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Optical dating of a Paleolithic site near the eastern coastal region of Shandong, northern China

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ABSTRACT

Many archaeological sites were excavated in China, but rarely in the adjacent coastal areas. An archaeological site at Huangniliang in the coastal area of Shandong Province, northern China was excavated in 2013. Abundant stone artifacts including hammers, cores, flakes, chunks, and retouched tools are found in the silty aeolian sediments. In this study, optically stimulated luminescence (OSL) technique was employed to establish the chronological framework of the site. Medium-grained (45–63 μm) quartz was extracted from six sediment samples for dating. The equivalent doses obtained with the single-aliquot regenerative-dose (SAR) protocol are shown to increase with depth. Three samples from the stone-tool containing layer yield OSL ages ranging from 54 ka to 59 ka, providing the earliest geochronological evidence for the presence of humans in the eastern coast of Shandong peninsula during the early period of Marine Isotope Stage (MIS) 3.

1. Introduction

The Huangniliang Paleolithic site (N35°29′50″, E119°33′44″) is located in the eastern coastal area of Shandong Province in northern China about 4.2 km far away from Yellow Sea rim (Fig. 1). The site was discovered in 2006 and excavated in 2013. It is the newly and first formal excavation of Paleolithic sites in recent years in Shandong Province, which filled the void of Paleolithic archaeology in this area. The archaeological site flanking the coastline of Yellow Sea is the evidence for the long-term use of marine resources and coastal environments in human evolution and subsequent development, which is vital to understanding patterns of human subsistence.

The section of stratum in archaeological excavation at the Huangniliang site is loess deposit with a total thickness of ca. 4 m, the bottom of which is the weathering crust incorporating high amount of granite breccia. The site contains rich stone artifacts, and the types are various, including stone hammers, cores, flakes, chunks, and retouched tools (Fig. 2). More than 1600 *in situ* stone artifacts were unearthed from a 50 m^2 excavation pit, and no animal fossil was found. The strata with archaeological materials are covered by a sediment sequence up to 1.2 m thick, extending from 3 m to 4.2 m below the ground surface (Fig. S1, supplementary data), and stone artifacts are continuously distributed over the cultural layer in vertical.

The deposit lacked suitable material for ^{14}C dating, but the optically stimulated luminescence (OSL) technique beyond the limit of radiocarbon method has already become a widely-used and successful tool for estimating the ages of different types of sediments, especially for aeolian sediment. In this study, OSL signals from quartz by the single-aliquot regenerative-dose (SAR) protocol were carried out on the samples to determine the age of the Huangniliang site. In addition, we also propose to discuss the relationship between human coastal migrations and their associations with climate change.

2. Sample collection and measurements

OSL as a dating method has been revolutionized since the early 2000s, especially the development of a broadly-applicable single-

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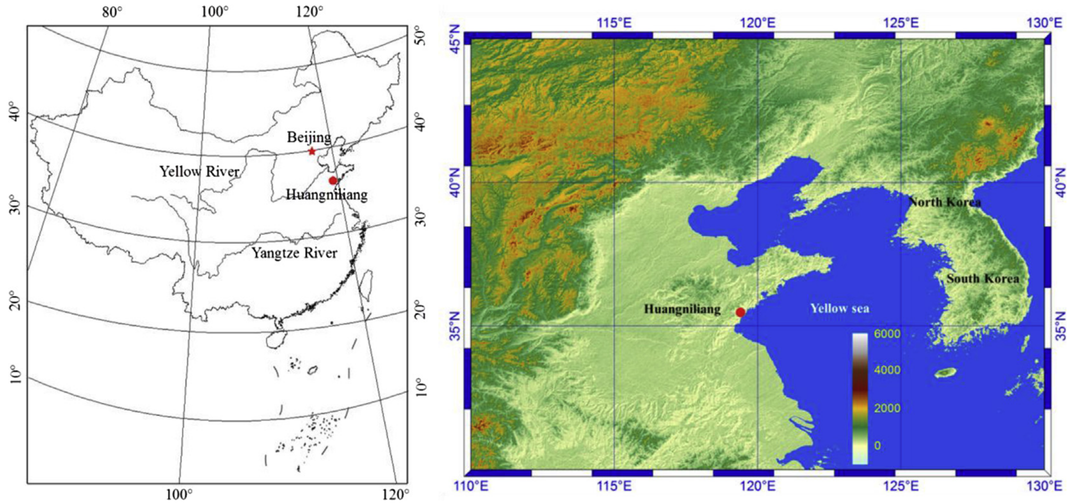

Fig. 1. Map showing the geographic location of the Huangniliang site.

aliquot regenerative-dose dating protocol for quartz is allowed to give very precise estimation of the equivalent dose (D_e) values (e.g. Wintle and Murray, 2006). The technique can be applied to estimate depositional age or burial age of the sediments in a variety of sedimentary environments (Atiken, 1998). In this study, quartz OSL signals were employed to determine the ages of the samples.

Six OSL samples were collected to provide a chronological frame for the sediment of the Huangniliang sequence, and the approximate positions of each sample are shown as red filled stars in Fig. S1 (supplementary data). Three samples (L2452-2454) from cultural layer covering the time interval of our interest and three samples (L2449-2451) from overlying sediment were all taken as a large block wrapped with aluminum foil and yellow plastic tape to provide protection against light and breakage during transportation. The number, depth below the ground surface, and U, Th, K concentration of the samples are listed in Table 1.

In order to understand the grain size distribution of the sediment, the OSL samples were analyzed by a Malvern Mastersizer 2000 laser particle-size analyzer. The samples were dissolved in 30% hydrogen peroxide (H_2O_2) and 10% dilute hydrochloric acid (HCl) to remove organic material and carbonates. 0.05 mol/L sodium hexametaphosphate ($(NaPO_3)_6$) solution was used to disperse the samples, and then the solutions were treated with ultrasonic impact and measured on the machine. The grain size distributions of the samples are expressed in Fig. 3. The results shown that all six samples follow a unimodal distribution, with the major peaks around 40–50 μm, so we chose medium grain (45–63 μm) fractions for OSL dating in this study.

Medium-grained quartz was extracted and used for age estimation. Sample preparation and OSL measurements were performed in subdued red light conditions using standard methods in the laboratory (Aitken, 1985, 1998). At least 3 cm external layers of the block samples were removed to ensure no contamination of the grains exposed to daylight during sampling, which were kept for dose rate measurements, and the etching procedures of the inner core with HCl (10%) and H_2O_2 (30%) were carried out to remove carbonates and organic material, following wet sieving to isolate 45–63 μm material. Medium-grained Quartz was extracted by silica-saturated hydrofluorosilicic acid (H_2SiF_6) (30%) treatment for three days, and then dissolved with 10% HCl to remove any fluorides and washed with distilled water several times. Medium grains were mounted on discs as a monolayer (~1.5 mm diameter) with silicone oil for OSL measurements. Infrared (IR) stimulation and 110 °C TL peak of the isolated quartz were used to check the purity. OSL measurements were performed using automated Risø-TL/OSL DA-20 reader with 7.5 mm Hoya U-340 filters (290–370 nm) in

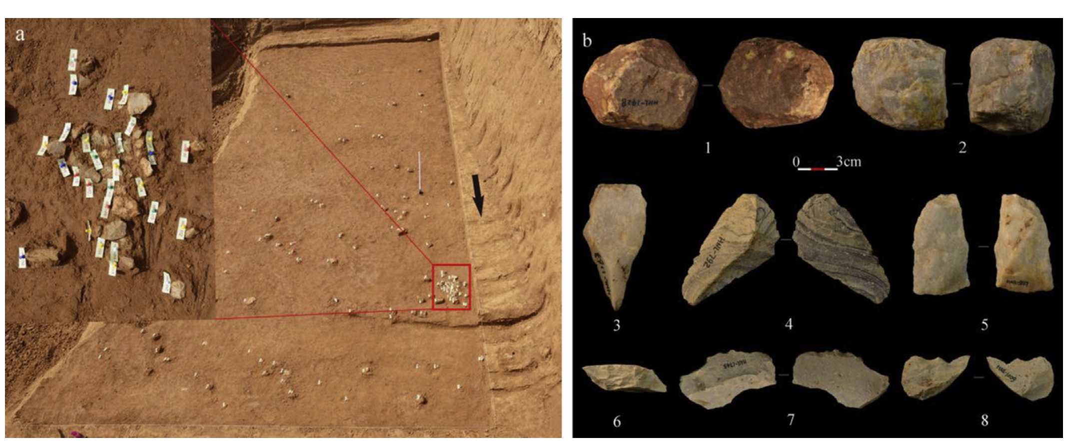

Fig. 2. a) The plane distribution of stone artifacts *in situ*. The scale is 1 m on the excavation plain. b) Stone artifacts from the Huangniliang site (1. Hammer stone; 2. Multi-platform core; 3–5. Flakes; 6. Scraper; 7–8. Denticulates).

Table 1
U, Th, K concentration and depth of the samples collected from the Huangniliang site.

Lab no.	U (ppm)	Th (ppm)	K (%)	Dose rate (Gy/ka)	Depth (m)	No. of aliquots	D_e (Gy)	Over-dispersion (%)	Age (ka)
L2449	1.82 ± 0.08	10.1 ± 0.28	2.39 ± 0.07	3.24 ± 0.18	1	10	142 ± 5	0.1 ± 0.03	44 ± 3
L2450	1.88 ± 0.08	10.6 ± 0.30	2.25 ± 0.07	3.16 ± 0.18	1.65	10	146 ± 4	0.08 ± 0.02	46 ± 3
L2451	1.87 ± 0.08	11.6 ± 0.33	2.5 ± 0.07	3.41 ± 0.19	2.55	10	171 ± 7	0.11 ± 0.03	50 ± 4
L2452	1.99 ± 0.08	11 ± 0.31	2.44 ± 0.07	3.34 ± 0.19	3.05	10	181 ± 5	0.05 ± 0.03	54 ± 4
L2453	1.78 ± 0.08	11.6 ± 0.33	2.33 ± 0.07	3.23 ± 0.18	3.6	10	188 ± 6	0.08 ± 0.02	58 ± 4
L2454	2.21 ± 0.08	10.9 ± 0.31	2.46 ± 0.07	3.44 ± 0.2	4.1	10	202 ± 4	0.04 ± 0.02	59 ± 4

front of an EMI 9235 QA photomultiplier tube. A calibrated $^{90}Sr/^{90}Y$ beta source (Bøtter-Jensen et al., 2003) was used for laboratory irradiation. Quartz grains were stimulated with blue light LED stimulation (470 ± 30 nm) set at 90% of 50 mW cm^{-2} full power.

Neutron activation analysis (NAA) was performed to determining the contribution of U, Th and K (Table 1). An alpha efficiency factor (α-value) of 0.04 ± 0.02 for quartz (Rees-Jones, 1995) was assumed to estimate the alpha contribution to the dose rate. Long-term water contents were assumed to 20% assigned uncertainties of ±5% to each value in age calculations. The calculation was performed using the 'AGE' program (Grün, 2009).

The SAR protocol (Murray and Wintle, 2000) was applied to the quartz OSL measurement, the D_e value was estimated by interpolation of the natural luminescence signal onto the growth curve, which was built with regeneration doses including a zero dose for monitoring thermal transfer effect and repeated first regeneration dose for testing the accuracy of the sensitivity correction. A fixed small test dose (13.3 Gy) after the natural/regenerative OSL measurement was used to correct for sensitivity change. A preheat at 260 °C (10 s) and a cut heat at 220 °C (0 s) were used, and then quartz OSL samples were stimulated by blue light at 125 °C for 40 s. The first 1.6 s integral of the initial OSL signal minus a background estimated from the last 8 s integral was used as a measurement of the last component for D_e estimation.

3. Results and discussion

In the medium SAR protocol, the linearity test was carried out, that is, a fixed regenerative dose (197.7 Gy) with 13.3 Gy test dose was repeated for ten cycles to check the sensitivity change with repeated heating and measurements (Wintle and Murray, 2006), the plot of the first 0.64 s quartz OSL signals of the regenerative doses vs test doses shows a linear relationship, indicating that the signals of test doses correlated well with that of regeneration doses (Fig. 4a). Dose recovery experiment (Murray and Wintle, 2003) was applied to the samples with medium quartz grains that were bleached twice by stimulation with blue LEDs for 1000 s at room temperature, with a pause of >10^4 s. The ratio of recovered dose to given dose (197.7 Gy) for selected sample L2453 was 0.95 ± 0.03 (n = 8) and its over-dispersion value is 0.06 ± 0.02%. The recycling ratios and recuperation values were within 0.95–1.05 and <2% respectively. The OSL signals were bright enough to measure, the rapid decay of the signals with stimulation time indicated that the signals were dominated by the fast component (Fig. 4b and c). Based on the results of preheat plateau test (Fig. 5), a preheat temperature of 260 °C for 10 s, and a cut-heat of 220 °C for 0 s were selected for OSL dose measurements. The SAR protocol with medium-grained quartz can be used to establish a reliable chronology for the deposit.

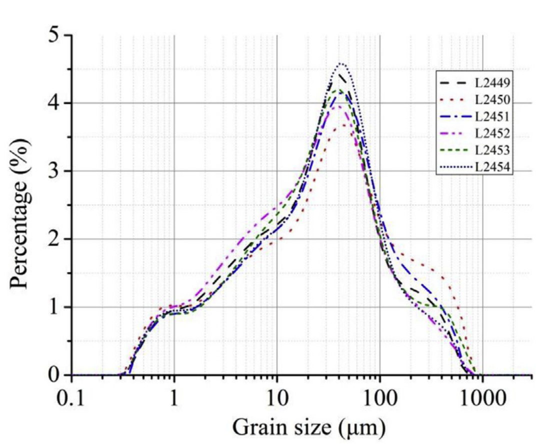

Fig. 3. Grain size distribution of the samples from the Huangniliang site.

Table 1 provides a summary of dose rate, D_e values, over-dispersion and ages of the six medium-grained quartz OSL samples with 10 aliquots measured of each sample. The results of dosimetry indicate very homogeneous sedimentary environments all along the sequence. The dose response curves of all the samples were fitted with single saturating exponential functions (the mean $D_e/2D_0$ values were below unity) and saturating exponential-plus-linear functions, and the D_e values from both functions were almost indistinguishable within error, however, the exponential-plus-linear functions gave better fittings, which were used for the interpolation of D_e (Fig. 4d). D_e values of samples L2449, L2450, L2451, L2452, L2453 and L2454 were 142 ± 5 Gy, 146 ± 4 Gy, 171 ± 7 Gy, 181 ± 5 Gy, 188 ± 6 Gy and 202 ± 4 Gy, respectively. D_e over-dispersion of the samples were in a similar range within experimental error (Table 1), and the over-dispersion between the dose recovery D_e values (well-bleached and homogeneous OSL behavior) and the natural D_e values were similar, suggesting that the samples were well bleached at the time of deposition and no spatially heterogeneous dose rates significantly affected the natural dose estimates. Although we must remain cautious about the proximity to saturation (especially for sample L2454), it is not likely that we underestimated the D_e values of the samples. At 1 m, 1.65 m and 2.55 m depths the three samples L2449, L2450 and L2451 above the cultural layer gave central OSL ages of 44 ± 3 ka, 46 ± 3 ka and 50 ± 4 ka, respectively. The central ages at 3.05 m, 3.6 m and 4.1 m are 54 ± 4 ka, 58 ± 4 ka and 59 ± 4 ka for the samples L2452, L2453 and L2454 from the cultural layer, respectively. The OSL ages show an increase with depth, which is consistent with the increasing storage time.

The artifact-bearing layer is ranged between 54 ± 4 ka and 59 ± 4 ka. Those ages are compatible with the MIS 3c suggesting that the Huangniliang area was occupied during this warm period (Fig. 6), demonstrating by stalagmite oxygen isotope record (Hulu cave, China) and linking North Atlantic climate with the meridional transport of heat and moisture from the warmest part of the ocean where the summer East Asian Monsoon originates (Wang et al., 2001).

Archaeological records are significant for understanding human subsistence patterns, mobility and dispersal (Bailey and Flemming, 2008). The main Paleolithic sites with absolute ages between 50 ka

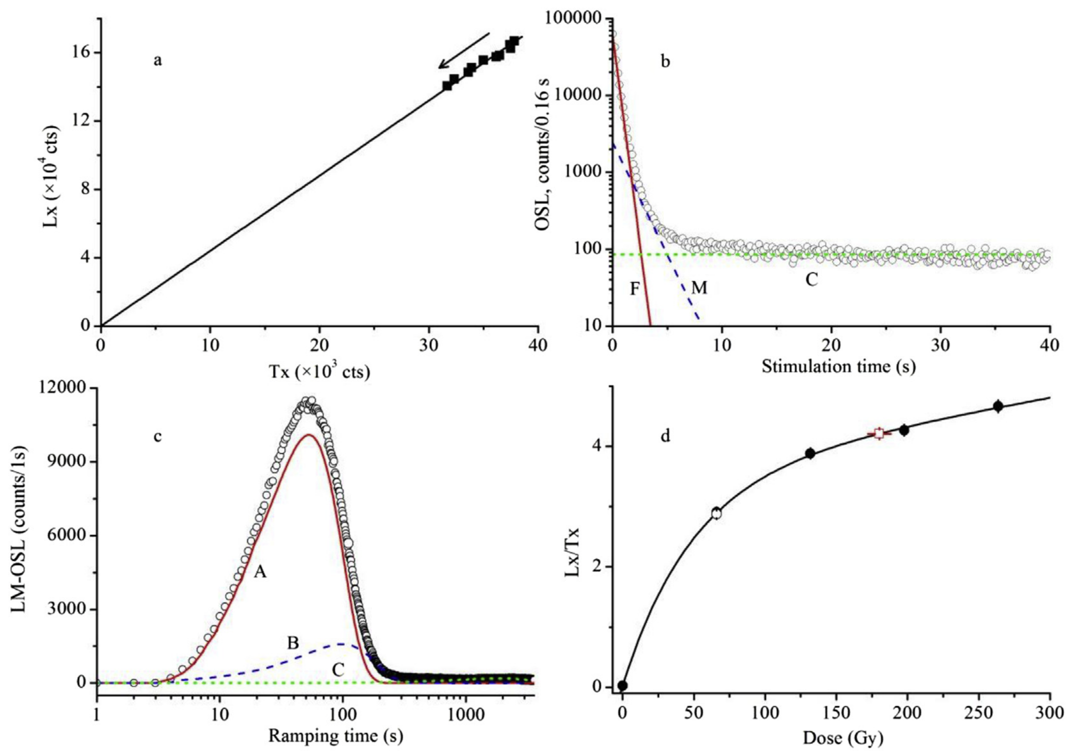

Fig. 4. a) OSL signal of repeated regenerative dose of 197.7 Gy versus the signal measured following a test dose of 13.3 Gy using the SAR protocol. b) Component separation of CW-OSL signals from natural quartz grains. F, M, and C are the fast, medium and constant components, respectively. c) Separation of natural quartz LM-OSL signals, which was measured at 125 °C for 3600s after preheating at 260 °C for 10s. Three components named A, B and C were obtained on the basis of the fitting equation (2) from Choi et al. (2006). d) Dose response curve obtained with quartz SAR protocol. L_x/T_x represents the sensitivity-corrected luminescence intensity to the test dose. Solid and open circle symbols represent regeneration doses and repeated dose, respectively. The open square symbol is the equivalent dose. a)–d) were based on the typical sample L2453.

and 100 ka are shown in Table S1 and Fig. S2 (supplementary data), most dates were obtained by U-series, as well as OSL, TL, ^{14}C and $^{230}Th-^{234}U$. In fact, the ages of some sites are questionable or controversial due to restrictions of dating techniques at that time, uncertain locational or stratigraphic context of dated samples, or complicated sediment environments. A discussion of these ages is beyond the scope of this paper and we will not discuss it in detail here, for a fuller discussion see Norton and Jin, 2009, Keates, 2010 and Gao, 2014. Huangniliang is the only coastal site of the above-mentioned Paleolithic sites listed in Table S1 (supplementary data).

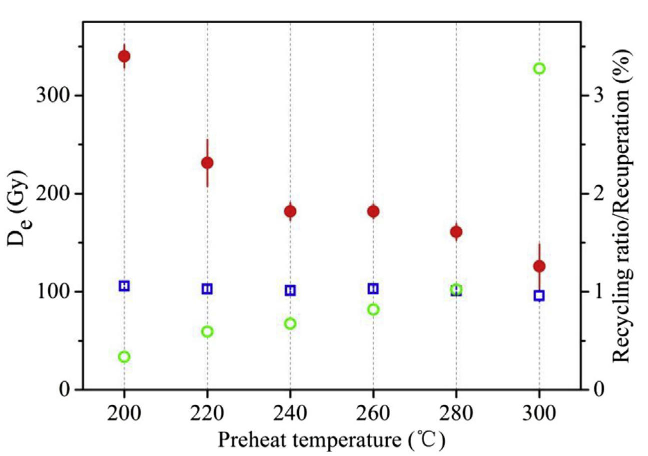

Fig. 5. Solid circles display equivalent dose as a function of preheat temperature for the medium-grained quartz (cut heat temperature = 220 °C). Open squares and circles refer to the recycling ratios and recuperation values for the sample L2453, respectively.

The Huangniliang site is close to the coastal area of Yellow Sea and is the oldest well-dated Paleolithic site in coastal area of China at present, which had powerful effects on human life, providing a suitable living environment for living beings during MIS 3c, and

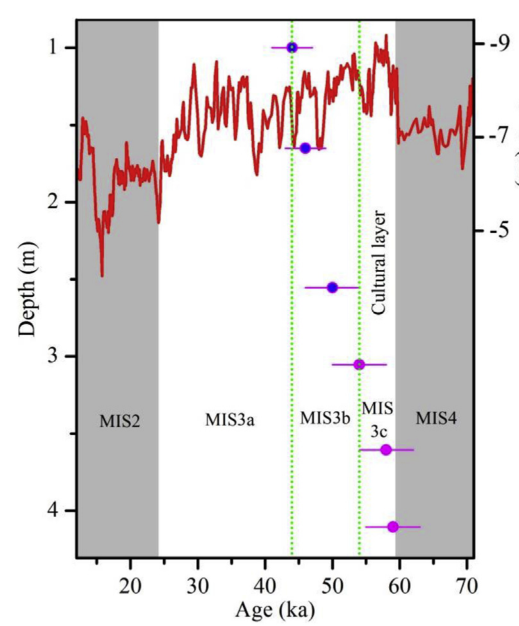

Fig. 6. The relationship of OSL ages and oxygen isotope at the Huangniliang site. Solid circles represent the ages obtained by the SAR protocol. Stalagmite oxygen isotope records derive from Wang et al. (2008).

human could have sufficient water and food. Unfortunately due to absence of animal bones and other remains representing food exploitation choices, we cannot reconstruct subsistence strategy of the Huangniliang occupants now. During the cold period MIS 3b temperature was ca. 5 °C lower than today (Yang et al., 2004), it was perhaps main factor of human dispersal, and finally human moved out from this area.

The origin and evolution of modern humans is a controversial issue today, and the timing of the dispersal of modern humans from Africa is a continuing and lively topic of debate. It is now general consensus that Pleistocene Homo sapiens from Ethiopia by 195,000 years ago represent the probable ancestor of modern humans (Clark et al., 2003; White et al., 2003; McDougall et al., 2005), and they dispersed from Africa and arrived in Eurasia about 40–50 ka (e.g. Cann et al., 1987; Coon, 1962; Endicott et al., 2009; Klein, 2008; Oppenheimer, 2012). However, the recent discoveries of human remains in central and south China implied that the anatomical features of modern humans occurred much earlier than current genetic evidence (e.g. Liu et al., 2010a, 2010b), but some researchers have questioned the dates and their associations with human fossils at the Zhiren Cave in Guangxi Province (e.g. Dennell, 2010). Other scholars proposed an alternative hypothesis of the origin of modern humans as "Continuity with Hybridization" (e.g. Wolpoff et al., 1984; Wu, 1990, 1998, 2006; Gao et al., 2010) which suggested that modern humans were developed from *Homo erectus* in different regions, especially in China. To solve the controversial subject above, appropriate dating techniques, sufficient fossil and archaeological records are elementary for the research of modern human origin and dispersal in China. The Huangniliang site located on the loess tablelands of Yellow Sea coastal area is a Paleolithic site with plentiful stone artifacts and reliable ages older than 50 ka, and subsequent detailed technological analysis will be helpful to clarify the controversies between hypotheses of origins of Modern humans. The new dates linking the occupation of the Huangniliang hominids to a relatively warm period and will also help us to understand interactions between the Huangniliang occupants and the local environment in coastal areas with more evidence of future environmental reconstruction. We further will establish reliable chronologies for more coastal Paleolithic sites to discuss the possible migration routes of human populations in coastal area of East Asia.

4. Conclusions

Human evolution in East Asia is still controversial because of the scarcity of a reliable chronology of archaeological sites. In this study we presented the OSL ages obtained from the medium-grained quartz at the Huangniliang site along the coast of the Shandong Peninsula. SAR measurements of loess deposit from the site yielded internally and stratigraphically consistent ages within errors, indicating adequate reliability of the technique. The results showed that the ages of loess deposit associated with the abundant stone artifacts were varied from 54 ± 4 ka to 59 ± 4 ka. The period linked to MIS 3c suggesting a potentially favorable and moist environment, that is, ancient humans have occupied this area within the period. Huangniliang is a coastal Paleolithic site, which is significant to investigate the subsistence patterns and migration routes of hominids in the coastal regions. However, at present we could not reliably compare the stone tools with other neighboring Paleolithic assemblages, due to the scarcity of precise and more robust chronologies for constraining and correlating stratigraphic successions. More dating work is required, and our further work will focus on constructing accurate chronologies for the Paleolithic sites in coastal East Asia to explore questions about human dispersal and human–environment interactions.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.quageo.2015.02.009.

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