J. Geogr. Sci. 2019, 29(3): 406-416
DOI: https://doi.org/10.1007/s11442-019-1606-1

Cosmogenic nuclide burial dating of Liuwan Paleolithic site in the Luonan Basin, Central China

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Abstract: The Luonan Basin is a key region of early human settlement in Central China with more than 300 discovered Paleolithic sites. Artifact layer 1 of the Liuwan site was dated to approximately 0.6 million years (Ma) based on correlation with the well-dated loess–paleosol sequence of the central Chinese Loess Plateau. This study reassessed the age of the Liuwan artifact layer via an absolute dating method, namely, ^{26}Al/^{10}Be burial dating. We determined the burial age of artifact layer 1, which was most likely at least 0.60 ± 0.12 Ma (1σ), using three simple burial ages. The new burial age confirmed the previous estimated age and provided a considerably accurate age range. Therefore, we suggest the use of the ^{26}Al/^{10}Be burial dating method in thin loess-covered Paleolithic sites around the Qinling Mountain Range is helpful to understand the early human behavior.

Keywords: Luonan Basin; loess; Paleolithic artifact; Middle Pleistocene; Cosmogenic nuclides

1 Introduction

China is a key area of early human settlement in East Asia during the Pleistocene. The Qinling Mountain Range (QMR) in Central China is a recognized center of early human occupation (Woo, 1964, 1966; Xue, 1987; Li and Etler, 1992; Wang *et al*., 1997, 2004, 2005, 2008; SPIA *et al*., 2007, 2008; Lu *et al*., 2007, 2011a, 2012, 2017; Wang and Lu, 2014, 2016; Sun *et al*., 2017), which can correspond to the well-known Nihewan Basin in North China (Chia and Wei, 1978; Zhu *et al*., 2001; Deng *et al*., 2006). The QMR is highlighted by hundreds of Paleolithic open-air sites and characteristic artifacts, such as hand axes, spheroids, picks, and heavy-duty scrapers (Wang *et al*., 2005; SPIA *et al*., 2007).

A few Paleolithic sites in the QMR, such as Liuwan (Lu *et al*., 2007, 2011b; Sun *et al*.,

Received: 2018-01-13 **Accepted:** 2018-05-30
Foundation: National Natural Science Foundation of China, No.41572155, No.41690111; The Global Change Program of the Ministry of Science and Technology of China, No.2016YFA0600503
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2014), Qijaojiayao (Lu *et al.*, 2007, 2011a), Yaochangwan (Sun *et al.*, 2012; Wang *et al.*, 2014), and Luojiacun (Sun *et al.*, 2017), have been dated approximately 0.60 Ma. The period of dense hominin settlement in the QMR was approximately 0.60 Ma. However, only several hominin fossil sites were found during this period (approximately 0.50–0.80 Ma) in China based on credible archaeological data. These archaeological sites were distributed between 20°N and 40°N latitude (Figure 1). Therefore, hominin settlement and distribution in the QMR during approximately 0.60 Ma are important for understanding early human evolution in China.

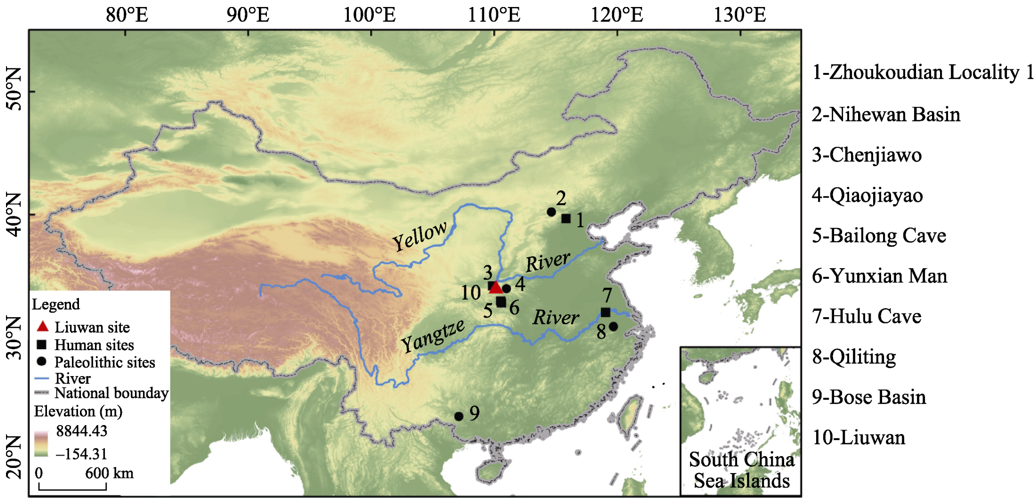

Figure 1 Locations of the Liuwan Paleolithic site in China and other Middle Pleistocene (approximately 0.50–0.80 Ma) hominin fossil and Paleolithic sites

An old and deeply rooted question is whether climate change shapes human evolution (Potts, 1998, 2012). Landforms and elevation are two important influential factors in human evolution. Hominin settlements were mainly located in riverine areas or fluvial terraces, particularly in medium-sized fluvial basins in China (Lu *et al.*, 2017), such as the Nihewan Basin (Zhu *et al.*, 2001, 2007) and the Bose Basin (Hou *et al.*, 2000). Many Middle Pleistocene hominin fossil and Paleolithic sites, such as Zhoukoudian Locality 1 (Shen *et al.*, 2009), Chenjiawo (An and Ho, 1989), and Yunxian Man (Li and Etler, 1992; Chen *et al.*, 1997), are on the second step or the transition between the second and third steps in China (Lu *et al.*, 2017) (Figure 1). Although Hulu Cave (Zhao *et al.*, 2001; Liu *et al.*, 2005) and Qiliting (Archaeology and Office, 2009) are in the Middle–Low Yangtze Plains, these areas are in hilly and mountainous locations near rivers (Figure 2).

The QMR in Central China is a natural barrier that functions as a boundary between the southern and northern climatic regimes and is a sensitive area for climate change controlled by the Asian monsoon. The QMR is a representative zone for the middle latitude, intermountain basins, and warm areas. Thus, Paleolithic discoveries are found in the Luonan and Lushi Basins along the South Luohe River Valley in eastern QMR, in the Hanzhong, Ankang, Yunxian, and Danjiang basins along the Hanjiang River Valley in southern QMR, and in the Lantian Basin along the Bahe River Valley in northern QMR (Figure 2). All these Paleolithic sites are covered with thin loess deposits along medium-sized rivers.

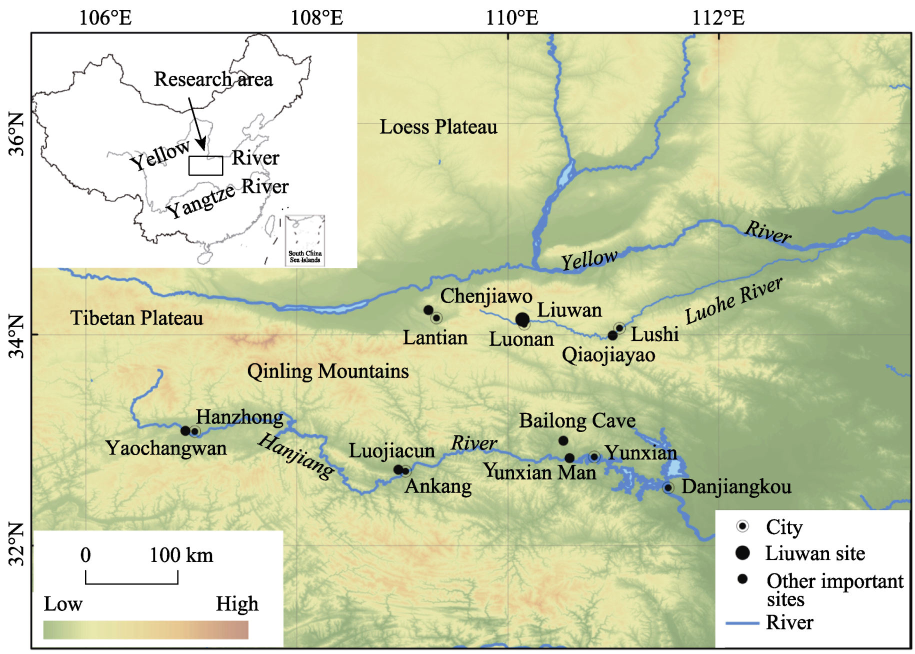

Figure 2 Location of the Liuwan Paleolithic site in the Luonan Basin and other Paleolithic sites approximately 0.60 Ma in the Qinling Mountains Range

We dated many Paleolithic sites (Lu *et al*., 2007, 2011a, 2011b; Sun *et al*., 2012, 2014, 2016, 2017) in the QMR using pedostratigraphic correlation with the well-dated loess–paleosol sequence of the Luochuan loess section of the central Chinese Loess Plateau (CLP). We identified a certain range of age error in these Paleolithic sites due to restrictions imposed by the paleomagnetic dating method and the problematic magnetic susceptibility used in thin and atypical loess deposition areas.

An absolute age control from a radioisotopic dating method is required to assess previously established chronologies of these Paleolithic sites. In the current study, we attempted to date the Liuwan Paleolithic site in the Lunan Basin by using the ^{26}Al/^{10}Be burial dating method. This method is a relatively new radioisotopic dating technique based on the built-up and radioactive decay of two cosmogenic nuclides.

2 Geographical, archaeological, and stratigraphic settings

2.1 Geographical setting

The Luonan Basin is an intermountain depression in the upper drainage of the South Luohe River (Figure 2). The terrace system of this river is composed of alluvial and thin loess deposits along the flanks of the river valleys. The Liuwan Paleolithic site (34°08′37″N, 110°08′13″E; 948 m above sea level) is located 6 km north of Luonan City. This site is situated on the second terrace of the Maping River, which is the main tributary of the South Luohe River (Figure 2). Low hills with an average elevation of 1000 m are found along both sides of the Maping River. This river is short, and the Liuwan site is only approximately 10 km from its headstream.

2.2 Archaeological setting

More than 20,000 lithic artifacts in over 300 Paleolithic sites have been discovered in the Luonan Basin from low to high terraces along the South Luohe River, thereby making the latter the richest Paleolithic artifact basin in Central China (Wang *et al*., 2005; SPIA *et al*., 2007).

The Luonan Basin was first determined to be attractive to the prehistoric community in the 1990s after a few Paleolithic artifacts were individually collected on the surface (by SJ Wang) from various landforms in this region. Thereafter, several sites with in situ artifacts, including Liuwan (locality 1), which is the most representative and contains distinct alternations of loess and paleosol, have been excavated and studied (Lu *et al*., 2007, 2011a, 2011b). In 2009, Sun *et al*. (2014) revisited the site and found two additional localities (localities 2 and 3) (Figure 3). Two in situ stone artifact layers were identified and more than 200 artifacts, including stone hammers, scrapers, points, cores, flakes, debris, and chunks, were unearthed during the trial excavation.

The loess deposit is approximately 500 cm thick in Liuwan locality 3, and three loess units interleaved with four paleosol complexes were identified (Figure 3). Artifact layer 1 lies approximately 160 cm below layer 2. This artifact layer is between 445 cm and 450 cm deep and contains 33 artifacts. Artifact layer 2 is situated at 305 cm and has a thickness of 5 cm. A total of 107 artifacts were excavated in this locality.

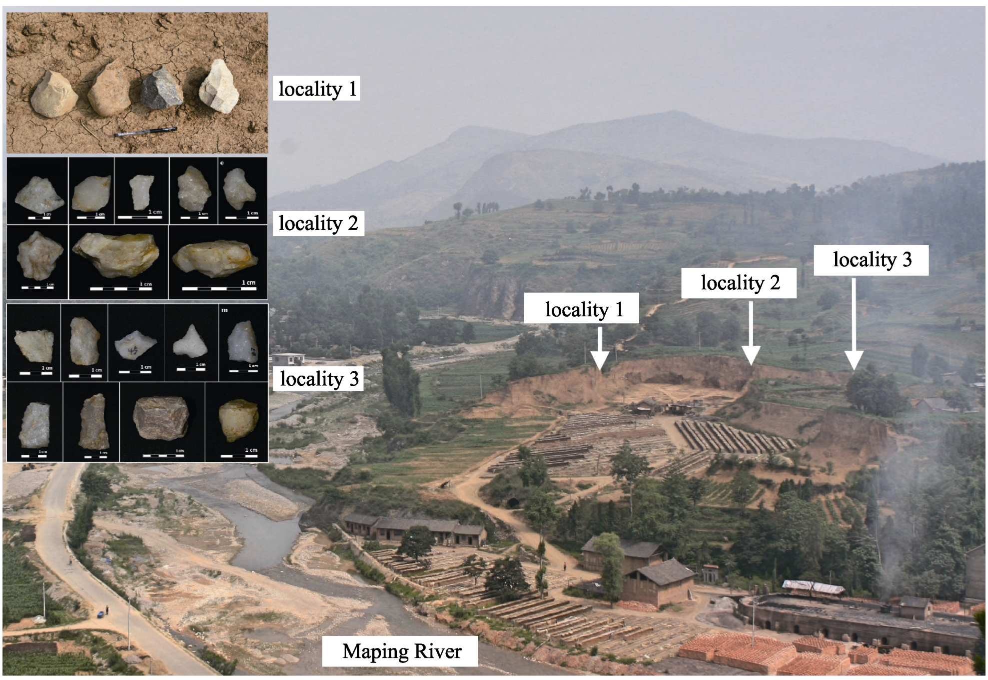

Figure 3 Positions and Paleolithic artifacts of localities 1, 2, and 3 in the Liuwan site. Collected hand axes in locality 1, excavated Paleolithic artifacts in artifact layer 1 in locality 2, and excavated Paleolithic artifacts in artifact layer 2 in locality 3 (Lu *et al*., 2007; Sun *et al*., 2014).

2.3 Stratigraphic setting

We studied the pedostratigraphy and magnetic susceptibility of Liuwan localities 2 and 3 (Sun *et al*., 2014) and compared their results with those of locality 1 and a typical loess–paleosol sequence in Luochuan (Lu *et al*., 2007). The result is still comparable although magnetic susceptibility, as a paleoclimate proxy in the Liuwan site, differs from the magnetic susceptibility recorded in the loess–paleosol sequence of the CLP (Lu *et al*., 2007; Sun *et al*., 2014). Nevertheless, we significantly correlated localities 1, 2, and 3 using the L5 layer as a prominent marker in the pedostratigraphy and magnetic susceptibility records (Sun *et al*., 2014). We found S5, L5, S4, S3, L3, S2, L2, S1, and L1 layers in locality 2. Only S5, L5, and the combined S4 and S3 layers were found in locality 3 for the upper part of the loess deposition transferred by local farmers for brick making. Although the loess deposit is thin, no evident hiatus was generally observed in the section. As shown in Figure 3, the loess–paleosol sequence in the Liuwan section is a condensed and "mini" type of the Luochuan loess–paleosol sequence.

Artifact layer 2 was suggested to be located in paleosol unit S5SS2, whereas artifact layer 1 is located in the S5SS3 soil unit based on the pedostratigraphy and magnetic susceptibility of localities 1, 2, and 3 (Sun *et al*., 2014). Therefore, the approximate ages of the two layers are 0.60 Ma (Lu *et al*., 1999).

3 Method and experiments

3.1 Sampling

We collected three sets of Paleolithic artifacts from artifact layer 1 at the bottom of the Liuwan site for $^{26}Al/^{10}Be$ burial dating. This area was the lower artifact layer. Each set of samples comprised four to five pieces of vein quartz chunks (Figure 4). These samples were collected in situ from the loess deposits.

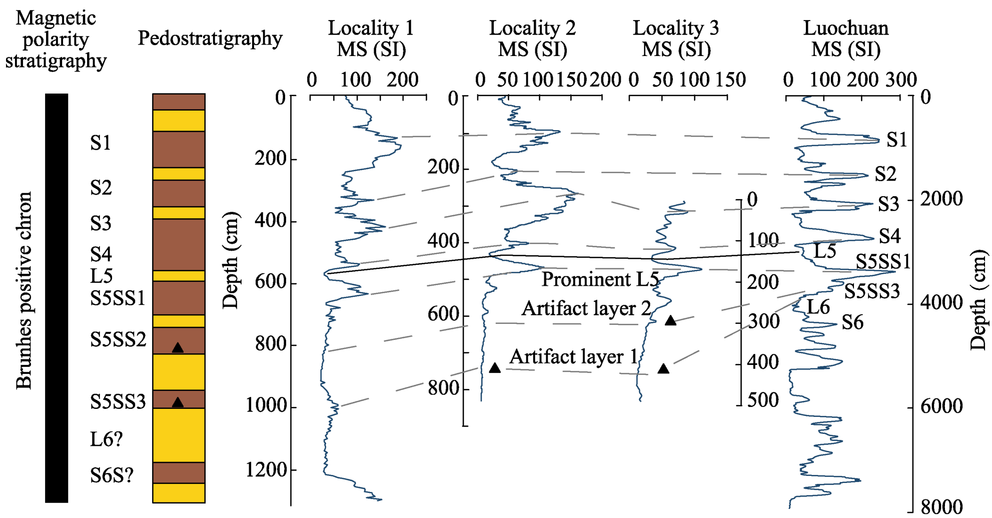

Figure 4 Positions of ^{26}Al and ^{10}Be burial dating samples in artifact layer 1 (black triangles) in the Liuwan localities and magnetostratigraphy, pedostratigraphy, and magnetic susceptibility records correlated with the Luochuan loess–paleosol sequence in Central CLP (Lu *et al*., 2007; Sun *et al*., 2014)

3.2 $^{26}Al/^{10}Be$ burial dating

The $^{26}Al/^{10}Be$ burial dating technique is an important method for dating quartzose deposits buried in the past 0.3–5.0 Ma and is particularly used in regions where K/Ar (Ar/Ar) is inapplicable. After its first application to cave deposits (Granger *et al.*, 1997), the $^{26}Al/^{10}Be$ burial dating method has been successfully used in river fluvial deposits (Granger and Smith, 2000), lacustrine sediments (Kong *et al.*, 2009), and conglomerate deposits (Kong *et al.*, 2011; Tu *et al.*, 2017).

The basic theory of $^{26}Al/^{10}Be$ burial dating has been elucidated by Granger and Muzikar (2001) and Granger (2014). The theory assumes that quartz mineral is gradually exposed in an outcrop that erodes steadily, and certain amounts of ^{26}Al and ^{10}Be will be produced by secondary cosmic rays. The ratio of the production rates of the two nuclides is basically constant and is typically assumed to be 6.8. If quartz is buried by meters of sediments or rushed into caves, then cosmogenic ^{26}Al and ^{10}Be production drastically decelerates. The inherited nuclides decay according to their specific half-lives, whereas a continued slow accumulation of nuclides through muon-induced reactions may persist. For samples that inherited a high concentration of nuclides and were buried deeply and rapidly, post-burial production is minimal and can be safely disregarded. In this case, the calculated age is referred to as the simple burial age. However, the reliability of simple burial dating will be affected in two cases. In the first case, the sample was not buried rapidly and deeply enough to be shielded quantitatively against cosmic rays; thus, the obtained age result will be underestimated. In the second case, the sample experienced prior burial before the last deposition; thus, the age result will be overestimated.

3.3 Sample preparation and measurements

The samples were pretreated in Nanjing University and Nanjing Normal University in China. Raw samples were crushed into submillimeter grains, and samples between 0.2 mm and 0.5 mm were sieved out and purified through hydrofluoric acid (HF) leaching and magnetic and gravimetric separations. Then, the samples were analyzed in the Australian Nuclear Science and Technology Organization (ANSTO). The purified quartz (50–100 g) was dissolved in HF/HNO_3 and spiked with approximately 0.3 mg ^{9}Be carrier. After HF volatilization, Fe was removed by pH-controlled precipitation. Al and Be were separated by ion exchange chromatography, and Be was further purified using precipitation and chromatography. Al and Be were then precipitated as hydroxides and transformed into oxides in a furnace at 800°C. Al_2O_3 and BeO were loaded into cathodes for $^{10}Be/^{9}Be$ and $^{26}Al/^{27}Al$ measurements using an

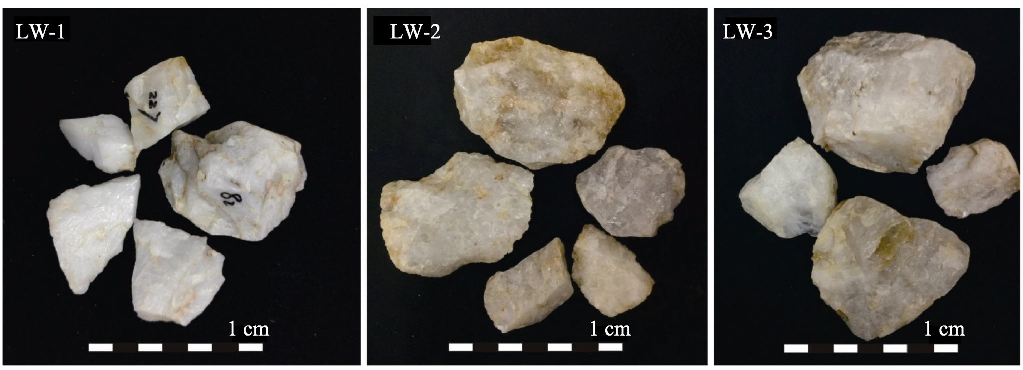

Figure 5 Dating samples from artifact layer 1 in Liuwan locality 3 (each group is a mixture of several vein quartz chunks)

accelerator mass spectrometer at the ANTARES Accelerator Mass Spectrometry (AMS) Facility, ANSTO.

4 Result

The AMS measurement of the three samples from the Liuwan site resulted in relatively good precision, namely, 2%–4% for $^{26}Al/^{27}Al$ and approximately 2% for $^{10}Be/^{9}Be$. The integrated precision percentages in nuclide concentration were 5%–6% and approximately 3% for ^{26}Al and ^{10}Be, respectively. Table 1 lists the nuclide concentrations and the corresponding simple burial ages. The simple burial ages (i.e., LW-1: 0.14 ± 0.11 Ma; LW-2: 0.20 ± 0.13 Ma; and LW-3: 0.60 ± 0.12 Ma) were calculated by assuming that the samples did not experience prior burial and did not undergo post-burial production.

Table 1 Cosmogenic nuclide concentrations and simple burial ages of the vein quartz chunks from artifact layer 1 in Liuwan locality 3

Sample	Description	Burial depth (m)	^{10}Be concentration ($\times10^6$ at g^{-1})	^{26}Al concentration ($\times10^6$ at g^{-1})	$^{26}Al/^{10}Be$	Minimum age (Ma)	Burial age (Ma)
LW-1	Paleolithic artifacts	4.5	0.2314 ± 0.0062	1.4401 ± 0.0719	6.223 ± 0.352	0.144 ± 0.115	0.144 ± 0.115
LW-2	Paleolithic artifacts	4.5	0.1895 ± 0.0062	1.1502 ± 0.0686	6.068 ± 0.412	0.201 ± 0.137	0.201 ± 0.137
LW-3	Paleolithic artifacts	4.5	0.4182 ± 0.0113	2.0541 ± 0.1106	4.912 ± 0.296	0.599 ± 0.122	0.599 ± 0.122

Minimum ages are obtained by assuming that the samples are completely shielded from cosmic rays after burial, without considering the nuclides produced during and after the depositional process caused by insufficient shielding against cosmic rays.

The accuracy of the simple burial ages highly depends on the validation of the two assumptions. First, we considered that the Liuwan samples were unlikely to have prior burial history because the Liuwan site is situated on the second terrace of the Maping River, which is approximately 10 km to the headstream of the river. The artifacts, including our dating samples, were most likely made of raw materials collected from the river bed at the time of human occupation. No depositional basin is present upstream; hence, the possibility that our samples might have been buried previously elsewhere is unlikely. Accordingly, the overestimation of the simple burial ages is improbable.

Second, the dating samples were buried in approximately 4.5 m-thick loess–paleosol. The overburden was insufficiently thick to shield quantitatively against cosmic rays, and the sedimentation rate of these aeolian deposits was relatively slow. Thus, the nuclides produced during and after the sedimentation process could not be completely disregarded. Moreover, the simple burial ages of the samples should have been underestimated. Although the three samples are supposed to be of the same true age and have identical post-burial components, their apparent ages vary significantly. One reason is that the underestimation degree of different samples depends on their inherited nuclide concentrations, i.e., samples with high concentrations yielded old apparent simple burial ages. The Liuwan samples followed this tendency within the error range.

The three simple burial ages are most likely minimal estimates; therefore, we suggest to use the oldest one (LW-3: 0.60 ± 0.12 Ma) to mark the minimal age of cultural layer 1 in the Liuwan site.

5 Discussion

5.1 Dating problems in the QMR

With loess–paleosol alternations contained loess. Loess is considered a significant archive of past environmental changes and provides a precise time series for studying hominin evolution. However, issues remain despite our successful application of optically stimulated luminescence (OSL) dating, thermally transferred OSL (TT-OSL) dating of quartz, post-infrared infrared stimulated luminescence (post-IRIRSL) dating of K-feldspars, magnetostratigraphic analyses, and pedostratigraphic correlation with the well-dated loess–paleosol sequence of the CLP to set a basic time series for several sites (Sun *et al.*, 2012, 2013, 2016, 2017).

The loess deposited in the southern and eastern Qinling Mountain areas differs from the typical loess deposited in the CLP. Accordingly, the thin loess deposit in a few high terraces is merely a short segment that contains the old part of the entire loess–paleosol sequence, whereas the young part on the surface has been eroded (Guo *et al.*, 2013; Sun *et al.*, 2017). This scenario presents a major obstacle to directly comparing the incomplete loess–paleosol sequence with the typical sequence of the CLP for the missing upper loess–paleosol sequence. In a few sites that are younger than 0.2 Ma, the use of the reference OSL, TT-OSL, and post-IRIRSL ages and magnetic susceptibility will enable the correlation of the incomplete loess–paleosol sequence with the complete loess–paleosol sequence in the CLP (Sun *et al.*, 2017).

However, the surface part of the loess section has already exceeded the dating ranges of OSL, TT-OSL, and post-IRIRSL, and no paleomagnetic alternation occurred for the span between approximately 0.20 Ma and 0.78 Ma or older than 0.78 Ma. These conditions are considerably common in the Middle to Early Pleistocene Paleolithic sites in the southern and eastern Qinling Mountains, particularly on high terraces. Moreover, suitable materials for the K/Ar dating and $^{230}Th/^{234}U$ dating in the loess-deposited areas are rarely available. Although we determined a few loess–paleosol segments, no reference age was obtained. The correlation of the incomplete loess–paleosol sequence with the complete loess–paleosol sequence in the CLP is questionable without any radiometric age control.

5.2 Use of burial dating method

In this study, the simple burial ages of loess-covered artifacts provided a minimal age control (0.60 ± 0.12 Ma) for Liuwan locality 3, thereby allowing the correlation of the incomplete loess–paleosol sequence of the site with the complete loess–paleosol sequence in the CLP and supporting the previous age estimate from Sun *et al*. (2014). Combined with the normal polarity of the entire strata (Figure 3), which should correspond to the Brunhes Chron (<0.78 Ma), artifact layer 1 in the Liuwan Paleolithic site most likely dates back to 0.60–0.78 Ma. This result reconfirmed that hominins occupied the Luonan Basin since at least 0.60 Ma.

Restricted by the unignorable post-burial produced cosmogenic nuclides, a simple burial dating method cannot yield an accurate age for loess-covered samples. Nonetheless, the method can provide a minimal age control to the site, which is valuable in correlating the incomplete paleomagnetic and pedostratigraphic records with the reference ones.

A simple burial dating method is still recommended only to date samples that were rapidly and deeply buried or loess-covered samples with relatively high inherited nuclides and thick burial depth. For loess-covered samples, an alternative and more reliable approach for dating is the isochron burial dating method (Erlanger *et al*., 2012). This approach provides a means to determine the true burial age of shallowly buried samples and is proven useful in dating loess-covered samples by analyzing a set of clasts (or stone artifacts) from the same horizon level (Tu *et al*., 2017).

Many Early–Middle Pleistocene Paleolithic sites in China were recovered from aeolian deposits, such as Xiashu loess, reticulate red clay, and Quaternary red clay. Most of these sites, including the Chenshan Paleolithic sites in Xuancheng, Anhui Province (Yang *et al*., 1997; Fang *et al*., 1997), the Qiliting Paleolithic sites in Changin, Zhejiang Province (Xu 2008; Liu *et al*., 2014), and the Huxushan Paleolithic site in Chishandao, Hunan Province, were dated using the paleomagnetic dating method or stratigraphic correlation. Only a few of these sites have been accurately dated. Therefore, we hope that the application of cosmogenic burial dating can contribute to the chronological framework of Early–Middle Pleistocene human evolution in East Asia.

6 Conclusions

Three groups of vein quartz artifacts in artifact layer 1 in Liuwan locality 3 were dated in situ using the simple ^{26}Al and ^{10}Be burial dating method. The simple burial dating ages were 0.14 ± 0.11, 0.20 ± 0.13, and 0.60 ± 0.12 Ma. The post-burial production of these samples was not negligible, and the simple burial ages should be considered as minima given the relatively slow sedimentation rate and thin burial depth. Therefore, artifact layer 1 in locality 3 should be older than 0.60 ± 0.12 Ma. The magnetostratigraphical record also indicates that the site is most likely dated to 0.60–0.78 Ma. Therefore, the application of the minimum age of the simple ^{26}Al and ^{10}Be burial dating method is useful in dating Paleolithic artifacts in thin loess-covered Paleolithic sites in the QMR and in Southern China.

Acknowledgment

We thank Prof. David Fink for his help in the laboratory work, and Prof. Guanjun Shen for his help and encouragement.

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