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# 1 Luminescence Dating of Holocene Siliciclastic Sediments in Eastern Dahomey Basin,

# 2 Southwestern Nigeria

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# 20 Abstract

- 21 Several attempts at reconstructing geological settings and palaeoclimatic changes of the
- 22 siliciclastic deposits of the Dahomey Basin, SW Nigeria, using relative age dating and
- 23 correlation methods, have resulted in serious discrepancies on the ages. Therefore, a
- chronology framework established by an absolute age dating method is requisite to constrain
- the geological interpretation. This research focuses on quartz optically stimulated
- 26 luminescence (OSL) dating of the upper siliciclastic sediments to help bridge the lacuna that
- 27 arose from previous relative geologic dating. Ten sub-surface sediment samples were
- collected from the eastern part of the basin, and quartz OSL dating using single-aliquot
- 29 regenerative-dose protocol was conducted for all the samples. The OSL signals appear well
- 30 bleached prior to deposition and the OSL ages are reliable and robust. Through the
- 31 application of OSL, the age framework of the uppermost part of sediments in the study area

32 was established. The OSL dating results revealed that these depositional periods fall within

the Holocene and are concentrated during two groups: 3.52 ka–1.55 ka, and 0.64 ka–0.05 ka.

- 34 The samples with ages of 3.52 ka–1.55 ka distribute in the belt-like inland zone
- 35 approximately parallel to the coastline. This deposition episode appears to be caused by the
- 36 decrease in relative sea level during late Holocene. Thus, this study sheds light on the
- 37 understanding past coast dynamics in the region.

38 Keywords: Luminescence dating; Quaternary; Holocene; Dahomey Basin; Nigeria

### 39 **1. Introduction**

Various research work has been conducted within the Dahomey Basin due to their geologic peculiarity and economic importance (Adegoke, 1980). Exploration of bitumen, limestone, glass sands and phosphates (Nton et al., 2006) has been undertaken with discovery of crude oil in 1908 (Billman, 1982). This has sprung up great geological interest for further research in the basin for hydrocarbon potential (Elueze and Nton, 2004). The knowledge of age of the deposits will provide crucial information for future exploration of these natural resources and understanding the evolution of coastal environment in the Dahomey Basin.

47 Previous researches had postulated ages for the sediments in the Dahomey Basin through the 48 application of series of relative age dating approaches, including age correlation of the basin 49 (Jones and Hockey, 1964; Reyment, 1965), dating of available pollen (Agagu, 1985) and 50 paleontological approach (Adegoke, 1969; Olabode and Mohammed, 2016). This has resulted 51 in serious age discrepancies ranging from Maastrichtian to Quaternary ages (Olabode et al.,

52 2016). Hence, there is an urgent need to adopt absolute age dating method to resolve and

53 bridge the lacuna arising from the various age differences of the siliciclastic deposits.

54 Optically simulated luminescence (OSL) is one of the most intensively and veritably used

numerical dating techniques to determine the age of Late Quaternary sub-surface sediments

56 (Murray and Wintle, 2000, 2003; Wintle and Murray, 2006; Chen et al., 2015; Zhang et al.,

57 2018). OSL dating techniques (Duller, 2004; Olley et al., 1998, 1999) work well not only on

sediments where grains have adequate exposure to sunlight at the time of deposition (Rhodes,

59 2007), but also on samples which were poorly bleached prior to sedimentation (Zhao et al.,

60 2015, 2017). It is applied by estimating the impact of radiation on the crystalline structure of

61 quartz mineral mostly presented in all sedimentary environments isolated from light (Wintle,

62 1997, 2008; Murray and Wintle, 2000, 2003). The OSL signal observed from quartz revealed

- 63 several exponentials relative to different levels of traps (Bailey et al., 1997) and successful
- 64 OSL dating is dependent on a fast signal component (Murray and Wintle 2000). This research
- aims to determine the age of the uppermost alluvial deposits in the eastern Dahomey Basin to
- 66 help bridge the long touted age disparities of the formation in previous research (Jones and
- 67 Hockey, 1964; Agagu, 1985; Omatsola and Adegoke, 1981).

# 68 2. Geologic and tectonic settings of the Dahomey Basin

The Dahomey Basin, also called Benin Embayment, is located on the shore of West Africa 69 (Whiteman, 1982), with coordinate of latitude 6°10'N to 6°25' N and longitude 4°30' E to 70 4°50' E respectively (Fig. 1). It is a rift or marginal pull-apart basin (Whiteman, 1982) or 71 marginal sag basin (Kingston et al., 1983), part of the West African pre-cratonic basins 72 (Mpanda, 1997) which was initiated during the early Cretaceous separation of the South 73 American and African plates, and opening of the South Atlantic Ocean (Adegoke, 1980; 74 Omatsola and Adegoke, 1981). The Dahomey Basin is a combination of inland, coastal, 75 offshore settings that stretches from southeastern Ghana through Togo and the Republic of 76 Benin to southwestern trending along the east-west direction in Nigeria, with Cretaceous 77 strata along the shore estimated to be about 200 m thick (Okosun, 1990; Olabode and 78 79 Mohammed, 2016). The geologic, stratigraphic, sedimentological and organic geochemical studies of different parts of the alluvial deposits in the Dahomey Basin have been reported 80 (e.g., Idowu et al, 1993; Adekeye, 2004; Adekeye et al., 2006; Nton et al., 2006; Ikhane et al., 81 2014). 82

The study area is located in eastern part of the Dahomey Basin, in the area or vicinity of Ilaje 83 84 community in coastal environment of Ondo state (Fig. 1). The coastal vegetation along the beach is dominate mangrove (Awosika and Folorunsho, 2010) with some coconut trees, 85 palms, sedges and climbers. The study area is a barrier lagoon coastal complex (Woodroffe 86 and Horton, 2005; Awosika and Folorunsho, 2010), stretching from around Agerige 87 community where the coastline starts a southward variation. It consists of beach ridges 88 adjoined with a foreshore of more than 50 m above the sea level similar to that of modern 89 coast. The beach crest elevation generally ranges from 3 to 4 m above mean water level. 90 Relief ranges from sea level along the coast backed by a wide expanse of tidal flat with the 91 coastal plain relief rising gently from 2 m to about 50 m above mean sea level. This barrier 92 lagoon bar experiences tides, current waves and predominantly of long shore currents 93 generated by south-westerly breaking waves at various degrees. Though the tidal range is 94

95 relatively small, the effects of tides on the general morphology of the coastline are very96 significant.

97 The alluvial sediments here were previously inferred a Pleistocene or Holocene age by

relative dating (Omatsola and Adegoke, 1981; Agagu, 1985). The alluvial plain is

99 lithologically indistinguishable from typical coastal plain sands strata. The high-level terraces

are rarely exposed, although few sections were seen around Ofada and Moloki on the Ogun

101 River bank. The sediments are made up of medium to slightly fine grained, well sorted to

102 moderately well sorted sands. Quaternary sediments mainly consist of recent alluvium

103 (Agagu, 1985), underlain by siltstone/ mudstone described by Omatsola and Adegoke (1981).

### 104 **3. Materials and methods**

#### 105 **3.1 Materials**

In this study, 10 representative samples were collected from the study area in the eastern 106 Dahomey Basin (Fig. 1). The samples are from sub-surface and unconsolidated in nature. 107 Texturally, the samples are fine to coarse grained sand with an average 85.0% of the samples 108 109 being medium grained sand. The colour ranges from white to light greyish and brownish red, an indication of high impurities. During sampling, surface sediments were removed, then the 110 stainless-steel tube with 30 cm long and 5 cm diameter was hammered to allow the sample 111 112 fill the tube. The tube was sealed at both ends using aluminum foil and wrapped with the black nylon to prevent it from sunlight reflection. Then each of the samples was properly 113 114 preserved in a sealed opaque container and clearly labelled for laboratory analysis.

### 115 **3.2 OSL dating method**

116 The single-aliquot regenerative dose (SAR) protocol was widely adopted to determine the

equivalent dose (D<sub>e</sub>) of quartz samples (Murray and Wintle, 2000; Roberts and Duller, 2004;

Lai and Wintle, 2006; Rodnight, et al., 2006; Wang et al., 2006; Roberts, 2007; Lai, 2010;

119 Chen et al., 2012). This can then be calculated from the ratio of the natural and regenerated

120 luminescence signals (Murray and Wintle, 2003; Yang et al., 2014). Both natural and

regenerative signals (Buylaert et al., 2008; Kang et al., 2013) are normalized with a

successive test dose which are used to monitor and correct for a potential sensitivity change.

123 This radiation  $(\alpha, \beta \text{ and } \gamma)$  from the radionuclides in the mineral and its natural environment

determined by either gamma spectrometry, neutron activation analysis, or alpha counting

125 (Aitken, 1985; Guérin et al., 2012) can be converted into alpha, beta and gamma dose rate

using conversion factors (Stokes et al., 2001; Chen et al., 2012).

#### 127 **3.2.1 Sample pretreatment**

Preparation of the samples and OSL experiments were conducted under a subdued red light in the luminescence dating laboratory, Key Laboratory of Tourism and Resources Environment in Universities of Shandong, Taishan University, Taian City, China. The two outer ends of each sample in the steel tube were removed for water content and dose rate analysis, while the remaining sediment was pretreated for luminescence measurement.

The remaining samples in the middle part of the tube were pretreated following the routine 133 134 procedures, including treatment with 10% HCl and 10% H<sub>2</sub>O<sub>2</sub> to remove carbonate and organic matter, respectively. After wet sieving, grains in the range of 150–250 µm were 135 extracted. The coarse fraction was then cleaned with 10% HF for 20 minutes to remove 136 coatings and the outer alpha irradiated layer, and rinsed in 10% HCl to remove any 137 precipitated fluorides. Subsequently, the remaining grains were separated by heavy liquid to 138 extract quartz grains ( $2.62 < \rho < 2.75 \text{ g/cm}^{-3}$ ). The purity of quartz was checked by IR 139 depletion ratio method (Duller, 2003) and 110 °C TL peak (Jain et al., 2004). The quartz 140 grains of each sample were mounted as small (2mm diameter) aliquots on a stainless-steel 141 discs, and twelve discs were measured for each sample. 142

### 143 **3.2.2 Dose rate determination**

144 Determination of the sediment dose rate involves assessment of concentrations of U, Th and K in the sample, water content and cosmic ray dose contribution. The environmental dose 145 rate was determined by the U, Th and K concentrations, measured by neutron activation 146 analysis (NAA) method in the Chinese Atomic Energy Institute. The U, Th and K 147 concentrations were converted to dose rates (Aitken, 1998). The total environmental dose 148 rate, including the contribution from cosmic radiation, was calculated according to Adamiec 149 and Aitken (1998). The cosmic ray dose contribution was estimated as a function of 150 longitude, latitude, altitude and depth (Prescott and Hutton, 1994). The in situ water content 151 was measured by weighing the sample before and after drying (mass of moisture/dry mass), 152 and was assigned an absolute uncertainty of  $\pm 5\%$ . 153

### 154 **3.2.3 Equivalent doses determination**

155 D<sub>e</sub> were estimated using the improved SAR protocol (Murray and Wintle, 2000, 2003; Wintle

and Murray, 2006; Rittenour 2008). Its determination includes irradiation, preheating and

- stimulating procedures. The procedures were conducted using an automated Risø TL/OSL
- 158 DA-20 reader equipped with blue LEDs (470 nm,  $\sim$ 80 mW cm<sup>-2</sup>) and infrared LEDs (875 nm,

~135 mW cm<sup>-2</sup>) (Bøtter-Jensen et al., 1994; Stokes, 1999). The irradiation procedure adopted 159 a  ${}^{90}$ Sr/ ${}^{90}$ Y beta source which was fitted on the reader with a dose rate of ~0.10 Gy/s to quartz 160 grains in stainless steel discs. The preheating was set at 160 °C for 20 s, based on the preheat 161 plateau and thermal transfer tests. The optical stimulation was conducted using blue light-162 emitting diodes (LEDs) for 40 s at 125 °C. This was detected using a 9235QA 163 photomultiplier tube through a 7.5 mm thick U-340 filter. The OSL signals for the  $D_e$ 164 determination were derived from the first 0.64 s stimulation minus a background of the 165 following 0.64 s of stimulation. 166

Preheat plateau test was carried out on samples MHN R2 and ARR R4 to show the variation 167 in dose with different preheats. The aliquots were bleached under blue light for 100 s at room 168 temperature, with an intervening gap of 1000 s, a set of four aliquots were later measured at 169 preheat temperatures from 160 to 260 °C, with a step of 20 °C. The results of the preheat 170 plateau tests (Fig. 2c) showed that the Des obtained by the SAR protocol are insensitive to 171 preheat temperature in range between 160 °C and 260 °C. For both samples (MHN R2 and 172 ARR R4), all the recycling ratios are close to 1, while the recuperation values are less than 173 174 3%, especially at 160 °C.

Young quartz samples may be affected by thermal transfer (Wintle and Murray, 2006). The 175 thermal transfer test as a function of preheat temperature was conducted for sample ARR R4 176 (Fig. 2d). The unheated aliquots were bleached twice under the blue light for 100 s at room 177 178 temperature with an intervening pause of 10 ks to allow charge optically transferred into the 110 °C TL peak to decay. For sample ARR R4, four aliquots were determined at each preheat 179 temperature between 160 °C and 260 °C (in increments of 20 °C). As shown in Fig. 2d, there 180 was no significant thermal transfer from 160 °C to 200 °C. Based on the preheat plateau and 181 thermal transfer tests, a preheat temperature of 160 °C was selected for the quartz D<sub>e</sub> 182 183 measurements.

184 The suitability of the procedure for  $D_e$  determination with the selected settings was checked

185 with a dose recovery test (Murray and Wintle, 2003). Nine natural aliquots of the sample

186 ARR R4 were stimulated twice by blue-light stimulation at 125 °C for 40 s. A laboratory dose

187 1.12 Gy was then administered, close to their expected natural  $D_{es}$ . The average dose

recovery ratio is  $1.01 \pm 0.01$ , which shows the selected SAR protocol is suitable for D<sub>e</sub>

189 determination.

#### 190 **4. Results**

### 191 4.1 Quartz luminescence characteristics

The dose response curve and natural OSL decay curve (insert) of the representative samples are shown in Fig. 2a and 2b. The decay curve of the samples resembles that of the calibration quartz sample. The blue-light stimulated OSL signals decreased very quickly, which indicates the OSL signals were fast-component dominated and that the sample was appropriate for OSL dating (Jain et al., 2003). The regeneration dose of 0 Gy was used to measure recuperation,

- 197 which was calculated by comparing the sensitivity corrected OSL signal of the zero dose to
- the sensitivity-corrected natural signal. Recuperation was in all cases <3% for all samples.
- 199 The recycling ratio, the consistency between the first regenerative dose and the repeated dose,
- suggested that the sensitivity changes during analysis were adequately corrected. The
- 201 recuperation and recycling ratio show the reliability of the SAR protocol.
- 202 The preheat plateau tests indicated that the Des obtained by the SAR protocol are independent
- 203 of preheat temperature (Fig. 2c). The thermal transfer test shows the variations in  $D_e$  were
- negligible from 160 °C to 200 °C, suggesting suitable preheating temperature range (Fig. 2d).
- 205 The  $D_e$  values are relatively low, and the majority of the  $D_e$  values fall in narrow range
- between 0.04 and 1.40 Gy. The Abanico plot of  $D_e$  distributions (Dietze and Kreutzer, 2019)
- 207 of two representative samples (MHN R2 and ODE R10) diplay normal and narrow
- distributions (Fig. 2e and 2f), indicating that the quartz particles were well bleached prior to
- 209 burial and the optical bleaching of the siliciclastic sediment in this study area was effective.
- 210 The study sediments may have experienced transportation mechanisms possibly from the
- tidal current in the adjacent Atlantic Ocean, coupled with flood and erosion from the adjoined
- 212 basement complexes. Therefore, all the  $D_e$  for OSL ages calculation were acquired using the
- average of twelve discs of each sample. The OSL dating results are summarized in Table 1.

### 214 **4.2 OSL age of the sediments**

- The results of OSL age dating, including the dose rates, equivalent doses and ages are listed
- in Table 1. The OSL ages are also displayed in the map (Fig. 3). The concentration of the
- radioactive elements in the siliciclastic sediments are 0.36–10.1 ug/g (Th), 0.17–1.98 ug/g
- 218 (U) and 0.011–0.469% (K) with mean values of 2.275 ppm for Th, 0.49 ppm for U and 0.135
- 219 % for K, respectively. The water content ranged from 1 to 20 %. The time-averaged water
- 220 content available in the sediment throughout its burial is an important, but a difficult
- parameter to assess (Cordier, 2010). Estimated value of the moisture content in these study

- sediments may not be well indicative of the entire period of burial, but may reflect the
- relative contribution to the effective dose rate. The dose rates range from  $1.54\pm0.066$  Gy ka<sup>-1</sup>
- to  $0.22\pm0.041$  Gy ka<sup>-1</sup>, which are typical for coastal sediments (Kunz et al., 2010), and the D<sub>e</sub>
- values, from  $1.4\pm0.03$  to  $0.04\pm0.01$  Gy. These yield OSL ages of the siliciclastic sediments
- ranging from  $3.52\pm0.34$  ka to  $0.05\pm0.01$  ka. The two oldest samples OKG R3 and MHN R2
- have OSL ages of  $3.52\pm0.34$  ka and  $3.01\pm0.39$  ka, respectively.
- 228 The OSL ages show that the deposits are of late Holocene. It was observed that the deposits
- are thicker in northern part of the study area. The extent of sampling depth was constraint
- 230 with shallow groundwater table and showed no spatial trend (Fig. 3). However, the spatial
- changes in age of the samples appear two belt-like distributions, with the exception of young
- age  $(1.55\pm0.15 \text{ ka and } 0.33\pm0.08, \text{ respectively})$  at the ORR and ZER barriers in the northeast
- part of the study area. The inland belt has ages ranging from  $3.01\pm0.39$  ka to  $1.55\pm0.15$  ka,
- while the coast belt has ages ranging from  $0.64\pm0.06$  ka to  $0.05\pm0.01$  ka. These two belts
- approximately parallel to the coastline (Fig. 3). The ages of samples in this area yield a rate
- of coastal progradation approximately 20 km in 3.5 ka, i.e. 5.7 km/ka.

#### 237 **5. Discussion**

Ages of the study samples range from 3.52 ka to 0.05 ka. The result of OSL dating are 238 reliable as evidenced by the good dose recovery and a signal dominated by a fast component. 239 The very young age for the sediments is in contrast with some previous researches (Omatsola 240 and Adegoke, 1980; Billman, 1982), in which they used relative age dating techniques to 241 adopt a Tertiary age for the sediments. The Tertiary sediment could have experienced 242 reworked processes, transportation and re-deposited in the late Holocene. The variations in 243 244 ages of these samples might be an indication of various depositional episodes during the Holocene epoch. 245

The thickness of the investigated siliciclastic sediments varies from 0.4 m to about 0.8
meters, while the deeper sediments could not be sampled in this study due to shallow
groundwater table in the region. The spatial changes in age of the samples appear two beltlike distributions, with the exception of young age of the two samples in the hinterland (Fig.
Geomorphologically, the belts parallel to the barrier bars along coastlines. It is highly
possible that the sediments with age of 0.64 ka–0.05 ka in the belt close to the coastline have
disturbed by modern tide activities. Therefore, we mainly focus on the belt with age range of

253 3.52 ka–1.55 ka.

254 The deposition episode of 3.52 ka–1.55 ka could have resulted from one or combination of two factors which include: 1) decrease in relative sea level; and 2) increase in flood activity. 255 Decrease in relative sea level is at least partly responsible for this deposition episode. 256 Decrease in relative sea level could be caused by basin uplift and/or decrease in sea level. 257 The knowledge of the tectonic movements in the region during the Holocene has been lacked. 258 Olabode (2015) analyzed tectonic evolution in the Dahomey Basin since Cretaceous, based 259 on data of one dimensional backstripping analysis on three offshore wells, and concluded that 260 the basin has been experienced accelerated tectonic subsidence during Quaternary period. 261 262 This may provide information of tectonic background for understanding the Holocene sedimentation, but is contrary to the deposition episode in the interval 3.52 ka-1.55 ka that 263

264 suggests basin uplift.

265 Decrease in sea level may also contribute to the formation of sediment in this older hinterland

belt. However, it is generally accepted that global sea level has been increasing in the

267 Holocene, and there is a progressive decrease in rise rate from 6.7 ka to recent time, within

which main rise occurred in the interval 6.7–4.2 ka (Lambeck et al., 2014). Many physical

269 processes, such as crustal rebound, continental levering, ocean syphoning, etc., produce

distinctive spatial and temporal patterns in relative sea level during late Holocene (Barnett et
al., 2019). It is still an open question what specific mechanism that drive relative sea level in
the study region.

273 Increase in flood activity that brings materials for the sediment may be another factor,

although the supply of reworked Tertiary sediments can not be precluded. The rivers (Ofara,

275 Oluwa, Talita and Alape Rivers) drains across the study area. Salzmann and Hoelzmann,

based on pollen and geochemical analysis, showed in southern Benin of Gulf of Guinea, an

episode of wetting climatic conditions occurred between 3.3 ka and 1.1 ka. This is timely

consistent with age of sediments in the range of 3.52 ka–1.55 ka. It is possible that increased

279 river flooding during the wet period had brought more sediments deposited in the hinterland

of the study area, in addition to the reworked older sedimentary rocks.

281 Presently, it is difficult to determine which factors are responsible for the deposition episode

during the period of 3.52 ka–1.55 ka. Understanding of previous coastal fluctuations is

283 crucial to envisage the present dynamics of the coasts within the framework of long-term

fluctuations (Masselink and Gehrels, 2014). Depositional changes in the coasts occur relative

to many factors which includes: sea level fluctuation, basin uplift, flood and sediment supply,

climate and human activity. Past changes associated with variations in these factors may

inform our understanding of future changes in the coast area. Further investigations with
large spatiotemporal coverage in the region are needed in future to better understand the past
dynamics of the coasts in the region.

#### 290 **6.** Conclusion

291 This study presents the first OSL dating of the coastal sediments, with large spatial coverage in the eastern Dahomey Basin. The OSL results revealed that the ages of the surficial 292 sediments in the eastern basin range from 3.5 ka to 0.05 ka, indicating that the deposits are 293 very young Quaternary deposits, specifically of Holocene Epoch. The young ages contradict 294 295 the assertion of some previous researcher (Billman, 1982) who adopt a Tertiary age for the sediments using relative age dating techniques. The age of the sediments varies spatially from 296 297 the northeast to southwest of the study area, indicating the sediments are result of the regressive depositional episodes that occurred in the study area. The decrease in relative sea 298 299 level and/or in river flooding during the wet climatic conditions may be responsible for the 300 elder deposition episode of 3.5-1.5 ka in the region. Therefore, this study provides first chronological constraints on evolution of the coast area and the linkage with relative sea level 301 changes (linked with both the sea level and tectonics) during the late Holocene. 302

### **303** Author contributions

304 Richard O. Fakolade: Investigation, Writing - Original Draft, Visualization. Philip R. Ikhane:

305 Conceptualization, Writing - Review & Editing, Supervision. Qiuyue Zhao:

306 Conceptualization, Methodology, Writing - Review & Editing, Supervision. Qingzhen Hao:

307 Conceptualization, Validation, Resources, Writing - Review & Editing, Supervision, Project

administration, Funding acquisition. Helena Alexanderson: Methodology, Editing. Zhengtang

309 Guo: Conceptualization, Validation, Resources, Writing – Review & Editing, Funding
310 acquisition.

### 311 Data availability

All data included in this study are available upon request by contact with the correspondingauthor.

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476									
477	Figure Captions								
478	Figure 1. Maps of the study area (Modified after NGSA, 1969).with locations of the OSL								
479	samples.								
480	Figure 2. Quartz luminescence characteristics of the representative samples. (a) and (b) Dose								
481	response curves of the typical samples OKG R3 and AGG R8, respectively. Inset shows								
482	the natural decay curves. (c) De values versus preheat plateau tests for two typical								
483	samples of MHA R2 and ARR R4. (d) Thermal transfer test of the typical sample ARR								
484	R4. (e) and (f) The Abanico plot of De distributions of two typical samples MHN R2 and								
485	ODE R10, respectively.								
486	Figure 3. Map of study area showing the OSL ages. The zone outlined by yellow line								
487	indicate the first episode of sedimentation in mostly of barrier bar, and the zone outlined								
488	by blue line indicate littoral zone.								
489									
490	List of Table								
491	Table 1. Summary of sample location, the burial depth, radionuclide concentrations,								
492	calculated dose rate, quartz De values and luminescence ages.								

Sample code	Latitude (°N)	Longitude (°E)	Altitude (m)	Depth (m)	Grain size (µm)	Water content (%)	U (µg/g)	Th (µg/g)	K (%)	D <sub>e</sub> (Gy)	Dose rate (Gy/ka)	Age (ka)
ATJ R1	6.31	4.75	6.00	0.80	150-200	4.0	0.32	1.61	0.054	0.93±0.03	0.39±0.035	2.40±0.23
MHN R2	6.20	4.78	3.20	0.50	150-200	3.0	0.17	1.25	0.011	1.17±0.02	0.39±0.049	3.01±0.39
OKG R3	6.38	4.76	7.60	0.60	200–250	14.0	0.35	1.55	0.011	1.40±0.03	$0.40 \pm 0.037$	3.52±0.34
ARR R4	6.34	4.49	1.46	0.45	200–250	6.0	1.98	10.70	0.216	$0.08 \pm 0.01$	1.54±0.066	$0.05 \pm 0.01$
AKD R5	6.26	4.77	1.97	0.60	150-200	14.0	0.58	1.19	0.090	1.08±0.02	0.49±0.036	2.21±0.17
ORR R6	6.38	4.74	8.50	0.60	150-200	1.0	0.29	2.01	0.024	0.76±0.03	0.49±0.043	1.55±0.15
ZER R7	6.38	4.78	9.40	0.80	150-200	18.0	0.21	0.49	0.011	$0.07 \pm 0.01$	0.22±0.041	0.33±0.08
AGG R8	6.31	4.63	10.40	0.40	150-200	8.0	0.34	0.36	0.175	1.00±0.03	$0.50 \pm 0.044$	2.02±0.19
UGB R9	6.15	4.79	27.20	0.45	200–250	12.0	0.24	1.91	0.469	$0.04 \pm 0.01$	0.79±0.039	$0.05 \pm 0.01$
ODE R10	6.29	4.62	8.50	0.50	150–200	20.0	0.42	1.50	0.299	0.39±0.03	0.61±0.034	0.64±0.06



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### **Declaration of interests**

 $\boxtimes$  The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

□The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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