grain $D_e$ distributions, in agreement with sedimentological interpretations at the site.

Pseudo single-grain $D_e$ measurements made on discs containing ~3 grains per hole for HCl10-1 (Fig. 1b) yield very similar $D_e$ distribution characteristics to their single-grain counterparts; in both cases the FMM identifies the same number of dose components, as well as consistent mixing proportions and $D_e$ values for the dominant dose population (Table 1). Pseudo single-grain $D_e$ measurements made on discs containing ~30 grains per hole, however, produce a noticeably different $D_e$ distribution composed of a more uniform, symmetric spread of values (Fig. 1c). For this sample, the sigmoid shape of the ranked single-grain $D_e$ estimates has become obscured by grain-hole averaging effects, though the amount of overdispersion and number of identified FFM components remains unchanged (Table 1). The original clustering of $D_e$ values at ~130 Gy is now completely absent in the pseudo single-grain $D_e$ distribution and can no longer be identified as a discrete dose component by the FMM. Instead, the dominant FMM dose component has shifted towards the midpoint of the $D_e$ distribution at ~275 Gy (Table 1). These averaging effects are even more pronounced in the ~80-grain aliquot $D_e$ distribution for sample HCl10-1 (Fig. 1d), resulting in both a reduction in the number of FMM components and a shift in the dominant dose component to ~400 Gy.

The same averaging influences are evident in the pseudo single-grain and ~80-grain aliquot $D_e$ distributions for HCl10-2, HCl10-3 and HCl10-4 (e.g. Fig. S3). For each of these samples, pseudo single-grain $D_e$ measurements made on discs containing ~30 grains per hole also reveal an additional FFM dose component compared to their single-grain counterparts (Table 1), akin to the phantom dose components identified by Arnold and Roberts (2009). This trend suggests that the discrete dose populations identified in the single-grain $D_e$ distributions have become distorted and mixed together at the pseudo single-grain scale of analysis. Interestingly, all 4 samples also display a significant upward shift in the dominant FMM component at the pseudo single-grain scale of analysis. In part, this reflects the increased uniformity of the resultant $D_e$ distributions, which is an inevitable outcome of multi-grain averaging effects for $D_e$ datasets containing discrete dose populations and large amounts of $D_e$ dispersion. However, this trend becomes even more exaggerated for the ~80-grain aliquot $D_e$ distributions and is accompanied by both a contraction and an upward displacement in the range of $D_e$ values (Fig. 1 and Fig. S3). The latter, in particular, would suggest an additional causal factor contributes to the complex averaging effects observed with these samples. Closer inspection of the single-grain datasets indicates this upward shift likely reflects the influence of rejected grains with particularly high sensitivity-corrected natural signals, namely non-intersecting, extrapolated and saturated grains (Table S3; Fig. S5e and f). The number of rejected grains falling into these categories amounts to ~50% of the accepted grain populations for each sample. These aberrant grains also account for 42–70% of the single-grain (natural test dose) cumulative light sums for each sample. While these large populations of undesirable grains are identified and rejected in the single-grain analysis, they cannot be excluded from the pseudo single-grain and multi-grain aliquot $D_e$ measurements and, thus, are likely to have exerted a significant averaging influence in the final $D_e$ datasets.

3.2. Inherent luminescence properties

Grain-hole averaging effects arising from pseudo single-grain measurements also influence statistical assessments that are routinely used to characterise quartz luminescence properties. In particular, cumulative brightness distributions (Duller et al., 2000; Fig. 2a) and plots of inter-grain variations of absolute signal intensities (Duller, 2006; Fig. 2b) obtained using pseudo single-grain OSL measurements display distinctly different trends to those obtained using single-grain OSL measurements. The site-averaged single-grain cumulative brightness plot for the Hotel California samples reveals 27% of all measured grains yield signals that are statistically indistinguishable from background and that a further ~25% of the measured grains contribute to 95% of the total OSL signal. In contrast, the site-averaged pseudo single-grain cumulative brightness plot for these samples implies that there are far fewer weakly luminescent grains (7% of pseudo single-grain OSL signals are indistinguishable from background) and that ~50% of the pseudo single-grain measurements contribute to 95% of the total OSL signal. Pseudo single-grain brightness distributions are clearly not suitable for providing direct assessments of inherent signal properties in this instance because they are dictated by the
frequency of luminescent grain-hole positions rather than luminescent quartz grains, and the average intensity of each grain-hole signal.

Significant differences are also evident in the classification of accepted/rejected grain types when comparing the single-grain and pseudo single-grain SAR measurements (Table 2). Specifically, the average proportion of accepted $D_e$ values for pseudo single-grain SAR measurements ($\sim 30$ grains per hole) is almost 2.5 times higher than for single-grain measurements. This is accompanied by large dissimilarities (i.e., by a factor of up to 8) in the respective proportions of single-grain and pseudo single-grain SAR measurements that are rejected for having either weak OSL signals, saturated or non-intersecting natural signals, extrapolated natural signals or anomalous dose-response curves (Table 2). These statistics would suggest that pseudo single-grain assessments of grain types are strongly biased by grain-hole averaging effects and cannot reliably be used as a diagnostic tool for examining intrinsic luminescence properties.

Additional single-grain SAR measurements made on 90–100 $\mu$m quartz grains revealed broadly consistent grain brightness distributions (Fig. 2) and grain type classification statistics (Table 2) with those of single-grain SAR measurements made on 212–250 $\mu$m grains. This confirms that the aforementioned differences between single-grain and pseudo single-grain measurements are not merely the outcome of a grain-size dependence in the luminescence properties of these samples.

4. Discussion

Burial dose estimates obtained using the most statistically suitable age models (i.e., according to the statistical criterion outlined in Arnold et al., 2009 — see discussions in Supplementary Information) are up to $\sim 3$ times higher when measuring $\sim 80$-grain aliquots compared to individual quartz grains for the Hotel California samples (Table 1). Analysis of the $D_e$ distribution characteristic at these two scales of analysis strongly suggests that the systematic $D_e$ offset is attributable to multi-grain averaging effects, which renders the use of traditional multi-grain aliquot techniques unsuitable in this archaeological context. Pseudo single-grain burial dose estimates derived using the dominant FMM components are up to $\sim 2.5$ times higher than their single-grain counterparts (Table 1). These results confirm that grain-hole averaging effects arising from pseudo single-grain measurements can also be sufficient to induce significant, systematic deviations in our burial doses estimates.

The inability to identify the dominant single-grain dose components in the pseudo single-grain or multi-grain aliquot $D_e$ distributions, and the presence of additional phantom doses at the pseudo single-grain scale of analysis, are both consistent with the results obtained by Arnold and Roberts (2009) for simulated multi-grain aliquots consisting of a few tens of grains. Furthermore, our empirical results reveal that grains that are routinely rejected during single-grain analysis can potentially exert additional averaging effects on pseudo single-grain or multi-grain aliquot $D_e$ distributions — a factor that could not easily be constrained in earlier modelling investigations. Our broader investigations also demonstrate that multi-grain averaging effects extend beyond $D_e$ datasets and are apt to influence other types of statistical assessments for these samples, particularly those commonly utilised to characterise intrinsic luminescence properties (e.g., Duller et al., 2000, Arnold and Roberts 2011). Significant inaccuracies are shown to ensue if the pseudo single-grain OSL measurements of the Hotel California samples are wrongly used to provide direct assessments of quartz signal characteristics (e.g., Fig. 2) or grain type classifications (e.g., Table 2). Such biases can have important consequences for interpretations of OSL datasets and could prevent reliable comparisons of luminescence properties between different samples, sites, or studies.

It is worth highlighting that the $D_e$ distribution characteristics and FMM burial doses of these particular samples remain largely unaltered when making pseudo single-grain measurements on discs that contain $\sim 3$ grains per hole (c.f., Fig. 1a and 1b). The pseudo single-grain $D_e$ distributions only deviate significantly from their single-grain counterparts when standard-sized discs are loaded with a few tens of grains per hole (e.g., Fig. 1c). It would be unwise, however, to make broader generalisations about the expected severity of grain-hole averaging effects at different scales of pseudo single-grain analysis on the basis of this single empirical dataset. The extent of pseudo single-grain averaging effects will inevitably vary from sample to sample as a function of the amount and type of extrinsic/intrinsic $D_e$ scatter, grain brightness distributions, inter-grain variations in absolute signal intensities, and systematic scatter, grain brightness distributions, inter-grain variations in absolute signal intensities,
inter-grain variability in luminescence responses to the chosen measurement conditions, and proportions/behaviours of rejected grains. Importantly, many of these variables cannot be accurately constrained without undertaking prior single-grain analysis, thus underscoring the need to exert caution when interpreting pseudo single-grain \( D_e \) datasets. Likewise, it is not possible to generalise about the expected nature of grain-hole or multi-grain averaging effects from this study alone. Burial dose underestimation, for instance, may be just as likely an outcome of multi-grain averaging as the apparent overestimations witnessed in this study (e.g., Demuro et al., 2008). Such an outcome does not necessarily contradict the findings reported here as it could simply reflect the interplay of different types of aberrant grains that would otherwise be rejected during single-grain. Some samples may also be relatively insensitive to systematic shifts in burial dose estimates at the pseudo single-grain or multi-grain scale of analysis — particularly if they display very low amounts of single-grain \( D_e \) scatter and do not contain populations of rejected grains with anomalous high or low sensitivity-corrected natural signals.

Though the ages of these samples are not independently known — and, hence, it has not been possible to evaluate the accuracy of the various burial doses obtained in this study — we still place great confidence in the single-grain \( D_e \) estimates since the OSL characteristics of each grain have been individually evaluated against objective quality assurance criteria, and only grains considered reliable contribute to the final burial dose estimate. We would also argue that the absence of age control does not undermine our interpretations of multi-grain \( D_e \) averaging effects because the same basic averaging influences clearly affect both the quartz luminescence characteristics (Fig. 2) and grain type classifications (Table 2) of these samples. The latter observations of averaging effects do not necessitate independent age control for their validation. It is also doubtful that multi-grain averaging could have selectively affected certain parameters of the OSL datasets without having exerted any influence on the pseudo single-grain and multi-grain aliquot \( D_e \) datasets derived from the same experimental measurements. Even if the multi-grain aliquot \( D_e \) distributions could be considered reliable (i.e., representative of the natural \( D_e \) variability affecting these samples) and not unduly influenced by averaging effects, it would still be necessary to explain away the highly structured and overdispersed single-grain \( D_e \) distributions as artefacts of the measurement procedure. This seems unlikely given (i) the sedimentological evidence at this site, which suggests that these grains have experienced complex depositional and post-burial histories (see Supplementary Information) and, thus, are likely to display extrinsic \( D_e \) scatter, and (ii) the stratigraphic consistency of the resultant single-grain FMM ages and their agreement with independent age control at two neighbouring Atapuerca open-air sites (Arnold et al., in press).

An obvious solution to the problem of having to undertake pseudo single-grain \( D_e \) measurements on samples that only contain very fine (<180 \( \mu \)m) quartz fractions, is to use custom discs that have been drilled with smaller sized grain holes. Custom-made discs containing arrays of 200 \( \mu \)m × 200 \( \mu \)m cylindrical holes will provide single-grain resolution for 180—200 \( \mu \)m quartz grains and near single-grain resolution (~2 grains per hole) for 125—180 \( \mu \)m quartz grains (Table 51). When dealing with even finer, 90—100 \( \mu \)m quartz fractions, use of custom discs drilled with, for instance, 300 \( \mu \)m × 100 \( \mu \)m holes (Fig. 51a) will ensure that each grain-hole position contains only ~3 grains, compared to ~30 grains for standard-sized discs (Table 51). Fig. 3 and Fig. S4 show that \( D_e \) distributions obtained using the latter type of customised disc loaded with 90—100 \( \mu \)m quartz grains are indistinguishable from the corresponding single-grain \( D_e \) distributions obtained using standard discs loaded with 212—250 \( \mu \)m fractions (Fig. 1 and Fig. S2). For both types of measurements, the FMM also identifies the same number of dose components, as well as consistent mixing proportions and \( D_e \) values for the dominant dose population (Table 1). These results support the suitability of custom discs for samples that contain only small grain fractions, and validate the aforementioned observation that pseudo single-grain aliquots containing ~3 grains per hole yield the same \( D_e \) distribution characteristics and FMM burial doses as their single-grain counterparts. The consistency of the \( D_e \) datasets for the 90—100 \( \mu \)m and 212—250 \( \mu \)m fractions also confirms that the grain-hole averaging effects reported in Section 3.1 were not merely the outcome of measuring different grain-size fractions as part of the single-grain and pseudo single-grain comparisons.

5. Conclusions

The results of this study support the recent modelling predictions of Arnold and Roberts (2009) by demonstrating that multi-grain averaging effects for very small-sized aliquots, namely pseudo single-grain aliquots containing a few tens of grain per hole, can significantly alter \( D_e \) distribution characteristics and resultant FMM burial dose estimates. The nature and severity of grain-hole averaging effects are likely to vary considerably from sample to sample. Indeed, many samples may be relatively insensitive to such influences. However, empirical observations from the Hotel California samples serve as a cautionary tale and suggest that care may be needed when interpreting certain pseudo single-grain OSL chronologies derived from complex depositional settings. In light of the potential complications that can arise from grain-hole averaging effects, we would recommend using custom single-grain discs drilled with appropriately sized grain holes in all routine single-grain dating studies.

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Appendix. Supplementary data

Supplementary data related to this article can be found online at doi:10.1016/j.radmeas.2012.02.005.

References


