Using post-IR IRSL and OSL to date young (< 200 yrs) dryland aeolian dune deposits

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ABSTRACT

Determining the most appropriate luminescence protocol, coupled with suitable data processing methods, for dating recently deposited sediments (< 200 years) is important for identifying episodes of sediment movement and interpreting historical landscape dynamics. Issues of partial bleaching, dim luminescence signals and the incorrect application of rejection criteria, can lead to inaccurate and imprecise ages of recent sediment deposition. This study first compares the performance of quartz optically stimulated luminescence (OSL) and K-feldspar post-IR IRSL (pIRIR) measurements in a series of dose recovery preheat plateau, bleachability and remnant dose tests. Sediments of known historical age are used to identify the most suitable aliquot size and age model choice for further application on near-surface aeolian dune sediments from the Nebraska Sandhills. Results show that the ideal conditions for measuring these aeolian sediments are small aliquots (2 mm) of either quartz or K-feldspar coupled with the relevant protocols (OSL130 pIRIR250) and the unlogged-CAM and unlogged-MAM respectively. Results of 4 ± 7 years (quartz) and 4 ± 8 years (K-feldspar) are in excellent agreement with 6 years. Additionally, we find a revised set of rejection criteria is useful for accurately identifying the appropriate aliquots or grains for reliable age estimation. Sensitivity testing of recuperation rejection criteria highlights the caution that should be taken to avoid arbitrarily applying rejection criteria and biasing towards age overestimations.

1. Introduction

Methodological and technological advances in luminescence dating (e.g. Better-Jensen et al., 2000; Murray and Wintle, 2000), and age model modifications (e.g. Arnold et al., 2009; Combès et al., 2015; Cunningham et al., 2015; Cunningham and Wallinga, 2012; Guérin et al., 2017), have meant that the accuracy of dating recent deposition events (e.g. < 200 yrs) has greatly improved over recent decades. As such, a range of existing studies have successfully applied luminescence dating techniques to young and known-age sedimentary samples (e.g. Bailey et al., 2001; Ballarini et al., 2003; Banerjee et al., 2001; Cunningham and Wallinga, 2012, 2009; Madsen et al., 2007; Olley et al., 1999; Riedesel et al., 2018) with the majority applying optically stimulated luminescence (OSL) techniques to the quartz fraction to date the age of deposition. The rapid bleaching of the fast component of the quartz luminescence signal (Wintle and Murray, 2006) minimises the likelihood of partial bleaching, and coupled with a seemingly apparent absence of anomalous fading (Huntley and Lamothe, 2001), quartz-focused studies have dominated young luminescence measurements.

Nevertheless, the application of feldspars to luminescence dating has many advantages. First, feldspars are widely abundant and can be found in a variety of sedimentary settings. Second, unlike quartz crystals, feldspars can be selectively measured under infrared excitation despite the presence of other minerals (Huntley and Lamothe, 2001). Third, a larger proportion of potassium-rich feldspar (hereafter referred to as ‘K-feldspar’) grains emit a detectable luminescence signal, which is generally brighter than the signal emitted from quartz grains (Duller et al., 2003). Finally, the advantage of high internal dose rates for K-feldspar sediments aids in producing a brighter luminescence signal (Reimann et al., 2012; Smedley et al., 2016), which improves the signal-to-noise ratio of luminescence measurements, boosting the capacity to measure young dim samples.

Until recently, however, comparatively few studies had used the K-feldspar fraction for dating sedimentological samples. As noted in Brill et al. (2018), when dating Holocene sediments, K-feldspar IRSL techniques have generally been disregarded in favour of quartz OSL measurements. One of the reasons for the dominance of quartz usage has been because of the instability of some of the signals in the feldspar grains, which results in a greater degree of anomalous fading (Wintle, 1973) than that found in quartz crystals, resulting in estimated age...

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undertakings (Buylaert et al., 2012; Roberts, 2012) unless corrected for. Previously, users of IRSL techniques corrected for the effects of anomalous fading with the g-value approach (e.g. Huntley and Lamothe, 2001), however, the introduction of the pIRIR protocol proposed by Thomsen et al. (2008), which identifies a low fading signal, has led to a re-exploration of K-feldspar luminescence potential. A series of recent studies have sought to test the application of different pIRIR protocols in a range of settings including dating modern and young sediments (Brill et al., 2018; Madsen et al., 2011; Reimann and Tsukamoto, 2012; Riedesel et al., 2018), comparing the utility of the pIRIR and IRSL signal against quartz OSL in attempting to extend the luminescence age range (Colarossi et al., 2015) and investigate the impact of varying measurement conditions to minimise residual doses and fading of the luminescence signal (Buylaert et al., 2009; Colarossi et al., 2018; Jain et al., 2015; Riedesel et al., 2018; Roberts, 2012).

Whilst the pIRIR signal has been shown to be more stable over time than previously used IRSL protocols, existing research has also suggested that the signal bleaches more slowly and thus results in greater residual signals than equivalent IRSL and quartz OSL measurements (Buylaert et al., 2012; Colarossi et al., 2018, 2015; Li and Li, 2011; Riedesel et al., 2018). Experiments conducted by Li and Li (2011) demonstrated the effect of measurement temperature on signal stability and bleachability (multi-elevated-temperature post-IR IRSL – MET pIRIR), exploring the capacity to target more stable traps within feldspars through higher stimulation temperatures. A summary of published pIRIR residual results in Smedley et al. (2015) suggests that under specific measurement conditions, remnant doses from multigrain aliquots have been recorded < 1 Gy, with the youngest reported age of a known modern sample at 48 ± 6 years (Madsen et al., 2011). More recently, Brill et al. (2018) have reported feldspar ages of 8 ± 2 years and 10 ± 6 years for pIRIR150 for modern storm deposits in Thailand. These results hint at the potential routine application of pIRIR protocols when measuring young (e.g. < 200 yrs) sediment samples, particularly those that demonstrate a dim quartz component.

Aside from identifying suitable mineralogical properties and protocol conditions for dating recently deposited sediments, the selection of appropriate post-measurement rejection criteria and age model usage is essential to calculating accurate age estimates of sediment movement and subsequent deposition over the last 200 years. For example, naturally high recuperation levels as a percentage of equivalent dose are expected when dating young sediments due to the noisy nature of the signals and the close proximity of the natural and zero dose points. Secondly, as shown by Arnold et al. (2009), the application of modified age models (i.e. un-logged versions of the Central Age and Minimum Age Models (Galbraith et al., 1999)) is more appropriate for samples with equivalent doses within errors of zero where logged-age models result in age overestimations.

Combined, identification of the most suitable mineral, measurement conditions, and post-measurement data analysis is required in all examples of luminescence dating to ensure that the technique has produced the most accurate age estimates for the sediment samples in question. In relation to recently buried sediments (i.e. < 200 years), the importance of deducing the optimum suite of luminescence methods is even more important when increases in precision and accuracy can allow us to apply luminescence techniques to answer questions of historical landscape dynamics.

With this in mind, this study aims to identify a protocol for the luminescence dating of recently deposited (< 200 yrs) aeolian dune sediments taken from the Nebraska Sandhills. In this study, the remnant dose (i.e. the dose that remains in the sample at the time of burial) of known-age near-surface aeolian sediments is used to test the suitability of the OSL and pIRIR signals when calculating the D0 of young known-age aeolian sediments. Quartz and K-feldspar fractions have been extracted and measured against blue OSL and pIRIR protocols to identify how the different minerals perform against a variety of preheat, dose recovery, anomalous fading and remnant dose experiments. Whilst previous studies have tested the optimum pIRIR conditions for fluvial and coastal samples (e.g. Colarossi et al., 2018; Reimann et al., 2012, 2011; Reimann and Tsukamoto, 2012), this study tests the application of the pIRIR method to young aeolian sediments extracted from a dryland location which typically experiences favourable bleaching conditions. With bleaching conditions considered non-limiting, it is anticipated that measuring the D0 of known-age sediments should identify the lowest potential remnant dose that we can expect to find when luminescence dating K-feldspars.

With the choice of mineral and measurement conditions explored, this study also outlines appropriate age model selection and rejection criteria application for measuring D0’s that are within errors of 0 Gy, exhibit noisy decay curves and typically have large uncertainties. With advances in age model development and a range of considered recuperation thresholds discussed in the literature, this study uses a series of sensitivity-testing results from known-age sediments to identify the most suitable combination of rejection criteria and data processing tools for the near-surface dune sediments presented.

2. Methods and instrumentation

2.1. Samples

Sediment samples GSL15/1/2, GSL15/1/3 and BBR15/1/1, extracted from the Nebraska Sandhills in July 2015 as part of a wider investigation into the recent landscape dynamics of the aeolian dune-field, are used in this study. GSL15/1/2 and GSL15/1/3 are quartz-rich samples extracted from the backwall of Yao’s Blowout in the Gudmundsen Sandhills Laboratory (42.08627°N, 101.36721°W). 20 cm black opaque plastic tubing was hammered horizontally into the exposed backwall at Yao’s Blowout at 54 cm and 97 cm depth below the surface of the dune crest to extract samples GSL15/1/2 and GSL15/1/3 respectively. BBR15/1/1 is a 50 cm vertical sediment core extracted from a lunette dune which has formed on the Barta Brothers Ranch since 2009 AD (42.24580°N, 99.65433°W). BBR15/1/1 was split lengthwise during sample preparation and sub-sampled at centimetre scale (sub-samples are labelled according to depth below the surface – i.e. BBR15/1/1/X = X cm down core from surface). Sub-samples from BBR15/1/1 should therefore be of a young age (post-2009 AD c.5–6 years) and provide a suitable test case for determining remnant doses between the different minerals and protocols.

2.2. Sample preparation and instrumentation

All samples were treated in an excess of hydrochloric acid and hydrogen peroxide to remove carbonates and organics prior to wet-sieving to the appropriate size fraction (multigrain aliquots: 125–180 μm, single-grain: 180–210 μm). Quartz fractions were isolated using sodium polytungstate density separation at 2.72 and 2.62 g/cm3 followed by a 45-min etch with concentrated hydrofluoric acid. K-feldspar fractions were isolated using sodium polytungstate density separation at < 2.58 g/cm³. All pIRIR experiments referred to in this study were applied to the K-feldspar fraction of the sediment sample. All multigrain luminescence measurements were made using an automated Risø TL/DA 15 reader, equipped with infra-red (IR) (870 nm) and blue (470 nm) LEDs and 90Sr/90Y beta sources for irradiations. A convex lens placed in front of the photomultiplier tube was used to focus the signal and increase the number of counts recorded. For quartz OSL measurement, luminescence was detected in the UV region using an EMI 9635Q alkali photomultiplier tube fitted with a 7.5 mm Hoya U-340 filter, whilst IRSL detection was achieved through a combination of Corning 7-59 and Schott BG39 filters. Multigrain experiments were conducted on small aliquots (2 mm mask) and large aliquots (8 mm mask), with c.109 grains (small) and c.1760 grains (large) expected on each aliquot based on the grain size fraction 125–180 μm (calculated in R Studio 0.10.153 (Buron, 2017)).
Single quartz and K-feldspar grains were measured on single grain discs with hole diameters of 300 µm. Single grain measurements were completed on the 180–210 µm size fraction to ensure only single grains were present within each of the single grain disc holes. Single grain measurements were performed using an automated Risø TL/DA 15 reader, equipped with infra-red (IR) (870 nm) and green (523 nm) lasers and 90Sr/90Y beta sources for irradiations. For quartz OSL measurement, luminescence was detected in the UV region using an EMI 9635Q alkali photomultiplier tube fitted with a 7.5 mm Hoya U-340 filter, whilst IRSL detection was achieved through a combination of Corning 7-59 and Schott BG39 filters. All feldspar signals were separated from stimulation light using an interference filter with peak transmission at 410 nm.

3. Selecting appropriate measurement parameters for quartz and feldspar

Luminescence measurements were made following the Single Aliquot Regenerative (SAR) protocol (Murray and Wintle, 2000) under a range of preheat and stimulation temperatures. Preheat conditions preceding the measurement of the natural, zero, or regenerative dose signals were the same as those used prior to the test dose signal measurement (Blair et al., 2005).

Recycling ratio tests and an IR-depletion ratio point were included to monitor sensitivity and identify any feldspar contamination in the quartz OSL experiments (Duller, 2003). Recuperation levels were recorded and analysed alongside the range of preheat and measurement temperature variations studied. Given the young nature of the sediments used in this study, dose response curves were fitted against a linear function and individual equivalent dose estimates were calculated using interpolation of the natural signal onto this line. Uncertainties were calculated following 1,000 Monte Carlo fits of the curve and propagated with a 2.5% measurement error.

Typical quartz OSL and K-feldspar IRSL decay and dose response curves are shown in Fig. 1. Based on the young nature of the sediments, the relatively noisy decay curve and the large contribution from the medium and slow components, quartz De’s were calculated using integration limits which captured a large signal with an early background subtraction following the recommendation of Cunningham and Wallinga (2010). Final integration limits used were 0–1.5 s followed by an immediate background interval 1.6–6 s for quartz, and 0–5 s followed by a late background interval covering the last 20 s of measurement time for feldspar. Sample equivalent doses were calculated using the un-logged Minimum Age Model (MAM) and Central Age Models (CAM) (Galbraith et al., 1999) given the young nature of the samples measured (Arnold et al., 2009).

Initial dose recovery preheat tests (Murray and Wintle, 2003) were performed for both combinations (quartz OSL and K-feldspar pIRIR) to determine optimum measurement conditions prior to testing for levels of anomalous fading and remnant doses. Both quartz and K-feldspar fractions were bleached prior to dose recovery preheat plateau experiments using blue diodes for two periods of 100 s at room temperature (20 °C) and with a 7.5 mm Hoya U-340 filter fitted. Given the potential for variable results from daylight bleaching of K-feldspars, bleaching using blue diodes offers a controlled means for applying equal bleaching conditions to all grains analysed. Filter combinations were subsequently changed prior to the given dose and measurement of the K-feldspar fraction during the SAR cycle and IRSL and pIRIR measurements.

Dose recovery preheat plateau tests were completed on two sizes of aliquot: small (2 mm mask) and large (8 mm mask). As demonstrated through single-grain measurements (Duller, 2008; Duller and Murray, 2000; Rhodes, 2007), there is great variability in the luminescence properties between individual grains within and between different samples. Rhodes (2007) noted that within a sample there is large variation in the brightness of the OSL signal as well as the proportion of grains having a detectable OSL signal between samples. As such, larger aliquots, with a higher volume of grains, produce a greater averaging of the luminescence characteristics between grains, whilst smaller aliquots produce a signal dominated by a small proportion of grains from the overall sample. Dose recovery preheat plateau tests were completed on small and large aliquots to ensure the measurement conditions selected were not based solely on an ‘average’ luminescence signal.

3.1. Quartz

Quartz OSL dose recovery preheat tests were completed using sample GSL15/1/3 with preheats ranging from 160 to 250 °C on small
and large aliquots. All OSL measurements were made at 130 °C for 100 s with a 6.5 Gy test dose and 0.65 Gy given dose (Table 1).

Dose recovery ratios from the large aliquots are in line with unity within errors for all preheat temperatures used, with a slight increase in the ratio and spread of results found at higher temperatures, suggesting some evidence of thermal transfer in the signal. As expected, results from the small aliquots yielded more variability than the large aliquots due to reduced averaging of individual signals (SI: Appendix A). Results across all preheat temperatures demonstrate an ability to recover the given dose within errors, yet a tighter clustering of results is identified at 200 °C. Large variability and error margins are found on the recuperation levels across all preheat temperatures, but a general increase with preheat temperature beyond 200 °C is observed.

High recuperation is anticipated since a small given dose was used (c. 0.65 Gy – low dose replicates a similar signal size to the natural $D_e$) and the recuperation level is proportionally dependent on the size of the signal. High levels of recuperation as a percentage of the natural dose are also expected when dating young samples and a normal distribution and the recuperation level is proportionally dependent on the size of the aliquot suggest that 200 °C is an appropriate preheat temperature for dating the quartz fraction of these sediments.

### 3.2. K-feldspar

Existing pIRIR studies have used preheat temperatures ranging from 150 °C–290 °C. Thomsen et al., (2008) recommended a pIRIR$_{225}$ protocol to reduce fading rates, yet a range of studies dating notably young sediments have suggested the use of a lower preheat and stimulation temperature when measuring the pIRIR signal (Madsen et al., 2011; Reimann et al., 2012, 2011; Reimann and Tsukamoto, 2012). The basic trade-off when selecting pIRIR temperature is that lower temperatures bring increased bleachability at the cost of higher athermal fading rates.

Dose recovery preheat plateau experiments were completed using samples GSL15/1.2 and GSL15/1.3 with 10 s preheats (160–255 °C) on large and small aliquots. IRSL measurements were made at 50 °C for 100 s followed by a pIRIR measurement for 100 s with a 6.5 Gy test dose and 0.65 Gy given dose (Table 1). Based on the method described in Roberts (2012), all pIRIR measurement temperatures were 30 °C cooler than the corresponding preheat temperatures.

Large aliquot results show that when preheated 160–200 °C the dose recovery ratio is in agreement with unity, yet increases with temperature beyond this range, indicating the gradual de-trapping of progressively less bleachable traps that may not be fully bleached by 100 s exposure to blue diodes at room temperature. Likewise, recuperation as a percentage of the natural appears to increase as a function of temperature beyond 220 °C (increasing beyond 5%). This result is further corroborated when $D_e$ is measured on small aliquots (SI: Appendix A).

Accordingly, 200 °C was selected as the most appropriate pIRIR preheat temperature to use for all further experiments of the protocol – coupled with IRSL$_{50}$ and pIRIR$_{170}$ measurements (Table 1). These results are in agreement with other studies (Madsen et al., 2011; Reimann et al., 2012, 2011; Reimann and Tsukamoto, 2012) which have demonstrated that a lower preheat temperature than pIRIR$_{225}$ is appropriate for dating young samples. Furthermore, with existing studies suggesting that the size of the residual dose increases with the stimulation temperature of the pIRIR measurement (Chen et al., 2013; Kars et al., 2014), a moderate stimulation temperature is more appropriate to reduce the size of residual signal and the likelihood of age over-estimations.

Additionally, results demonstrate that a 290 °C hot bleach is required at the end of each SAR cycle to remove any remaining charge from the test-dose and prevent transfer to the following $L_x$ measurement in the SAR cycle (Smedley et al., 2015). Results across both small and large aliquot suggest that 200 °C is an appropriate preheat temperature for these samples, with IRSL$_{50}$ and pIRIR$_{170}$ measurements coupled with a hot bleach at the end of each SAR cycle.

### 4. Comparing rates of anomalous fading

Anomalous fading is a key issue affecting the luminescence dating of most feldspar grains (Wintle, 1973), potentially resulting in an overall age underestimate of the sediment if not appropriately accounted for. To determine levels of anomalous fading, short-term fading experiments were completed for both combinations of sediments (quartz GSL130 and pIRIR$_{170}$) by bleaching large aliquots of sample GSL15/1.3 and subsequently providing a known irradiation (Auclair et al., 2003). $L_x/T_x$ measurements were taken immediately following irradiation and at various time intervals (up to 236 hours) after irradiation. Samples were preheated following irradiation prior to storage (Auclair et al., 2003) and anomalous fading results were quantified by the $g$-value (Aitken, 1985). Calculation of the $g$-value allows for luminescence ages for fading sediment samples to be corrected if the $D_e$ lies within the linear dose response range (Auclair et al., 2003; Huntley and Lamothe, 2001), which is typical of young sediments.

Whilst pIRIR studies have shown the pIRIR signal to be more stable than the IRSL signal, we would expect pIRIR$_{170}$ to fade more than pIRIR$_{225}$. For this purpose, testing the stability of the luminescence signal to ensure that there is minimal anomalous fading provides greater confidence in the natural luminescence signal measured. Fading results for the K-feldspar pIRIR$_{170}$ and quartz GSL130 fractions of sediment sample GSL15/1.3 are presented in Fig. 2. Results show that short-term fading does not appear to be a problem across either protocol, with both values within uncertainties of zero. Quartz is thought to not suffer from anomalous fading and values in the region $1.3 ± 0.3%/\text{decade}$ have previously been considered as a laboratory artefact (Thiel et al., 2011) and suggest minimal fading. Equally, the low values demonstrate that there is minimal short-term fading of the pIRIR$_{170}$ signal in these samples and therefore the K-feldspar fraction.

| Step | Treatment | Measured | Step | Treatment | Measured |
|------|-----------|----------|------|-----------|----------|
| 1    | Dose (Natural, 0 Gy, 0.45 Gy, 0.85 Gy, 0.45 Gy) | -        | 1    | Dose (Natural, 0 Gy, 0.45 Gy, 0.85 Gy, 0.45 Gy) | -        |
| 2    | Preheat (200 °C for 10 s) | -        | 2    | Preheat (200 °C for 10 s) | -        |
| 3    | OSL 100 s @ 130 °C | $L_x$    | 3    | IRSL 100 s @ 50 °C | -        |
| 4    | Test dose (6.5 Gy) | -        | 4    | IRSL 100 s @ 170 °C | $L_x$    |
| 5    | Preheat (200 °C for 10 s) | -        | 5    | Test dose (6.5 Gy) | -        |
| 6    | OSL 100 s @ 130 °C | $T_x$    | 6    | Preheat (200 °C for 10 s) | -        |
| 7    | IRSL 100 s @ 50 °C | -        | 7    | IRSL 100 s @ 170 °C | $T_x$    |
| 8    | IRSL 50 s @ 290 °C | -        | 8    | IRSL 100 s @ 170 °C | $T_x$    |
| 9    | IRSL 50 s @ 290 °C | -        | 9    | IRSL 50 s @ 290 °C | -        |
should not give an age underestimation if used to date these young samples. By comparison, as is expected with the IRSL50 signal, a greater degree of fading is noted in the measurements with a g-value c.4%/decade.

5. Comparing the bleaching rate of quartz and feldspar

Residual bleaching tests were used to identify the residual dose in the quartz and K-feldspar grains following various periods of exposure to bleaching conditions. Identifying complicating factors such as residual doses, is imperative to determining the accuracy of the resultant natural equivalent doses, and thus in addressing the aim of identifying an appropriate measurement method for dating young samples.

Experiments were completed for both the quartz and K-feldspar fraction of sediment sample GSL15/1/3. Large aliquots were prepared of each mineral fraction and exposed to bleaching conditions for various time intervals, before being measured for any residual luminescence signal. Sediments were placed outside on a flat windowsill for different periods of daylight exposure (1–236 hours). Small aliquot quartz OSL measurements suggest a natural De of 1.7 ± 0.06 Gy for sample GSL15/1/3 and can be used for comparison with the residual dose measured in the quartz and K-feldspar fractions.

Quartz results show that after all bleach times tested, all of the luminescence signal had been depleted and De’s indistinguishable from zero at 1σ were measured (Table 2). In comparison, the pIRIR170 signal of the K-feldspar fraction retained a residual dose after 1.5 (0.276 Gy) and 8 h (0.152 Gy), but was reduced to zero following an extended 236 h of daylight exposure. Based on a dose rate of 2.891 ± 0.176 Gy/ka these results suggest that in each pulse of potential sediment activation and deposition, K-feldspar grains may be retaining a residual dose upwards of c. 100 yrs; a significant over estimation when measuring young samples for age of deposition (Table 2). These results are in agreement with previous work which has suggested that the pIRIR signal from feldspars bleaches more slowly during exposure to daylight than the OSL signal from quartz (Buylaert et al., 2012) and typically has a hard-to-bleach component (Kars et al., 2014).

6. Comparison of remnant doses

To test the likelihood of sediments retaining a remnant dose post-deposition in the natural landscape, remnant dose experiments were completed for both quartz and K-feldspar fractions on known-age sediment sample BBR15/1/1. Samples were measured for their natural De following standard SAR OSL130 and pIRIR170 protocols. Since these samples are of known age (post-2009 AD), any luminescence signal (> 5–6 years) measured is indicative of a remnant dose. A remnant dose test which uses samples of a known age provides insight into how well individual minerals have been bleached in the natural environment, under more complex bleaching conditions, which is key to interpreting natural equivalent doses and luminescence ages.

This experiment was completed for a range of aliquot sizes: large, small and single grain to identify whether any remnant dose is restricted to a few isolated grains and can be excluded from the overall De calculation, or whether it is more commonly found amongst the grains; identifying the optimum mode for measuring future natural luminescence signals. Comparing the remnant dose found at a large aliquot,
small aliquot, and a single grain scale is essential to identifying the degree of partial bleaching of sediment samples and should be used to inform the appropriate mode of measuring natural equivalent doses from other samples in the region.

Results from the remnant dose test are shown for both protocols (OSL130 and pIRIR170) against a variety of aliquot sizes, with equivalent doses calculated following both the unlogged-Minimum Age Model (MAM-3) and unlogged-Central Age Model (CAM) age models following the recommendation of Arnold et al. (2009) (Fig. 3 and Table 3). The results are used to explore whether it is possible to identify an appropriate combination of measurement protocol, mineral and age model selection which reduces the remnant dose to the expected level.

For the pIRIR170 signals measured, the D_e distributions are broadly unimodal, with a multi-modal peak in the large aliquot results potentially a factor of a small sample size or a residual dose associated with a particular aeolian event. D_e distributions increase in width as the number of grains measured decreases (i.e. from large aliquot → small aliquot → single grain measurements). K-feldspar pIRIR170 results vary greatly between the single grain and multigrain measurements, especially when modelled through the two different unlogged-age models. Large aliquot (both MAM_UL and CAM_UL) results and small aliquot CAM_UL results are dominated by a small remnant dose with age estimates ranging from 40 ± 6 to 60 ± 6 years. By contrast, when the MAM_UL is applied to the pIRIR170 small aliquot results, an estimated age 4 ± 8 years highlights the significant potential of pIRIR protocols when dating young sediments and applying MAM age models in depositional settings with incomplete bleaching. Results from section 5 have previously demonstrated the slower bleaching rate of the K-feldspar pIRIR170 signal relative to the quartz OSL130 signal; justifying the choice of the minimum age model in this setting. In contrast with previously published data, the remnant dose measured at the small aliquot scale in this study is smaller than some previously dated modern analogue sediments (e.g. Buylaert et al., 2009; Madsen et al., 2011; Reimann et al., 2012; Thomsen et al., 2008) and compares well with more recent studies (e.g. Brill et al., 2018) which have highlighted the potentially very low remnant doses attainable using pIRIR protocols. These significant results demonstrate the potential application of pIRIR optical dating of K-feldspar grains to aeolian sediments in semi-arid locations with the confidence that accurate age estimates are achievable when moderate stimulation temperatures are coupled with appropriate age models.

When measured at the single grain scale, coupled with the unlogged minimum age model, a negative D_e suggests that this combination is not able to recover the known age of the sediment with

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**Table 3**

| Mineral   | Sample | Protocol | Hot Bleach | Age ± 1σ (years) | Small Aliquot | Large Aliquot |
|-----------|--------|----------|------------|------------------|---------------|---------------|
| K-Feldspar| BBR15/1/1/5 &/10 | pIRIR170 | Y          | −18 ± 7 n = 49  | 52 ± 29 n = 49 | 4 ± 8 n = 11  |
|           |        |          |            | −51 n = 7       | −51 n = 7     | 40 ± 6 n = 49 |
|           |        |          |            | n/a             | n/a           | 60 ± 6 n = 6  |
| Quartz    | BBR15/1/1/10 | OSL130   | N          | −51 n = 7       | −51 n = 7     | 4 ± 7 n = 27  |
|           |        |          |            | 40 ± 6 n = 11   | 30 ± 2 n = 6  | 29 ± 42 n = 6 |

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estimated $D_e$ dominated by the negative values associated with a handful of individual grains. A greater volume of measured single grain measurements could potentially improve this result and yield an estimated age closer to the known age of the sediment. Whereas, results from the single grain K-feldspar dating coupled with the CAMUL equally produce age estimates within 2σ errors of the known sediment burial date (i.e. 2009 AD) and demonstrate the potential for the technique to be applied as a geochronological tool in historical environmental and archaeological research. However, the large spread in the single grain $D_e$’s, coupled with the large uncertainty estimates, (likely driven by the noisy nature of the single grain decay curve) restricts the capacity to calculate more precise age estimates and use in investigating environmental dynamics on timescales at the sub-decadal scale.

For the OSL signals measured, the $D_e$ distributions are unimodal across the three combinations with the results from the large aliquots producing the narrowest spread, but also with the greatest remnant dose due to a bigger combined signal of grains (c.1760 grains for large aliquot, c.109 grains for small aliquot) with varying levels of bleaching, and an individual aliquot which produced a larger $D_e$ outside of the general unimodal distribution. In comparison, single grain results, coupled with the minimum or central age model both demonstrated the lowest residual dose with age estimates indistinguishable from zero. It is likely that the very young (i.e. 2009 AD) age of the sediments means that despite the relatively high dose rates found in the sediments (c.2.2 Gy/ka), individual luminescence decay curves are too dim to extract a measurable decay curve amongst the noise and very few grains are emitting a measurable signal (i.e. only 6 grains out of 400 grains measured produced decay curves for the largest regeneration dose point).

As discussed in Ballarini et al. (2007), the signal levels released by individual grains from recently deposited samples can be much lower than in older sedimentary deposits, a function of a smaller absorbed dose and potentially reduced sensitivity if extracted from newly eroded material, and coupled with a small percentage of grains giving rise to a $D_e$ value (e.g. Duller et al., 2006; Duller and Murray, 2006; Jacobs et al., 2013). Nevertheless, existing studies have equally shown that single grain quartz luminescence dating can successfully be applied to recently deposited sediments, yielding ages within the last 200 years when applying a modified SAR protocol (incorporating an IR wash prior to OSL stimulation) (Olley et al., 2004), the minimum age model, and simulating synthetic small aliquots (e.g. 10 grain aliquots) (e.g. Brooke et al., 2008; Olley et al., 2004).

As Fig. 4 highlights, if all grains provided the same luminescence signal, a linear line through the origin would be plotted. However, results from BBR15/1/1/10 show that over 90% of the OSL signal originates from less than 10% of the grains, suggesting the majority of grains do not contribute to the overall luminescence signal. Likewise, the luminescence signal from aliquots of GSL15/1/3, a much older sediment, is largely driven by less than 40% of the total number of grains.

The most appropriate aliquot size and age model combination for luminescence dating these particular sediments is therefore identified as small aliquots (2 mm) of either quartz OSL coupled with the un-logged central age model which produced an age estimate of 4 ± 7 years or K-feldspar pIRIR70 with the un-logged minimum age model which produced an age estimate of 4 ± 8 years, both of which are in good agreement with the known-age of 5-6 years of the sediments. Given the aeolian dune context of these sediments, we expect quartz sediments to be well-bleached prior to deposition and thus the central age model presents the most appropriate age model for quartz analysis (Bailey and Arnold, 2006). Aeolian sediment deposition in dryland dune environments is likely followed by rapid further burial from deposited sand grains, relying on the bleaching of the luminescence signal to occur during transportation and immediate deposition. The requirement for K-feldspars to be exposed to sunlight for much longer periods of time (e.g. up to 30 days – Table 2) to fully remove the pre-burial dose cannot be guaranteed in the natural environment. Thus, whilst K-feldspar pIRIR70 has reproduced $D_e$ estimates within errors of the known sediment age, the results from the quartz OSL measurements may be more reliable in a context where we cannot guarantee long periods of exposure to sunlight. Alternatively, the most well-bleached population of estimated $D_e$’s could be extracted from pIRIR70 results if combined with the MAM.

Results in this section have suggested that small aliquots of both quartz OSL and K-feldspar pIRIR70 measurements can yield the expected age when aliquot size and incomplete bleaching are taken into consideration. Dose distribution results suggest a tighter clustering of results in the OSL example, whilst a larger spread in $D_e$ is found with pIRIR70 results (Fig. 3) – likely attributed to a range of incomplete bleaching. Consequently, the pIRIR70 MAM age estimate (4 ± 8 years) is largely driven by the $D_e$ associated with a single aliquot (Fig. 3). Whilst the single aliquot has incidentally yielded the expected age in this example, it would not be advisable to assume that the MAM of pIRIR70 results would always yield the correct age unless multiple discs from this age population could be measured. This being said, the K-feldspar results shown are promising and offer an alternative method to OSL protocols across a range of settings. For example, in regions of rapid bedrock erosion, local quartz sediments that have not been sensitised over numerous cycles of bleaching and deposition may be too dim to produce useful luminescence signals for dating. By comparison, as noted earlier, the naturally bright K-feldspar grains coupled with high internal dose rates offers an alternative approach when partially bleached signals can be removed using appropriate age model selection.

7. Selection of appropriate rejection criteria for young samples

Whilst section 6 has identified that both quartz and K-feldspar have the potential to accurately date these young aeolian sediments, this section discusses the details of the appropriate rejection criteria and analysis methods for studying young, dim luminescence signals. In addition to standard recycling and IR-depletion ratio tests, recuperation thresholds and $D_e$(T)-plot analysis of quartz signals were used to identify the most appropriate criteria for including individual aliquots in overall
suggest that rejection based on recuperation rejection criteria is minimal, it has a bleached samples as discussed in section 6), with moisture content 3 ± 2% and overburden density 1.9 g/cm³ used for all samples.

Alternatively, a recuperation threshold can be applied as a % of the largest Regeneration dose. De's have been calculated according to the un-logged Central Age Model with moisture content 3 ± 2% and overburden density 1.9 g/cm³.

Table 4

| Sample     | Aliquots measured | Passed recycling ratio & IR-depletion ratio | Passed recuperation as % of natural (5%) | Passed recuperation as absolute value (1 s) | Passed recuperation as % of largest regen (5%) | Passed recuperation as % of largest regen (5%) |
|------------|-------------------|---------------------------------------------|----------------------------------------|------------------------------------------|-----------------------------------------------|-----------------------------------------------|
| GSL15/1/3  | 48 b              | 37 aliquots                                 | 31 aliquots 1.65 ± 0.063               | 21 aliquots 0.023 ± 0.013                | 27 aliquots 1.7 ± 0.06                       | 37 aliquots 1.7 ± 0.06                       |
| BBR15/1/10 | 30 a              | 27 aliquots                                 | 13 aliquots 0.02 ± 0.018               | 27 aliquots 0.008 ± 0.01                 | 21 aliquots 0.008 ± 0.01                     | 37 aliquots 0.008 ± 0.01                     |

a Small aliquots of fully-prepared quartz grains measured.  
b Recycling ratio and IR-depletion ratio within ± 10% of unity.  
c c.0.0647 Gy based on and beta source conversion 0.0647 Gy/s.  
d Largest regeneration dose ~ c.9 Gy.  
e One aliquot of GSL15/1/3 and three aliquots of BBR15/1/10 were rejected due to partial bleaching. See section 7.3 for rationale.  
f Denotes aliquots that passed recuperation test in addition to the recycling ratio or IR-depletion ratio.

denote that the natural De is not modern or close to the threshold set; prior knowledge of the expected age is required. Alternatively, a recuperation threshold can be applied as a % of the largest regeneration dose (King et al., 2013).

When applied to the quartz luminescence fraction, a comparison of the different recuperation thresholds (Table 4) highlights that whilst the impact on the overall De of older sediments is minimal, it has a much greater influence on the overall De of a very young sediment age where the De is more than doubled when recuperation is applied as a percentage of natural or an absolute value is applied. As noted above these are not appropriate parameters to reject young luminescence signals and can lead to an estimated age overestimation. Whereas, the recuperation rejection when based on a percentage of the largest regeneration does not highlight any additional aliquots that needed to be rejected. Since minimal recuperation has been identified in these particular signals, it is not considered a key rejection criteria to apply to these sediments when measured according to the OSL130 protocol.

The results from the K-feldspar pIRIR170 analysis (Table 5) equally identify aliquots that passed recuperation test in addition to the recycling ratio or IR-depletion ratio is unsuitable for these very young sediments. When measured using the small aliquot population identified as yielding accurate age estimates (4 ± 8 years), application of a recuperation threshold as either 5% of the natural or largest regenerative dose leads to a rejection of all of the aliquots.

When the recuperation threshold is increased to 7% of the largest regenerative dose, 64% of the aliquots are accepted and the revised MAMUL ages overestimate the known age of the sediment. The aliquots yielding the lowest individual De's have been rejected (0.01 ± 0.01 Gy and 0.05 ± 0.01 Gy) due to proportionally high recuperation levels, yet when assessed as an absolute value, the recuperation of these aliquots is indistinguishable from the remaining aliquots. These results therefore agree with the analysis from the quartz fraction, that recuperation does not appear to systematically vary between the aliquots and a threshold value runs the risk of arbitrarily biasing the overall age estimate towards the larger De's measured.

Table 5

K-feldspar Dₐ ± error calculated according to the application of the different recuperation threshold options. Recycling ratio rejection criteria is applied to all five combinations, and subsequently combined with a different threshold based on % of Natural dose, absolute value (seconds), and % of largest Regeneration dose. De's have been calculated according to the un-logged Minimum Age Model (Identified as the most appropriate age model for these partially-bleached samples as discussed in section 6), with moisture content 3 ± 2% and overburden density 1.9 g/cm³ used for all samples.

| Sample     | Aliquots measured | Passed recycling ratio | Passed recuperation as % of natural (5%) | Passed recuperation as absolute value (1 s) | Passed recuperation as % of largest regen (5%) | Passed recuperation as % of largest regen (5%) |
|------------|-------------------|------------------------|------------------------------------------|------------------------------------------|-----------------------------------------------|-----------------------------------------------|
| BBR15/1/10 | 11                | 11 aliquots 0.01 ± 0.02 | 0 aliquots n/a                            | 11 aliquots 0.01 ± 0.02                 | 0 aliquots n/a                                | 7 aliquots 0.08 ± 0.01                       |

a Small aliquots of fully-prepared K-feldspar grains measured.  
b Recycling ratio within ± 10% of unity.  
c c.0.0647 Gy based on and beta source conversion 0.0647 Gy/s.  
d Largest regeneration dose ~ c.9 Gy.  
e Denotes aliquots that passed recuperation test in addition to the recycling ratio.
than the remaining aliquots are also those which display rising $D_e(t)$-plots and Z-values > 1 (Fig. 5). Z-values refer to the ratio of the $D_e$ from the final integral to that of the first channel (Bailey, 2003). By replacing the natural dose with a regeneration dose point, it is possible to test whether this rising $D_e(t)$-plot is driven by partial bleaching, or a dominant slow component within these sediments. Under controlled bleaching conditions, we would not expect a rising $D_e(t)$-plot associated with the regenerative dose point unless the signal has been dominated by the slow component in these samples with an overall low luminescence signal.

When calculated with a regeneration dose point, results suggest aliquots ‘B’ and ‘C’ were partially bleached (Z-value < 1) (Table 6); it is likely that these aliquots contain a couple of grains that were not well-bleached in the reactivation event. Whilst the majority of grains may be well-bleached, the near-surface nature of the samples may experience post-depositional mixing (Bateman et al., 2007) or partial-bleaching of due to deposition at night (insufficient ambient light) or during a rapid process (e.g. dust storm), preventing every grain from being fully-bleached. Meanwhile, aliquot ‘A’ continues to demonstrate a rising $D_e(t)$-plot despite being bleached within the SAR cycle prior to further irradiation and measurement.

Through component fitting of the regenerative dose luminescence decay curve for aliquot ‘A’, the contribution of each of the individual components of the OSL decay curve can be calculated. Fig. 6 highlights that the slow and medium components of the signal are contributing to almost 60% of the total luminescence signal in the first instance, and rapidly rise to 100% of the overall signal within 2 s of measurement time.

The reason for the rise in $D_e(t)$ remains unclear, but may be associated with incorrect background subtraction (e.g. if there is significant decay of the slow component in the time prior to background

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**Table 6**

| Aliquot | Z-value (Natural) | Z-value (Regen point) |
|----------|------------------|----------------------|
| A        | 1.67             | 1.3                  |
| B        | 16               | 0.42                 |
| C        | 4.60             | 0.77                 |

Fig. 5. (a) Z-value vs. $D_e$ (based on initial integration window) plot based on $D_e(t)$-plots of Bailey (2003). Dashed line: aliquots inside dashed circle represent partially-bleached signals that contain a greater proportion of the pre-burial dose. Dotted line: aliquots inside dotted circle are those with large Z-values, but low $D_e$ values. (b) Z-value vs. $D_e$ (based on initial window) and $D_e$ (based on final integration window). Aliquots with typically high Z-values and low $D_e$ are shown to have over-lapping initial and final $D_e$ values. The large uncertainties associated with these aliquots is driven by the noisy dim signal and requires further analysis than > 1 Z-value to qualify as partially bleached. Likewise, some aliquots with high Z-values are artificially driven by negative final $D_e$ values and equally are not partially bleached despite Z-value > 1. Three aliquots highlighted in red (‘A’, ‘B’, ‘C’) depict those which show Z-values which suggest partial bleaching – initial and final $D_e$ values do not overlap, Z-values > 1, and all $D_e$’s > 0. For the remaining samples with high Z-values, error bars associated with the Z-values and $D_e$ estimates demonstrate that whilst displaying high Z-values, the $D_e$’s at various integration intervals are within errors and therefore the > 1 Z-values have not been analysed further. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
measurement). As a conservative measure, we suggest rejecting all aliquots that display this signal feature in these sediment samples, to reduce the likelihood of ‘false-positives’. Aliquots which display these characteristics should either be rejected since they will lead to a significant age overestimation of young samples, or individual fast components need to be extracted from the overall luminescence decay and De’s calculated accordingly for that individual component. Previous curve fitting experiments (e.g. Jain et al., 2003; Tsukamoto et al., 2003) have demonstrated that the fast component shows less recuperation due to thermal transfer when preheating than the slow and medium components; another reason why luminescence signals with a strong fast component should be selected, especially for young samples with a comparatively small De.

8. Conclusions

In this study, sediments extracted from a very young lunette dune of known-age (5–6 years) provided a reliable test case for identifying the most suitable measurement and analysis combinations for dating young aeolian dune sediments. Given the young nature of the sediments, identifying the most rapidly bleached signal is imperative to ensure low remnant doses and a more accurate chronology is produced. Whilst K-feldspar rich samples showed slower bleachability results when left to bleach under natural conditions versus quartz counterparts, when measured using the pIRIR170 protocol and paired with MAMUL, age estimates of a known age sediment were both accurate and equally as precise as the quartz OSL130 and K-feldspar pIRIR170 measurements. pIRIR protocols have increased the suitability of K-feldspar luminescence dating to a range of research projects, identifying and stimulating deeper electron traps which exhibit reduced levels of anomalous fading, yet require much longer bleaching periods to reduce residual doses and improve dating accuracy when measuring young sediments in a dynamic environment. Results from this study are in agreement with those reported by Buylaert et al. (2012) which show that the pIRIR signal bleaches more slowly than that from quartz when exposed to sunlight, increasing the likelihood and size of a residual dose; reducing the applicability of this protocol to young aeolian sediments if not paired with the most appropriate age model.

Single-grain pIRIR measurements yield more varied results, likely attributed to a small sample size and relatively low photon count. Nonetheless, the success of the small aliquot dating is significant and demonstrates that pIRIR methods have the scope to produce accurate luminescence ages with reduced levels of fading than IRSL equivalents. As expected, both large and small aliquots of quartz OSL130 measurements produce De values in line with the expected age based on historical data. Due to fast bleaching rates the un-logged central age model provides the most appropriate age model to use when calculating age estimates.

Additionally, we find a revised set of rejection criteria is useful for accurately identifying aliquots/grains for reliable age estimation. In particular, sensitivity testing a range of recuperation rejection criteria highlights the caution that should be taken to avoid arbitrarily applying rejection criteria and biasing towards age overestimations. Problems of incomplete bleaching or slow component dominance of the occasional grain have highlighted the importance of selecting the most appropriate aliquot size for measurement and rejection criteria, analysis and age-model selection post-measurement.

Investigations into aliquot sizes show that without a widespread issue with partial bleaching in the quartz fraction, single grain analysis is not needed for De calculation and small aliquots have an advantage of producing greater signal sizes. Whilst large aliquots would provide an even greater signal, they are susceptible to partial bleaching or slow component dominance which lead to an overall age overestimation. Thus, a trade off option that allows us to maximise the signal but not miss the aliquots that hide issues of partial bleaching is needed. In this study, small aliquots of both quartz OSL130 and K-feldspar pIRIR170 have demonstrated the most promising results, allowing us to identify partial bleaching of K-feldspar through bleachability tests, dose distribution plots and the MAM, whilst partially bleached quartz grains are identified through the use of De(t)-plots and Z-values, but without the weak noisy signals of the single grain analysis. When markedly larger De’s are identified within individual aliquots, D(t)-plots and component-fitting analysis can be used to identify the most well-bleached quartz aliquots and those which have a dominant fast component.

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Appendix A. Supplementary data

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