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Coupled ESR and U-series dating of fossil teeth from Yiyuan hominin site, northern China

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ABSTRACT

Coupled ESR and U-series analyses of mammalian fossil teeth were carried out on two localities of Yiyuan hominin site (Locality 1 and 3) in northern China. The U-migration history of the fossil samples could be reconstructed by the combination of the two techniques, and overcome the limitation of stand-alone ESR and U-series age estimation. We obtained a combined ESR/U-series age (AU model) range from ~420 to 320 ka from nine teeth recovered from the two localities, which pinpoints the deposition of hominin layer of Yiyuan site to MIS 11 to 9. The age results in this study places Yiyuan site at the same time range of Zhoukoudian Locality 1 and Hexian *Homo erectus* sites. Comparing with other hominin sites, this study of Yiyuan *Homo erectus* site highlights the possibility of coexistence between *Homo erectus* and archaic *H. sapiens* in China.

1. Introduction

In 1981 and 1982, a fragment of human cranial vault bone, two fragments of supraorbital part of the frontal bone and seven human teeth were discovered from the two localities of Yiyuan site – Yiyuan Locality 1 (YYI) and Locality 3 (YYIII, ~56 m south of YYI) during the excavation in a fissure deposit on Qizianshan limestone hill, Yiyuan County, Shandong Province, China (118°09′E, 36°12′N) (Lu et al., 1989; Wu and Poirier, 1995) (Fig. 1). The cranial fragment from YYI was recovered from small fragments of the parietals, frontal, and the occipital bones. Based on a morphological comparison, Lu et al. (1989) suggesting that both Yiyuan cranial fragments and teeth are similar to those of Zhoukoudian, and were attributed to *Homo erectus*. Additionally, a recent study of interproximal grooves on Yiyuan human teeth shows the evidences of tooth-picking behavior, probably as one of the earliest in eastern Asia (Sun et al., 2014).

YYI is a small limestone cave site filled with fluvial deposits, and YYIII nearby is a fissure site with the same kind of deposition as YYI. Five out of seven human teeth were unearthed from YYIII during the excavation in 1980s (Lu et al., 1989). However, due to road construction, the section of YYIII is completely covered by modern sediments, only YYI could be observed at the present time. Lu et al. (1989) made a detailed description of each layer of two localities, and pointed out that the stratigraphy of YYI and YYIII could be correlated. Five stratigraphic layers were hence recognized at the two localities (Fig. 2). The human remains and abundant faunal remains were unearthed from layer 3. The association of Bovinae, *Ursus arctos*, *Equus sanmeniensis*, *Sus lydekkeri* and *Megaloceros pachyosteus* in the Yiyuan hominin layer was also recognized at

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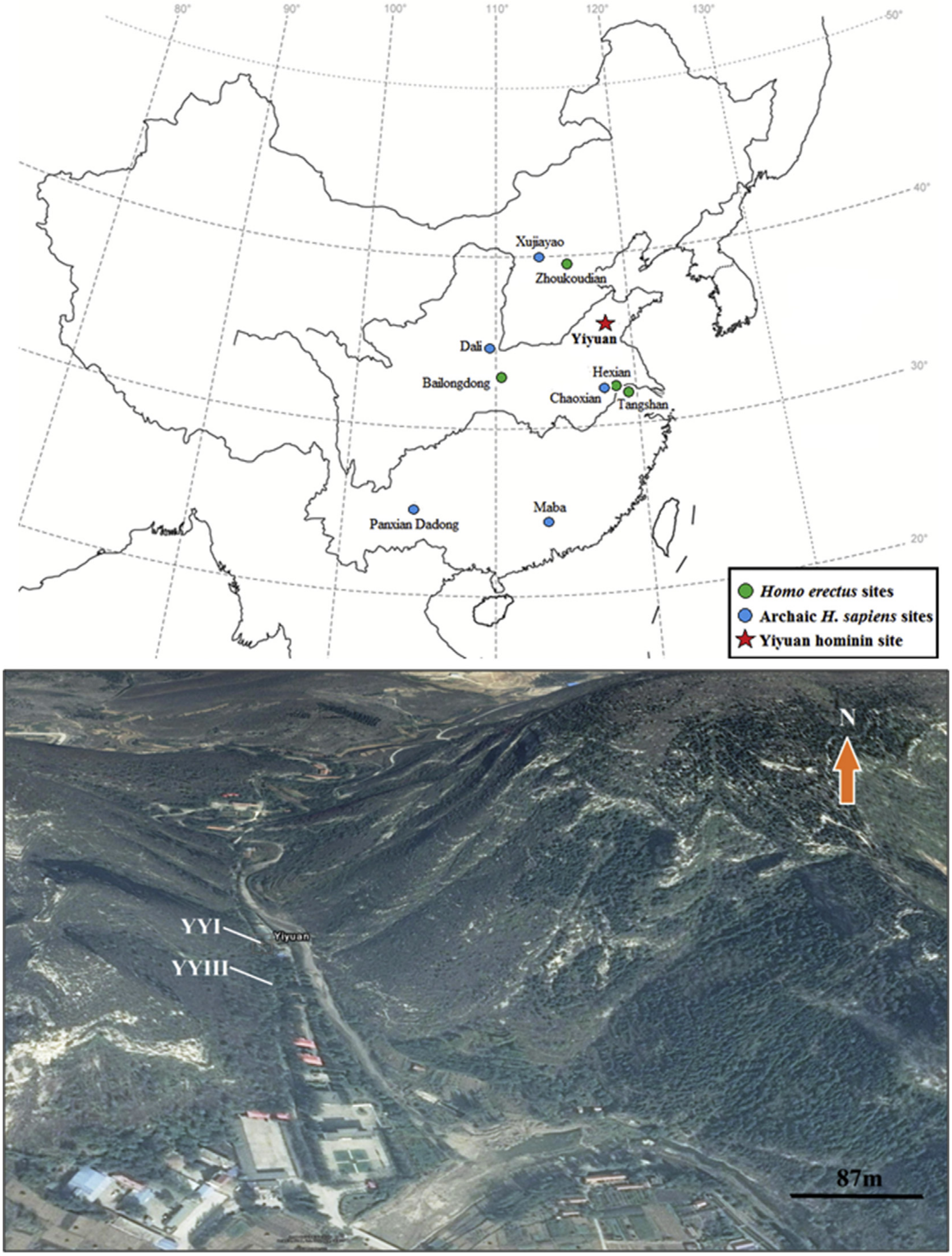

Fig. 1. Location of Yiyuan site and other hominin sites mentioned in this study (Bird's-eye view of Yiyuan Locality 1 and Locality 3 came from Google Earth Pro).

Hexian and in layers 4–5 of Zhoukoudian locality 1, indicating a Middle Pleistocene age (Lu et al., 1989).

Despite the significance of the Yiyuan site, few chronological studies were carried out, and no numerical dating results were present since the discovery of the site. ESR/U-series method is a useful tool for dating fossil teeth from early human sites, as it could reconstruct the uranium migration history in the fossil teeth. Grün et al. (1988) proposed to couple ESR and U-series data to describe the U-uptake history in the different dental tissues (US-ESR model). The US-ESR model allows the calculation of an U-uptake parameter (p-value) for each dental tissue, but it is restricted to $p \geq -1$, i.e. U-loss could not be modeled (Grün, 2009). In the case of U-leaching, an Accelerating Uptake (AU) model (Shao et al., 2012) was recently introduced which describes the U-uptake into dental tissue as an accelerating process. This model is able to reconstruct a process combining incorporation followed by

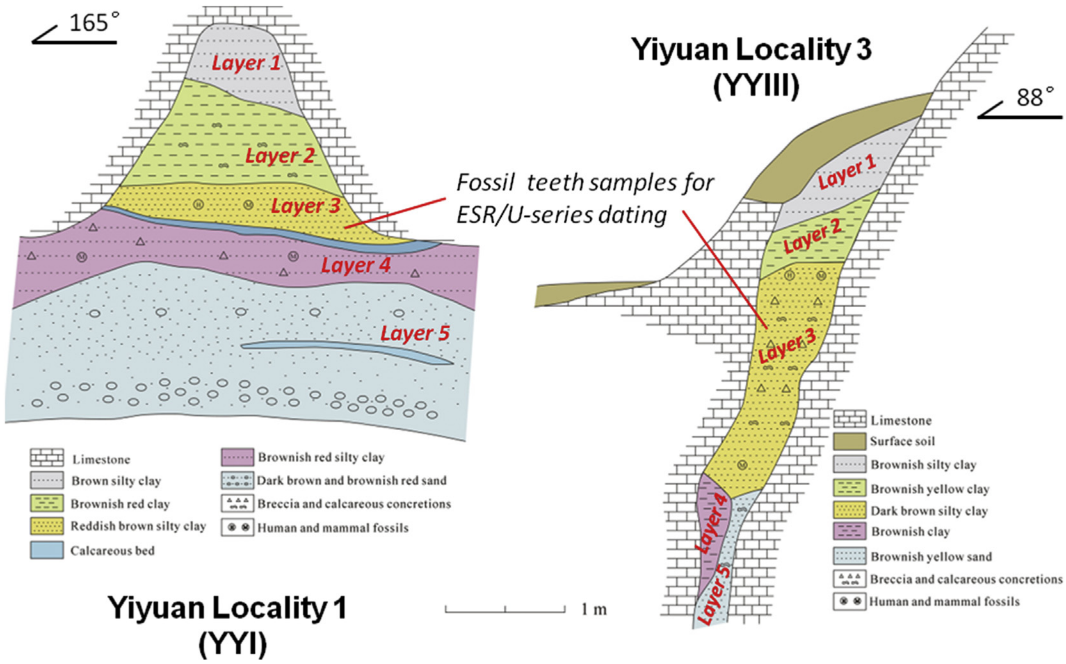

Fig. 2. Stratigraphy of Yiyuan Locality 1 (YYI) and Locality 3 (YYIII) after Lu et al. (1989).

leaching with two parameters, initial uptake rate, and acceleration of this uptake rate. It introduces also another uptake parameter, n-value, which corresponds to the ratio of acceleration of ^{238}U uptake rate and its initial uptake rate. Dating study of the Mauer site in Germany shows the AU model age obtained from the fossil teeth with U-leaching evidence is consistent with other independent age control (Shao et al., 2012).

In order to apply US-ESR or AU model for dating fossil samples, U-series analysis must be coupled with ESR measurement. The coupled ESR/U-series dating method was applied to date fossil teeth in a number of Early and Middle Pleistocene sites in Europe and Africa (e.g. Falguères et al., 1999, 2010; Curnoe et al., 2001; Bahain et al., 2007; Duval et al., 2012), as well as several hominid sites in China, including Hexian (Grün et al., 1998), Panxian Dadong (Jones et al., 2004), Dali (Yin et al., 2011), Chuifeng (Shao et al., 2014) and Longgupo (Han et al., in press). In the present study, nine fossil teeth from two localities (YYI and YYIII) of Yiyuan hominin site were analyzed by the ESR and U-series methods for establishing the chronology of the Yiyuan site.

2. Method and materials

The fossil samples in this study were all collected during the early excavation of Yiyuan localities (YYI and YYIII) in 1980s. The fossil samples of YYI and YYIII were collected from the reddish-brown sandy clay layer 3 where the hominin fossils were unearthed (Fig. 2, after Lu et al., 1989). Although the precise position of the samples were not recorded in the early excavation, as the hominin remains were recovered from the top of layer 3, the fossil age should be contemporary or slightly older than the hominin fossils.

In this study, fossil teeth unearthed from hominin layers of YYI (n = 3) and YYIII (n = 6) were analyzed with the ESR and U-series methods (Table 1). All of the tooth samples except YYI-3 (Suidae) are herbivore molars. Samples YYIII-5a and YYIII-5b are two cervid molars derived from the same mandible fragment. The samples are well preserved in general and have both enamel and dentine tissues. These dental tissues initially were separated mechanically with dentist tools. The enamel was then ground and sieved into fine powder (100–200 μm) and split into 10 or 15 aliquots for gamma irradiation from 32 to 12,500 Gy in the National Key Laboratory of Metrology and Calibration Technology, China Institute of Atomic Energy. The uranium concentration and isotopic ratios of each dental tissue were analyzed by the U-series method with a Nu-Plasma MC-ICP-MS in the Radiogenic Isotope Facility, the University of Queensland, and combined with HpGe gamma spectrometer measurements in MNHN, Paris in order to evaluate possible radon loss from the dental tissues (Bahain et al., 1992).

Table 1
Basic information and U-series measured data of Yiyuan fossil teeth.

Sample no.	Species	Tissue	Thickness enamel (μm)	Removed enamel (S1/S2[a]) (μm)	U-content (ppm)	^{234}U/^{238}U	^{230}Th/^{234}U	^{222}Rn/^{230}Th[a]
YYI-2	Bovid	Enamel	1347	86/70	6.064 ± 0.004	1.8548 ± 0.0019	1.2421 ± 0.0030	0.627
		Dentine			16.808 ± 0.014	1.8904 ± 0.0018	1.5674 ± 0.0045	0.332
YYI-3	*Sus*	Enamel	2052	110/97	2.398 ± 0.005	1.5192 ± 0.0036	1.1430 ± 0.0088	0.560
		Dentine			27.352 ± 0.336	1.8686 ± 0.0060	1.6945 ± 0.0412	0.233
YYI-4	Cervid	Enamel	1003	45/45	1.260 ± 0.001	1.6320 ± 0.0022	1.2071 ± 0.0031	0.934
		Dentine			18.489 ± 0.011	1.8983 ± 0.0015	1.5941 ± 0.0035	0.391
YYIII-1	Equid	Enamel	1239	66/47	1.593 ± 0.001	1.6573 ± 0.0018	1.1199 ± 0.0036	0.707

(continued on next page)

Table 1 (*continued*)

Sample no.	Species	Tissue	Thickness enamel (μm)	Removed enamel (S1/S2[a]) (μm)	U-content (ppm)	$^{234}U/^{238}U$	$^{230}Th/^{234}U$	$^{222}Rn/^{230}Th$[a]
		Dentine			34.231 ± 0.017	1.8022 ± 0.0010	1.3134 ± 0.0028	0.318
YYIII-2	Cervid	Enamel	1102	59/57	0.975 ± 0.001	1.7170 ± 0.0025	1.1733 ± 0.0028	0.707
		Dentine			19.868 ± 0.016	2.0574 ± 0.0026	1.7967 ± 0.0040	0.318
YYIII-3	Cervid	Enamel	1169	87/52	1.493 ± 0.001	1.7484 ± 0.0016	1.1019 ± 0.0027	0.707
		Dentine			16.896 ± 0.009	1.9801 ± 0.0018	1.5674 ± 0.0042	0.318
YYIII-4	Cervid	Enamel	920	160/82	2.032 ± 0.001	1.6839 ± 0.0016	1.2001 ± 0.0028	0.707
		Dentine			30.075 ± 0.015	1.8485 ± 0.0015	1.4425 ± 0.0032	0.318
YYIII-5a	Cervid	Enamel	951	85/90	1.867 ± 0.001	1.6259 ± 0.0020	1.2118 ± 0.0025	0.707
		Dentine			38.471 ± 0.021	1.8666 ± 0.0020	1.4890 ± 0.0049	0.318
YYIII-5b	Cervid	Enamel	988	55/52	2.308 ± 0.001	1.5351 ± 0.0013	1.1494 ± 0.0027	0.707
		Dentine			38.365 ± 0.025	1.8660 ± 0.0022	1.4719 ± 0.0046	0.318

[a] **Note:** S1, S2 represent the dentine and sediment sides attached to enamel respectively; Rn loss was calculated with combined U-series data measured by MC-ICP-MS and gamma spectrometers, and the mean values of Rn loss of both enamel and dentine tissues of YYI samples were applied on YYIII.

The ESR intensity of enamel samples was measured by peak-to-peak amplitudes (T1-B2) of the ESR signal (Grün, 2000). Equivalent doses (D_E) were determined by exponential plus linear (EPL) fitting function using Origin 8.5 software, with weighting by $1/I^2$. Two other fitting functions single saturation exponential (SSE) and double saturation exponential (DSE) were also tested and compared with EPL. The SSE function was traditionally used for D_E determination in ESR dating. However, this function will cause systematic D_E overestimation for fossil teeth samples (Duval et al., 2009). Although DSE function fits the dose points better than the SSE function, and could described the different dose responses of two CO_2^- radicals in tooth enamel (Grün et al., 2008), we observed meaningless fitting parameters of DSE function in D_E determination (the saturation level is below the natural aliquot measured intensity), and large uncertainty of D_E up to 105%. EPL was simpler than DSE, and gives compatible D_E values with rational fitting parameters in this study. It also has similar adjusted R-square (Adj. R^2) to the DSE function and relatively smaller uncertainty (3%–15%). Therefore, we choose EPL functions for D_E determination of Yiyuan fossil samples.

In situ gamma dose rate of YYI was measured by Ortec DigiDART portable gamma spectrometer with a NaI detector using the threshold method (Mercier and Falguères, 2007). Although the stratigraphic section of YYIII could not be observed, the unearthed position of the tooth samples were recorded from the reddish-brown sandy clay fossil layer 3 as YYI which contains the hominin remains. In order to better constrain the external dose rate, we measured three different places in the fossil layer of YYI, and took the mean value of three points (3.12 ± 0.58 Gy/ka) for external gamma dose rate of both YYI and YYIII. The sediments were collected and analyzed at MNHN by laboratory HpGe gamma spectrometer for external beta dose rate determination, and a sediment water content of 15 ± 5% was assumed for fossil age calculation (Table 2).

Table 2
Dose rate components and ESR/U-series ages (AU model) of Yiyuan teeth samples.

Sample no.	Species	Tissue	D_E (Gy)[a]	Internal dose (α + β) enamel (μGy/a)	β dentine (μGy/a)	β sediment (μGy/a)	Total dose rate (μGy/a)[b]	n-values (w.u.)	AU-ESR age (ka)[c]
YYI-2	Bovid	Enamel	2205.37 ± 104.55	3049 ± 480	170 ± 27	186 ± 10	6525 ± 573	−0.0042 ± 0.00038	338 ± 25
		Dentine						−0.0045 ± 0.00039	
YYI-3	*Sus*	Enamel	1507.81 ± 52.73	872 ± 235	164 ± 44	115 ± 10	4271 ± 393	−0.0039 ± 0.00041	353 ± 30
		Dentine						−0.0044 ± 0.00043	
YYI-4	Cervid	Enamel	1865.59 ± 122.34	672 ± 361	288 ± 155	259 ± 22	4339 ± 502	−0.0031 ± 0.00036	430 ± 41
		Dentine						−0.0034 ± 0.00038	
YYIII-1	Equid	Enamel	1989.56 ± 278.50	682 ± 742	317 ± 345	105 ± 6	4224 ± 876	−0.0026 ± 0.00052	471 ± 72
		Dentine						−0.0029 ± 0.00053	
YYIII-2	Cervid	Enamel	1471.49 ± 99.68	437 ± 321	284 ± 209	115 ± 6	3956 ± 494	−0.0036 ± 0.00048	372 ± 39
		Dentine						−0.0041 ± 0.00051	
YYIII-3	Cervid	Enamel	1665.20 ± 216.00	633 ± 700	188 ± 208	120 ± 7	4061 ± 794	−0.0030 ± 0.00059	410 ± 60
		Dentine						−0.0036 ± 0.00063	
YYIII-4	Cervid	Enamel	1650.33 ± 241.99	942 ± 880	340 ± 318	144 ± 14	4546 ± 986	−0.0038 ± 0.00076	363 ± 58
		Dentine						−0.0041 ± 0.00078	
YYIII-5a	Cervid	Enamel	1846.79 ± 89.74	857 ± 268	502 ± 157	126 ± 12	4605 ± 440	−0.0034 ± 0.00034	401 ± 33
		Dentine						−0.0036 ± 0.00035	
YYIII-5b	Cervid	Enamel	2068.98 ± 227.34	946 ± 641	497 ± 336	129 ± 12	4692 ± 788	−0.0029 ± 0.00047	441 ± 56
		Dentine						−0.0033 ± 0.00049	

Note:
[a] Paleodoses of fossil samples were determined by single saturation exponential plus linear (EPL) fitting function.
[b] *In situ* dose rates' mean value of 3.12 ± 0.58 Gy/ka was taken for external gamma dose rate.
[c] The AU-ESR ages of the samples were calculated using a program based on MATLAB software (Shao et al., 2012) with the following parameters and assumptions: alpha efficiency of 0.13 ± 0.02 (Grün and Katzenberger-Apel, 1994); dose rate conversion factors of Adamiec and Aitken (1998); Monte-Carlo beta attenuation factors (Marsh et al., 2002) based on the thickness of the enamel layer, before and after its preparation; water contents of 3 wt% in the enamel, 7 wt% in the dentine and 15 wt% in sediment were assumed.

3. Results and discussion

The uranium contents and U-series isotopic ratios ($^{234}U/^{238}U$ and $^{230}Th/^{234}U$) of Yiyuan fossil teeth samples were shown in Table 1. The U contents range between 0.97 and 6.06 ppm in enamel samples, and from 16.81 to 38.47 ppm in dentine tissue. The measured $^{234}U/^{238}U$ and $^{230}Th/^{234}U$ ratios of both dental tissues vary from 1.519 to 2.057 and 1.102 to 1.797, respectively, and the beyond equilibrium ratios of $^{230}Th/^{234}U$ (>1.04) indicate uranium leaching. The US-ESR model could not be applied for age calculation, and the AU model was therefore systematically used in this study.

The n values calculated for the Yiyuan samples are all negative (Table 2), as a result of the uranium-series open system which has evolved beyond equilibrium state. The dose rate components calculated with the AU model and their contributions to the total dose rate are shown in Table 2. For Yiyuan samples, the gamma and beta dose rates from the sediments are more than half of the total dose rates, which range from 51% to 82%. The percentages of internal dose rate from enamel are no more than 20% of the total dose rates, except for YYI-02 (47%).

The AU model ages of two samples of three from YYI show good agreement, and give a weighted mean age of 344 ± 19 ka. The six teeth from YYIII are consistent in the error range, and their weighted mean AU model age is 400 ± 19 ka. The main uncertainty of age calculation of YYIII comes from the external dose rate which cannot be reconstructed directly at present due to the construction, and we used the measured values of coeval layer of YYI (layer 3) instead. Although the mean fossil ages of YYIII are ~60 ky older than YYI, the result of the two sites are in agreement with the faunal evidence which indicating a middle period of Middle Pleistocene age (300–600 ka, Dong, 2016), between MIS 11 and 9 (Fig. 3).

Comparing with the human remains from other *Homo erectus* sites in China, Lu et al. (1989) suggested that the Yiyuan cranial fragments are morphologically closer to Zhoukoudian than to Hexian, and the degree of postorbital constriction is intermediate between the Zhoukoudian and Hexian specimens. In this study, the

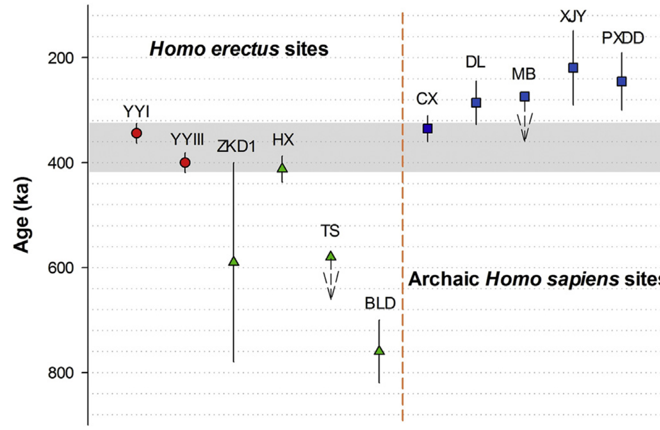

Fig. 4. The combined ESR/U-series ages of Yiyuan site obtained in this study compared with other recently dated hominin sites (*H. erectus* and archaic *H. sapiens*) in China (YYI, YYIII – Yiyuan Locality 1 and 3 (this study)), ZKD1 – Zhoukoudian Locality 1 layers 1–10 (Grün et al., 1997; Shen et al., 2001, 2009), HX – Hexian (Grün et al., 1998), TS – Tangshan (Zhao et al., 2001), BLD – Bailongdong (Liu et al., 2015), CX – Chaoxian (Shen et al., 2010), DL – Dali (Yin et al., 2011; Sun et al., in press), MB – Maba (Shen et al., 2014), XJY – Xujiayao (Tu et al., 2015; Xing et al., 2015), PXDD – Panxian Dadong (Jones et al., 2004; Zhang et al., in press)).

dated two Yiyuan localities were compared with nearby *Homo erectus* sites and some other recently dated archaic *Homo sapiens* (Middle Pleistocene hominins that morphologically and behaviorally fall between *H. erectus* and modern *H. sapiens* (Stringer, 2002; Bae, 2013)) sites in China (Fig. 4). According to the chronological study by multiple dating techniques, the hominin presence at

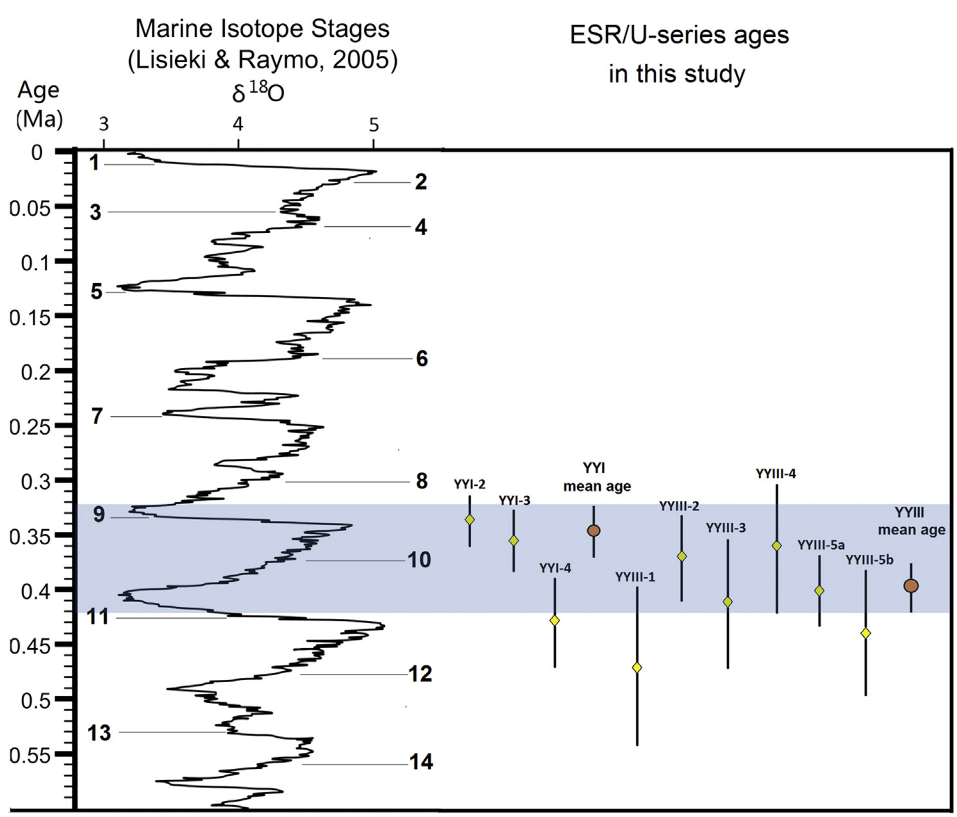

Fig. 3. Coupled ESR/U-series ages of Yiyuan site obtained in this study and the corresponding marine isotopic stage (light blue shadow indicates the time range of weighted mean age with error of fossil teeth samples from Yiyuan Locality 1 (YYI) and Locality 3 (YYIII)) (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.).

Zhoukoudian Locality 1 is constrained to a range of 0.40–0.78 Ma (Grün et al., 1997; Shen et al., 2001, 2009). The combined ESR/U-series ages of both YYI and YYIII obtained in this study fall within the total time range (layers 1–10) of Zhoukoudian Locality 1 (ZKD1), which agrees with the fauna evidences which show similarity of YYI, YYIII and layers 4–5 of ZKD1 (400–600 ka, Shen et al., 2009); The age of Yiyuan sites were also consistent with Hexian *Homo erectus* site nearby (412 ± 25 ka) (Fig. 4). Although the Yiyuan cranial fragments are morphologically closer to ZKD1 specimens than Hexian (Lu et al., 1989), these variations are attributed to regional differentiation of *Homo erectus* evolution in China, as they are all in the same time range (Xing et al., 2014).

Compared with some archaic *Homo sapiens* sites in China, such as Chaoxian (310–360 ka) (Shen et al., 2010), Dali (258–327 ka) (Yin et al., 2011; Wu and Athreya, 2013; Sun et al., in press) and perhaps Maba (no younger than 278 ± 4 ka) (Shen et al., 2014; Xiao et al., 2014), which were dated with updated techniques or protocols, a potential overlap could be observed (Fig. 4). This suggests the possibility of an earlier interface between *H. erectus* and archaic *H. sapiens* in China, mentioned repeatedly based on chronological studies on several Chinese sites (Chen and Zhang, 1991; Shen et al., 2010, 2014). High-resolution U-series dating of speleothem calcites and combined ESR/U-series dating of fossil tooth directly coupled with other sediment and stone artifacts dating methods, such as OSL and $^{26}Al/^{10}Be$ burial dating, will provide more evidence to resolve the question of the coexistence of these two *Homo* species. This dating study also places the Yiyuan hominin as one of the earliest chronological evidence of tooth-picking by hominins in China and East Asia, earlier than the Huanglong Cave evidence (81–101 ka) (Liu et al., 2010; Shen et al., 2013; Sun et al., 2014).

4. Conclusion

Coupled ESR and U-series dating was applied on nine teeth from two localities of Yiyuan *Homo erectus* site (YYI and YYIII) in China. Uranium leaching of all the samples was indicated by U-series analysis and the AU model has been used for fossil age calculation. Nine fossil teeth samples from YYI and YYIII give an age range from ~320 to 420 ka. This places the deposition of hominin layers of Yiyuan site to the time between MIS 11 and 9. The dating places Yiyuan site in the same time range with Zhoukoudian Locality 1 and Hexian *Homo erectus* sites. Compared with other recently dated hominin sites, this dating study of Yiyuan *Homo erectus* site highlights the possibility of coexistence of *Homo erectus* with archaic *H. sapiens* in China.

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