

## Research Paper

## A combined luminescence and radiocarbon dating study of Holocene lacustrine sediments from arid northern China

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## ABSTRACT

Arid northern China is an ideal place for the reconstruction of palaeoenvironmental changes, for which the chronology is a key issue. To test the applicability of optically stimulated luminescence (OSL) dating methods to Holocene lake sediments and to evaluate the hard water reservoir effect of <sup>14</sup>C dating in arid northern China, 12 OSL samples and 12 radiocarbon samples were dated. These samples were from an 8 m long lacustrine section (Qingtu Lake, QTL) in the Zhuyeze Palaeolake in arid northern China. Tests of luminescence characteristics (preheat temperature, laboratory dose recovery, OSL decay and growth curve, and equivalent doses distribution) confirm that the OSL signal of silt-sized quartz (38–63 μm) from the QTL section was fully reset before burial, and that OSL dating has considerable potential for improving the dating of Holocene lake sediments in the arid land of northern China, especially in those cases where there is a significant hard water 'old carbon' problem for <sup>14</sup>C dates. The apparent agreement between OSL and <sup>14</sup>C dating for QTL section suggests that the hard water reservoir effect of <sup>14</sup>C samples in the study area is much smaller than that in other lakes in northern China, which is also supported by analyses on two <sup>14</sup>C samples using different dating materials for each of the two individual samples.

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## 1. Introduction

Lake sediments are important recorders of palaeoenvironment information (Cohen, 2003). Accurate and reliable dating of lacustrine deposits is of crucial importance in both the reconstruction of palaeolake and palaeoclimate evolutionary history and the understanding of the mechanisms for climate changes, especially abrupt changes of regional-hemispheric hydrological circulation (Zhang et al., 2006). Radiocarbon dating is the most commonly used method for establishing chronologies of lake sediments. However, <sup>14</sup>C dating of such sediments could be problematic because they often suffer from contamination from sources of 'old carbon' (hard water reservoir effect), which appears common in the radiocarbon dating of lacustrine sediments in the arid regions of China (Liu et al., 2009). This reservoir effect for lacustrine samples from arid northern China vary from several hundred to thousands of years (e.g. Bangong Lake: 6670 a, Fontes et al., 1996; Qinghai Lake: 1039 a, Shen et al., 2005; Ahung Lake: 600–700 a, Morrill et al., 2006; Bositeng Lake: 1140 a, Chen et al., 2006; Zigetang Lake: 2010 a, Herzschuh et al., 2006; Chaka Salt Lake: 1700 a, Liu et al., 2008a;

Wulun Lake: 760 a, Liu et al., 2008b; Kusai Lake: 3400 a, Liu et al., 2009; Hurleg Lake: 2758 a, Zhao et al., 2010). Therefore, the evaluation and calibration of <sup>14</sup>C reservoir effect is critical for enabling reliable age control for the lake sediments from arid northern China.

With respect to <sup>14</sup>C dating, terrestrial plant macrofossils from the lake sediments have been used as dating materials in order to avoid the 'old carbon' errors associated with bulk samples (Edwards and Whittington, 2001). However, such terrestrial plant macrofossils are very difficult to find in lake sediments, especially in arid lands. Other methods for reservoir effect calibration have also been employed (Shen et al., 2005; Herzschuh et al., 2006; Liu et al., 2008a, 2008b, 2009). In an analysis of lake sediments from Qinghai Lake, Shen et al. (2005) used a linear regression function for organic accelerator mass spectrometry (AMS) <sup>14</sup>C ages with depth to infer a radiocarbon reservoir effect of 1039 a. These authors then assumed a constant <sup>14</sup>C reservoir effect over their 18 000 a lake record and subtracted 1039 a from all <sup>14</sup>C ages prior to calibration. Herzschuh et al. (2006) determined a <sup>14</sup>C reservoir effect of 2010 a for a modern sample from Lake Zigetang on the Tibetan Plateau, using AMS <sup>14</sup>C dating of organic sediments from the surface sample of a core. They then assumed a constant <sup>14</sup>C reservoir effect over their lake record and subtracted 2010 a from all

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radiocarbon samples prior to calibration. Discrepancies between  $^{210}\text{Pb}$ - &  $^{137}\text{Cs}$ -based ages and ages determined by AMS  $^{14}\text{C}$  dating of bulk organic samples from lake cores have also been assessed as an equivalent to the reservoir effect offsets (Liu et al., 2008b, 2009; Zhao et al., 2010). However, it is likely that the  $^{14}\text{C}$  reservoir effect could change over time with the variations of hydrochemical conditions in a lake (Wang et al., 2007), beyond the limits of  $^{210}\text{Pb}$ - and  $^{137}\text{Cs}$  dating. If discontinuous deposition occurred at the top part of the lake core (Morrill et al., 2006) it could also complicate determinations of the reservoir effect by the measurement of  $^{210}\text{Pb}$  and  $^{137}\text{Cs}$  or  $^{14}\text{C}$  dating of surface sediments. Furthermore, Liu et al. (2008a) accounted for the  $^{14}\text{C}$  reservoir effect at Chaka Lake in the Qaidam Basin by correlating a major decrease in the total organic carbon (TOC) and total nitrogen (TN) of lake sediments, indicative of colder and drier conditions, with the globally recognized Younger Dryas event, which has been recorded as a cold event in Greenland (Stuiver et al., 1995). However, such peak matching has inherent assumptions and it is possible that some global climate events could not be imprinted in lake sediments in such localities. For lacustrine sediments from arid China, a dating technique is thus needed, whose chronology could be (a) independent of variations in lake reservoir effect, (b) convenient for extracting dating materials, (c) independent of the variations in the lake hydrochemistry, and (d) independent on the assumed climate events.

To provide the optimum chronological framework of lake sediments for palaeoenvironment research, optically stimulated luminescence (OSL) dating had been applied to dating lake sediments in order to overcome the  $^{14}\text{C}$  dating problems (Lang, 1994; Magee et al., 1995, 2004; English et al., 2001; Lang and Zolitschka, 2001; Berger and Doran, 2003; Bubbenzer and Hilgers, 2003; Thomas et al., 2003, 2009; Olley et al., 2004; Argyilan et al., 2005; Cupper, 2006; Madsen et al., 2008; Shen et al., 2008; Lee et al., 2009; Telfer et al., 2009; Fan et al., 2010; Liu et al., 2010; Sun et al., 2010). Using infrared-stimulated-luminescence (IRSL), Lang (1994) shows dating results for subaerially exposed lacustrine sediments from South Germany that are consistent with calibrated  $^{14}\text{C}$  ages. Lang and Zolitschka (2001) reported results of IRSL investigations carried out on annually laminated (varved) sediments from lake Holzmaar, Eifel, West Germany, suggesting that IRSL ages of their varved sediments are accurate only for clastic-rich horizons and IRSL standard techniques seem to work well. Thomas et al. (2003) presented infrared (IR), post-IR blue and blue OSL characteristics of the fine grain sediments from Lake Xiniias in central Greece and a comparison with independent ages by  $^{14}\text{C}$  AMS dating of terrestrial plant macrofossil remains from peat and clayey peat. They concluded that (1) OSL dating of quartz silt-sized grains has considerable potential in the dating of lacustrine sediments, (2) quartz is well-bleached compared to the age range under investigation, and (3) the growth curve permits age estimation up to 300 ka. It has been shown that silt-sized lake sediments have been adequately bleached (Lang, 1994; Lang and Zolitschka, 2001; Thomas et al., 2003). Shen et al. (2008) applied OSL dating using fine silt quartz, together with  $^{14}\text{C}$  dating of terrestrial plant macrofossils, which do not have the problem of 'old carbon' error, to establish a chronology for a lake sediment core from Crummock Water, NW England. Their results indicated a good agreement between OSL and  $^{14}\text{C}$  ages, suggesting that the OSL dating method can be used to date lake sediments in the British Isles. Moreover, it is showed that OSL dating is a credible alternative to  $^{14}\text{C}$  method in playa lacustrine sediments where  $^{14}\text{C}$  methods have been proved problematic (Argyilan et al., 2005; Bubbenzer and Hilgers, 2003; Cupper, 2006; English et al., 2001; Magee et al., 1995, 2004; Olley et al., 2004; Telfer et al., 2009). Using OSL dating of lake shoreline sediments, chronologies of lake evolution since late Pleistocene have recently been

constructed, even for the time periods beyond the  $^{14}\text{C}$  dating limit (Madsen et al., 2008; Thomas et al., 2009; Lee et al., 2009; Sun et al., 2010; Fan et al., 2010; Liu et al., 2010).

However, OSL dating method has rarely been applied to the lake sediments of arid northern China. This region is currently characterized by many palaeolakes within the surrounding deserts, with chronologies constrained only by large numbers of  $^{14}\text{C}$  ages published as part of intensive palaeoenvironmental studies (e.g. Zhang et al., 2004). For lakes in arid area, it is likely that the presence of abundant quartz grains in the lacustrine sequences, probably blown into the basins from surrounding dune fields, makes them suitable for OSL dating (Cupper, 2006; Long et al., 2010a). The key aim of the current study is firstly to test the applicability of optical dating methods to silt-sized lake sediments from arid northern China, and then to evaluate the reservoir effect of  $^{14}\text{C}$  dating through the comparison of OSL and  $^{14}\text{C}$  chronologies.

## 2. Study area, section and samples

There are many palaeolakes in arid northern China (with annual precipitation of <200 mm, Fig. 1a) (Wang and Dou, 1998). Among them, Zhuyeze is a terminal lake of the Shiyang River (see Fig. 1b for its location). The river originates from the Qilian Mountains up to 5000 m above sea level, and runs northward to Zhuyeze Lake in the Tengger Desert for a distance of about 260 km (Fig. 1b). The river has a catchment of  $4.16 \times 10^4 \text{ km}^2$ , with an average runoff of  $14.7 \times 10^8 \text{ m}^3/\text{a}$  (Chen and Qu, 1992). The modern hydrometeorological data suggest that the major runoff in this drainage is from monsoonal precipitation, and only 7% runoff is from the melt water of glaciers from the Qilian Mountains (Ding and Wang, 2001). This region is dominated by the summer monsoon from July to September, which brings most of the moisture for the whole year. In winter, cold and dry continental air mass prevailed in the area. The mean annual temperature and precipitation of Zhuyeze is  $7.8 \text{ }^\circ\text{C}$  and 110 mm respectively, while the annual evaporation is over 2600 mm. The patchy vegetation, consisting of grass and shrubs, is mainly distributed in places where the groundwater level is close to the ground surface. Because of the aridity and the diversion of the river flow for irrigation during the past century, the Zhuyeze palaeolake shrank significantly, and even dried up (Wang et al., 1999).

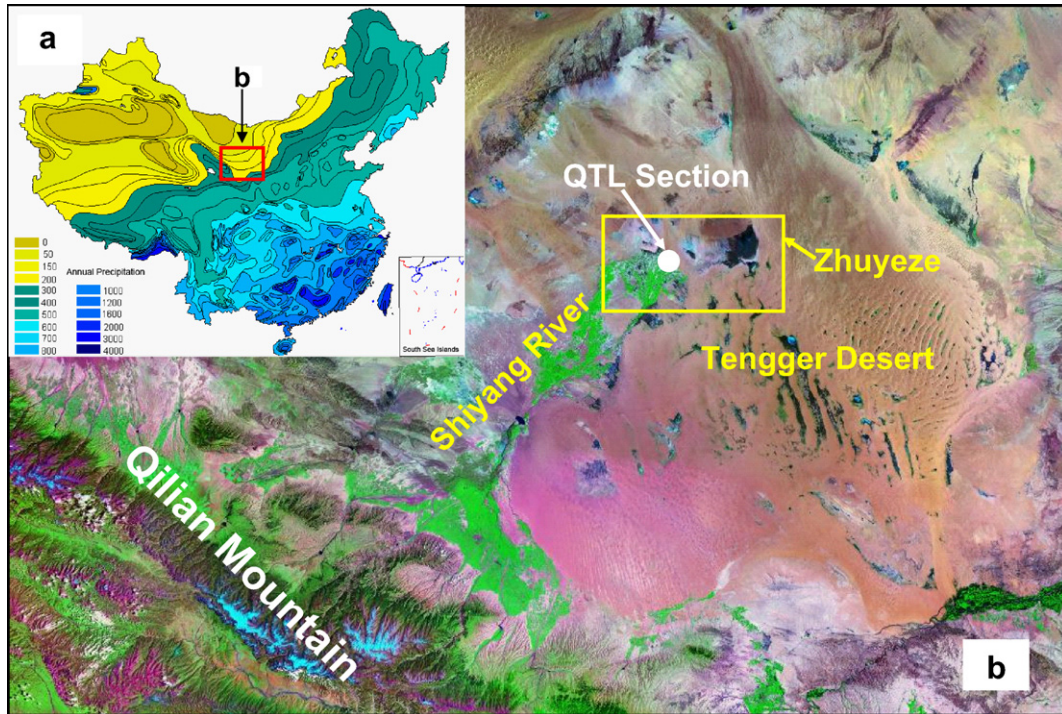
Qingtu Lake (QTL) section ( $39^\circ 03' \text{N}$ ,  $103^\circ 40' \text{E}$ ) lies in the central part of the Zhuyeze palaeolake (Fig. 1b), and has a thickness of 800 cm (Fig. 2a). Geological records suggested that a megalake with a surface area of more than 20 000  $\text{km}^2$  during Late Pleistocene existed in the study area (Zhang et al., 2004). Historical documents indicate that the palaeolake during 1849–1949 AD covered an area of >400  $\text{km}^2$  in the Zhuyeze region, and dried up during the last decades (Wang et al., 1999). Twelve OSL samples and twelve  $^{14}\text{C}$  samples were taken from the QTL section for the construction of a chronology. All OSL samples were obtained by hammering steel tubes (20 cm long cylinders with a diameter of 5 cm) into a freshly dug vertical section (Fig. 2b). The tubes were then covered and sealed with aluminum foil, and then wrapped using plastic bags and tape to avoid light exposure and moisture loss. The samples for  $^{14}\text{C}$  dating include shell remains and bulk sediments collected from the strata with relatively abundant organic materials.

## 3. Methods

### 3.1. OSL sample preparation

In the laboratory, the sediments at each end of the tube were scraped and used for dose rate and water content measurement.



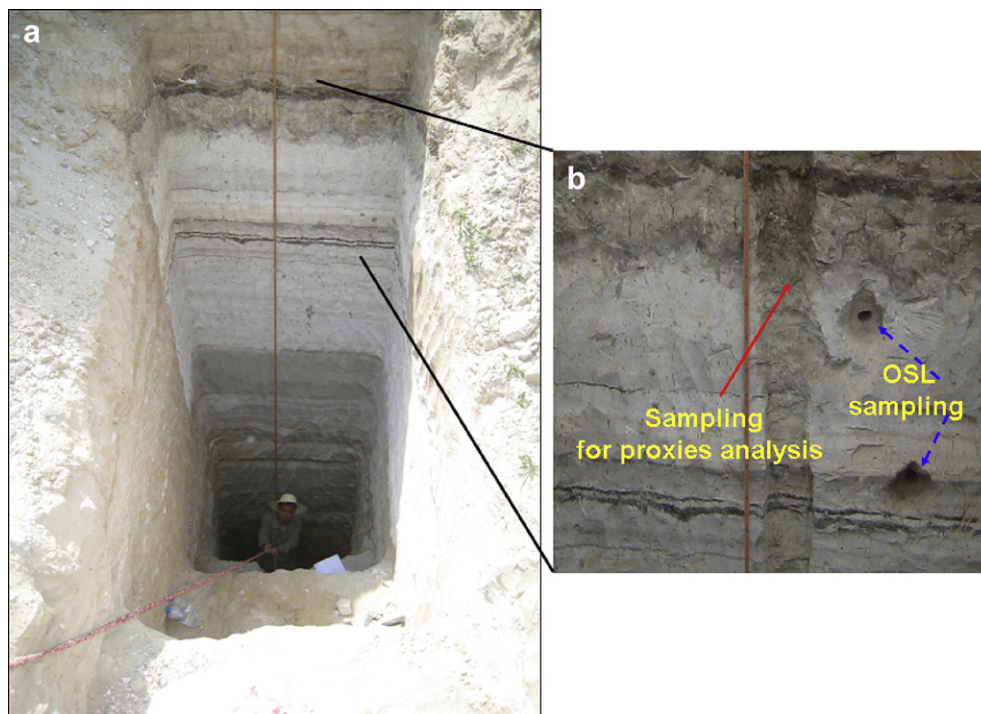


**Fig. 1.** (a) Map of annual precipitation in China. The areas with the annual precipitation of <200 mm are defined as arid land. Red square denotes the location of the study area (Zhuyeze palaeolake). (b) Topographic map of Shiyang River drainage and its terminal lake (i.e. Zhuyeze palaeolake denoted by the yellow square). The filled white circle marks the location of QTL section.

The non light-exposed materials in the middle part of the tube were pretreated for luminescence measurement.

Raw samples were treated firstly with 30% H<sub>2</sub>O<sub>2</sub> and 10% HCl to remove organic materials and carbonates. For each sample, the 38–63 μm grains were separated by wet sieving, and then treated with fluorosilicic acid (38%) for about 2 weeks to corrode feldspars,

followed by 10% HCl acid to remove fluoride precipitates (Lai et al., 2007a; Roberts, 2007; Lai, 2010). It is important to ensure that the feldspar contamination has been efficiently removed in order to avoid age underestimation (Roberts, 2007; Lai and Brückner, 2008). The purity of the isolated quartz was checked by infrared light stimulation (830 nm), and no obvious IRSL was observed in any



**Fig. 2.** (a) Field picture of digging the QTL section. (b) A picture showing OSL sampling holes in QTL section.

sample. The separated quartz grains were then mounted on the centre part (0.5–0.6 cm in diameter) of stainless steel discs (diameter of 10 mm) using silicone oil.

### 3.2. Equivalent dose ( $D_e$ ) determination

OSL measurements were performed in the Luminescence Dating Laboratory of the Qinghai Institute of Salt Lakes, Chinese Academic Sciences, using an automated Risø TL/OSL-20 reader. Stimulation was carried out by a blue LED ( $\lambda = 470 \pm 20$  nm) stimulation source for 40 s at 130 °C. Irradiation was carried out using a  $^{90}\text{Sr}/^{90}\text{Y}$  beta source built into the reader. The OSL signal was detected by a 9235QA photomultiplier tube through a 7.5 mm thick U-340 filter. OSL signals from the first 0.64 s stimulation were integrated for growth curve construction after background subtraction. The  $D_e$  was determined using both the single aliquot regenerative-dose (SAR) protocol (Murray and Wintle, 2000) and a standardised growth curve (SGC) method (Roberts and Duller, 2004; Lai, 2006; Lai et al., 2007b; Long et al., 2010b). The application of SGC could reduce significantly the machine time. For each sample, 6–8 aliquots were measured by SAR to build an SGC for each of these samples, and then 12–21 aliquots were measured by SGC. The final  $D_e$  for a sample is the average of all  $D_e$ s measured.

### 3.3. Dose rate determination

The concentrations of uranium, thorium and potassium were measured by neutron activation analysis in the China Institute of Atomic Energy in Beijing. The cosmic ray dose rate was estimated for each sample as a function of depth, altitude and geomagnetic latitude (Prescott and Hutton, 1994). According to the description about lake area changes of Zhuyeze (see Section 2), the lake sediments from QTL section should be saturated with water throughout the burial period. The observed (not saturated) water content (WC) is thus not the real WC of the sediments during the burial period. We measured the saturation water content of the samples by soaking the sediments until it was unable to hold more water. Uncertainties in water content were assigned to 5% for all samples. And the very low organic content in the lake sediments of QTL section (Long et al., 2010a) might confirm that the saturation water content have changed less through time due to compression and dewatering (Duller, 2008). In the field, we also measured their water content of modern lake sediment (still saturated). The results indicated that, where their grain size and sediment type are similar, the saturation water content of modern lake samples is close to that of samples from QTL section calculated by the above method. Finally, the elemental concentrations were converted into annual dose rate according to Aitken (1998).

### 3.4. $^{14}\text{C}$ dating

The materials used for radiocarbon dating include organic carbon (bulk samples) from the organic-rich sediments or peat and inorganic carbon from shells. To discuss the reservoir effect, we collected two sub-samples (one for organic carbon dating and the other one for inorganic carbon dating) at each of 315 cm and 425 cm depth for cross-checking. The bulk samples were dated by conventional  $^{14}\text{C}$  method in the Radiocarbon Dating Laboratory of Lanzhou University, and the shells were dated by AMS radiocarbon method in the Radiocarbon Dating Laboratory of Peking University. The  $^{14}\text{C}$  ages were converted to calendar years to compare them with the OSL ages. All radiocarbon dates were calibrated to calendar year (cal a BP or cal ka BP) using CALIB 5.1 program with INTCAL04 model (Reimer et al., 2004) after  $^{13}\text{C}/^{12}\text{C}$  adjustment. The details of

**Table 1**

Radiocarbon dating results for twelve samples from QTL section. Two sub-samples were collected from both depths of 315 and 425 cm.

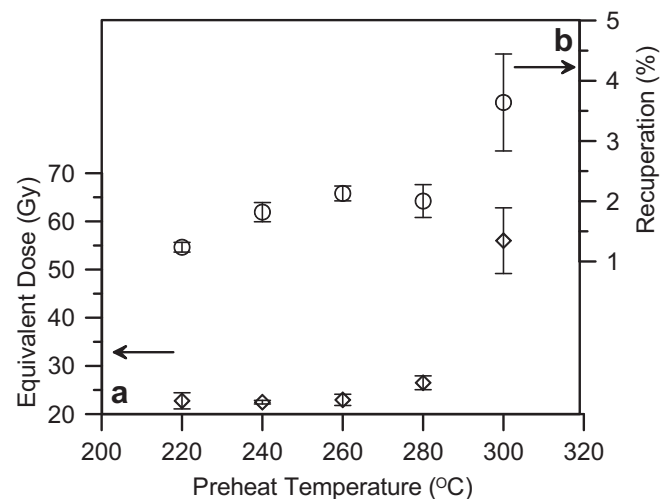
Sample ID	Depth (cm)	Dating material (dating method)	$\delta^{13}\text{C}$ (‰)	$^{14}\text{C}$ age (a BP)	Cal. age (2 $\sigma$ ) (cal a BP)
QTL-01	225	Peat organic material (Conventional $^{14}\text{C}$ dating)	-27.8	1550 $\pm$ 60	1434 $\pm$ 118
QTL-02	250	Peat organic material (Conventional $^{14}\text{C}$ dating)	-28.2	2470 $\pm$ 90	2546 $\pm$ 195
QTL-03	262	Shell (AMS)		3140 $\pm$ 40	3383 $\pm$ 66
QTL-04	290	Peat organic material (Conventional $^{14}\text{C}$ dating)	-28.5	3300 $\pm$ 90	3541 $\pm$ 185
QTL-05A	315	Peat organic material (Conventional $^{14}\text{C}$ dating)	-27.4	4130 $\pm$ 110	4632 $\pm$ 247
QTL-05B	315	Shell (AMS)		4160 $\pm$ 40	4701 $\pm$ 130
QTL-06	360	Bulk organic material (Conventional $^{14}\text{C}$ dating)	-25.7	4530 $\pm$ 80	5143 $\pm$ 187
QTL-07A	425	Bulk organic material (Conventional $^{14}\text{C}$ dating)	-24.1	5960 $\pm$ 65	6803 $\pm$ 151
QTL-07B	425	Shell (AMS)		5920 $\pm$ 40	6732 $\pm$ 74
QTL-8	446	Shell (AMS)		6550 $\pm$ 40	7468 $\pm$ 50
QTL-9	494	Shell (AMS)		6910 $\pm$ 40	7752 $\pm$ 81
QTL-10	537	Bulk organic material (Conventional $^{14}\text{C}$ dating)	-24.3	8412 $\pm$ 62	9412 $\pm$ 119
QTL-11	572	Bulk organic material (Conventional $^{14}\text{C}$ dating)	-24.7	9183 $\pm$ 60	10368 $\pm$ 134
QTL-12	600	Shell (AMS)		11175 $\pm$ 50	13070 $\pm$ 118

the  $^{14}\text{C}$  samples and the dating materials, as well as the dating results are listed in Table 1.

## 4. Luminescence characteristics

### 4.1. Preheat plateau test

Thermal transfer, or the transfer of charge from light-insensitive but thermally-stable traps into the light-sensitive traps by frequent stimulations and preheats, leads to the increase of the optical signal, which may result in  $D_e$  overestimation (Aitken, 1998). In order to select appropriate preheat conditions that minimise thermal transfer for  $D_e$  determination using the SAR protocol, a preheat temperature plateau test was conducted for sample ZY08-04. Preheat temperatures from 220 °C to 300 °C (for 10 s) with an interval of 20 °C were tested, with the cut-heat kept at 220 °C for 10 s, using the heating rate of 5 °C/s. Four aliquots were



**Fig. 3.** Preheat plateau test showing the dependence of equivalent dose (a), and recuperation (b) on preheat temperature. Each point represents the average of results from four aliquots.

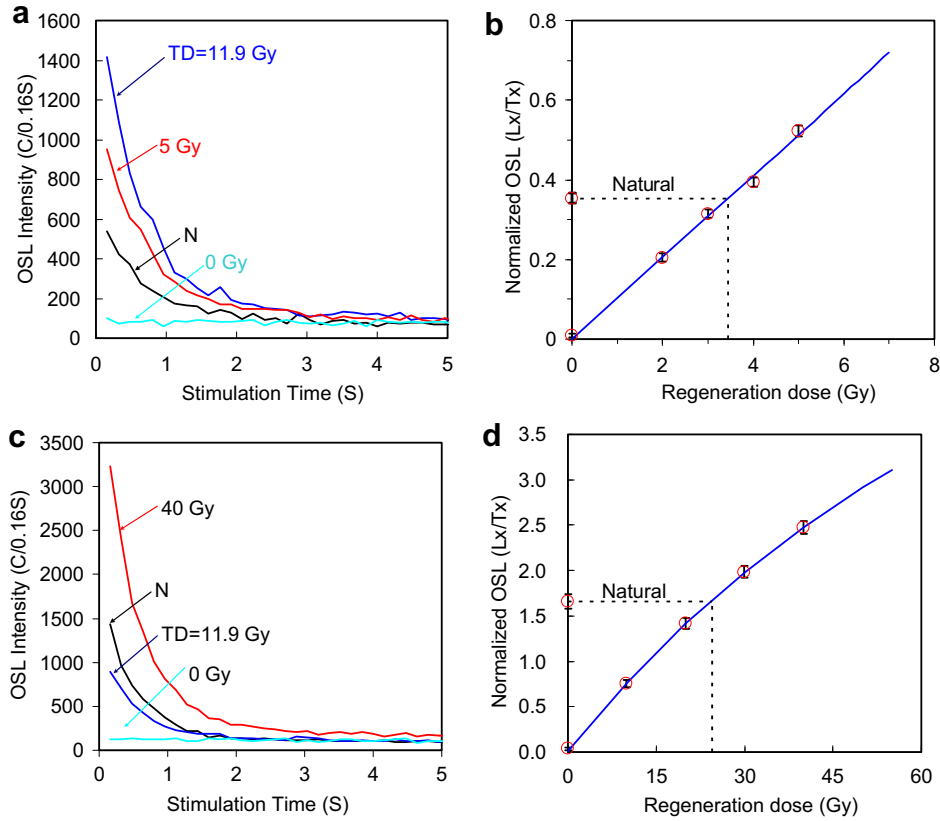


Fig. 4. OSL decay curves and growth curve for sample ZY08-12 (a, b) and ZY08-04 (c, d).

used for each temperature point. The result for each temperature point is the average of the four aliquots (Fig. 3a). A plateau was observed for temperatures from 220 °C to 260 °C, and the recuperation implied by the ratio of the sensitivity-corrected 0 Gy value to the sensitivity-corrected natural signal is small (<3%) for temperatures from 220 °C to 280 °C (Fig. 3b). Therefore, we select a preheat temperature 260 °C (for 10 s) for routine  $D_e$  determination.

4.2. Dose recovery tests

The suitability of SAR procedures for  $D_e$  determination was further checked with a ‘dose recovery test’ (Murray and Wintle, 2003). This test examines the combined function of all the

procedural conditions, such as preheat temperature and size of test dose. The dose recovery test was applied to 12 aliquots of each of the two samples, ZY08-04 and ZY08-05. The given laboratory doses were 23.8 Gy. The measured  $D_e$  values were  $22.1 \pm 0.2$  Gy and  $21.5 \pm 0.2$  Gy for ZY08-04 and ZY08-05 respectively. Thus, the ratios of the given doses to the measured doses were  $0.98 \pm 0.03$  and  $0.96 \pm 0.02$ , respectively. The results suggest that the SAR procedure is able to recover a laboratory dose.

4.3. OSL decay curves and growth curves

Fig. 4a and c shows typical OSL decay curves of natural dose (N), test dose (TD = 11.9 Gy), two regeneration doses for a relatively young sample ZY08-12 and a relatively old sample ZY08-04. Their

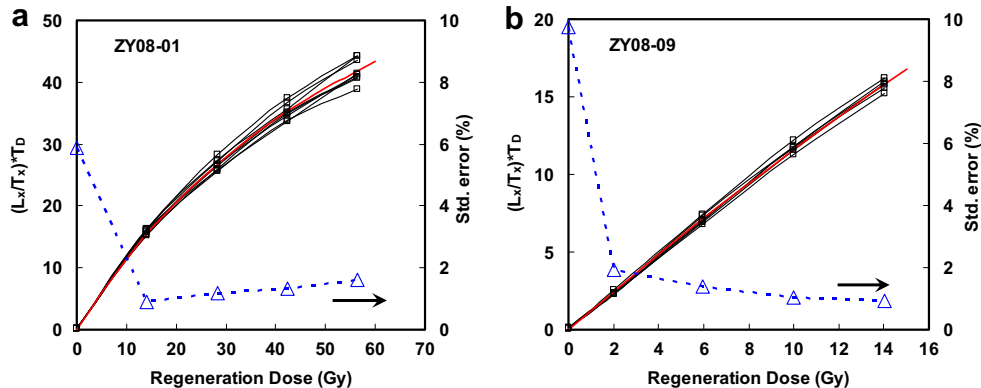


Fig. 5. The individual growth curves of eight aliquots for sample ZY08-01 (a) and of six aliquots for sample ZY08-09 (b). The SGC for each of two samples is shown by the red line. The standard error for the regenerative-dose points is shown as empty triangles (right-hand y-axis).



**Table 2**

Equivalent doses for twelve samples from QTL section are determined by both SAR and SGC.

Sample ID	Aliquot Number	$D_e$ SAR (Gy)	$D_e$ SGC (Gy)	$D_e$ SAR / $D_e$ SGC	$D_e$ (Gy)
ZY08-01	8 <sup>a</sup> + 12 <sup>b</sup>	36.59 ± 0.34	37.11 ± 0.67	0.99	36.90 ± 0.37
ZY08-02	8 <sup>a</sup> + 12 <sup>b</sup>	29.54 ± 0.54	30.74 ± 0.49	0.96	30.26 ± 0.31
ZY08-03	8 <sup>a</sup> + 12 <sup>b</sup>	26.46 ± 0.52	24.55 ± 0.50	1.06	25.41 ± 0.41
ZY08-04	6 <sup>a</sup> + 20 <sup>b</sup>	25.40 ± 0.86	23.90 ± 0.67	1.08	24.50 ± 1.00
ZY08-05	6 <sup>a</sup> + 12 <sup>b</sup>	22.57 ± 0.54	20.90 ± 0.69	1.08	21.60 ± 0.50
ZY08-06	6 <sup>a</sup> + 21 <sup>b</sup>	10.95 ± 0.40	11.87 ± 0.22	0.92	11.50 ± 0.30
ZY08-07	6 <sup>a</sup> + 12 <sup>b</sup>	11.78 ± 0.48	11.81 ± 0.47	1.00	11.80 ± 0.50
ZY08-08	6 <sup>a</sup> + 12 <sup>b</sup>	4.28 ± 0.17	3.98 ± 0.19	1.07	4.10 ± 0.30
ZY08-09	6 <sup>a</sup> + 12 <sup>b</sup>	6.55 ± 0.17	6.46 ± 0.09	1.01	6.50 ± 0.20
ZY08-10	6 <sup>a</sup> + 12 <sup>b</sup>	4.73 ± 0.15	4.68 ± 0.26	1.01	4.70 ± 0.10
ZY08-11	8 <sup>a</sup> + 12 <sup>b</sup>	4.97 ± 0.08	4.54 ± 0.12	0.95	4.70 ± 0.09
ZY08-12	6 <sup>a</sup> + 12 <sup>b</sup>	3.98 ± 0.18	4.18 ± 0.08	1.09	4.10 ± 0.10

<sup>a</sup> Aliquot numbers using SAR method.

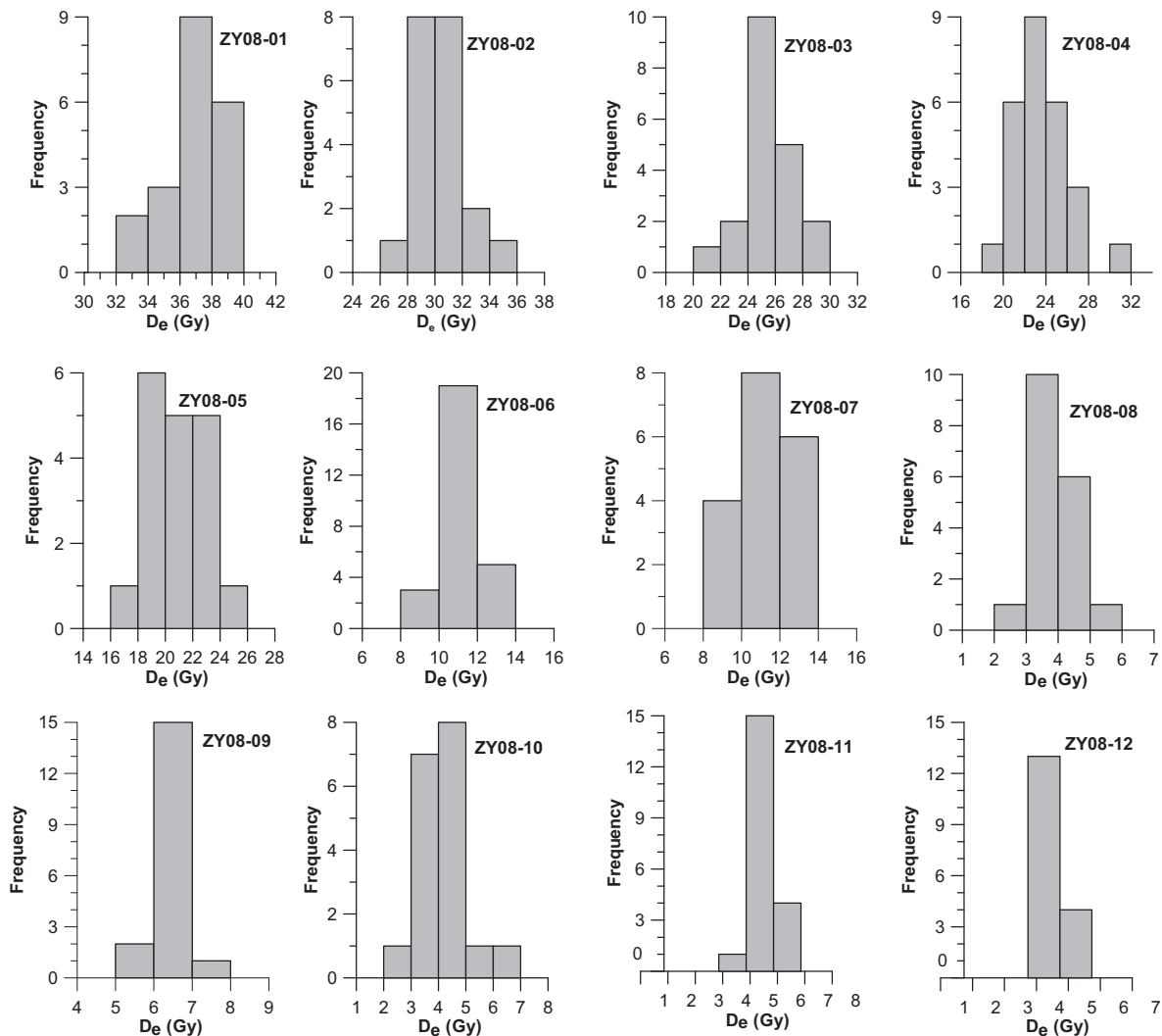
<sup>b</sup> Aliquot numbers using SGC method.

OSL signals decrease very quickly during the first second of stimulation, indicating that the OSL signal is dominated by the fast component. The regeneration dose of 0 Gy is used to measure recuperation, 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. Murray and Wintle (2000) introduced the 'recycling ratio' to check for sensitivity change correction. For most of the aliquots the recycling ratios fall into the range of 0.9–1.1. A few discs with a recycling ratio falling outside this range were rejected in the final  $D_e$  calculation. Fig. 4b and d shows two typical growth curves of two aliquot for samples ZY08-12 and ZY08-04, respectively. The growth curve can be well fitted using the exponential plus linear form.

#### 4.4. The applicability test of SGC

Long et al. (2010a) have showed that an SGC exists for lacustrine samples from arid northwestern China. However, it has been warned against the use of such 'universal' curves as the variation between growth curves is observed for quartz samples that show no signs of feldspar contamination (Burbidge et al., 2006; Stevens et al., 2007). In this study, the applicability of SGC thus needs to be further tested for the lacustrine samples from QTL section. Fig. 5 shows the individual growth curves of eight aliquots for sample ZY08-01 (a relatively old sample) and six aliquots for sample ZY08-09 (a relatively young sample). The growth curves of all aliquots for each of two samples are similar. For all dose points, the standardised dose response for the two samples display



**Fig. 6.** Histograms of  $D_e$  distribution for all 12 OSL samples.

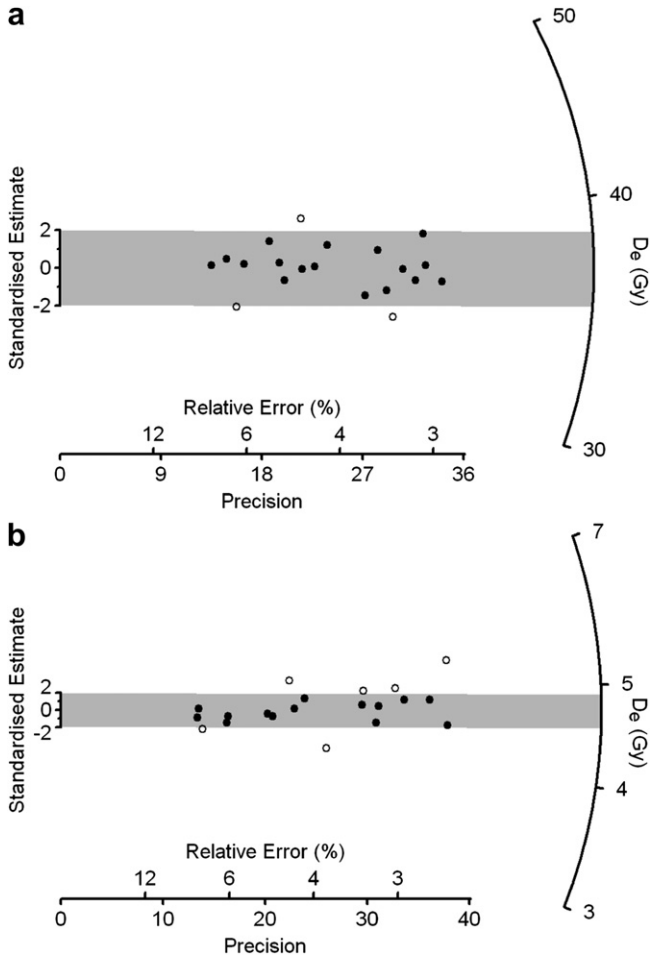


Fig. 7. Radial plots for the equivalent doses of samples ZY08-01 (a) and ZY08-11(b).

a scatter with a standard error of within 2% (except for the zero dose where the scatter is higher simply because of the low signal intensity). For the construction of the SGC for each individual sample, the average data of all aliquots measured by SAR is used. As can be seen in Fig. 5, the SGC for sample ZY08-01 and ZY08-09 can be fitted using the exponential plus linear form. For all samples, the  $D_e$  determined by SAR ( $D_{e\_SAR}$ ) is in agreement with  $D_e$  determined by SGC ( $D_{e\_SGC}$ ) within 10% (see Table 2). The SGC could be used for  $D_e$  determination for these samples from QTL section in study area.

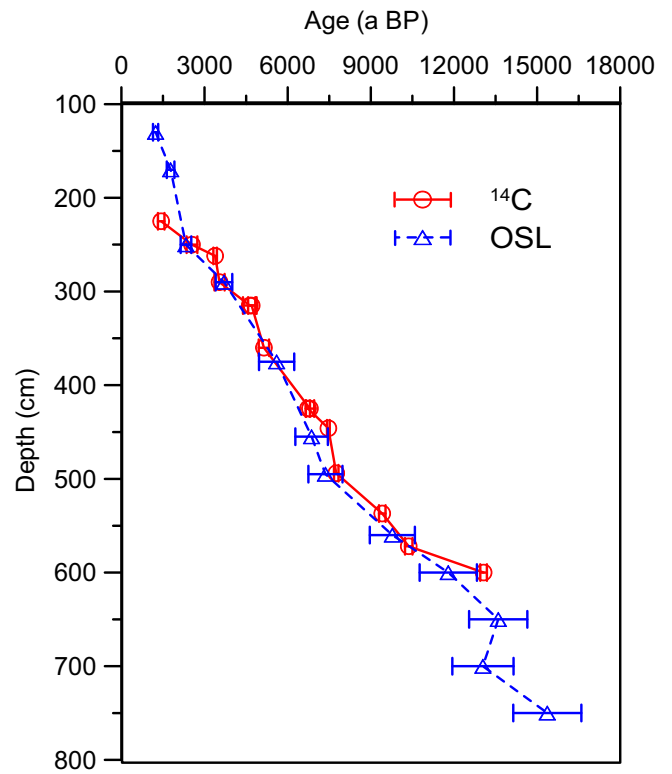


Fig. 8. Plot of OSL and  $^{14}C$  ages against the depth of QTL section.

#### 4.5. $D_e$ distribution

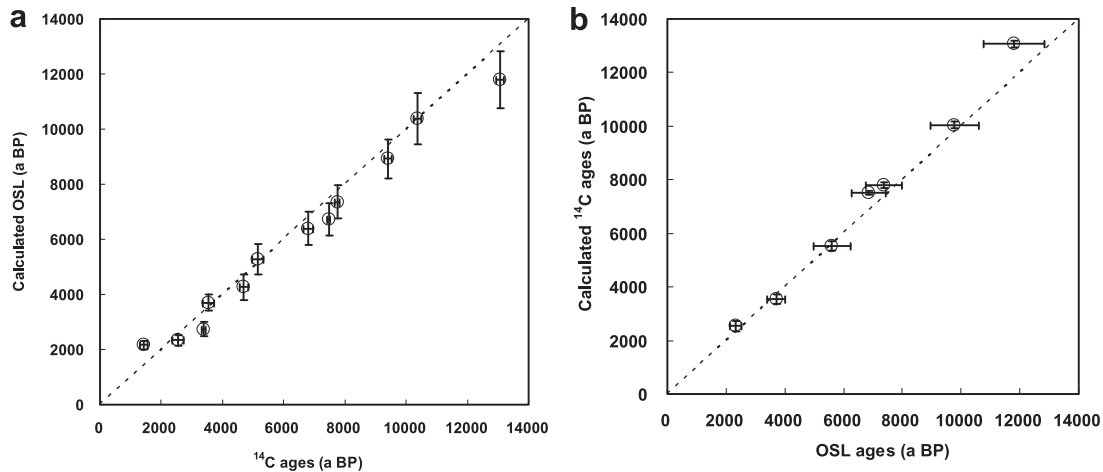
The  $D_{eS}$  for each of the 12 samples display an approximately normal distribution (Fig. 6). The mean equivalent doses for all samples are almost indistinguishable from their medians and in light of this the mean  $D_e$  is suggested to be suitable for age estimation. Significantly, no sample shows a long ‘tail’ towards the upper end of the distribution histogram. Thus there is no evidence that a significant portion of incompletely-bleached older grains have probably been inherited before burial (Lee et al., 2009). The radial plot for equivalent doses for two typical samples (ZY08-01 and ZY08-11) is also provided (Fig. 7).

### 5. Results and discussions

The OSL dating results are listed in Table 3, which shows the  $D_e$  values, ages, radionuclide concentrations, dose rates and water content for all 12 samples. Both the OSL ages (denoted by triangles) and  $^{14}C$  calendar ages (denoted by circles) are plotted against the

Table 3  
OSL age calculation for 12 samples from QTL section.

Sample ID	Depth (cm)	K (%)	Th (ppm)	U (ppm)	Water Content (%)	Dose Rate (Gy/ka)	$D_e$ (Gy)	OSL Age (ka)
ZY08-01	750	1.97 ± 0.06	4.42 ± 0.21	1.09 ± 0.20	15 ± 5	2.40 ± 0.19	36.90 ± 0.37	15.4 ± 1.2
ZY08-02	700	1.97 ± 0.06	5.31 ± 0.24	1.27 ± 0.20	15 ± 5	2.31 ± 0.19	30.26 ± 0.31	13.0 ± 1.1
ZY08-03	650	1.88 ± 0.06	7.13 ± 0.27	1.50 ± 0.21	35 ± 5	1.87 ± 0.14	25.41 ± 0.41	13.6 ± 1.1
ZY08-04	600	1.46 ± 0.05	7.63 ± 0.27	7.74 ± 0.30	50 ± 5	2.08 ± 0.16	24.50 ± 1.00	11.8 ± 1.0
ZY08-05	560	1.17 ± 0.04	5.48 ± 0.25	10.65 ± 0.35	51 ± 5	2.21 ± 0.18	21.60 ± 0.50	9.7 ± 0.8
ZY08-06	495	0.97 ± 0.03	2.82 ± 0.17	8.10 ± 0.28	57 ± 5	1.49 ± 0.12	11.50 ± 0.30	7.7 ± 0.7
ZY08-07	455	1.30 ± 0.05	2.93 ± 0.18	4.47 ± 0.23	43 ± 5	1.63 ± 0.12	11.80 ± 0.50	7.2 ± 0.6
ZY08-08	375	0.32 ± 0.02	1.70 ± 0.14	4.08 ± 0.21	58 ± 5	0.76 ± 0.07	4.10 ± 0.30	5.3 ± 0.6
ZY08-09	290	0.97 ± 0.04	4.34 ± 0.20	6.27 ± 0.27	45 ± 5	1.76 ± 0.13	6.50 ± 0.20	3.7 ± 0.3
ZY08-10	250	0.58 ± 0.03	2.65 ± 0.16	11.19 ± 0.35	50 ± 5	2.02 ± 0.16	4.70 ± 0.10	2.3 ± 0.2
ZY08-11	170	1.93 ± 0.06	6.04 ± 0.27	1.11 ± 0.18	10 ± 5	2.65 ± 0.20	4.70 ± 0.09	1.8 ± 0.1
ZY08-12	130	1.93 ± 0.06	10.16 ± 0.35	2.50 ± 0.25	10 ± 5	3.32 ± 0.24	4.10 ± 0.10	1.2 ± 0.1



**Fig. 9.** (a) Comparison of twelve <sup>14</sup>C ages with twelve calculated OSL ages (each from a linear interpolation between the two adjacent optical ages) at the same depth of the corresponding <sup>14</sup>C sample. (b) Comparison of seven OSL ages with seven calculated <sup>14</sup>C ages (each from a linear interpolation between the two adjacent radiocarbon ages) at the same depth as the corresponding OSL sample.

sampling depth in QTL section for comparison (see Fig. 8). The results suggest that the agreement between the OSL and <sup>14</sup>C ages is good within errors. Good agreement between quartz OSL and <sup>14</sup>C ages has also been recently reported for aeolian samples from arid China (Lai et al., 2009). For a further comparison, an OSL age at the depth for each of the twelve individual <sup>14</sup>C samples has been calculated using a linear interpolation between the two adjacent OSL ages. Similarly, a <sup>14</sup>C age at the depth for each of the seven individual OSL samples (from the depth of 230 cm–600 cm) has been calculated using a linear interpolation between the two adjacent radiocarbon ages. The comparison of the so-calculated twelve OSL ages with the twelve <sup>14</sup>C ages at the same depth is shown in Fig. 9a, and the comparison of the so-calculated seven <sup>14</sup>C ages with the seven OSL ages at the same depth is shown in Fig. 9b. Good agreement was observed in both cases. In Fig. 9a the average ratio of the <sup>14</sup>C ages to the calculated OSL ages is 1.04, close to unity. Likewise, in Fig. 9b the average ratio of the OSL ages to the calculated <sup>14</sup>C ages is 0.96, also close to unity. The consistency of luminescence and radiocarbon ages could suggest that the hard water reservoir effect of <sup>14</sup>C dating could be small in the study area.

For evaluating the suggestion that the reservoir effect of <sup>14</sup>C dating could be small in the study area, two <sup>14</sup>C samples using different dating materials for each of the two individual samples have been dated. It is generally accepted that the dating of peat sample is hardly affected by reservoir effect and peat bulk <sup>14</sup>C dates are reliable (Blaauw et al., 2004). As shown in Table 1, for the two sub-samples (QTL-05A and QTL-05B) at the same depth of 315 cm, the <sup>14</sup>C dating result ( $4130 \pm 110$  <sup>14</sup>C a BP) of organic carbon (from peat) is in agreement with that ( $4160 \pm 40$  <sup>14</sup>C a BP) of inorganic carbon (from shells). Thus, we assume that the age of shells from sample QTL-05 is not affected by 'old carbon'. The <sup>14</sup>C age ( $5960 \pm 65$  <sup>14</sup>C a BP) of organic carbon for sample QTL-07A from the organic-rich sediments of bulk sample is also consistent with the age ( $5920 \pm 40$  <sup>14</sup>C a BP) of inorganic carbon for sample QTL-07B from shells at the depth of 425 cm. Therefore, the reservoir effect is not significant for samples under study, and the <sup>14</sup>C dating is robust. The consistency of luminescence and radiocarbon ages could also suggest that the reservoir effect of <sup>14</sup>C dating could be very small in Zhuyeze Lake, which contrasts with findings from other lakes (mentioned in Introduction) in arid northern China (Fontes et al., 1996; Shen et al., 2005; Morrill et al., 2006; Chen et al., 2006; Herzschuh et al., 2006; Liu et al., 2008a, 2008b, 2009; Zhao et al., 2010).

The agreement of the OSL ages with <sup>14</sup>C ages for QTL section gives considerable confidence that the OSL signal was fully reset before

burial, and therefore that OSL dating could be able to provide reliable ages for lacustrine sediments in similar depositional environments in arid northern China. Although some partial bleaching may occur in many water-lain sediments (Murray et al., 1995; Li, 2001), previous investigations into the sources of lake sediments in arid northern China have shown that the majority of the fine sand or silt quartz grains in the lacustrine sequences have been blown into the lakes from the surrounding dunes (Long et al., 2010a; Liu et al., 2009), which as a consequence will have been well-bleached before deposition.

## 6. Conclusions

- (1) Tests of luminescence characteristics (preheat temperature, lab dose recovery, OSL decay and growth curve, and equivalent doses distribution) confirm that the OSL signal of lacustrine sediments from QTL section was fully reset before burial, and that OSL dating could have considerable potential for improving the chronology of Holocene lake sediments in the arid northern China, especially in those cases where there is a significant 'old carbon' problem when using <sup>14</sup>C dating.
- (2) The good agreement between the OSL and <sup>14</sup>C ages suggests that the reservoir effect of <sup>14</sup>C samples in the study area is very small. This is also supported by the analysis on two <sup>14</sup>C ages of two sub-samples from each of the two depths 315 and 425 cm using different dating materials (shells which are assumed to be affected by reservoir effect, and peat organic matter which is assumed not to be affected by reservoir effect).

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