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Comparisons of equivalent dose values obtained with different protocols using a lacustrine sediment sample from Xuchang, China

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ABSTRACT

Comparisons of seven protocols for equivalent dose (D_e) determination using the conventional optically stimulated luminescence (OSL) signal and the recuperation OSL (ReOSL) signal were made on a sample that is representative of a series of samples taken from a lacustrine sedimentary sequence at the archaeological site of Xuchang Man. Fine-grained quartz (4–11 μm) was extracted and the OSL signal was found to be dominated by the fast component. The D_e values obtained using different protocols varied from 222 ± 4 Gy to 368 ± 8 Gy. The dose response curves were all fitted with a single saturating exponential function except in the case of the multiple-aliquot additive dose protocol with sensitivity correction for the recuperated OSL signal (ReMAAD). The characteristic saturation dose (D_0) values obtained with the conventional OSL signal varied between 107 ± 6 Gy and 154 ± 7 Gy and showed no relationship with D_e values. The ReMAAD dose response curve was linear, and yielded an equivalent dose of 269 ± 12 Gy. The ReMAAD protocol may potentially serve as a means of dating relatively older samples provided the bleaching at the time of deposition was complete.

1. Introduction

Optically stimulated luminescence dating of quartz grains has been demonstrated to be a powerful tool for establishing chronological frameworks for Quaternary sequences (Wintle, 2008). Recently, a series of reviews on various applications to different types of sediment has been published, including studies on water-lain sediments (Jacobs, 2008; Rittenour, 2008), on aeolian deposits (Lancaster, 2008; Roberts, 2008), and glaciation-related deposits (Fuchs and Owen, 2008; Houmark-Nielsen, 2008). While different techniques are applied in OSL dating of quartz, many of the recent studies have employed the single aliquot regenerative dose (SAR) protocol (Murray and Wintle, 2000). However, comparative studies involving different OSL dating protocols for cross checking the reliability and internal consistency of the D_e values have been rarely reported. In this study, a variety of OSL dating protocols using both conventional OSL signal and recuperated OSL signal is applied to a sample from the fossil bearing layer of the archaeological site where broken pieces of the fossil skull of the Xuchang Man had been found.

Apart from the SAR protocol (Murray and Wintle, 2000), six other protocols were also applied to the sample. A special consideration was given to the possible dating range during the selection of the protocols. We applied the sensitivity-corrected multiple-aliquot regeneration dose (SC-MAR) protocol, which was developed by Zhou and Shackleton (2001) as Lu et al. (2007) successfully applied this protocol to a loess section at Luochuan and obtained OSL ages up to 100 ka. With the consideration of different OSL responses to natural and laboratory irradiation, Bailey (2004) and Bailey et al. (2005) examined the effects of a protocol with the laboratory irradiation administered in steps on the regeneration dose response curves, and hence the D_e values. This approach has been further tested by Qin and Zhou (2009) who suggested that the effect of the stepped irradiation may lead to significant increase of the D_e values. While many of the protocols for D_e determination suffer from the effect of saturation around 200 Gy (Wintle and Murray, 2006), the thermally transferred optically stimulated luminescence (TT-OSL) signal used by Wang et al. (2006) was shown to have a very large characteristic saturation dose, providing the potential of extending the age limit to nearly 1 Ma. Therefore, we have chosen to test the stepped irradiation approach and the TT-OSL protocol.

2. Sample, analytical facilities and protocols

Over 20 fragments of a broken fossil human skull were excavated from a lacustrine sedimentary sequence at the Lingjing site in Xuchang of Henan Province (N34°04′08.6″, E113°40′47.5″), central China. There was no independent age control for the sequence. Samples were collected for OSL dating. Initial OSL measurements

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Table 1
The SAR, SC-MAR[a], SI-SAR[b] and SI-SC-MAR[a,b] protocols (cf. Murray and Wintle, 2000).

Step	Treatment
1	Give dose, D_i[c]
2	Preheating at 260 °C for 10 s
3	Blue stimulation at 130 °C for 40 s
4	Give test dose, D_t (19.6 Gy)
5	Cut heat at 220 °C for 0 s
6	Blue stimulation at 130 °C for 40 s
7	Return to 1

[a] For the multiple-aliquot, the average OSL from three aliquots were applied in each regenerative dose. The natural signal is firstly measured for the set of aliquots before various regenerative doses administered on different aliquots.
[b] A stepped irradiation strategy with a unit dose of 24.5 Gy and 10 s heating at 240 °C between two dosing steps was used.
[c] For the natural sample, $i = 0$ and D_0 is the natural dose.

on four samples from the site showed that their OSL signals were extremely bright and yielded similar apparent equivalent doses. This study was focused on one of the samples, L1199.

Sample preparation was undertaken under subdued red light. Hydrochloric acid (10%) and hydrogen peroxide (30%) were used to remove carbonate and organic matter, respectively. The fine-grained fraction (4–11 μm) was obtained by settling in 0.01 N sodium oxalate solution. Quartz grains were extracted with an H_2SiF_6 (30%) treatment for 3 days, and dispersed in acetone and then deposited on aluminum discs (with about 0.8 mg sample on each disc). The purity of the isolated quartz was checked by determining the ratio of IRSL to OSL (<3%) and the characteristic 110 °C TL peak of quartz.

Luminescence measurements were carried out with an automated TL/OSL reader (Risø-TL/OSL-20) equipped with blue light (470 ± 30 nm) emitting diodes (LEDs). The OSL signal was detected by an EMI 9235QA photomultiplier tube with a 7.5 mm Hoya U-340 filters (290–370 nm) in front of it. A $^{90}Sr/^{90}Y$ beta source was used for irradiation (Botter-Jensen et al., 2003). All OSL measurements were made at 130 °C with the blue light LED stimulation set at 90% of the full power (50 mW cm^{-2}).

The seven protocols employed in this study include the SAR (Murray and Wintle, 2000), SC-MAR (Zhou and Shackleton, 2001), stepped irradiation SAR (SI-SAR) (Bailey, 2004; Qin and Zhou, 2009), stepped irradiation SC-MAR (SI-SC-MAR), SAR protocol for the recuperated OSL signal (ReSAR) (Wang et al., 2007), stepped irradiation ReSAR (SI-ReSAR) (Wang et al., 2006) and ReMAAD protocol (Sun et al., 2008). Tables 1 and 2 give a summary of the experimental conditions of the protocols. For the stepped irradiation, a unit dose of 24.5 Gy and pre-annealing for 10 s at 240 °C between any two dose steps were applied. The ReOSL signal of the test dose administered (Table 2, Part 2) was reported to contribute to the subsequent TT-OSL measurements (Tsukamoto et al., 2008). To avoid such a signal build-up, we applied a preheat at 260 °C for 10 s followed by blue stimulation at 125 °C for 100 s at the end of each cycle (Table 2). There is no significant net ReOSL signal for the zero regeneration dose.

3. Results and discussion

Linearity tests were carried out to check the validity of the sensitivity correction by test dose measurements (Wintle and Murray, 2006). For the SAR and SI-SAR protocols with a preheat of 260 °C for 10 s and a 220 °C cut heat, a linear relationship between the regenerative and test dose OSL signals exists (Figs. 1a,b), indicating that the OSL test dose signal correlates well with the regeneration OSL signal of the SAR and SI-SAR protocols. For the ReSAR and SI-ReSAR protocols, which utilize the ReOSL signal for equivalent dose measurements, the sensitivity change of the TT-OSL signal can be corrected by the OSL signal measured following a small test dose (Fig. 1c). However, this correction does not work well when the regenerative dose was applied in a stepped mode, which is indicated by the poor linearity shown in Fig. 1d. Therefore no further test of the SI-ReSAR protocol was undertaken.

As shown in Fig. 2, a D_e plateau can be identified for the preheat temperature in the range 230 °C to 280 °C with a cut heat temperature of 220 °C for the SAR protocol. Thus, a preheat of 260 °C for 10 s combined with a cut heat of 220 °C for 0 s was employed.

Dose recovery experiments (Murray and Wintle, 2003) were performed for the SAR, SI-SAR and ReSAR protocols. For the SAR and SI-SAR, the natural signal was bleached by blue light for 100 s at room temperature twice, with a delay of 10^4 s before the second one, while for the ReSAR, the natural signal was bleached with sunlight. The ratio of recovered dose to the given dose of 245 Gy is 0.94 ± 0.02 for the SAR protocol and 0.87 ± 0.01 for the SI-SAR protocol. However, the recovered dose is overestimated by 45–55% for the ReSAR. Therefore the ReSAR protocol was not investigated further.

Ten aliquots were used to obtain D_e values for the single aliquot protocols while the average OSL signals from three aliquots for each dose point have been used for the multiple-aliquot protocols. Dose response curves obtained using all the protocols were fitted with a single saturating exponential function except the ReMAAD protocol to which a linear curve was fitted.

Figs. 3a,b depict the growth curves obtained and Table 3 shows the D_e and D_0 values determined using the different protocols. The D_e value derived from the SAR protocol is 234 ± 3 Gy, while that for the SC-MAR is 222 ± 4 Gy. We should note that for early Late Pleistocene loess samples, the value of D_e obtained with the SAR

Table 2
The ReSAR, SI-ReSAR and ReMAAD[a] protocols.

Part 1: TT-OSL		Part 2: BT-OSL[b]	
1–1	Give dose, D_i[c]	2–1	Annealing to 300 °C for 10 s
1–2	Preheating at 260 °C for 10 s	2–2	Blue stimulation at 130 °C for 100 s
1–3	Blue stimulation at 130 °C for 300 s	2–3	Preheating at 260 °C for 10 s
1–4	Preheating at 260 °C for 10 s	2–4	Blue stimulation at 130 °C for 100 s; $L_{BT\text{-}OSL}$
1–5	Blue stimulation at 130 °C for 100 s; $L_{TT\text{-}OSL}$	2–5	Give test dose, D_t (4.9 Gy)
1–6	Give test dose, D_t (4.9 Gy)	2–6	Cut heat at 220 °C for 10 s
1–7	Cut heat at 220 °C for 10 s	2–7	Blue stimulation at 130 °C for 100 s; $T_{BT\text{-}OSL}$
1–8	Blue stimulation at 130 °C for 100 s; $T_{TT\text{-}OSL}$	2–8	Annealing to 300 °C for 10 s
		2–9	Blue stimulation at 130 °C for 100 s
		2–10	Return to 1–1

[a] ReSAR is carried out on a single aliquot, while ReMAAD is applied to the average OSL measured from three aliquots following each additive dose. Regenerative dose administered in step 1 is in stepped irradiation mode for SI-ReSAR like SI-SAR and SI-SC-MAR.
[b] Basic transfer optical stimulated luminescence (BT-OSL). The sensitivity-corrected ReOSL value (Corrected L_{ReOSL}) was obtained using the following equation: *Corrected* $L_{ReOSL} = (L_{TT\text{-}OSL}/T_{TT\text{-}OSL}) - (L_{BT\text{-}OSL}/T_{BT\text{-}OSL})$ (Wang et al., 2006).
[c] For the natural sample, $i = 0$ and D_0 is the natural dose. For the ReMAAD protocol, D_i was added to the nature dose.