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$^{40}\text{Ar}/^{39}\text{Ar}$ and ESR/U-series data for the La Polledrara di Cecanibbio archaeological site (Lazio, Italy)

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Abstract

La Polledrara di Cecanibbio (20 km from Rome) discovered in 1984, is a famous Middle Pleistocene site of the Italian peninsula (Lazio) mainly thanks to its excellent preservation state and its richness in archaeological and faunal remains. This deposit presents a typical Aurelian faunal assemblage with a large number of “elephants” specimens. A human tooth and scavenging traces on “*elephants*” bones were also pointed out. Palaeontological and archaeological remains are located in deposits characterized by its high percentage of white volcanic material blocks embedded in a fine matrix, laying on a fluvial volcano-sedimentary sequence. This archaeological sequence belongs to the Aurelia Formation, and was correlated to the MIS 10-9. To refine this age assignment we provided a multi-methods investigation including both $^{40}\text{Ar}/^{39}\text{Ar}$ and ESR/U-series methods. $^{40}\text{Ar}/^{39}\text{Ar}$ measurements made on single K-feldspars minerals extracted from white pumices within the archaeological horizon suggest a deposition age of 325 ± 6 ka while the ESR/U-series analyses applied on fossil enamel teeth highlighted a minimum age of 304 ± 25 ka, in agreement with the $^{40}\text{Ar}/^{39}\text{Ar}$ ages. These results allowed us to place with confidence the occupation as well as palaeontological assemblage of La Polledrara di Cecanibbio contemporaneously to the middle part of the MIS 9 period just after the interglacial optimum *s.s.* La Polledrara di Cecanibbio is hence now the most precisely and accurately dated Lower Palaeolithic site among the numerous sites from the Lazio province.

Keywords

$^{40}\text{Ar}/^{39}\text{Ar}$ on single crystal, ESR/U-series, MIS 9, Italy

1. Introduction

La Polledrara di Cecanibbio is one of the numerous Middle Pleistocene sites of the Roman area, Italy. It was discovered in 1984 through a program of territorial surveys led by the “Soprintendenza per i Beni Archeologici di Roma”. After thirty years of excavation campaigns, La Polledrara di Cecanibbio is currently considered as one of the richest deposit

of the Lazio province but also as one of the landmark sites from the Italian Middle-Pleistocene period (Anzidei et al., 2004, Anzidei et al., 2012, Santucci et al., 2016). > 20,000 palaeontological fossils and at least 1100 lithic tools and bones artefacts were identified. It is worth mentioning that La Polledrara di Cecanibbio is characterized by the absence of bifacial tools (Anzidei et al., 2012). Furthermore, several *Palaeoloxodon antiquus* carcasses trapped in muddy sediments demonstrate hominids scavenging traces highlighting the opportunistic behaviours of the Pre-Neanderthal populations. These impressive numbers of well-preserved fossils and tools as well as the testimony of opportunistic behaviours with respect to elephants' skeletons underline the exceptional nature of this deposit within the corpus of Italian's Lower Palaeolithic sites (Anzidei et al., 2012, Santucci et al., 2016). Since 2000, a building covering 900 m² of the site palaeo-surface is accessible to the public and allows the site protection.

Current geological and palaeontological constraints allowed the attribution of La Polledrara di Cecanibbio to the Aurelian formation. This site was estimated by biochronology between 340 and 320 ka, corresponding to marine isotopic stage (MIS) 9 (Anzidei et al., 2012). However, the chronology of La Polledrara must be refined by geochronological data derived from absolute dating technics as without accurate chronological data the comparison of La Polledrara di Cecanibbio with the numerous nearby sites contemporaneous with this time period remains still hazardous (e.g. Villa et al., 2016). In 2012, in the frame of an Italian/French collaboration, we started a large program of geochronology that aims at creating a more robust chronological framework for the corpus of Italian's Lower Palaeolithic sites. We will present in this contribution the detail results obtained for La Polledrara di Cecanibbio using the ⁴⁰Ar/³⁹Ar on single-grains and the ESR/U-series methods on dental tissues; replacing this site in a precise palaeo-climatological context and in the frame of the Roman area's Lower Palaeolithic sites corpus.

2. Geological setting

2.1. Geological context of the Lazio province

La Polledrara di Cecanibbio is located in the Lazio, close to Rome. This region, hosting the delta of the Tiber River since the beginning of the Middle Pleistocene (e.g. Milli, 1997, Marra and Florindo, 2014, Marra et al., 2016), is particularly rich in Middle Pleistocene archaeological sites such as Torre in Pietra, Malagrotta, Castel di Guido or Casal de'Pazzi (e.g. Manzi et al., 1990, Anzidei et al., 1999, Boschian and Saccà, 2010, Villa et al., 2016, Fig. 1). In fact, this particular geological context allows the deposition of lacustrine fluvio-sedimentary successions, in response to the strictly interaction among the glacio-eustatic variations, the tectonic uplift, and the volcanism along the Latium Tyrrhenian margin, so allowing a good correlation with the marine isotopic stages (Milli, 1997, Milli and Palombo, 2005; Marra et al., 2016, Marra et al., 2014). The continued uplift of this region since 800 ka, allows a good exposure of sedimentary successions making this region one of the richest of the Italian peninsula harboring mammalian fauna as well as testimonies of human activities (Marra et al., 2014).



Fig. 1. Geological map showing the location of La Polledrara di Cecanibbio and the neighbouring archaeological sites of Torre in Pietra, Casal de'Pazzi, Castel di Guido and Osteria moricone (Villa et al., 2016, Manzi et al., 1990, Anzidei et al., 1999, Barbattini et al., 1982, Saccà, 2012 and Ceruleo et al., 2015) and with the active volcanic complexes of the Middle Pleistocene (Karner et al., 2001, Sottili et al., 2010, Marra et al., 2011).

These specific archaeological deposits are, according to Milli and Palombo (2005), related to the informal sequence stratigraphic unit known in the literature as Ponte Galeria Sequence (PGS). Oriented northwest-southeast on 30 km along the Tyrrhenian Sea margin, this sequence was subdivided in twelve depositional sequences limited above and below by unconformity surfaces (Milli et al., 2016). La Polledrara di Cecanibbio was attributed in the literature to the informal lithostratigraphic unit Aurelia formation, MIS 10/9 whose limits are equal to the PG6 sequence-stratigraphic unit, mainly composed of fluvial and fluvio-palustrine deposits rich in volcanic material (Milli, 1997, Milli and Palombo, 2005).

The volcanic materials found at La Polledrara and in the major part of the Lazio's successions come from Roman volcanic districts very active between 450 and 300 ka (Karner et al., 2001, Sottili et al., 2010, Marra et al., 2011). La Polledrara di Cecanibbio like the neighbouring sites of Torre in Pietra and Castel di Guido is located in the marginal sector of the Monti Sabatini volcanic district and thus presents both reworked volcanic materials and primary fallouts rich in K-Feldspars allowing the use of the $^{40}\text{Ar}/^{39}\text{Ar}$ radio-isotopic method. Furthermore, Castorina et al. (2015) suggested, on the basis on Sr isotopes analyses, that volcanic materials originated from Monti Sabatini have contributed to the sediments found at La Polledrara di Cecanibbio.

2.2. La Polledrara di Cecanibbio stratigraphy

The site of la Polledrara di Cecanibbio displays a 2 m thick stratigraphic succession composed of fluvial deposits rich in volcanic material (80 m a.s.l) (Fig. 2). The base of the stratigraphy is made of cross-laminated deposits characteristic of a small river channel (oriented W-NW to E-SE Anzidei et al., 2004). A pit made outside the protected area demonstrated that this fluvio-lacustrine deposit overlaid a white volcanic level discovered 3 m below the fossiliferous area (POL 13-01, Fig. 2). This small river channel is currently filled by about 2

m of sediments rich in graded volcanic material, mainly composed by white altered graded blocks of pyroclastic material (Fig. 2). These blocks are very rich in sanidines, clinopyroxenes and apatites. Large size volcanic material blocks are at the base of the layer and the thinnest ones at the top of the level.



Fig. 2. a) Schematic stratigraphy of La Polledrara di Ceganibbio highlighting the sampling positions for the $^{40}\text{Ar}/^{39}\text{Ar}$ and ESR/U-series dating. b) Photo highlighting the white volcanic block where sample 12-03 was collected. c) Photo demonstrating sample 12-01 and the cross-laminated sediments (sample 12-02)

The archaeological layer, characterized by its high composition in volcanic material, is described as fluvial-flood sediments by Anzidei et al. (2012). Fossils and artefacts were deposited following the river stream and piled when the flow decayed enough. In the higher part of the fossiliferous level, devoid of visible large volcanic material blocks, were discovered three *Palaeoloxodon antiquus* skeletons found in anatomical articulation (Santucci et al., 2016). It proves that some animals were probably trapped later when this deposit became swampy (Anzidei et al., 2012) and was not totally consolidated (Anzidei et al., 2012). The exceptional preservation state of the archaeological and palaeontological remains is explained by the preponderance in this deposit of volcanic material rich in fluorine (Anzidei

et al., 2012, Castorina et al., 2015). In fact, the high percentage of fluorine encompassing the bones allows the rapid exchanges of calcium to form fluoro-apatite (Anzidei et al., 2004).

2.3. Palaeontological record

As mentioned above, > 20,000 vertebrate fossils were recovered at La Polledrara (Anzidei et al., 2004, Anzidei et al., 2012). The most represented taxa are *Bos primigenius* and *Palaeoloxodon antiquus*. Some other species like *Stephanorhinus hemitoechus*, *Equus ferus*, *Sus scrofa*, *Cervus elaphus* and *Bubalus murrensis* are also represented. Only few carnivores (*Canis lupus* and *Vulpes vulpes*) and primates (*Macaca sylvanus*) are mentioned. Concerning the micro-mammals species, arviculids and murids were identified. Arviculids species are *Iberomys* cf. *I. breccensis*, *Pliomys* cf. *P. episcopalis* and *Arvicola* sp. The only murid species found is *Apodemus sylvaticus* (Anzidei et al., 2004, Anzidei et al., 2012). The large palaeontological assemblage of La Polledrara di Cecanibbio is associated to the Torre in Pietra faunal unit related to the early “Aurelian mammals ages” (Milli and Palombo, 2005).

2.4. Archaeological record and evidence of human activities

Only one human tooth attributed to “*Homo heidelbergensis*” after the estimated age of the site (Manzi et al., 2011) was found. This tooth is a deciduous second maxillary molar, belonging to a child aged between 5 and 10 years old (Anzidei et al., 2012). > 1100 artefacts were unearthed, 500 pieces were collected mainly at the bottom of the archaeological layer and about 600 flint implements were found around the butchered elephant (Anzidei et al., 2012). Concerning the tools discovered in both base and top of the fossiliferous deposit it is worth mentioning that they were made using flint and, more rarely, siliceous limestone little pebbles. The assemblage includes cores, pebble tools, flakes and retouched tools like denticulates, notches and scrapers. Like at Castel di Guido some tools are made of *Palaeoloxodon antiquus* bones (Anzidei et al., 2012). One particularity of tools assemblages from La Polledrara di Cecanibbio is the lack of handaxes, present in other nearby sites of the Lazio (e.g. Torre in Pietra or Castel di Guido; Fig. 1, Anzidei et al., 2012, page. 181). Another characteristic of La Polledrara is the highlighting of the opportunistic nature of the Pre-Neanderthal populations. In fact, the most well-preserved elephant carcass is associated with a lithic industry that was produced in situ (cores, tools, flakes and working debris). Some of the flint implements associated present wear traces of use on animal tissues, document human activity related to the exploitation of the carcass by scavenging (Anzidei et al., 2012, Santucci et al., 2016). Furthermore, the femurs show fracture morphology referable to human activity, indicating that these elements were exploited. The fracturing mode should be attributed to the shaping of the raw material and not just to simple fracturing for marrow exploitation. Finally, some bones of carcass are missing after the butchering episode because of natural factors, or by human activity (Santucci et al., 2016).

2.5. Palaeoenvironmental data

$^{87}\text{Sr}/^{86}\text{Sr}$ and stable isotopes ($\delta^{13}\text{C}$ and $\delta^{18}\text{O}$) analyses were made by Palombo et al. (2005) on *Palaeoloxodon antiquus* dental enamel (twenty-three samples). These analyses confirmed the proximal origin of the volcanic products and suggested an intermediate diet for these elephants (herbs, leaves, bark) in agreement with a temperate and/or hot climate (i.e. interglacial conditions). New $^{87}\text{Sr}/^{86}\text{Sr}$ analyses performed on the matrix of the archaeological deposit by Castorina et al. (2015) suggested different sources from the Monti Sabatini volcanic complex. However, the fact that they did not investigate the white volcanic blocks

but only the matrix may explain the diversity of sources highlighted by this study for the archaeological level.

3. Material and methods

3.1. $^{40}\text{Ar}/^{39}\text{Ar}$ on sanidines single grains

Three different layers were sampled for $^{40}\text{Ar}/^{39}\text{Ar}$ dating. The first one is the white volcanic layer located 1 m below the deepest archaeological remains (POL 13-01) (Fig. 2). The second sample was taken just under the archaeological layer within the reddish cross-laminated fluvial horizon very rich in volcanic material (POL 12-02). Finally, the last samples were taken within the archaeological layer itself (Anzidei et al., 2012). For this layer (around 1.5 m thick, see Fig. 2) we decided to collect pieces of white volcanic material blocks in two different places (see Fig. 2). A large one (10 cm in diameter) was taken at the base of the archaeological layer (POL 12-03, Fig. 2) whereas smaller ones found 10 cm apart (POL 12-01) were taken closer to the top of the layer (i.e. 3 cm in diameter).

All the $^{40}\text{Ar}/^{39}\text{Ar}$ analyses were performed at the LSCE laboratory, Gif-sur-Yvette (France). Volcanic materials extracted from the archaeological deposit were separately prepared crushed and sieved. Clear and non-altered sanidine crystals ranging from 500 μm up to 1 mm in size were concentrated for each sample and handpicked under a binocular microscope. These sanidines were then leached for 5 min in a 7% HF acid solution in order to remove the possibly attached groundmass and then repeatedly rinsed with distilled water in an ultrasonic bath. At least 30 crystals were then chosen for each sample and separately loaded in aluminium disks. Samples POL 13-01 and POL 12-02 were irradiated for 40 min (IRR 77) in the $\beta 1$ tube of the OSIRIS reactor (CEA Saclay, France) and samples POL 12-03 and POL 12-01 were irradiated for 1 h (IRR 66). After irradiation between 15 and 20 crystals for each sample were individually loaded in a copper sample holder. The sample holder was then put into a double vacuum Cleartran window. Each crystal was then fused using a Synrad CO_2 laser at 10 to 15% of nominal power (c.a. 25 W). The extracted gas was then purified for 10 min by two hot GP 110 and two GP 50 getters (ZrAl). Argon's isotopes (^{36}Ar , ^{37}Ar , ^{38}Ar , ^{39}Ar and ^{40}Ar) were analysed by mass spectrometry using a VG5400 equipped with an electron multiplier Balzers 217 SEV SEN coupled to an ion counter. Neutron fluence J for each sample is calculated using co-irradiated Alder Creek Sanidine (ACs-2) standard with an age of 1.193 Ma (Nomade et al., 2005) and the total decay constant of Steiger and Jäger's (1977). For the irradiation 77, mean J -values computed from standard grains are $J = 0.00024170 \pm 0.00000145$ for POL 13-01 and $J = 0.00037885 \pm 0.00000189$ for POL 12-02 and for irradiation 66; $J = 0.00036470 \pm 0.00000182$ and $0.00038070 \pm 0.00000190$ for POL 12-03; POL 12-01 respectively. Mass discrimination was estimated by analysis of Air pipette throughout the analytical period, and was relative to a $^{40}\text{Ar}/^{36}\text{Ar}$ ratio of 298.56 (Lee et al., 2006). Procedural blank measurements are computed after every two or three unknown samples. For typical 10 min static blank, typical backgrounds are about $2.0\text{--}3.0 \times 10^{-17}$ and 5.0 to 6.0×10^{-19} mol for ^{40}Ar and ^{36}Ar respectively. The precision and accuracy of the mass discrimination correction was monitored by weekly measurements of air argon of various beam sizes. Recent revisions of standards and decay constants suggest values of about $\pm 1\%$ than the one we used (e.g. Kuiper et al., 2008, Renne et al., 2011, Phillips and Matchan, 2013, Rivera et al., 2013, Jicha et al., 2016, Niespolo et al., 2016). However, the difference implied for the final ages obtained is here negligible, well within the full-propagated reported uncertainties.

3.2. ESR/U-series method

Seven bovid teeth (aurochs), recovered directly on the palaeosurface, were analysed both by ESR and U-series in order to estimate their age using ESR/U-series approach by US model (Grün et al., 1988).

ESR measurements were performed on EMX Bruker ESR spectrometer using the following parameters: 10 mW microwave power, 0.1 mT modulation amplitude, room temperature, 10 mT scan range, 4 min scan time and 100 kHz frequency modulation, at least four measurements for each dose on different days. The ESR intensities were extracted from the $g = 2.00018$ enamel ESR signal through peak-to-peak (T1-B2) amplitude measurements according to Grün (2000).

The teeth were prepared following the protocol proposed by Bahain et al. (2012). A part of an external enamel layer was extracted from each tooth; the enamel layer was then cleaned on each side by any contamination by sediment or dentine. After grounding and sieving, the 100–200 μm grain-size fraction was split into 10 aliquots. Nine of them were irradiated using ^{60}Co calibrated source (LNHB, CEA, CEN Saclay, France) at doses ranging from to 260 to 20,000 grays (Gy), then the ESR intensities I of the whole set of aliquots were measured using a Bruker EMX spectrometer. The equivalent doses D_E were then extrapolated from the obtained dose growth curve using an exponential plus linear fitting function (E + L, according with Shao et al., 2015) with a $1/I^2$ weighting using Origin Pro 8.0 software. For La Polledrara samples, these D_E are similar to those obtained with double saturation exponential function (DSE) generally preconized for high D_E values (Duval and Grün, 2016), but with smaller associated uncertainties. U-series analyses were performed on each dental tissue by both gamma and alpha spectrometries (following the chemical protocol of Bischoff et al., 1988) in order to evaluate eventual radium and radon losses (Bahain et al., 1992) and to determine the U-uptake parameters necessary to the dose rate contributions and age calculations (Shao et al., 2015).

The beta dose contributions were corrected from the enamel part destroyed on each side of the enamel layer during the preparation process (according to Brennan et al., 1997) (Table 1).

Samples	Tissue	U content (ppm)	$^{234}\text{U}/^{238}\text{U}$	$^{230}\text{Th}/^{232}\text{Th}$	$^{230}\text{Th}/^{234}\text{U}$	$^{222}\text{Rn}/^{230}\text{Th}$	Initial thicknesses (μm)	Removed thickness internal side (μm)	Removed thickness external side (μm)
Po2012-01	Dentine	342.83 ± 9.12	1.215 ± 0.008	20	0.658 ± 0.030	0.351	1270 ± 159	172 ± 21	32 ± 4
	Enamel	9.89 ± 0.26	1.131 ± 0.021	> 100	0.760 ± 0.034	0.612			
Po2012-02	Dentine	294.18 ± 9.96	1.172 ± 0.033	48	0.617 ± 0.027	0.395	1323 ± 165	116 ± 14	6 ± 1
	Enamel	10.11 ± 0.33	1.148 ± 0.039	13	0.798 ± 0.039	1.000			
Po2012-03	Dentine	283.76 ± 8.81	1.215 ± 0.031	85	0.575 ± 0.025	0.390	1124 ± 140	191 ± 24	86 ± 11
	Enamel	23.52 ± 0.83	1.153 ± 0.044	46	0.629 ± 0.032	0.321			
Po2012-04	Dentine	326.73 ± 8.79	1.209 ± 0.026	> 100	0.646 ± 0.026	0.426	1394 ± 174	43 ± 5	114 ± 14
	Enamel	13.80 ± 0.49	1.387	53	0.704 ± 0.034	0.497			

	mel		± 0.053						
Po2012-05	Dentine	273.00 ± 9.22	1.239 ± 0.036	97	0.633 ± 0.033	0.533	1238 ± 155	109 ± 14	78 ± 10
	Enamel	9.98 ± 0.28	1.185 ± 0.023	88	0.699 ± 0.033	0.557			
Po2012-06	Dentine	340.53 ± 11.61	1.252 ± 0.034	> 100	0.503 ± 0.027	0.385	1339 ± 167	225 ± 28	155 ± 19
	Enamel	8.92 ± 0.25	1.164 ± 0.024	> 100	0.679 ± 0.037	0.650			
Po2012-07	Dentine	396.22 ± 13.70	1.062 ± 0.030	> 100	0.654 ± 0.035	0.279	1376 ± 172	138 ± 17	75 ± 9
	Enamel	5.72 ± 0.24	1.473 ± 0.061	> 100	0.724 ± 0.035	0.780			

Table 1. U-series data (α -spectrometry) and samples thicknesses determined on teeth from La Polledrara di Cekanibbio (Lazio, Italy). Analytical uncertainties are given with $\pm 1\sigma$.

The radioelement contents of 10 sediment samples associated to the archaeological level were determined by gamma spectrometry and in situ gamma dose rate (5 measurements) was also measured using TL Al_2O_3 dosimeters. The cosmic dose rate was estimated using the formula of Prescott and Hutton (1994). The following mean values were then used for any age calculation: $U = 12.63 \pm 0.35$ ppm; $Th = 47.10 \pm 0.61$ ppm; $K = 1.27 \pm 0.03\%$; $D_{\gamma + \text{cosm}} = 2716 \pm 40 \mu\text{Gy/a}$. As the excavation was open since a long time and covered by a structure, it was not possible to directly measure reliable water contents from the sediments. A fixed value of $15 \pm 5\%$ was therefore assumed for the age calculations. Were also used the following parameters: a k-value (α efficiency) of 0.13 ± 0.02 (Grün and Katzenberger-Appel, 1994); water content of 0 wt% in the enamel and 7 wt% in the dentine; conversion contents-doses factors from Adamiec and Aitken (1998). The “USESR” computer program (Shao et al., 2012) was used for the age calculation.

4. Results

4.1. $^{40}\text{Ar}/^{39}\text{Ar}$ on sanidines single grains

Full analytical details for individual crystals are given in the supplementary dataset. In Fig. 3 results are presented for each level as probability diagrams (Deino and Potts, 1990). A homogeneous population is considered relevant when the weighted mean of these crystals has a probability fit ≥ 0.1 . The weighted average ages are calculated using IsoPlot 3.0 (Ludwig, 2001) and given at 2σ (95% of probability) including J uncertainty throughout the text. The full-propagated uncertainties (including the total ^{40}K decay constant) are given between brackets with the statistical characteristics. Individual crystals results are presented in the supplementary dataset in tables S1 to S4.

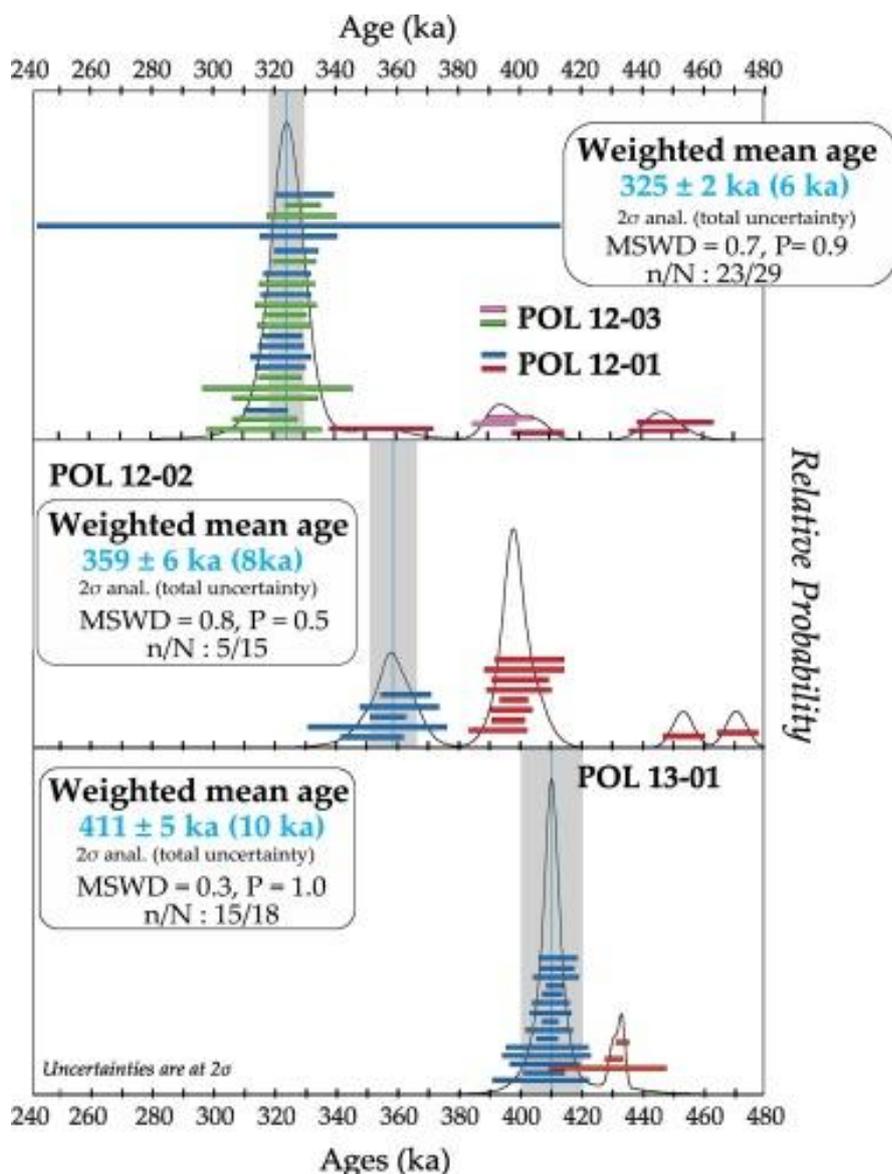


Fig. 3. $^{40}\text{Ar}/^{39}\text{Ar}$ results for La Polledrara di Ceganibbio presented as probability diagrams. MSWD = Mean Square Weighted Deviation, P = Probability fit. For the archaeological layer two different samples were taken (POL 12-01 and POL 12-03) and combined in the same probability diagram.

- POL 13-01

Eighteen sanidines were individually dated. This white volcanic deposit, characterized by a very homogenous granulometry, is a primary volcanic deposit, however it displays a bimodal probability diagram. The volcanic nature of this layer is attested, however as the sample was taken in a borehole, it is not determined if it corresponds to a pyroclastic-flow or to an air-fall deposit. Nevertheless, the homogeneity of the deposit and its lithology suggests that it may correspond to a primary fine ash fall. The main population (juvenile one) of crystals is made of fifteen on the eighteen measured crystals. The three oldest crystals are centred at an age around 433 ka (Fig. 3). The juvenile population of sanidine crystals gives a weighted mean age of 411 ± 5 ka (10 ka, MSWD = 0.3 and P = 1). The $^{40}\text{Ar}/^{36}\text{Ar}$ initial ratio given by the inverse isochron for this population is 295 ± 9 (2σ analytical uncertainties) equivalent within uncertainties to the atmospheric ratio of 298.56.

- POL 12-02

We dated fifteen sanidines extracted from the cross-laminated fluvial deposit (Fig. 3). As expected for reworked volcanic deposit the probability diagram is multimodal. The main population of sanidines is made of eight crystals centred at 400 ka. The youngest mode, composed of five crystals, allows the calculation of a mean age of 359 ± 6 ka (8 ka, MSWD = 0.8 and P = 0.5). Two older crystals are also highlighted in the probability diagram, dating respectively to 454 ± 7 ka and 471 ± 7 ka. Their presence underlines the presence of much older eruptions reworked within the layer. The inverse isochron calculated using these 5 crystals displays a low scattered and $^{40}\text{Ar}/^{36}\text{Ar}$ initial ratio of 249 ± 41 (2σ , analytical uncertainties). Even if this ratio is very imprecise it is still within uncertainties equivalent to the atmospheric ratio.

- POL 12-01 and 12-03

A total of twenty-nine crystals extracted from the white volcanic material blocks found in the archaeological level were measured for POL 12-01 and POL 12-03 samples (fifteen for POL 12-01 and fourteen for POL 12-03, Fig. 3).

We pooled the results obtained for the two samples in a single diagram, as separated results were within uncertainties equivalent (Fig. 3). The pumices displayed similar results with one main juvenile population including 23 of the 29 crystals dated (Fig. 3). The weighted mean age obtained for this juvenile population is 325 ± 2 ka (6 ka, MSWD = 0.7 and P = 0.9). The six xenocrysts are scattered between 350 ka and 450 ka (Fig. 3).

4.2. ESR/U-series method

The ESR/U-series data (calculated using US model), dose rate contribution and ages are displayed Table 2.

Sampl es	Tissue	D_E (Gy)	U uptake parameter p	D_a α intern al ($\mu\text{Gy/a}$)	$D_a \beta$ ($\mu\text{Gy/a}$)	D_a (γ + cosm) ($\mu\text{Gy/a}$)	D_a total ($\mu\text{Gy/a}$)	ESR/U- series ages US model (ka)
Po2012 -01	Dentine	1664.92	-0.3600 ± 0.1509	1779	708	2716	5203	320 \pm 44
	Enamel	± 168.64	0.2803 ± 0.2477	± 868	± 183	± 40	± 888	
Po2012 -02	Dentine	1901.67	-0.5926 ± 0.027	2691	1150	2716	655	290 \pm 28
	Enamel	± 104.66	0.2210 ± 0.1719	± 696	± 212	± 40	± 729	
Po2012 -03	Dentine	1845.44	0.2083 ± 0.2341	2361	1074	2716	6151	300 \pm 29
	Enamel	± 140.61	0.5801 ± 0.2872	± 487	± 52	± 40	± 702	
Po2012 -04	Dentine	1717.57	-0.6826 ± 0.1120	3008	1902	2716	7626	183 \pm 23
	Enamel	± 48.33	-0.5380 ± 0.1346	± 955	± 530	± 40	± 1093	
Po2012 -05	Dentine	1420.99	-0.0762 ± 0.0883	1472	1230	2716	5418	317 \pm 20
	Enamel	± 89.41	0.3044 ± 0.1143	± 314	± 200	± 40	± 375	
Po2012 -06	Dentine	1499.71	-0.1283 ± 0.1365	1388	863	2716	4968	286 \pm 28
	Enamel	± 103.76	1.0761 ± 0.2634	± 528	± 233	± 40	± 578	
Po2012 -07	Dentine	1248.43	-0.6405 ± 0.0680	1566	1928	2716	6211	201 \pm 15
	Enamel	± 34.97	-0.4890 ± 0.0836	± 338	± 360	± 40	± 495	

Table 2. Dose rates and ESR/U-series ages obtained on teeth from La Polledrara di Cecanibbio, Lazio, Italy. Analytical uncertainties are given with $\pm 1\sigma$.

Fig. 4 presents the U-uptake kinetics reconstructed from corresponding U-uptake p parameters for each tooth and associated modelled dose-contributions. The modelled U-incorporation timing indicates for all the analysed tissues a post-burial uptake, ranging from relatively early uptake (P-values comprised between -1.0 and -0.6 to recent uptake ($P > 0$). It leads mechanically to a relatively great impact of the environmental dose rate contribution (gamma plus cosmic) on the age calculation.

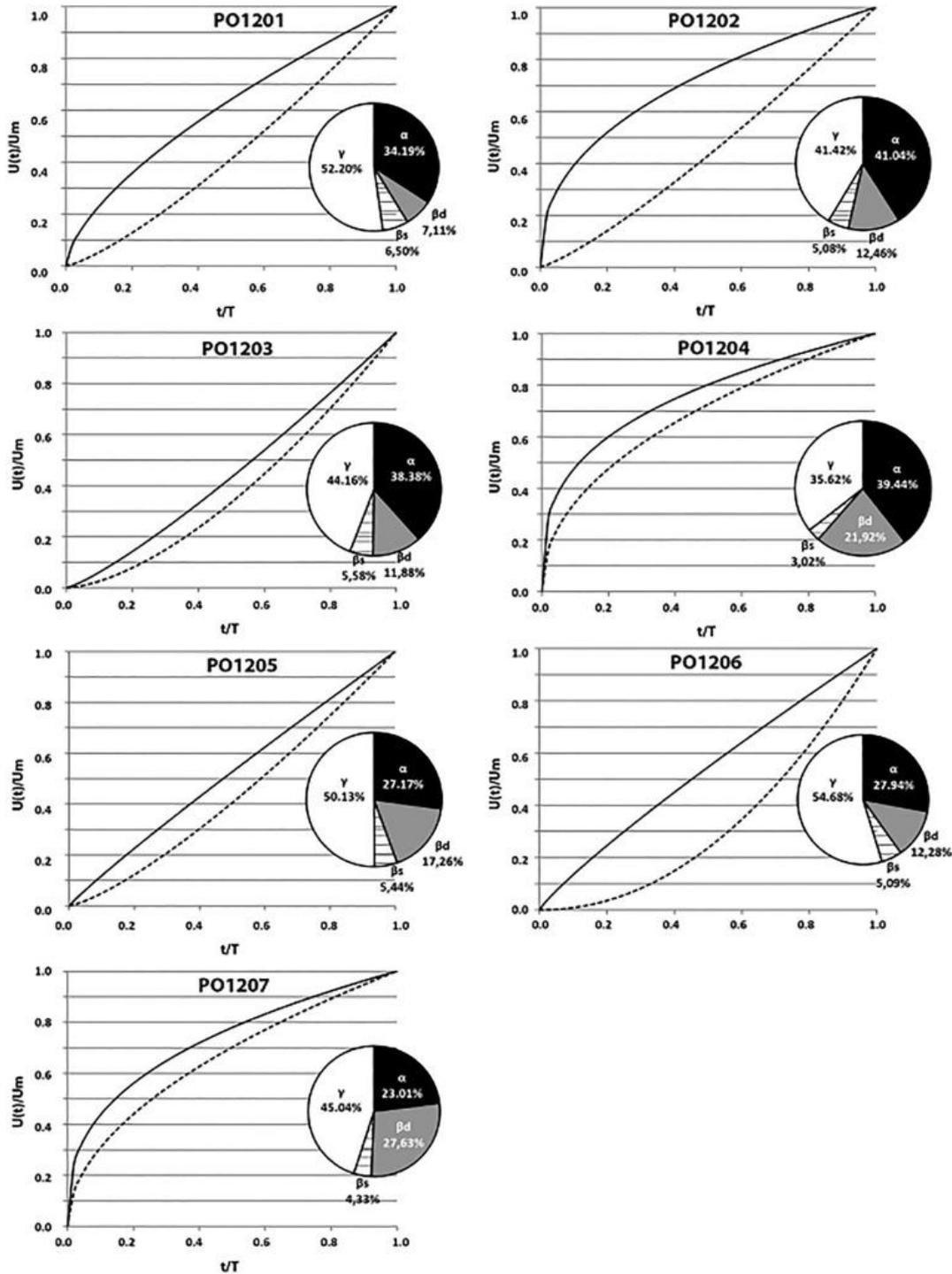


Fig. 4. U-uptake kinetics and contributions to the dose rate modelled for teeth from La Polledrara di Cecanibbio, Lazio, Italy from p parameters determined by US-ESR dating approaches.

In supplementary data are presented information allowing the reader's better comprehension: in Fig. S1, ESR growth curves of the Polledrara tooth enamel samples and corresponding (Exp + Lin) D_E extrapolation for the 7 teeth are shown; In Table. S5, equivalent doses obtained for the Polledrara tooth samples using Exponential + Linear (E + L), Double Saturating Exponential (DSE) and Single Saturating Exponential (SSE) functions respectively are presented; Fig. S2, demonstrates the comparison of the equivalent doses obtained for the Polledrara tooth samples using Exponential + Linear (E + L) and Double Saturating Exponential (DSE) functions respectively. In Table S6 are displayed the radioelement contents of the Polledrara sediments samples determined by low background high purity γ -spectrometry. Table S7 shows the gamma dose rate measured by TL (α -Al₂O₃:C) dosimeters at La Polledrara (by courtesy of H. Valladas, LSCE, France). Finally, Fig. S3 represents the map of La Polledrara site with locations of the sampled teeth, sediments and dosimeters.

Two groups of dates can be observed on the age density plot (Fig. 5). The first one (5 teeth), around 304 ± 25 ka, fits well with $^{40}\text{Ar}/^{39}\text{Ar}$ dates, while the second one, centred at 196 ± 25 ka, seems greatly underestimated. These two teeth correspond to those for which the earliest U-uptake reconstructions were obtained.

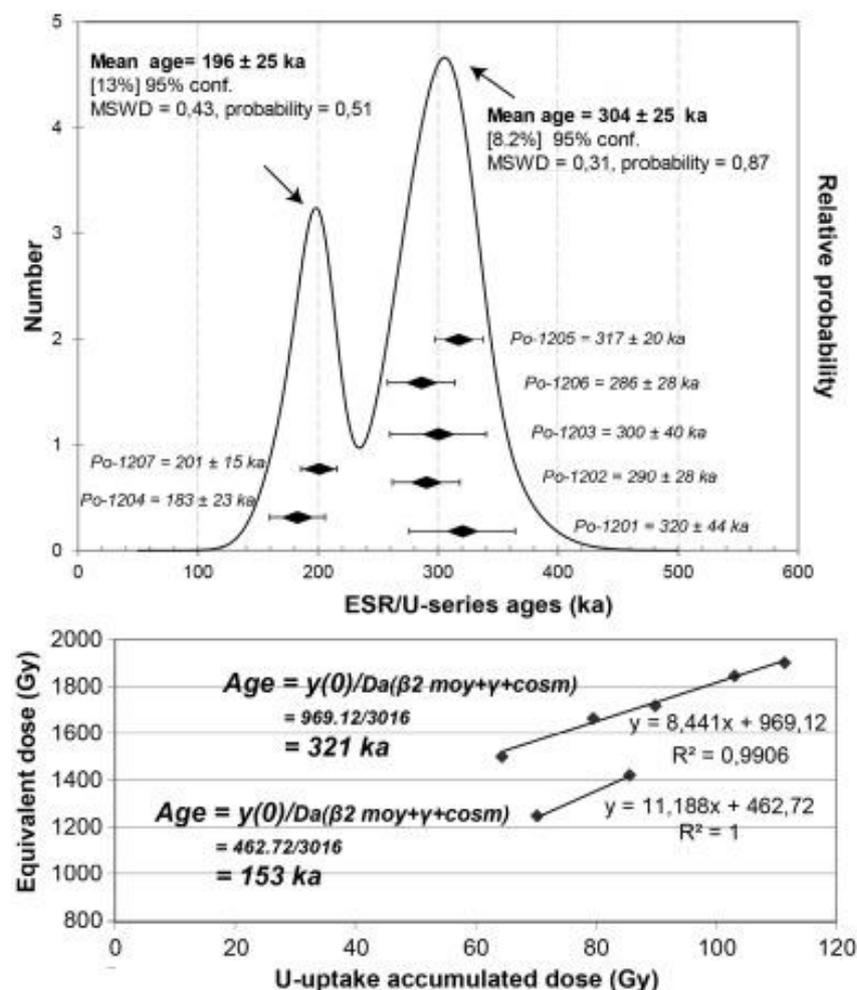


Fig. 5. Age density probability plots (top) and isochron curves (bottom) obtained for teeth from La Polledrara di Cecanibbio, Lazio, Italy. Analytical uncertainties are given with $\pm 1\sigma$.

Isochronous plot showing measured equivalent dose values vs internal doses modelled was therefore build for the La Polledrara teeth (Fig. 5). The teeth corresponding to the oldest group are highly correlated ($R^2 > 0.99$), indicating that the measured environmental dose rate seems geologically representative for them. The intercept of the obtained correlation line equation with the Y-axis provides then the part of the D_E related to this external dose. By dividing this value by the environmental dose rate including the external beta dose rate related to the sediments, an age estimate of ca 320 ka is obtained. This estimate is close to the age of the oldest population and agrees with the $^{40}\text{Ar}/^{39}\text{Ar}$ date of the archaeological layer.

On the other hand, the two other teeth are completely out of the line. It could indicate for them an underestimation of the D_E , which could be related to the presence of unstable components in the ESR spectrum (Joannes-Boyau and Grün, 2011). It could be also due to an inaccurate dosimetric reconstruction, in relation with a local change of the environmental dose rate caused by the relatively late U-uptake modelled for the whole set of samples, as evoked recently for teeth from another Italian Middle Pleistocene site, Isernia la Pineta, by Shao et al. (2012).

5. Discussion

5.1. About the age of the archaeological remains

The $^{40}\text{Ar}/^{39}\text{Ar}$ method has yielded to the measurement of four different samples for the Polledrara belonging to three different stratigraphic levels (Fig. 2). The white volcanic layer that is a primary volcanic deposit, located below the site is dated to 411 ± 5 ka (9 ka) whereas the fluvial unit 1 m above, reworks several volcanic eruptions. The youngest one, that brings chronological information for the layer, is dated to 359 ± 6 ka (8 ka) (see Fig. 2, Fig. 3).

This date leads us to believe that this fluvial layer is younger than 359 ± 6 ka (8 ka) and encourages us to make a correlation with the phase occurred during glacial termination IV at the onset of MIS 9 that led to the Aurelia Formation deposition (Anzidei et al., 2012). The results for POL 12-01 and POL 12-03 samples confirm the geological uniformity of the volcanic material blocks found in the archaeological deposit, suggesting a unique origin, as demonstrated, within uncertainties, by the same age (see Tables S1 to S4 in supplementary dataset) on both largest and thinnest pumices taken in two different locations. We combined the results for these two samples and obtained a pooled age of 325 ± 2 ka (6 ka) (twenty-three on twenty-nine crystals measured). This age should be thus considered, within uncertainties, close to the deposition of the entire layer. As a consequence, the archaeological remains and palaeontological assemblages found at the bottom of this volcano-derived horizon may be considered as well constrained by the age of sanidines extracted from the white blocks. The *Palaeoloxodon antiquus* skeletons trapped in anatomical articulation in the upper part of the deposit are probably slightly younger, but within uncertainties also dated around 325 ± 2 ka (6 ka). In conclusion, $^{40}\text{Ar}/^{39}\text{Ar}$ results refine the previous hypothesis that placed La Polledrara di Ceganibbio contemporaneous with the interglacial period of the MIS 9 (Anzidei et al., 2012). We can now with confidence place the site occupation slightly after the MIS 9 interglacial optimum (see Fig. 6).

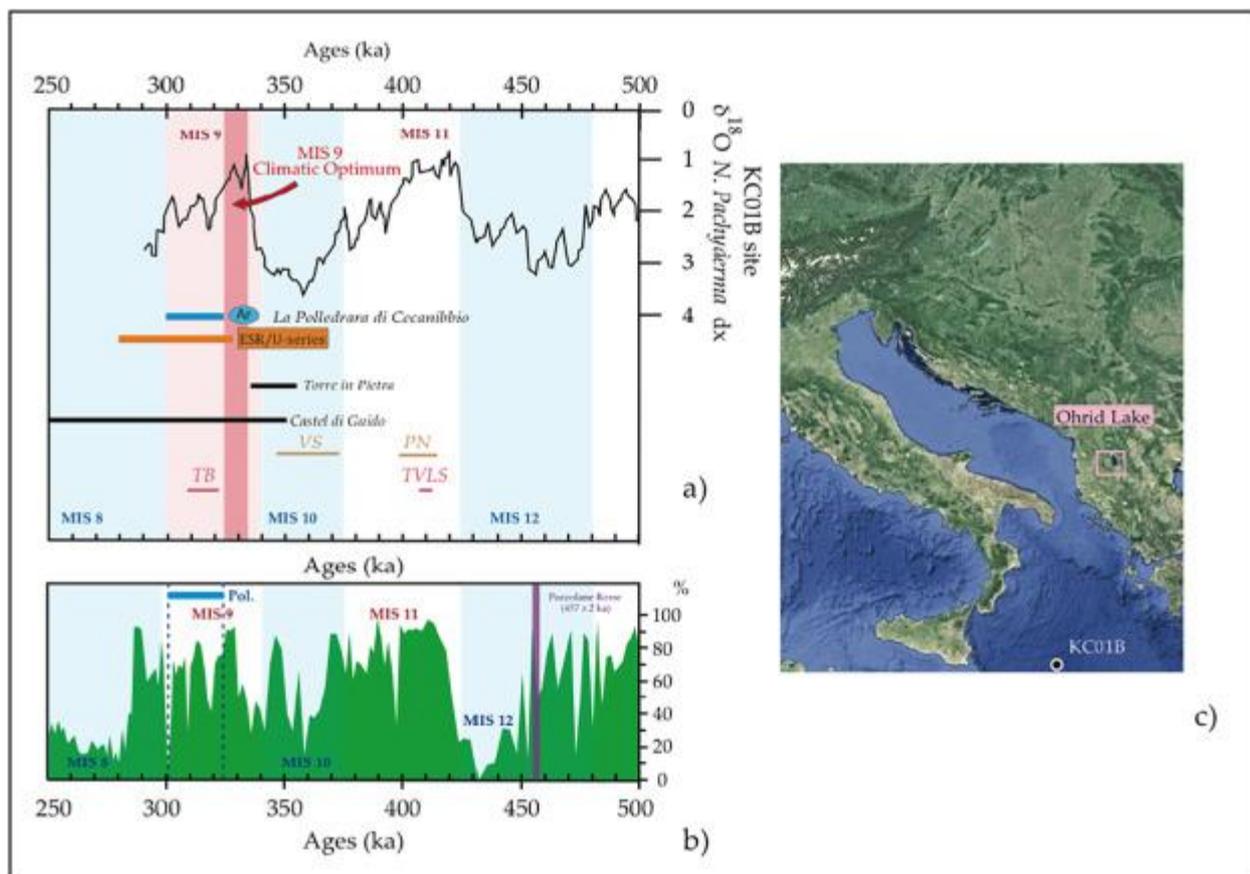


Fig. 6. a) $^{40}\text{Ar}/^{39}\text{Ar}$ and ESR/U-series chronological constraint obtained for La Polledrara di Cecanibbio in correlation with the estimated age at Torre in Pietra for the Acheulian archaeological level and at Castel di Guido (Villa et al., 2016, Michel et al., 2008). Major volcanic eruptions of the Lazio (Colli Albani (CA) and Monti Sabatini volcanic district (SVD)) between 450 and 300 ka are correlated to the archaeological sections. CA: Pozzolane Nere (PN), Villa Senni sequence (VN). SVD: Tufi Stratificati Varicolori di La Storta (TVLS) and Tufo di Bracciano (TB) (Karner et al., 2001, Sottili et al., 2010). These results and eruptions are compared with the $\delta^{18}\text{O}$ climatic curve of KC01b obtained by the analyses of *N. Pachyderma dx* foraminifera (Capotondi et al., 2016). b) Pollen diagram obtained by Sadori et al., 2015 based on the analyses of the Ohrid Lake sequence (Macedonia and Albania), pinus excluded. c) Locations of the KC01b core site and Ohrid Lake.

Results obtained by ESR/U-series analyses on aurochs teeth are in agreement with the $^{40}\text{Ar}/^{39}\text{Ar}$ on single-crystals ages. A mean age of 304 ± 25 ka for five on seven teeth dated was obtained, supported by an isochronous age estimated around 320 ka (see Fig. 5). The two other teeth gave a mean age of 196 ± 25 ka probably due to problems on palaeodose determination or on the palaeodosimetric reconstruction. However, the $^{40}\text{Ar}/^{39}\text{Ar}$ and ESR/U-series results are complementary and reflect a chronological coherence in the geological deposition of the site, refining the age proposed by Anzidei et al., 2012 (i.e. 340–320 ka). We now with confidence propose that the fossiliferous level can be attributed to the end of MIS 9 (probably after the climatic optimum, Fig. 6). It is worth mentioning that the same chronological coherence between the ESR/U-series and $^{40}\text{Ar}/^{39}\text{Ar}$ dating methods was recently demonstrated for the archaeological site of Guado San Nicola (Molise, Pereira et al., 2016) underlying the pertinence of combining ESR/U-series and $^{40}\text{Ar}/^{39}\text{Ar}$ dating methods for precisely and accurately dating Middle Pleistocene sites.

5.2. Volcanic origins of the eruptions recorded

The Lazio during the Middle Pleistocene was subjected to intense and regular volcanism. Several volcanoes, such as the Monti Sabatini, the Colli Albani, the Vico and the Vulcini complexes, were very active between 700 and 250 ka (e.g. Karner et al., 2001, Marra et al., 2011, Marra et al., 2014) and may have led to the deposition of a significant amount of volcanic material in the Tiber delta during the Middle Pleistocene as evidenced by the compositional data of the PGS sequence (Tentori et al., 2016). La Polledrara di Cecanibbio is located in the marginal sector of the influence area of Sabatini volcanic district of the 1800 km² of the Sabatini Volcanic District (SVD) (Fig. 1), making this volcanic complex the most probable source for the volcanic material found in the site like previously suggesting by the presence of large sanidines crystals (Conticelli et al., 1997) and by the ⁸⁷Sr/⁸⁶Sr measurements obtained by Castorina et al. (2015).

The new ⁴⁰Ar/³⁹Ar ages obtained in this study bring new and more precise chronological information. They contribute to refine the origin of the volcanic materials found in La Polledrara by making correlations with well-known eruptions from the Roman province.

5.2.1. About the white volcanic layer origin

Firstly, the age of 411 ± 5 ka (9 ka) for the white volcanic tuff located below the archaeological sequence is comparable chronologically to known eruptions coming both from the Monti Sabatini and Colli Albani volcanic districts. In fact, for example the “*Tufi Stratificati Varicolori di La Storta*” (TVLS), correspond to a corpus of Monti Sabatini volcanoes eruptions dated to 410 ± 2 ka (Karner et al., 2001). This eruption could be the source of the white volcanic layer found 3 m below La Polledrara di Cecanibbio site (411 ± 5 ka (9 ka)).

Another possibility that deserves to be mentioned is the Pozzolane Nere (PN) eruption dated to 407 ± 8 ka (Karner et al., 2001, see Fig. 6). This volcanic event is also recorded 50 km Southeast from the site, at Fontana Ranuccio in the Frosinone province (Lazio)(S. Nomade, personal communication, Pereira et al. *in prep*). However, according to the known dispersion of this eruption it seems less probable that the Pozzolane Nere reached La Polledrara di Cecanibbio area.

It is also interesting to mention the fact the Vico volcano is also very close from the site and one of the most prominent eruptions for this volcano is the Vico α eruption dated to 412 ± 2 ka (Marra et al., 2014). Chronologically the age obtained at La Polledrara for this level is compatible (411 ± 5 ka (9 ka)) with the Vico α age. It is thus important not to overlook this possibility for the volcanic origin of the level.

5.2.2. Origin of the volcanic material contained in the cross-laminated fluvial deposit

The age of 359 ± 6 ka (8 ka) provided for the youngest sanidines population recorded inside the cross-bedded fluvial layer has also (within uncertainties) been identified on the nearby Middle Pleistocene site of Torre in Pietra (Fig. 1, Fig. 6). This site unlike La Polledrara di Cecanibbio presents typical Acheulian assemblages rich in handaxes (recently dated around 354 ± 7 ka, Villa et al., 2016). However, we noticed that the palaeontological assemblage found in Torre in Pietra (level *m*) is almost equivalent to the assemblage highlighted at La Polledrara di Cecanibbio. These two ages are within uncertainty chronologically close to the

“*Villa-Senni eruptive sequence*” (VS) of the Colli Albani, which correspond to several eruptions very close in age (Karner et al., 2001). According to Marra et al. (2011) this eruptive sequence is currently dated between 351 ± 4 ka and 366 ± 7 ka. Future $^{87}\text{Sr}/^{86}\text{Sr}$ analyses on clino-pyroxenes (Cpx) from these two sites as well from the VS sequence are necessary to confirm or not our hypothesis concerning the origin of the volcanic material found in these layers at La Polledrara di Cekanibbio and Torre in Pietra.

5.2.3. Origin of the volcanic material included into the archaeological layer

During the period around 325 ka only few eruptions from the Sabatini volcanic district have been identified (Sottili et al., 2010, Marra et al., 2011, Marra et al., 2014). The main known eruption that could correspond to the age of the white volcanic material (blocks and pieces) embedded into the archaeological layer is the *Tufo di Bracciano* eruption characterized by its richness in white pumices that formed the Bracciano Lake located only 25 km North of the archaeological site (Fig. 1). The volcanic event was dated in 2010 by Sottili et al. and two different ages were proposed: 307 ± 5 ka and 316 ± 6 ka (95% of probability). The nature of this volcanic deposit and the published ages support the correlation with the white blocks embedded in the archaeological layer at La Polledrara.

5.3. About the climatostatigraphic position of La Polledrara di Cekanibbio

In Fig. 6 we reported the age obtained for the archaeological layer interpreted as the age of La Polledrara di Cekanibbio deposit. This age is compared to the $\delta^{18}\text{O}$ variations obtained using planktonic foraminifera (*N. Pachyderma dx*) from the KC01B core (Capotondi et al., 2016). Human presence at La Polledrara occurred contemporaneously to the middle part of the interglacial stage 9 but as highlighted Fig. 6, after what is considered to be the interglacial optimum s.s. We tried to further refine the palaeo-environmental condition during this specific period in Mediterranean realm thanks to the recent pollens diagram provided by Sadori et al. (2015) from the Ohrid Lake (Macedonia/Albania border). This record covers the last fifteen marine isotopic stages and confirms the presence of mainly arboreal taxa in Western Europe after the MIS 9 climatic optimum. This is in agreement with the faunal and microfauna assemblages of La Polledrara di Cekanibbio suggesting rather hot and humid conditions typical of interglacial environment (Anzidei et al., 2004). We would also like to notice that, even if this paleo-environmental record is not located in Italy, it remains the only continuous well-dated continental record in the Mediterranean realm for this time period. In fact, almost a dozen of tephras layers coming from Italian volcanic provinces, like the Pozzolane Rosse eruption (457 ± 2 ka, Fig. 6), are recorded in the Ohrid sequence (Leicher et al., 2016). In Fig. 6, is indicated the recently proposed age for Torre in Pietra acheulian level *m* (Villa et al., 2016) compared with the new age proposed for La Polledrara di Cekanibbio. This comparison suggests that level *m* of Torre in Pietra is slightly older (MIS10/MIS9) than La Polledrara di Cekanibbio (MIS 9). However, according to the Ohrid pollen data (Sadori et al., 2015) and $\delta^{18}\text{O}$ climatic curve (Capotondi et al., 2016) of the KC01B site, it seems that climatic conditions weren't dramatically different between these two sites; probably rather drier and colder at Torre in Pietra (Fig. 6). These climatic conditions are related to the beginning of the MIS 9 interglacial phase and, as said previously, to the early aggradational phase of the Aurelian formation linked to the Mediterranean Sea level rise in response to the improving of the climatic conditions at the end of MIS 10 (Marra et al., 2016). We also compare La Polledrara di Cekanibbio and Torre in Pietra to the proposed age for the neighbouring archaeological deposit of Castel di Guido. This site presents bones tools industries, and a similar paleontological assemblage. Unlike la Polledrara and Torre in Pietra

and due to the large uncertainty on the age proposed for Castel di Guido makes any conclusion premature. In fact, no $^{40}\text{Ar}/^{39}\text{Ar}$ date is currently available for this site and the ESR/U-series ages obtained by Michel et al. (2008) (Fig. 6) on auroch teeth have displayed a large dispersion of ages between 327 and 260 ka. This time range is too large to currently discuss the chronological position of this site within the Aurelia formation.

6. Conclusion

For the first time, direct $^{40}\text{Ar}/^{39}\text{Ar}$ and ESR/U-series constraints were obtained for La Polledrara di Cecanibbio archaeological site. $^{40}\text{Ar}/^{39}\text{Ar}$ measurements on sanidines extracted from volcanic material blocks found within the archaeological deposit suggest a deposition age of 325 ± 6 ka. ESR/U-series analyses on 5 teeth highlighted a minimum age of 304 ± 25 ka, in agreement with the $^{40}\text{Ar}/^{39}\text{Ar}$ age. These results allowed us to place with confidence the hominin occupation as well as faunal record of La Polledrara di Cecanibbio during MIS 9 slightly after the interglacial optimum. Despite the fauna similarities, La Polledrara di Cecanibbio is younger than the nearby archaeological site of Torre in Pietra (i.e. acheulian level *m* MIS 10/MIS 9 transition).

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Appendix A. Supplementary data

Supplementary material in annex

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