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Comparison of paired quartz OSL and feldspar post-IR IRSL dose distributions in poorly bleached fluvial sediments from South Africa

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ABSTRACT

A comparative study using quartz optically stimulated luminescence (OSL) and feldspar post-infrared infrared stimulated luminescence (post-IR IRSL) was undertaken on Quaternary fluvial sediments from an unnamed tributary of the Moopetsi River in South Africa. The aim is to assess whether the post-IR IRSL signal can be used to date incompletely bleached sediments. Several post-IR IRSL signals using varying stimulation and preheat temperatures were investigated; of these the post-IR IRSL₂₂₅ signal was deemed most appropriate for dating because it bleached most rapidly. The feldspar post-IR IRSL₂₂₅ equivalent dose (D_e) values from this site are consistently larger than those from quartz OSL, probably due to differences in the bleaching characteristics of the two signals. Additionally, the post-IR IRSL₂₂₅ D_e values within a sample showed less variation in precision than the quartz D_e data, possibly due to greater averaging between grains in the feldspar small aliquots. The agreement between ages based on the OSL and post-IR IRSL₂₂₅ signals was better for younger samples (<20 ka) than for older ones (>50 ka); the cause of this variation is unclear.

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1. Introduction

Across the South African interior, numerous dongas (gullies and badland-type environments) have formed within Quaternary colluvium and alluvium. Within these dongas, exposures of up to 30 m in thickness reveal multiple phases of sedimentation, palaeosol formation and erosion. Luminescence dating techniques have been used to determine the chronology of sediment accumulation and to explore linkages between episodes of deposition and palaeoenvironmental changes reconstructed from various other proxy records (e.g. Botha et al., 1994). At many sites, the focus has been on late Quaternary deposition prior to widespread late Holocene erosion and the basal parts of many successions remain undated, limiting the reconstruction of important palaeoenvironmental changes earlier in the Pleistocene. Most recent investigations using luminescence dating in South Africa have used quartz optically stimulated luminescence (OSL) (e.g. Lyons et al., 2013, 2014) with derived ages extending over the last ~50 ka. Saturation of the quartz OSL signal may limit the ability to obtain older ages (e.g. Chapot et al., 2012), therefore we need to consider a different

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luminescence chronometer.

Feldspars have been suggested as a dosimeter that can be used to extend luminescence dating beyond the range covered by quartz OSL (e.g. Buylaert et al., 2012). The post-infrared infrared stimulated luminescence (post-IR IRSL) protocol was proposed by Thomsen et al. (2008) to minimise the impact of anomalous fading. The aim of this paper is to assess whether the post-IR IRSL signal from feldspars can be used to date poorly bleached fluvial sediments, and also to extend the age range of luminescence dating beyond that which is possible using the OSL signal from quartz. A site in north-eastern South Africa comprised of fluvial sediments is used in this study; a suite of 10 luminescence ages based upon the OSL signal from quartz already exists (Lyons, 2012) and can be compared with new data obtained using feldspar post-IR IRSL signals.

2. Study site

The Moopetsi River is a small tributary (catchment area \sim 228 km²) of the Steelpoort River which in turn joins the Limpopo River. The catchment lies \sim 280 km north-east of Johannesburg near the town of Steelpoort in Limpopo Province. The south-westerly part of the area is underlain by mafic lithologies of the Bushveld Complex. In this part of the catchment dongas have developed

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along many of the valleys (Fig. 1a), cutting through thick (up to ~30 m) alluvial and colluvial sedimentary sequences down to, and locally into, bedrock (Lyons, 2012). River channel bank and donga exposures reveal a sedimentary succession consisting of a series of stacked fluvial sediments ranging up to gravelly sand, intercalated with two main palaeosols (Fig. 1b). The small size of the Moopetsi catchment means that fluvial transport distances are short (<5 km), and this may limit the opportunities for exposure of sediments to daylight prior to deposition.

As part of a wider geomorphological study of the Moopetsi catchment (Lyons, 2012), a suite of ten samples was collected from an unnamed tributary of the Moopetsi River (Fig. 1) at various locations along a 0.5 km reach. This approach was taken due to variations in unit thickness, and to ensure that the age of each unit was bracketed. Samples were dated using measurements of the OSL signal from small aliquots of coarse-grained quartz (Lyons, 2012). The OSL ages are in stratigraphic order, with one exception, and range from 0.20 \pm 0.02 ka to 119 \pm 7 ka (Table 1). As expected for fluvial samples that have experienced short transport distances, the quartz OSL signal has been heterogeneously bleached prior to deposition. This was demonstrated by the high overdispersion (110%) calculated for the quartz D_e values for the youngest sample (Aber170/MPT8); ages for the four youngest samples were calculated using the minimum age model (MAM) to overcome the impact of this heterogeneous bleaching. For older samples, the impact of any residual dose remaining at the time of deposition was small, and for these samples the central age model (CAM) was used. Previous work on use of the post-IR IRSL signal from feldspars has indicated that this signal bleaches more slowly during exposure to daylight than the OSL signal from quartz (Buylaert et al., 2012), and so using post-IR IRSL methods to date these fluvial sediments may be challenging. The suite of ten samples from the unnamed tributary site provides an excellent opportunity to compare results obtained using the OSL signal from quartz and the post-IR IRSL signal from feldspars.

3. Methods and instrumentation

Samples were collected by hammering metal tubes into fluvial deposits exposed along the tributary channel margins and returned to the laboratory so that preparation could be completed under red light conditions. Carbonates and organic material were removed with a 10% v.v. dilution of concentrated (37%) hydrochloric acid (HCl) and 20 vols hydrogen peroxide (H₂O₂) respectively. Samples were dry-sieved, and the 180-212 µm fraction was retained for all further preparation and measurements. Mineral fractions were separated on the basis of density using sodium polytungstate solutions with densities of 2.62 g/cm³ and 2.70 g/cm³ for quartz, and 2.53 g/cm³ and 2.58 g/cm³ for K-rich feldspar. As part of the previous study (Lyons, 2012), quartz grains were etched in 40% hydrofluoric acid (HF) for 45 min in order to remove the alphairradiated surface of the grains and any remaining feldspars prior to resieving. K-rich feldspar grains for the present study were not etched in HF due to concern about the anisotropic removal of the surface

Luminescence measurements were undertaken using automated Risø TL/OSL-DA-15 and DA-20 readers. Any guartz stimulation in this study used blue LEDs (470 nm; 80 mW/cm²; Bøtter-Jensen et al., 2010) and detection used an EMI 9635 QA photomultiplier fitted with 7.5 mm thickness of Hoya U-340 filter. Infrared stimulation of feldspar used an IR LED array (870 nm; 135 mW/cm²; Bøtter-Jensen et al., 2010) and detection in the blue region of the spectrum using a combination of 2 mm BG-39 and 2 mm Corning 7-59 glass filters. Laboratory irradiations were made using two calibrated ⁹⁰Sr/⁹⁰Y beta sources, with a dose rate of 0.097 Gy/s for the source used for feldspar measurements and 0.038 Gy/s for the source used for guartz measurements. Measurements on guartz were made using the single aliquot regenerative dose (SAR) protocol described by Murray and Wintle (2000), whilst a post-IR IRSL procedure based on Thomsen et al. (2008) was used for feldspars exploring a range of stimulation temperatures from 225 to 290 °C. The protocols used for measurements of quartz



Fig. 1. a) Map of the Moopetsi River catchment showing the location of the sampling site and the contact between the lithologies of the meta-sedimentary Transvaal Supergroup (stippled) and the mafic Bushveld complex. Inset shows the location of the study area in South Africa. b) The location of samples within the composite stratigraphy, ~10 m at its maximum thickness. Sample ID is preceded by Aber170/MPT or Aber162/MPT in the text, i.e. Aber170/MPT8 represents the youngest sample from the suite.

Table 1

Equivalent dose (D_e) distribution data obtained using quartz OSL and feldspar post-IR IRSL₂₂₅ measurements, and ages calculated for the ten samples using both minerals (shown to 3 significant figures). Also shown is the number of aliquots used in the D_e estimate (n), the overdispersion of the D_e distribution (OD) and the age model used to derive each D_e value.

Sample ID ^b	Quartz OSL ^a					Feldspar post-IR IRSL ₂₂₅				
	n	OD (%)	Age model ^c	D _e (Gy)	Age (ka)	n	OD (%)	Age model ^c	Measured D_e (Gy)	Age ^d (ka)
MPT8	44	110	MAM	0.37 ± 0.04	0.20 ± 0.02	33	45	MAM	11.5 ± 1.3	0.20 ± 0.68
MPT12	48	31	MAM	8.18 ± 0.62	3.45 ± 0.30	31	37	MAM	32.2 ± 1.3	6.72 ± 0.62
MPT13	43	59	MAM	9.05 ± 0.51	4.11 ± 0.30	39	30	MAM	22.6 ± 1.9	3.88 ± 0.78
MPT14	42	59	MAM	7.29 ± 0.31	4.53 ± 0.28	39	27	MAM	18.4 ± 2.6	3.10 ± 1.24
MPT3	47	19	CAM	11.3 ± 0.3	6.72 ± 0.35	39	33	MAM	29.1 ± 1.0	7.38 ± 0.71
MPT4	48	17	CAM	20.6 ± 0.5	9.15 ± 0.47	31	11	CAM	46.1 ± 1.0	11.7 ± 0.9
MPT10	38	24	CAM	103 ± 4	50.6 ± 3.0	37	15	CAM	257 ± 7	88.2 ± 4.4
MPT9	42	25	CAM	158 ± 6	106 ± 6	5	10	CAM	608 ± 45	268 ± 23
MPT6	49	16	CAM	161 ± 4	90.8 ± 4.7	4	0	CAM	664 ± 34	258 ± 17
MPT7	36	25	CAM	170 ± 8	119 ± 7	15	13	CAM	643 ± 29	292 ± 18

^a Quartz data are based on Lyons (2012).

^b The full laboratory codes for these samples are Aber170/MPT8, Aber170/MPT13 etc., except for Aber162/MPT3, Aber162/MPT4 and Aber162/MPT6.

^c A sigma-b value of 0.15 was used for the minimum age model calculations using both quartz OSL and feldspar post-IR IRSL₂₂₅ measurements.

 d Ages using feldspar post-IR IRSL₂₂₅ measurements were calculated after subtracting a residual dose of 11.0 \pm 1.3 Gy from the D_e as described in the text.

OSL and feldspar post-IR IRSL signals in this study are detailed in Table S1. All feldspar measurements in this study were made on small aliquots (2 mm diameter) to enable direct comparison with the quartz OSL ages previously determined (Lyons, 2012). Feldspar post-IR IRSL D_e values were determined by integrating the initial 4 s of the decay curve after subtraction of a late background, taken from the last 20 s of the decay curve. Acceptance criteria for the feldspar data required individual aliquots to have recycling ratios within 10% of unity, and recuperation less than 5% of the natural signal.

Dose rates were calculated using a combination of thick-source alpha counting and GM-beta counting. The dose rate for K-feldspar grains includes an internal beta dose calculated assuming a K-content of $12.5 \pm 0.5\%$ (Huntley and Baril, 1997), and because the grains were not etched in HF an external alpha dose rate was calculated from the U and Th concentrations of the surrounding sediment. After correction for the a-value and grain size attenuation, the alpha dose rate ranged from 0.044 to 0.109 Gy/ka. Conversion factors were taken from Adamiec and Aitken (1998). Dose rate data are presented in Table S2.

4. Comparing the bleaching rate of various signals from quartz and feldspar

In order to measure the rate at which different luminescence signals from quartz and feldspar decrease due to exposure to light, a bleaching experiment was conducted using nine small aliquots of sample Aber162/MPT4, three for each signal (quartz OSL, feldspar post-IR IRSL₂₂₅ and post-IR IRSL₂₉₀). For the feldspar aliquots, the IRSL signal at 50 °C (IRSL₅₀) was also measured (step 3 in Table S1b). To reduce sensitivity change between the various measurement steps and to ensure reproducibility of the signal, aliquots were measured repeatedly prior to the bleaching experiment following the protocols shown in Table S1, using a dose of ~45 Gy in step 1. The experiment then continued by giving a beta dose of ~45 Gy and measuring the luminescence signal remaining after exposure to light in a Honlë SOL-2 for varying amounts of time. All measurements were normalised to the measurement where no exposure to the SOL2 had occurred (Fig. 2).

As expected from previous work (Buylaert et al., 2012), the quartz OSL signal bleached more rapidly than both the feldspar IRSL₅₀ and the post-IR IRSL signals (Fig. 2). After 10 s of bleaching, the quartz OSL signal was reduced to 5% of the original signal whilst the post-IR IRSL₂₂₅ signal was only reduced to ~86%, and the post-IR



Fig. 2. Bleaching of the quartz OSL signal, and feldspar post-IR IRSL₂₂₅, post-IR IRSL₂₉₀ and associated IRSL₅₀ signals by exposure to the Honlë SOL-2 solar simulator. Data are normalised to the response when no exposure has occurred.

IRSL₂₉₀ signal showed negligible reduction compared to the original signal. The post-IR IRSL₂₂₅ signal required 4 days of bleaching in the SOL-2 to reduce the signal to 5% of the original signal, whilst the post-IR IRSL₂₉₀ signal took 14 days to reach this level.

It is interesting to note that over a period of exposure to the SOL-2 in excess of 1 million seconds (14 days), all of the luminescence signals show a monotonic decrease; there is no indication of a residual post-IR IRSL signal that cannot be removed by exposure to this light source. However, Fig. 2 does emphasise the slow rate at which the post-IR IRSL signals bleach in the laboratory in comparison with the quartz OSL signal. Given the high overdispersion (110%; Table 1) of the quartz dataset for the youngest sample (Aber170/MPT8), and the slower bleaching rate of feldspar relative to quartz (Fig. 2), it is likely that large residuals will be observed in post-IR IRSL signals of these samples from the unnamed tributary of the Moopetsi.

5. Selecting appropriate measurement parameters for feldspar

A variety of preheat temperatures and post-IR IRSL measurement temperatures are possible when applying the post-IR IRSL method (e.g. Thomsen et al., 2008; Buylaert et al., 2009; Thiel et al., 2011; Roberts, 2012). A series of experiments was undertaken to identify the most appropriate post-IR IRSL measurement conditions for these samples.

5.1. Using a dose recovery test to assess the residual signal

A dose recovery test was undertaken on sample Aber162/MPT4. A total of 20 small aliquots were prepared and bleached in a Honlë SOL-2 for 48 h, in order to remove trapped charge previously accrued during burial. The aliquots were split into four groups, each containing five aliquots. Within each group, two aliquots were given no dose, while three aliquots received an irradiation of ~45 Gy. Each of the four groups used a different preheat and post-IR IRSL stimulation temperature for the post-IR IRSL protocol shown in Table S1b. Stimulation temperatures (step 4, Table S1b) were 225 °C, 250 °C, 270 °C or 290 °C, and the preheat temperature (step 2, Table S1b) was 30 °C higher than the stimulation temperature, except for the post-IR IRSL₂₂₅ signal where the preheat was 25 °C higher so as to be consistent with previous protocols (e.g. Buylaert et al., 2009).

As anticipated from Fig. 2, the residual dose remaining in the bleached aliquots that were given no dose increases with increased preheat temperature (Fig. 3a). The ratio of the measured to given dose (after subtraction of the residual dose shown in Fig. 3a) was within 10% of unity for post-IR IRSL₂₂₅₋₂₇₀ signals measured following preheats of 250 °C-300 °C, and all four signals (post-IR IRSL₂₂₅₋₂₉₀) passed the dose recovery test within errors (Fig. 3b), suggesting that any of the measurement protocols would be suitable for dating this material.

5.2. Anomalous fading

A major issue affecting the use of feldspars in luminescence dating is the effect of anomalous fading of the luminescence signal (Wintle, 1973), which typically results in age underestimation if it is not corrected for. A fading test was conducted on sample Aber162/MPT4 for the same post-IR IRSL_{225–290} signals used in the dose recovery test. Five aliquots were prepared for each of the four signals. After measuring the natural D_e, aliquots received a dose of ~45 Gy, were preheated immediately after irradiation following Auclair et al. (2003), and stored for varying periods of time before



Fig. 3. Dose recovery test (given dose ~45 Gy) showing a) the measured residual signal after a 48 h bleach in the Honlë SOL-2 and b) the ratio of the measured dose to the given dose. Shown in c) are the fading rates (g-values) calculated for the same sample using Eq. (4) of Huntley and Lamothe (2001) and normalised to 2 days.

measurement. The delay between irradiation and measurement ranged from 10 min to 47 days. Irrespective of the stimulation temperature used, g-values are all less than 1.5%/decade (Fig. 3c). Thiel et al. (2011) have suggested that very low fading rates such as this may be a laboratory artefact. In support of this argument, they presented a g-value of $1.3 \pm 0.3\%$ /decade that they had measured from quartz, which is thought not to suffer from anomalous fading. The low g-values measured for the unnamed tributary samples imply that there is minimal fading of the post-IR IRSL signal and hence again any of these signals could be used to date these samples.

In summary, all of the investigated post-IR IRSL signals exhibit minimal fading and have been shown to recover a laboratory-given dose, to recycle within errors, and to show recuperation of less than 5% of the natural signal. The post-IR IRSL₂₂₅ signal was selected for measurement of the D_e distribution of the ten unnamed tributary samples because it bleaches most rapidly in the SOL-2, giving the lowest residual dose (Fig. 3a). Working with the most rapidly bleaching of these post-IR IRSL signals is important given the fluvial depositional context of the study site.

6. Comparison of D_e distributions

Equivalent dose (D_e) values were measured using the protocol in Table S1b, using between 31 and 39 small aliquots of K-feldspar for the seven uppermost samples in the composite stratigraphy (Table 1). The number of replicate D_e measurements (n = 4 to 15) for the three lowermost samples was smaller because limited material was available for some samples. Fig. 4 plots the D_e values obtained from feldspar along with those from quartz for the youngest sample (Aber170/MPT8). Fig. S1 presents similar comparisons for all ten of the samples in this study and the data are summarised in Table 1.

As seen for quartz, overdispersion (OD) values of the feldspar De



Fig. 4. Quartz OSL and feldspar post-IR $IRSL_{225}$ D_e distributions for the youngest sample, Aber170/MPT8. Quartz OSL data are shown as closed circles and feldspar post-IR $IRSL_{225}$ data as open triangles. The grey lines represent the D_e values for quartz and feldspar calculated using the minimum age model, 0.37 Gy and 11.5 Gy respectively.

data are generally larger for the younger samples (Table 1), and in cases where the OD was greater than 20%, the MAM was applied to extract the population with a signal that had been bleached most completely at deposition. For the five oldest samples, the OD in the feldspar data was less than 20% and hence the CAM was applied (Table 1). It is interesting to note that although the feldspar post-IR IRSL₂₂₅ signal of Aber162/MPT3 is poorly bleached the quartz signal is well bleached, as shown by the D_e distributions (Fig. S1) and the OD values of 19% for quartz and 33% for feldspar. This observation is in agreement with the different bleaching rates for the quartz and feldspar signals shown in the bleaching experiment (Fig. 2).

Comparison of the De distributions from the two minerals (Fig. 4; Fig. S1 and Table 1) shows that the feldspar post-IR IRSL₂₂₅ signal gives consistently higher De values than those obtained using quartz OSL. This offset is much greater than can be explained by the difference in the dose rate using these two minerals, and hence ages calculated using the post-IR IRSL₂₂₅ signal (Fig. 5) are consistently higher than those from quartz OSL signals (Fig. 5). This difference in apparent age is most likely to result from differences in the rate at which these two signals bleach (Fig. 2). The MAM De value for the youngest sample (Aber170/MPT8) is 0.37 ± 0.04 Gy for guartz and 11.5 ± 1.3 Gy for feldspar (Fig. 4; Table 1). If one assumes that the age calculated using the MAM for guartz is accurate, and that both minerals are dating the same depositional event as they are taken from the same physical sample, then the magnitude of the post-IR IRSL₂₂₅ residual De at the time of deposition can be calculated, giving a value of 11.0 ± 1.3 Gy.

The geomorphic context and sedimentology of the samples in this study suggest that the depositional environment has remained similar over time, and it may therefore be reasonable to assume that a similar residual D_e (11.0 ± 1.3 Gy) was present in all samples at the time of deposition. After subtraction of this residual value, ages calculated using the post-IR IRSL₂₂₅ signal are broadly similar to those from quartz for the six youngest samples (Fig. 5; Table 1).



Fig. 5. Comparison between ages calculated using signals from quartz OSL (circles) and feldspar post-IR IRSL₂₂₅ (triangles). The red triangles represent post-IR IRSL₂₂₅ ages without the residual subtraction and the yellow triangles represent the ages calculated after subtraction of a residual of 11.0 \pm 1.3 Gy. The inset is an enlarged version of the dashed box. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Excluding the youngest sample that was used to determine the residual, for three of the five samples (MPT13, MPT14 and MPT3) the quartz and post-IR IRSL ages overlap within one sigma, one sample (MPT4) overlaps at two sigma, and one sample (MPT12) is just beyond two sigma (inset to Fig. 5). The uncertainty in the ages calculated using the post-IR IRSL₂₂₅ signal is consistently larger than that from quartz, in part due to subtraction of the residual from the measured D_e which introduces additional uncertainty into the final age.

A feature that is common to all of the paired quartz OSL and feldspar post-IR IRSL₂₂₅ dose distributions (Fig. 4 and S1) is that there is less variation in the precision of D_e values for individual small aliquots of feldspar than is seen for small aliquots of quartz. This could result from a greater variation in luminescence efficiency for individual grains of quartz compared with feldspar, or it may be caused by a higher proportion of feldspar grains giving a luminescence signal than quartz (Duller et al., 2003) and thus greater averaging for the small aliquots of feldspar than for the same size aliquots of quartz.

The ages calculated for the four oldest samples using the two minerals differ dramatically (Fig. 5; Table 1) with the post-IR IRSL225 ages being much older than those from quartz OSL. At least two explanations may be suggested. The first is that the quartz ages are underestimated owing to the quartz OSL signal approaching saturation. Chapot et al. (2012) showed for samples from Chinese loess that natural and laboratory dose response curves for quartz OSL deviated above ~150 Gy. At the sampling site in this study, the three oldest samples have D_e values between 158 and 170 Gy (Table 1), all of which are above the threshold found by Chapot et al. (2012), but much lower than $2D_0$ for these samples, which is ~280 Gy. A second possibility is that the post-IR IRSL225 signal in these older sediments was not reset at deposition, and that the residual signal at deposition was not ~11 Gy as suggested previously, but of the order of ~450 Gy. A residual of this magnitude may be explained if these lowermost sediments were dominantly sourced from local bedrock by scouring rather than being derived from reworking of older fluvial sediments, but no sedimentological evidence exists to support this idea. At this time it is not clear which chronology is more accurate for these older samples.

7. Conclusions

Laboratory characterisation of the post-IR IRSL₂₂₅ signal from feldspars from an unnamed tributary of the Moopetsi River has shown that this signal is suitable for dating but the signal is also slow to bleach, taking ~4 days in the SOL-2 to reach <5% of the original signal. However, De distributions measured for quartz and feldspar on the same samples shows that they are offset from one another (Table 1; Fig. S1), with post-IR IRSL₂₂₅ De values consistently larger than those from quartz OSL. Analysis of a young sample (Aber170/MPT8) suggested that even when applying the MAM to D_e data obtained using small aliquots of feldspar, a residual dose of 11.0 ± 1.3 Gy remained at deposition. After subtracting this residual dose from the other De values determined at this site, broad agreement was found between quartz and post-IR IRSL225 ages for the six youngest samples. However, even after subtracting this residual dose, the four oldest samples gave post-IR IRSL ages that were up to 175 ka older than the corresponding quartz OSL ages. The cause of this discrepancy is unknown. Although the results from the younger samples are encouraging, it is not clear whether small aliquot measurements of feldspars will be useful for dating older sediments in geomorphological settings similar to the Moopetsi and its tributaries where fluvial transport distances are very short (<5 km).

The comparison of small aliquot quartz and feldspar D_e data in

this study highlights the impact of the different bleaching rates of these two minerals, and potentially the different degree of averaging that occurs when using small aliquots. Use of a post-IR IRSL signal may potentially extend the age range that can be dated with luminescence, but the relatively slow bleaching rate of the post-IR IRSL signal poses a challenge when considering samples where exposure to daylight at deposition may have been limited.

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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.2015.02.015.

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