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Combined US-ESR dating of fossil teeth from El Harhoura 2 cave (Morocco): new data about the end of the MSA in Temara region.

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Abstract

The study of the Middle Stone Age (MSA) in North Africa is essential to better understand the human dispersals during the Late Pleistocene. The timing of the transition from the MSA to the Later Stone Age (LSA) in the region is still debated due to the scarcity of sites and the limited chronological constraints available. Among the few existing MIS 5 to MIS 1 archaeological sites in Northwestern Africa, El Harhoura 2 cave (Morocco) has been extensively excavated during the last decade. It has provided MSA and LSA sequences from which human remains have been systematically discovered in association with faunal and lithic assemblages.

The combined US-ESR method has been applied to date five teeth from layer 3 and 4A, which correspond to the most recent MSA occupations at El Harhoura 2 cave. The results suggest the disappearance of the MSA at this site to have occurred around ~ 40 ka, at the end of MIS 3, which is consistent with the palaeoenvironmental interpretation derived from the faunal remains. These new results suggest that the last MSA human occupation are ~15 ka more recent than previously showed by OSL dating. Further investigation is required to fully understand the reason of such discrepancy between the two methods.

1. Introduction

During the last decades, research in Northwestern Africa has greatly improved the chronology of the North Africa Middle Stone Age (Barton *et al.*, 2009, 2015; Dibble *et al.*, 2012; Doerschner *et al.*, 2016; El Hajraoui *et al.*, 2012; Jacobs *et al.*, 2011, 2012; Klasen *et al.*, 2017; Mercier *et al.*, 2007; Richter *et al.*, 2017, 2010; Schwenninger *et al.*, 2010). These dating results have contributed to highlight the importance of this region in the study of the origin and dispersal of anatomically modern humans within and out of Africa.

In North Africa, the end of the Late Pleistocene is marked by a cultural and subsistence shift from the Middle Stone Age (MSA) to the Later Stone Age (LSA). The nature and timing of this transition from MSA to LSA remains poorly known due to the paucity of reliable dating data and a lack of well-stratified sites. This results in a large age uncertainty for this transition, dated to between 60 and 25 ka.

El Harhoura 2 cave (Fig.1), located in Rabat-Temara region (Morocco), is one the few sites in Northwestern Africa with a complete archeological sequence spanning from the MSA and the Neolithic (El Hajraoui *et al.*, 2012). This coastal area is well known for its rich Middle to Late Pleistocene (MIS 5 to MIS 1) human fossil record. The MSA and LSA human occupations are documented in Dar-es-Soltane 1, Dar-es-Soltane 2, El Harhoura 2, El Mnasra and Contrebandiers caves. This area has also delivered most of MSA humans remains (Debénath, 2000; El Hajraoui *et al.*, 2012). However, for some of the caves such as El Mnasra, the stratigraphic interpretation of the deposits is complicated by post-depositional perturbations (Lenoble, 2010). In contrast, the undisturbed archaeo-stratigraphic record observed at El Harhoura 2 makes the site a good candidate to chronologically constrain the disappearance of the MSA in the region.

In the present study, we applied the combined US-ESR dating method on hydroxyapatite from herbivore fossil teeth (Grün *et al.*, 1988) to directly date human occupation at El Harhoura 2. These results will contribute to the refine the end of the MSA of Rabat-Temara sites.

2. Geological, paleoenvironmental and archeological contexts

El Hahroua 2 cave (33°57'08.9''N, 6°55'32.5''W) is located in Rabat-Temara region, (Nespoulet *et al.*, 2008; El Hajraoui *et al.*, 2012). This karstic cave was dug in local calcarenites by the marine erosion of the Plio-Pleistocene coastal ridge, described as a succession of marine and continental deposits (Chahid *et al.*, 2016). Located 300 m from the shoreline and 20.6 m above the present NGM (actual sea level), the site length is estimated to 22 m, the depth around 9 m and the height ~ 8 m. El Harhoura 2 was discovered in 1977 and a preliminary excavation was conducted mainly in the entrance area (El Hajraoui *et al.* 2012). A first test pit of 8 m² was established in 1996, followed by a lateral extension excavated in 2007, which allowed to describe the complete archeological sequence at the entrance of the cave. In 2012, the excavation was further extended to cover a total area of more than 200 m².

The sedimentary cave infilling is homogenous and dominated by sandy-clay matrix (Boudad *et al.*, 2017). Four sedimentary units (Fig.2-A) were identified from bottom to top. Unit I is dominated by grey sandy clays. Unit II is defined by a higher proportion of clays and a lower content of silts. Unit III is described as an intermediate unit between clay and carbonates and the Unit IV described by a lower content of sands.

Archeostratigraphy (Fig.2-A) has been mainly described from the test-pit of the entrance area and subdivided into eleven levels (El Hajraoui *et al.*, 2012). Level 1 (Neolithic), level 2 (LSA), levels 3, 4, 6, 8 (MSA). Other levels (5, 7, 9, 10 and 11) were excavated only on a small surface (<3m²). For a total number of n=687 lithic artefacts founded in the MSA levels, this number is less than n=14 for levels 5, 7, 9, 10 and 11 (El Hajraoui *et al.*, 2012, p.92). In the current state of excavations, these levels are considered as archeologically sterile. Only the levels 3 and 4 are studied in this paper. The MSA industries of these levels are composed by Levallois and micro-Levallois knapping tools, laminar artifacts, side-scrapers and chopping tools. No tanged tools, characteristic of the Aterian, have been recovered, even in extensively excavated areas. Humans burials were

found in the level 1 and 2 and post-cranial isolated bones were found on the levels 3 and 9 (El Hajraoui et al., 2012).

Taphonomic studies on abundant and well-preserved micro-faunal remains confirm that no major disturbance occurred in these accumulations, and demonstrate the integrity of the archeological levels (Stoetzel et al., 2011). Zooarcheological studies (Campmas et al., 2015) show a diversified faunal assemblage (presence of Alcelaphinae, Equidae, Bovinae and Rhinocerotidae). Faunal spectrum is dominated by *Gazella* sp. which represent >70% of the number of identified specimens (NISP) in levels 3 and 4A (and almost 57% in level 8). There is also a presence of carnivores (between 10% and 16% of the NISP for the levels 3 and 4A), which appear to be the main accumulators of large mammal remains in levels 2 to 5 (Campmas et al., 2015). These observations, combined with results from the lithic analysis (Stoetzel et al., 2014), show that human occupations in these levels were short and non-intensive. Considering palaeoenvironmental reconstructions, both large and small vertebrate assemblages from the whole archeological sequence show a succession of relatively humid (levels 3, 4A, 6 and 8) and arid (levels 2, 5 and 7) phases (Stoetzel et al., 2011, 2014).

3. Material and methods

Five fossil teeth (Fig.2-B-C) of *Equidae* (*Equus* sp.) and *Bovidae* (*Gazella* sp.) were selected for dating analyses from the Institut National des Sciences de l'Archéologie et du Patrimoine (INSAP, Rabat) collections (three from level 3, one near the limit between levels 3 and 4A and one from level 4A). These teeth were collected during excavations conducted between 2004 and 2010. Combined US-ESR dating analyses were performed at the Geochronology laboratory of the Museum National d'Histoire Naturelle (MNHN) in Paris.

Fossil teeth were prepared following the protocol described in Wagner et al. (2010). Dental tissues (enamel, dentine, cement) were mechanically separated. Enamel samples were cleaned using a dental drill then ground and sieved to 100-200 μ m. Depending on the amount of enamel powder available, each sample was split into 8, 9 or 10 aliquots (~50 mg each) and irradiated using a ^{60}Co γ -ray source at LABRA (CEA, Saclay, France) in 2014. ESR measurements were carried out at room temperature using EMX X-band Bruker spectrometer and the following acquisition parameters: 5 scans, 1024 points resolution, 1.013 mW microwave power, 9.80 GHz microwave frequency, 100 kHz modulation frequency, 0.1mT modulation amplitude, 20.48 ms conversion time and 5.12 ms time constant. The ESR measurements were repeated 4 times for each aliquot over different days. ESR intensities were extracted using the Bruker WINEPR System software from the asymmetric ESR signal between the T1-B2 (Grün, 1998) and normalized by the weight of each aliquot. Average ESR intensities from the repeated measurements were used for the evaluation of the equivalent dose (D_e) determination.

D_e values (Table 1) were obtained by fitting a single saturated exponential (SSE) function through the experimental data points (Yokoyama et al., 1985; Duval and Grün, 2016). Fitting was performed with Origin Pro8 software, with data weighted by the inverse of the squared ESR intensity (Grün and Brumby, 1994). All ESR dose response curves are

provided in Fig.1 SM. An example of ESR spectrum is displayed in Fig.2 SM. This spectrum shows a non-horizontal baseline which may potentially impact the D_e (Duval and Martín-Francés, 2017). This was evaluated on one sample (EH2-2730) by performing a baseline correction (using a cubic function) for each aliquot measured.

Alpha-ray spectrometry U-series analyses (Table 2) were carried out at the Musée de l'Homme U-series laboratory. Samples (0.3-1 g) were dissolved in HNO_3 (7 N) and spiked with ^{233}U , ^{236}U and ^{229}Th and charged onto anion exchange resin AG 1 $\times$ 8 (100-200 mesh chloride form) in order to separate and purify U and Th sources (Shao et al., 2011). The columns were rinsed with HCl (0.1 N) to collect uranium and purified using a UTEVA resin column (Horwitz *et al.*, 1992) in HNO_3 (7 N). Th was eluted with HCl (8 N). The purified U and Th were then evaporated by mixing TTA/benzene solution and deposited on an iron disk covered by aluminum paper. Alpha counting was run for 8 days.

Dose rate provided on Table 1 was determined *in situ* (Table 3) using a γ -ray portable spectrometer Inspector 1000 Canberra with NaI detector and following the threshold technique (Mercier and Falguères, 2007). Additional high resolution γ -spectroscopy measurements were performed at the Geochronology laboratory (Paris) on raw sediment samples (Fig.2-C, Table SM.1), using a high resolution low-background Ge detector, to determine the external β -dose rate from specific activity of U, Th, and K. Analyses were performed on about 90 g of homogenized sediment samples sealed in a plastic box during at least three weeks to ensure radon equilibrium.

The cosmic dose was evaluated according to Prescott and Hutton (1994). The spatial configuration of the cave in different orientations (North, South, West and East) was taken into account with a 3D model as in Richard et al. (2017).

Moisture content was measured in the laboratory by drying at 40°C for ten days. The water content was estimated from the weight difference sediment before and after drying. Sediments presented low water contents, below 5 %, which are quite similar to values presented by Jacobs et al. (2012). However, we suspect these values may underestimate the long-term water content, as sediment samples were collected in summer (June 2014) in relatively dry conditions (archeological sections exposed to sunlight). Consequently, we assumed instead a higher value of $10 \pm 5\%$ for age calculation (Grün, 1989).

Combined US-ESR ages were calculated using DATA program (Grün, 2009) and the following parameters: α -efficiency of 0.13 ± 0.02 (Grün and Katzenberger-Apel, 1994), a water content of 5 ± 3 wt% in dentine and cement and null in the enamel. β -attenuation factors are from Brennan et al. (1997) and dose rate conversion factors from Guérin et al., (2011). Age results are given at 1σ confidence level. A cement/enamel/dentine configuration was used for the age calculation of sample EH2-2993, whereas we used a sediment/enamel/dentine configuration for the other samples.

4. Results and discussion

Equivalent doses, doses rates, p -values, and US-ESR ages calculated for each sample are given in Table 1. U-series data and enamel thickness are presented in Table 2.

U-series data

Uranium content in the enamel ranges between 0.170 and 0.060 ppm, i.e. close to the detection limits of α -ray spectrometry (see Falguères et al., 2018). U-content in cement and dentine is between 4 and 8 ppm. $^{234}\text{U}/^{238}\text{U}$ ratios are systematically higher than 1 in dentine and cement tissues. In enamel, this ratio is associated with large relative errors (between 10 and 20%), particularly for samples in which U content is very low. P -values for dental tissues are superior than -1 and $^{230}\text{Th}/^{234}\text{U}$ values are lower than unity (between 0.03 and 0.81): they suggest that there is no uranium leaching in the dental tissues.

Radioelements content

Specific activities of ^{238}U , ^{226}Ra , ^{222}Rn and ^{232}Th in the surrounding sediments are presented on Table SM.1. No significant disequilibrium in the U-238 decay chain is observed for EH2-sed 1 and EH2-sed 2 sediments. In contrast, a 17 % loss of Ra and 26 % of Rn-loss observed for EH2-sed 3 sample. The origin of these disequilibria is unknown.

Equivalent doses

D_e values range between 20 and 31 Gy. The ESR spectrum displayed in Fig.SM.2 of the sample EH2-2730 presents a non-horizontal baseline. A sensitivity test performed on sample EH2-2730 shows that baseline correction has, however, a very limited impact on the D_e value ($< 3\%$). Following Duval and Grün (2016), the influence of the maximum irradiation dose ($D_{\max}$) on the D_e estimate was also evaluated by successively removing the last irradiation points (until 220-240 Gy). Results displayed in Fig.SM.4 show a minor impact of $D_{\max}$ in the present data set, as the difference between each resulting D_e does not exceed the error range. Finally, repeated measurements ($n=4$) show relatively limited variability of the individual D_e values derived from each independent measurement ($<8\%$ for a given sample; Table SM.3). All these evidence consistently illustrate the robustness of the ESR data set collected.

Dose rate

Results show that 3/4 teeth display homogeneous dose rates, except for EH2-2993 sample that exhibits a lower value by about 10%. This difference could be explained by the presence in this tooth of a cement layer, which produced a lower β dose rate compared to that from the surrounding sediment.

Gamma dose rates significantly contribute to the total dose rate by 50-60 % (Table 1, Fig. SM.3), while cosmic dose rates represent 15-20% of the total dose rate. Consequently,

these two parameters significantly impact the age calculation. The correspondence between sediment and *in situ* measurements with tooth samples is specified in table SM.1. The comparison of our data with *in situ* gamma dose rates measured by Jacobs et al. (2012) and those measured with TL-dosimeters by Janati-Idrissi et al., (2012) does not show significant variability (Table 3). For level 3, values overall range from 368.3 ± 18.4 to 310.0 ± 15.5 $\mu\text{Gy/a}$. The present work and Jacobs et al. (2012) obtained mean values of 363.9 ± 18.2 and 346.7 ± 17.3 $\mu\text{Gy/a}$, respectively, which representing a variability $< 5\%$. Similar observations can be made for level 4A: values range from 348.0 ± 17.4 to 385.4 ± 19.3 $\mu\text{Gy/a}$, with a variability of about 10% (Table 3). As these mean *in situ* gamma values are very close to the individual *in situ* measurements, the latter were chosen to calculate ages. To evaluate the potential impact of the moisture content on the calculated age, simulations were performed by taking into account different moisture content ($3 \pm 1\%$, $5 \pm 1\%$, $8 \pm 3\%$, $10 \pm 5\%$). Some of water content, as $3 \pm 1\%$, $5 \pm 1\%$, $6 \pm 2\%$, $7 \pm 2\%$ and $9 \pm 2\%$ have been used by Jacobs et al. (2012). Results are summarized on Fig.SM.5 and show a difference of $< 1\%$. Consequently, whatever the water content used (between 3 and 10 %), the moisture content has virtually no impact on the calculated ages.

Combined US-ESR ages

Combined US-ESR range from 39 to 48 ka for the level 3 (with a mean age of 43.7 ± 4.5 ka) and from 46 to 49 ka for the level 4A.

EU-ESR ages, considered as minimum ages (Table SM.2), were also calculated using DATA software for comparison. EU-ESR results are very close to US-ESR ages, with differences ranging between 3 and 6 ka (between 7-15% of variability). This is due to the fact that uranium content in dental tissue from El Harhoura 2 samples generated low dose rate from teeth (accounting for 1-15 % of the total dose rate, Fig. 2 SM). Consequently, U-uptake modelling has a negligible impact on the calculated ages, as often expected for Late Pleistocene tooth samples (Richard, 2015). Combined US-ESR age results appear to be overall stratigraphically consistent.

Comparison with previous results

Our ages are in agreement with previous combined US-ESR results obtained by Janati-Idrissi et al. (2012) for the level 4A but they are younger than previous OSL dates. Only two of OSL ages overlap with the combined US-ESR ages for the level 3 (Fig.3). Indeed, OSL ages locating the age of the latest MSA human occupations around 57 ± 4 ka as a mean age (Jacobs *et al.*, 2012) and the age of the level 4A at 74 ± 4 ka.

Regarding these data, the difference observed cannot be explained by the gamma dose rates, which are quite similar for ESR and OSL samples. Considering the equivalent doses, many remarks can be made. First of all, the D_e values determined using SG-SAR protocol by Jacobs et al. (2012) lead to the identification of 2-3 quartz grain populations using Finite Mixture Model (FMM). The presence of different components could be explained

either as mixing grains from underlying/overlying levels even if “mixing mechanism is not clear”, or “by the effect of variations in the beta dose rate to individual grains” (Jacobs *et al.*, 2012, p.385-386). It is difficult to understand the meaning of these different population grains in term of geology. In fact, the sedimentary processes in karstic coastal caves of this area are not well understood in terms of chronology and origin, due to the impossibility to differentiate quartz from different origins (aeolian, marine, peeling of the cave)(Niftah *et al.*, 2005). The final OSL D_e chosen for age calculation correspond to the quartz grain population statistically well represented (for level 3, between 63.7 ± 4.7 to $48.5 \pm 5\%$ of representation). Consequently, the resulting OSL age estimates are about 20% higher than our combined US-ESR ages. Using instead the D_e from the less representative quartz grain population (between 5.0 ± 2.7 to $19.2 \pm 4.2\%$ of representation) would allow to recalculate OSL ages between ~ 19 ka and 38 ka for level 3. For the level 4A, D_e from the less representative quartz grain population ($8.7 \pm 3.7\%$ of representation) provides an OSL age of about 42 ka. This result would be closer to the combined US-ESR ages.

The discrepancy of $\sim 20\%$ between previous OSL ages (Jacobs *et al.*, 2012) and combined US-ESR ages could also be explained by an underestimation of the D_e values obtained for the fossil teeth, due to the presence of unstable radicals in the hydroxyapatite as observed in Joannes-Boyau and Grün (2011) and Richter *et al.* (2017) on enamel fragment. However, ESR dose estimate were carried out in the present study on enamel powder, which results in randomly spatial distribution of the enamel crystals that renders difficult such a comparison. The impact on these unstable components remains nevertheless unclear and needs to be further investigated. It is indeed sample dependent, and some teeth are simply not affected by this issue (e.g. Dirks *et al.*, 2017).

Combined US-ESR ages presented in this work suggest that the last MSA occupation in Rabat-Temara is dated to around 40 ka. Other Northwestern African sites display similar chronologies for late MSA occupations: ~ 29 ka using radiocarbon method in Taforalt (Barton *et al.*, 2015) and 30.9 ± 2.5 ka with OSL (Weisrock *et al.*, 2008) for the end of MSA without tanged tools at Wadi Noun.

In contrary, others sequences in the Rabat-Temara region show different chronologies: for example, the latest MSA in Contrebandiers cave is older than 90 ka by OSL and 70 ka by TL (Dibble *et al.*, 2012). However, erosion surface and the disruption in the deposits of the Contrebandiers cave (Aldeias *et al.*, 2014) limit the discussion. Nevertheless, these combined US-ESR ages of El Harhoura 2 cave match with previous TL ages obtained for the end of MSA in El Harhoura 1 cave placed between 32.15 ± 4.8 ka and 41.16 ± 3.5 ka (Debénath *et al.*, 1986).

5. Conclusion

The numerical ages obtained in the present study correlate the latest MSA human occupations at El Harhoura 2 to the end of the MIS 3 (Fig.3). This attribution is in good agreement with paleoenvironmental data derived from the study of micro fauna (Stoetzel *et al.*, 2011), which position these levels in humid phase and associated to a tree-filled meadow environment.

Combined US-ESR dating of fossil teeth provides a direct chronological constraint on the archaeological levels at El Harhoura 2 and suggest that the latest MSA human

occupation may have occurred at least 15 ka after the OSL ages previously estimated by Jacobs et al. (2012). The origin of the discrepancy between the results from the two methods should however be further investigated in the future.

The direct dating of the last MSA human occupation at El Harhoura 2 cave provides an important contribution to a better understanding of the timing of Late Pleistocene human dispersals in the coastal environments of Northwestern Africa. Future investigations at Rabat-Temara sites should focus on both the revision of old chronological data and new dating application studies of well-identified and well-contextualized archeological material in order to improve the existing chronological framework of the area.

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Figure captions

Figure 1: Location of El Harhoura 2 cave in Rabat-Temara region, Northwestern Africa (Map modified from El Hajraoui *et al.*, 2012).

Figure 2: Synthetic stratigraphy (A) of the El Harhoura 2 site (modified from El Hajraoui *et al.*, 2012), including the location of the relative area where the sediments and tooth samples come from (B), their depth distribution projected on the North-South profile (C). Photo: R. Nespoulet and C. Falguères.

Figure 3: Synthesis of the US-ESR and OSL ages obtained for the level 3 and 4A opposite to the $\delta^{18}\text{O}$ data from Lisiecki and Raymo (2005).

Table captions

Table 1: Equivalent dose (D_e), Dose rate and combined US-ESR age estimation presented at 1 σ confidence level. Key: e= enamel; d=dentine; c=cement. The dose rate components presented are: internal dose rate ($\alpha + \beta$ of the enamel), β_1 (beta contribution from the dentine), β_2 (beta contribution from cement or sediment), gamma and cosmic dose rates.

Table 2: U-series data obtained by alpha-spectrometry analyses of El Harhoura 2 tooth samples. Data are presented with 2 σ . Key: D= dentine; C= cement; E = enamel; side 1 =dentine and side 2 =sediment or cement. For enamel in low U-content, isotopic ratios (italic) are associated with large relative errors (between 10 and 20%).

Table 3: *In situ* gamma dosimetry measured with a NaI detector (Jacobs *et al.*, 2012; this work) and TL dosimeters (Janati-Idrissi *et al.*, 2012) in levels 3 and 4A. A mean value was calculated for each level. Error associated to mean value is the mean of error. A 5% error was assumed for each individual measurement. For level 3, 5 % variability was estimated from the two mean values (346.7 ± 17.3 and 363.9 ± 18.2). For level 4A, 10 % variability was estimated from the two values presented (370.0 ± 18.5 and 348.0 ± 17.4); *: initial value given was 438 $\mu\text{Gy/a}$ and the cosmic dose rate of 90 $\mu\text{Gy/a}$ was subtracted from this value.

Supplementary material

Table SM.1: ^{238}U , ^{226}Ra , ^{222}Rn , ^{232}Th and ^{40}K content (dpm/g or %) measured and converted using dose rate factors published in Guérin *et al.* (2011). Sample EH2-sed 3 has been measured but not used on this calculation because of its geographical distance from the teeth samples.

Table SM.2: EU-ESR and combined US-ESR ages comparison for tooth samples dated in this work. Errors are displayed at 1σ .

Table SM.3: D_e values over the repeated ESR measurements ($n=4$). The dispersion of the individual D_e per sample is not significantly important and remains rarely more than $\sim 8\%$ ($< 3\text{ Gy}$). RSD: relative standard deviation. Data are associated with 1σ .

Figure SM.1: ESR dose response curve for samples dated in this work computing by Origin Pro 8 software, using SSE function (Yokoyama *et al.*, 1985; Duval and Grün, 2016). All D_e presented range between 20 and 31 Gy.

Figure SM.2: ESR spectra measured in sample EH2-2730.

Figure SM.3: proportion of the different dose rate components. Key: I_{internal} = dose rate α + β contribution from the enamel, β_1 = beta contribution from the dentine, β_2 = beta contribution from cement or sediment.

Figure SM.4: Evolution of D_e as a function of D_{max} for all samples fitted with a SSE function. 1σ errors are displayed. The D_e value used for the US-ESR calculation is shown with a dash line.

Figure SM.5: recalculated combined US-ESR ages in taking account different water content of $3 \pm 1\%$, $5 \pm 1\%$, $8 \pm 3\%$ and $10 \pm 5\%$.

															Initial enamel		Enamel		Enamel		
Sample	Sample Level	Layer ±		Z (cm)	Tissue	U-content (ppm)				± Dose rate (²³⁴ U/ ²³⁸ U) (μGy/a)				± ²³⁰ Th/ ²³⁴ U		± US-ESR age ±	Thickness ±	removed side 1	removed side 2		
																	(μm)	(μm)	(μm)		
																		e	d	c	
EH2-2993	3	19.95	0.8	7	2	16	4	11	2	378	27	104	10	516	27		39	3	1.27 ± 0.33	0.58 ± 0.39	1.27 ± 0.33
EH2-3986	3	27.25	3.8	11	5	50	22	91	8	362	36	104	10	618	43		44	7	-0.64 ± 0.1	-0.43 ± 0.21	-
EH2-2730	3	28.83	0.8	0	0	26	6	108	10	368	39	104	10	606	41		48	4	19.56 ± 2.02	0.72 ± 0.38	-
EH2-10221	4A	30.19	1.7	17	14	30	6	90	9	389	22	93	10	619	31		49	5	-0.87 ± 0.09	0 ± 0.20	-
EH2-10984	4A	29.13	1.1	12	5	50	7	103	11	372	27	93	10	630	33		46	3	-0.74 ± 0.15	-0.22 ± 0.10	-

Table 1: Equivalent dose (D_e), Dose rate and combined US-ESR age estimation presented at 1 σ confidence level. Key: e= enamel; d=dentine; c=cement. The dose rate components presented are: internal dose rate ($\alpha + \beta$ of the enamel), β 1 (beta contribution from the dentine), β 2 (beta contribution from cement or sediment), gamma and cosmic dose rates.

<i>EH2-2993</i>	3	-415	D	4.72	0.17	1.05	0.04	0.13	0.01	830 ± 83	39 ± 4	57 ± 6
			C	4.78	0.22	1.14	0.06	0.03	0.03			
			E	0.17	0.02	<i>0.94</i>	<i>0.14</i>	<i>0.09</i>	<i>0.09</i>			
<i>EH2-3986</i>	3	-426	D	7.90	0.47	1.05	0.06	0.29	0.06	720 ± 72	43 ± 4	94 ± 9
			E	0.06	0.01	<i>0.90</i>	<i>0.21</i>	<i>0.25</i>	<i>0.82</i>			
<i>EH2-2730</i>	3	-437	D	7.44	0.44	1.06	0.06	0.14	0.02	690 ± 69	53 ± 5	40 ± 5
			E	0.07	0.01	1.29	0.26	<i>0.03</i>	<i>0.16</i>			
<i>EH2-10221</i>	4A	-458	D	6.59	0.29	1.05	0.05	0.75	0.17	710 ± 71	92 ± 9	64 ± 6
			E	0.15	0.04	1.05	0.35	0.81	0.32			
<i>EH2-10984</i>	4A	-470	D	7.98	0.31	1.16	0.05	0.18	0.02	700 ± 70	58 ± 6	54 ± 5
			E	0.09	0.02	<i>0.72</i>	<i>0.24</i>	<i>0.67</i>	<i>0.38</i>			

Table 2: U-series data obtained by alpha-spectrometry analyses of El Harhoura 2 tooth samples. Data are presented with 2σ . Key: D= dentine; C= cement; E = enamel; side 1 =dentine and side 2 =sediment or cement. For enamel in low U-content, isotopic ratios (italic) are associated with large relative errors (between 10 and 20%).

Gamma Dose rate ($\mu\text{Gy/a}$)

Layer	This work	Jacobs et al., 2012	Janati-Idrissi et al., 2012	% Variability
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3		±		±	5
	(dos 34)		310.0	15.5	
	368.3	18.4			
			360.0	18.0	
	(dos 35)				
	359.4	18.0			
			370.0	18.5	
	(dos 36)				
	364.0	18.2			
			350.0	17.5	
			350.0	17.5	
			340.0	17.0	
	Mean	363.9	18.2	346.7	17.3
4A	(dos 33)		370.0	18.5	10
	385.4	19.3		348.0*	
	Mean		367.8	18.8	

Table 3: *In situ* gamma dosimetry measured with a NaI detector (Jacobs *et al.*, 2012; this work) and TL dosimeters (Janati-Idrissi et al., 2012) in levels 3 and 4A. A mean value was calculated for each level. Error associated to mean value is the mean of error. A 5% error was assumed for each individual measurement. For level 3, 5 % variability was estimated from the two mean values (346.7 ± 17.3 and 363.9 ± 18.2). For level 4A, 10 % variability was estimated from the two values presented (370.0 ± 18.5 and 348.0 ± 17.4); *: initial value given was $438 \mu\text{Gy/a}$ and the cosmic dose rate of $90 \mu\text{Gy/a}$ was subtracted from this value.

- ESR/U-series ages (this work)
- SG-OSL ages (Jacobs et al., 2012)
- ESR/U-series ages (Janati-Idrissi et al., 2012)

A

El Harhoura 2

Archeostratigraphy

Sedimentology

Neolithic

Later Stone Age

Middle Stone Age

1

2

3

4A

4B

5

6

7

8

9

10

11

I

II

III

IV

Area not
excavated

1 m

Legend

powdered ashy layer

yellowish sandy-clay

brown red sandy-clay

brown grey sandy-clay

brown reddish sandy-clay

brown yellowish sandy-clay

brown red sandy-clay

brown reddish sandy-clay

limestone

manganese

ash

A

El Harhoura 2

Archeostratigraphy

Sedimentology

Neolithic

Later Stone Age

Middle Stone Age

1

2

3

4A

4B

5

6

7

8

9

10

11

I

II

III

IV

Area not
excavated

1 m

Legend

powdered ashy layer

yellowish sandy-clay

brown red sandy-clay

brown grey sandy-clay

brown reddish sandy-clay

brown yellowish sandy-clay

brown red sandy-clay

brown reddish sandy-clay

limestone

manganese

ash

B

C

Repartition of the samples (projection on the North-South profile)

B

C

Repartition of the samples (projection on the North-South profile)