High resolution OSL dating of the late quaternary loess from Central Shandong Mountains in eastern China and the paleoclimatic implications

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Abstract. The loess deposits widely distributed in the Central Shandong Mountains are significant terrestrial palaeoclimatic archives in the alluvial plain of eastern China. It is also sensitive records of East Asian monsoon changes for situating in a key region connected to the Eurasian plate and the Pacific Ocean. However, research into the climate changes in CSM loess is restricted by the lack of independent age control. High resolution samples of Optically Stimulated Luminescence (OSL) dating results are presented from a new Heishan loess section over ~34 ka. Our results reveal the following: (1) The boundary age defined by the sedimentation rates model between the Pleistocene and the Holocene at Heishan loess section is 10.9±0.6 ka. (2) A marked hiatus in the record is identified between ~30 and ~17 ka, probably resulting from deflation; this has never been raised in previous CSM loess researches and indicates that the study area is the wind erosion area during this time. (3) The relatively rapid sedimentation rate spanning 10.9±0.6 to 8.5±0.4 ka may be related to the post-depositional disturbance induced by more monsoon precipitation in the CSM region.

1. Introduction

The world loess deposits are mainly situated on the margins of deserts, mountains and ice-sheets[1], or distributed along the major rivers[2] and have the potential for detailed palaeoenvironmental archives [3-7]. Such deposits not only document local to global climate signals [3,8-9], but also record sedimentary or preservational conditions [10-11], even geomorphic history [12-13], especially when they are close to the major river systems in the world [2,11], such as the Yangtze River, Rhine River, Mississippi River, etc.

In the lower reaches of the Yellow River of China, loess deposits are widely distributed on the margin of the Central Shandong Mountains (CSM, Figure 1), and are valuable Quaternary palaeoclimatic archives for this currently warm and semi-humid region of eastern China [14-23]. Moreover, the CSM loess is very sensitive to the East Asian monsoon changes and hydrological changes of the Yellow River [20] for situating in the transition belt between Eurasia and Pacific Ocean. However, research into the climate changes in CSM loess is restricted by the lack of independent age control. Both the established chronological frame and the ages of same strata are not consistent [15,19].
Furthermore, high resolution chronological sequences have not yet been established, and detailed palaeoclimatic processes are still not clear.

Figure 1. (a) Digital elevation model (DEM) map of northern China showing the Yellow River and the North China Plain. The red letters indicate the boundary of the upper, middle and lower reaches of the Yellow River. The white circles indicate the loess sections referred to in the text. (b) Map showing the location of the research area in the west of Central Shandong Mountains. (c) Sampling section of Heishan in the field.

In this study, Systematic Optically Stimulated Luminescence (OSL) dating were performed at Heishan loess section in CSM and measured the magnetic susceptibility (MS) and median grain size of the loess. In examining this new loess exposure, the aim of this study was to establish the numerical chronology and estimate the dust accumulation rates based on high resolution coarse-grained quartz OSL ages of the loess-palaeosol sedimentation. Furthermore, we intend to reconstruct palaeoenvironmental changes using MS and median grain size.

2. Regional background
CSM is situated in the east of North China Plain (Figure 1a). The geomorphology of the CSM region is characterized by a series of low mountains and hills [24], with elevations ranging from 200 to 1545 m above sea level [20]. The region has a warm and semi-humid monsoon climate, which is characterized by obvious seasonal changes in temperature, precipitation and wind direction. The mean annual temperature is 12.6-14.5 °C and the mean annual precipitation vary between 550 mm and 950 mm, but most rainfall (60%~70%) occurs in summer from June to August. In winter, the climate is controlled by the Siberia High and the prevailing wind is northwesterly, and southeasterly winds in summer.

3. Materials and methods
3.1. Sample collection
The new Heishan (HS) loess section (36°10.05'N, 116°20.63'E) sampled from the south bank of lower reaches of Yellow River is in the southwest of CSM (Figure 1b, 1c). The exposed thickness of loess is about 5.5 m. The section can be sub-divided into three lithological units: 1) yellow plough horizon (0-1.0 m); 2) grayish black Holocene soil (1.0-2.9 m); 3) Malan loess (2.9-5.5 m). Fifteen OSL samples were collected using light-tight steel cylinders with 5 cm diameter 25 cm long from the ~ 5.5 m of a freshly dug vertical section. The tubes were sealed at both ends using aluminum and the OSL samples
were taken at 10-40 cm intervals. A total of 220 samples were collected at 2.5 cm intervals for MS analysis and median grain-size measurements.

3.2. OSL dating

In the laboratory, the outer layers at both ends of the tube were extracted for water content and dose rate measurements. Grain sizes of 63-90 µm were obtained from the exposed non-light inner part of the tube by wet sieving. Carbonates and organics was removed by 10% HCl and 10% H₂O₂, respectively. The remaining material was prepared by Sodium polytungstate heavy liquid at a density between 2.58 and 2.70 gcm⁻³ to separate the quartz-rich grains. The quartz-rich grains were then treated with 40% HF to remove remaining feldspar and out layers irradiated by alpha particles. 10% HCl were used to dissolve any precipitated fluorides. Finally, the samples were mounted on stainless steel discs with silicon oil. Considering the contamination with incompletely dissolved feldspar can affect the equivalent dose (Dₑ) and the shape of growth curves [25], infrared-stimulated luminescence (IRSL) [26] and the 110 °C Thermo-Luminescence (TL) [27] peaks were measured to check the purity of the quartz grains.

All luminescence measurements were carried out by an automated Risø TL/OSL reader DA-20 system equipped with blue LEDs (470 nm, ~80 mW cm⁻²) and infrared LEDs (875 nm, ~135 mW cm⁻²) in the luminescence dating laboratory of Taishan University. A calibrated ⁹⁰Sr/⁹⁰Y beta source were used in all the measurements. Quartz OSL signals were detected through a 7.5 mm Hoya U-340 glass filter. The stimulation power was set at 90% of the maximum value and the heating rate is 5 °C/s in all thermal treatment. The De for quartz were determined by single-aliquot regenerative dose (SAR) protocol [28]. Typical dose response curves and decay curves are shown in Figure 2a. The blue-light stimulated OSL signals of HS-14 decrease very quickly during the first second of stimulation, indicating that the signal is dominated by the fast component [29]. To select appropriate preheat temperatures for De determination, preheat temperature plateau tests were used to check the De dependence on preheat temperature for sample HS-4 and HS-12 (Figure 2b). Preheat temperatures from 200 °C to 300 °C with an interval of 20 °C were tested. The results demonstrate that the De values are relatively stable at preheat temperatures from 240 to 300 °C. A preheat of 260 °C (10 s) and cut-heat of 220 °C combination was chosen for all De measurements. The suitability of SAR procedures for De determination was further checked by two dose recovery tests. The dose recovery ratio of HS-5 and HS-15 are 1.01±0.01 (n=8, Given dose=36Gy, Figure 3a) and 1.00±0.01 (n=10, Given dose=100Gy, Figure 3b), respectively. For all the samples, recuperation was <1% and the average recycling ratio is 1.003±0.002 (n=179) indicating that the adopted SAR protocol successfully corrects for sensitivity changes.

![Figure 2](image_url)

**Figure 2.** Coarse-grain quartz luminescence characteristics. (a) Representative small aliquot dose response curve for sample HS-14 showing recycling and recuperation (open symbols) and the interpolation of the sensitivity-corrected natural signal onto the dose response curve. Inset shows the natural decay curve. (b) Preheat plateau tests of samples HS-4 and HS-12. Three aliquots were measured at each temperature and error bars represent one standard error. The dashed line is drawn at the average De over the 200-300 °C interval.
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3.3. MS and median grain size

The MS and grain size were determined in the Laboratory of Environmental Evolution at Taishan University. The Bartington MS2B Magnetic Susceptibility reader (470/4700 Hz) was utilized to measure mass magnetic susceptibility. The naturally dried samples (10 g) packed in a nonmagnetic plastic sample box were weighted for MS analysis. The Malvern Mastersizer 2000 laser grain-size analyzer was used for grain-size measurements, which has a measurement range of 0.02-2000 µm with a precision of ±1%. About 200 mg of sediment from each air-dried, disaggregated sample was pretreated with 10 ml 10% H₂O₂ and 10% HCl to remove organic matter and carbonates. Detailed pretreatment are provided in the literature [20].

4. Results

4.1. Dose rates

Table 1. Summary of the burial depth, radionuclide concentrations, calculated dose rate, quartz De values and luminescence ages. The water content is assumed to be 15±5% based on the measured water content. (n) represents the number of aliquots contributing to the De. Uncertainties represent one standard error.

| Sample Code | Depth (m) | U (ppm) | Th (ppm) | K (%) | Dose Rate (Gy ka⁻¹) | De (Gy) | Aliquots (n) | Age (ka) |
|-------------|-----------|---------|---------|-------|---------------------|--------|-------------|---------|
| HS-1        | 1.1       | 2.16 ± 0.04 | 11.80 ± 0.03 | 2.13 ± 0.03 | 3.20 ± 0.05 | 26.9 ± 1.2 | 12 | 8.5 ± 0.4 |
| HS-2        | 1.5       | 2.31 ± 0.04 | 11.60 ± 0.03 | 2.09 ± 0.03 | 3.14 ± 0.05 | 24.3 ± 1.2 | 12 | 7.8 ± 0.4 |
| HS-3        | 2.1       | 2.20 ± 0.04 | 11.00 ± 0.03 | 2.13 ± 0.03 | 3.10 ± 0.05 | 28.4 ± 0.9 | 10 | 9.2 ± 0.3 |
| HS-4        | 2.5       | 2.42 ± 0.04 | 12.10 ± 0.03 | 2.14 ± 0.03 | 3.22 ± 0.05 | 35.1 ± 1.9 | 15 | 10.9 ± 0.6 |
| HS-5        | 2.6       | 1.96 ± 0.04 | 11.10 ± 0.03 | 1.97 ± 0.03 | 2.91 ± 0.10 | 35.4 ± 0.9 | 13 | 12.2 ± 0.6 |
| HS-6        | 2.7       | 2.04 ± 0.04 | 11.50 ± 0.03 | 1.95 ± 0.03 | 2.93 ± 0.05 | 43.9 ± 1.4 | 12 | 15.0 ± 0.6 |
| HS-7        | 2.8       | 2.17 ± 0.04 | 12.10 ± 0.03 | 2.01 ± 0.03 | 3.10 ± 0.10 | 51.9 ± 1.6 | 12 | 17.0 ± 0.8 |
| HS-8        | 2.9       | 2.40 ± 0.04 | 12.10 ± 0.03 | 2.18 ± 0.03 | 3.24 ± 0.05 | 55.5 ± 2.6 | 12 | 17.1 ± 0.9 |
| HS-9        | 3.1       | 2.07 ± 0.04 | 12.10 ± 0.03 | 2.02 ± 0.03 | 3.03 ± 0.11 | 48.9 ± 1.4 | 12 | 16.1 ± 0.8 |
| HS-10       | 3.3       | 2.01 ± 0.04 | 11.90 ± 0.03 | 1.86 ± 0.03 | 2.86 ± 0.11 | 97.1 ± 4.8 | 12 | 33.9 ± 2.1 |
| HS-11       | 3.7       | 2.31 ± 0.04 | 12.30 ± 0.03 | 2.09 ± 0.03 | 3.18 ± 0.12 | 95.5 ± 5.7 | 10 | 30.4 ± 1.9 |
| HS-12       | 4.2       | 2.45 ± 0.04 | 11.70 ± 0.03 | 1.73 ± 0.03 | 2.82 ± 0.05 | 88.4 ± 2.4 | 10 | 31.4 ± 1.0 |
| HS-13       | 4.6       | 2.44 ± 0.04 | 11.10 ± 0.03 | 1.99 ± 0.03 | 3.00 ± 0.05 | 96.2 ± 1.9 | 10 | 32.1 ± 0.8 |
| HS-14       | 5.1       | 2.95 ± 0.04 | 11.00 ± 0.03 | 1.95 ± 0.03 | 3.06 ± 0.05 | 99.6 ± 2.1 | 15 | 32.6 ± 0.9 |
| HS-15       | 5.5       | 2.54 ± 0.04 | 12.20 ± 0.03 | 1.97 ± 0.03 | 3.06 ± 0.05 | 103.1 ± 2.0 | 12 | 33.7 ± 0.9 |

Figure 3. Dose recovery tests of 36 Gy (a) and 120 Gy (b). Eight to ten aliquots were bleached with blue LEDs at room temperature for 100 s, followed by a pause of 10,000 s and another blue-light bleach for 100 s. The aliquots were then given a dose close to the natural, and measured with the SAR protocol.
The environmental dose rate was determined from the uranium, thorium and potassium concentrations, measured by neutron activation analysis (NAA) method in the Chinese Atomic Energy Institute. The concentrations of $^{238}$U and $^{232}$Th, and $^{40}$K are relatively constant for the whole section (Table 1; Figure 4a, 4b, 4c), with RSDs of 7, 12 and 3%, respectively. This may indicate the HS loess sequence has not been affected by radioactive disequilibrium possibly caused by radioactive element leaching and illuviation during post sedimentation. The historic mean water content of $15\pm5\%$ was assumed based on the measured water contents. Cosmic ray contribution to the dose rate was calculated from the geomagnetic latitude, longitude, the burial depth, and the altitude of the sample location [30]. Quartz internal dose rate of $0.06\pm0.03$ Gy/ka was assumed [31]. A summary of the uranium, thorium and potassium concentrations and the resulting dose rates of quartz grains are given in Table 1.

Researches demonstrated that the underestimation of the dose rate for the existence of the calcrete nodules at the base of the S1 layer in the Luochuan section [32]. In view of previous experience, we check the distribution of our dose rate (Figure 4d) and all of them are in the normal range.

![Figure 4](image_url)

**Figure 4.** Stratigraphy, MS, median grain size and OSL ages of the Heishan loess section.

### 4.2. Equivalent dose

The OSL De values and the number of aliquots used are given in Table 1. De gradually increases from ~24 to 103 Gy downwards at the Heishan section (Figure 4e). No obvious saturation of the dose response curve is observed, even for samples from the bottom of HS Section is ~103 Gy. The maximum coarse-grained quartz OSL De obtained in this research is also within the limits (~200 Gy) of reliable De determination using the SAR protocol [33].

### 4.3. OSL ages and sedimentation rate

A detailed overview about all luminescence dating results are shown in Table 1. The OSL ages of the HS profile increase with depth. The luminescence age estimates show that the HS Loess sequence covers the time range from ~8.5 ka to 33.7 ka (Figure 5a, 5d), including a chronological gap between 30 and 17 ka. The upper 1.0 m of this section was not sampled for OSL dating for apparent agricultural disturbance. The sedimentation rate can be estimated from the OSL ages, which demonstrates three stages of sedimentation and two turning points of sedimentary rate changes. Quartz SAR ages give a sedimentation rate of $54.8\pm1.1$ cm/ka during ~33.7-30.4 ka and of $5.5\pm0.8$ cm/ka during ~17.1-10.9 ka and of $32.0\pm5.5$ cm/ka during ~10.9-8.5 ka. The first point of sedimentation rate changed dramatically from $5.5\pm0.8$ cm/ka to $32.0\pm5.5$ cm/ka, at a depth of 2.5 m under the surface, dated to $10.9\pm0.6$ ka, corresponding to the boundary between marine oxygen isotope stage (MIS) 1 and MIS2. The second sedimentation rate turning point (from $54.8\pm1.1$ cm/ka to $5.5\pm0.8$ cm/ka) is at a depth of 3.7 m, dated to $30.4\pm1.9$ ka, which is equivalent to the upper layer of the Malan Loess (L1) in the CLP, corresponding to the boundary between MIS2 and MIS3.

### 4.4. Ms and grain size characteristics

MS of Chinese loess sediments is widely used to derive the palaeoclimatic changes information and mark the stratigraphy [4,7-8,34-36]. Its value is higher in paleosol than that of loess between paleosol [3]. At HS section, the MS values (1.0-2.9 m) range from 80 to 122 (mean of 99) in the Holocene
indicates strengthened summer monsoon. MS values (2.9-5.5 m) range from 63 to 93 (mean of 80) in the Malan loess, suggesting weak summer monsoon during the glacial time (Figure 5a,5b). The stratigraphic location marked by MS is consistent with field observation (Figure 5a).

Grain size variations of loess sediments in China are related to the varied intensity of the East Asian winter monsoon [37]. The median grain size is commonly used proxy for winter monsoon intensity (Xiao et al., 1995; Zhou et al., 1996). Median grain size for 1.0-2.9 m and 2.9-5.5 m within 12-21 µm (mean of 16 µm) and 12-24 µm (mean of 18 µm), respectively (Figure 5c). The results indicate a weak winter monsoon in the Holocene soil (0.9-2.9 m) and a strengthened winter monsoon in the Malan loess (2.9-5.5 m) during the glacial time.

![Diagram](image)

**Figure 5.** Stratigraphy, MS, median grain size and OSL ages of the Heishan loess section.

The changes in grain size of the HS section are reverse to those of the MS with decrease in grain size corresponding increase in MS in soil layer. This pattern is similar to that of the CLP [3]. Moreover, there are several peaks in MS during the Holocene soil, with corresponding valleys in grain size. This characteristic is consistent with the interglacial soils deposited in the Chinese Loess Plateau [3,6-7], which demonstrates that Shandong loess is a kind of typically accretion soil.

5. Discussion

5.1. Boundary age between the Pleistocene and the Holocene

MS of Chinese loess sediments is commonly used to mark stratigraphy [4,7-8,34,36]. The boundary defined by MS between paleosol and loess generally corresponds to the middle of the upper peaks and lower valleys. The boundary of L1/S1 is placed at the depth of 2.9 m by the MS records (Figure 5b), which is consistent with the observation of the field colour (Figure 5a). The boundary age of OSL defined by MS between the Pleistocene and Holocene is ~17.1 ± 0.9 ka. Nevertheless, a series of pedogenic penetration effects been considered to interpret the discrepancy in stratigraphic boundaries originated from variations of sedimentation rate, field colour and MS [38-41]. The OSL age of the Pleistocene and the Holocene boundary defined by the MS shift at Xifeng and Shiguanzhi is about 20 ka, they attributed it to a significant pedogenic overprinting of the late glacial loess [39]. Similar post-depositional diagenesis has also been tested at the Yuanbao sites [40]. They proposed to use the sedimentation rate model to define the boundary age between the Pleistocene and the Holocene. As noted above, the OSL boundary age between the Pleistocene and the Holocene is placed at 2.9 m (coarse quartz OSL age of 17.1 ± 0.9 ka) defined by the MS at HS section. In view of the possible pedogenic penetration effect, the L1/S1 boundary should be placed higher than a depth of 2.9 m. The turning point of the sedimentation rates is considered within the depth of 2.9 m [40]. Sedimentation rates varies from 5.5 ± 0.8 cm/ka (2.9-2.5 m) to 32.0 ± 5.5 cm/ka (2.5-1.5m) at the depth of 2.5 m and the corresponding quartz OSL age is 10.9 ± 0.6 ka.

5.2. Variation in sedimentation rates

The variation in sedimentation rate reveals an abrupt drop at ~30.4 ka, which is close to the MIS3/2 boundary. The dust accumulation rate decreases from 54.8 ± 1.1 cm/ka to 5.5 ± 0.8 cm/ka (Figure 5d).
The observed decreasing trend in HS loess accumulation rate from 30.4 ka is similar to many parts of Loess Plateau, such as Mangshan section (97.26 cm/ka, Qiu and Zhou., 2015), Luochuan Section (14.5-6.4 cm/ka) [42], Weinan section (16.7 cm/ka) [41] and Ledu section (89.47 cm/ka) [43]. The characteristic in loess accumulation rate across the Loess Plateau may imply that various areas including the studied region is an erosion area around 30.4 ka.

The loess sedimentation rate increases from 5.5±0.8 cm/ka (17.1-10.9 ka) to 32.0±5.5 cm/ka (10.9-8.5 ka) at 10.9±0.6 ka (Figure 5d), which is close to the MIS 2/1 boundary. The increasing trend of sedimentation rate in the early Holocene is different from most of the loess section in the Loess Plateau (e.g. Yuanbao section [40]; Jingbian section [44]) but is similar to the Weinan section in the south of the Weihe River at the southeastern margin of the CLP [41]. They attributed it to be possible pedogenesis process of palaeosol S0 or there was a relatively rapid dust sedimentation event at the beginning of the Holocene at Weinan.

The relatively rapid sedimentation rate spanning 10.9±0.6 ka to 8.5±0.4 ka may be attributed to the post-depositional disturbance. Higher sedimentation rate during the Holocene epoch (after about 11 ka), compared to the relatively low rate during the late glacial period, could be actually resulted from post-depositional disturbance [45-46]. The influence by post-depositional disturbance (i.e. bioturbation during pedogenesis) induced by more monsoon precipitation in the CSM region may be significant. It may homogenize sediments, disturb the underlying loess deposits and may offset the measured OSL age [46-47], the same sedimentary processes as the distal sites on the CLP behave [46].

5.3. Hiatus
A hiatus in the record is detected between ~30 ka and 17 ka (Figure 5d). However, it is not easily identified by eye in field investigations and via MS or grain size determinations (Figure 5a, 5b, 5c). We suggest caution when using the loess sediments in this area to obtain the information of palaeoenvironmental changes, especially during the Glacial stages such as the LGM. Recently, a hiatus was identified from 30 ka to 20 ka at Tuxiangdao (Xining, western of the CLP) loess section [43]. They emphasized the importance of high-resolution optical dating in Chinese loess research. A hiatus was also tested between 39 ka and 11 ka at the Hebei section in the northeast Tibetan Plateau by high resolution OSL dating, they attributed it to the deflation at the same time as the CLP is accumulating [48]. A gap was also detected in deposition between 25 and 2 ka at the Ledu loess section in the Huangshui river valley on the northeastern Tibetan Plateau by high resolution OSL dating, they attributed it to the erosion events or lower accumulation rate [49]. A depositional hiatus was detected between 70 and 20 ka containing the LGM at the Jingbian section by high resolution OSL dating, they demonstrate that the ice-volume forced erosion is the main reason for the sedimental hiatus [44]. There seems a sedimentation hiatus in the Mangshan loess during 30-20 ka [50] and Xiashu loess within the period of 30-0 ka [51]. Maybe there is still a hiatus between 40 and 20 ka at the section of Miaodao Island, Changshandao Island and Zibo in Shandong without high resolution dating results and they did not explicitly mention it [21,52]. It seems that loess close to desert boundaries, or adjacent to source regions of CLP, or neighbour the major river systems such as the Yangtze River or the Yellow River, often sedimentated episodically or was eroded, especially during the LGM.

We believe that an erosion hiatus at HS section is a more likely interpretation. Firstly, the recent researches indicate that the North China Plain including the Yellow River floodplain were the major dust source for the loess in the Central Shandong Mountains[20]. The deposits on the North China Plain is formed since 40 ka based on the data from the drilling samples [53]. It can be inferred that the wind-dust in Shandong Province is continuously transported and deposited on millennial time scale for the environmental pattern of the dust source area has been relatively stable at least since 40 ka. In addition, the simulated changes of atmospheric circulation in winter show that stronger winter monsoon and apparently strengthened westerlies at the LGM over the North China Plain [54]. The low accumulation rates seems less likely over the LGM, the alternative an erosional hiatus at the section mentioned above is a more plausible explanation.
6. Conclusions

The main conclusions of our study are:

1. The coarse-grained quartz SAR OSL method can be used to date the HS loess since ~34 ka in the lower reaches of the Yellow River.

2. The boundary age defined by the sedimentation rate model between the Holocene and Pleistocene is 10.9 ± 0.6 ka.

3. A marked hiatus in the record is identified between ~30 ka and ~17 ka at the HS, probably resulting from deflation, which indicates that it is an erosion area during 30-17 ka.

4. The relatively rapid sedimentation rate spanning 8.5 ± 0.4 to 10.9 ± 0.6 ka may be related to the post-depositional disturbance induced by more monsoon precipitation in the CSM region.

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