

Paleoenvironmental evolution of Picos de Europa (Spain) during marine isotopic stages 5c to 3 combining glacial reconstruction, cave sedimentology and paleontological findings

Daniel Ballesteros, Adrián Álvarez-Vena, Mael Monod-del Dago, Laura Rodríguez-Rodríguez, Jorge Sanjurjo-Sánchez, Diego Álvarez-Lao, Carlos Pérez-Mejías, Pablo Valenzuela, Irene Defelipe, César Laplana, et al.

▶ To cite this version:

Daniel Ballesteros, Adrián Álvarez-Vena, Mael Monod-del Dago, Laura Rodríguez-Rodríguez, Jorge Sanjurjo-Sánchez, et al.. Paleoenvironmental evolution of Picos de Europa (Spain) during marine isotopic stages 5c to 3 combining glacial reconstruction, cave sedimentology and paleontological findings. Quaternary Science Reviews, 2020, 248, pp.106581 -. 10.1016/j.quascirev.2020.106581 . hal-03493365

HAL Id: hal-03493365 https://hal.science/hal-03493365v1

Submitted on 17 Oct 2022

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers. L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial 4.0 International License

1 Paleoenvironmental evolution of Picos de Europa (Spain) during Marine Isotopic Stages 5c to 3

2 combining glacial reconstruction, cave sedimentology and paleontological findings.

3 Daniel Ballesteros (1), Adrián Álvarez-Vena (2), Mael Monod-Del Dago (3), Laura Rodríguez-

- 4 Rodríguez (4, 5), Jorge Sanjurjo-Sánchez (6), Diego Álvarez-Lao (2), Carlos Pérez-Mejías (7), Pablo
- 5 Valenzuela (8), Irene DeFelipe (9), César Laplana (10), Hai Cheng (7), Montserrat Jiménez-Sánchez
- 6 (2).
- 7 (1) UMR 6266 IDEES, University of Rouen-Normandie/CNRS. Mont Saint-Aignan CEDEX, France.
 8 daniel.ballesteros@univ-rouen.fr
- 9 (2) Department of Geology, University of Oviedo. c/ Jesús Arias de Velasco s/n, 33005, Oviedo.
- 10 adrian@picos-europa.com, dalao@geol.uniovi.es, mjimenez@geol.uniovi.es
- 11 (3) Université Paris 1 Panthéon-Sorbonne, 191 rue Saint Jacques, 75005 Paris. maelmdd@gmail.com
- 12 (4) Laboratoire de Géographie Physique (UMR 8591, CNRS). 1 Place Aristide Briand, 92195 Meudon
- 13 CEDEX, France. lelydriguez@gmail.com
- 14 (5) Departamento de Ciencias de la Tierra y Física de la Materia Condensada, Universidad de
- 15 Cantabria. Avenida de los Castros 48, 39005 Santander, Spain
- 16 (6) University Institute of Geology Isidro Parga Pondal, University of A Coruña. Campus de Elviña s/n,
- 17 15011, A Coruña, Spain. jsanjurjo@udc.es
- 18 (7) Institute of Global Environmental Change, Xi'an Jiaotong University, Xi'an, 710049, China.
 19 perezmegias@mail.xitu.edu.cn, cheng021@xitu.edu.cn.
- 20 (8) TRAGSATEC S.A. c/ Julián Camarillo 6B, 28037, Madrid, Spain.
 21 pablo.valenzuela.mendizabal@gmail.com
- 22 (9) Geosciences Barcelona (CSIC). c/ Lluís Solé i Sabaris s/n 08028 Barcelona.
 23 irene.defelipe@gmail.com
- 24 (10) Museo Arqueológico Regional de la Comunidad de Madrid. Pza. Bernardas s/n, 28801, Alcalá de
- 25 Henares, Spain. cesar.laplana@gmail.com

26 ABSTRACT

In glaciated areas, the environmental evolution before MIS 2 is usually poorly constrained mainly due to the later glacial erosion during the global Last Glacial Maximum (LGM). However, in carbonate areas, karst caves can preserve records of pre-LGM paleoenvironment interest. We studied a cave 30 (1350 m altitude) to establish the paleoenvironmental evolution of a glaciated karst area in Picos de 31 Europa (SW Europe). For this objective, a glacial reconstruction, cave sedimentology analyses, 32 macro- and micromammal remains are combined with ten U-Th, OSL and AMS ¹⁴C ages. The paleo-33 glacial reconstruction indicates glaciers descended down to 810-1040 m of altitude covering an area 34 in 36.18 km² of the surroundings of Covadonga Lakes during the glacial local maximum, with the 35 equilibrium line altitude located at 1524 \pm 36 m. The geomorphological study of the cave and the U-Th 36 and OSL dates reveal the presence of three allochthonous alluvial sediment sequences at 132-135, 37 98-60 and ca. 36 ka. These last two sequences would come from the erosion of fluvioglacial 38 sediments including teeth fragments of *Pliomys coronensis* (=*P. lenki*), an unusual species in NW 39 Spain in high areas during the Upper Pleistocene. In addition, found remains of chamois (Rupicapra 40 pyrenaica) dated in 37-33 cal ka BP constitutes the oldest evidence of chamois above 800 m asl in 41 the region. All the presented data indicate the development of alpine glacier-free areas covered by 42 fluvioglacial sediments at ca. 1450 m altitude at 98-60 and 37-33 ka, corresponding to glacial retreat 43 stages.

44

45 KEYWORDS

46 Karst, glacial reconstruction, paleoenvironment, *Pliomys coronensis*, *Rupicapra pyrenaica*.

47 1. INTRODUCTION

Paleoenvironmental records corresponding to Marine Isotope Stages (MIS) 4–3 are relatively scarce in glaciated mountains, mainly due to glacier erosion associated to the Last Glacial Maximum (LGM) of MIS 2. In areas made of limestone or another soluble rock, karst caves often preserve abundant additional paleoenvironmental evidence sheltered from surface weathering (Häuselmann, 2013). Cave evidence can be compared with surface landforms and sedimentary logs in order to reconstruct the past environmental evolution of a territory, improving our understanding on glacial advances and retreats (Mylroie and Mylroie, 2004; Bočić et al., 2012; Weremeichik and Mylroie 2014).

In glaciated areas, endokarst sedimentary records are mainly represented by speleothems and fluvial deposits, commonly originated during the Pliocene–Upper Pleistocene (Audra et al., 2006). Among the speleothems, stalagmites frequently grow during interglacial periods, when high CO₂ concentrations related to warm environments favors carbonate dissolution and precipitation in the karst conduits 59 (Isola et al., 2019; Tîrlă et al., in press). However, its growth is not only limited to interglacials, as can 60 be seen in a compilation of stalagmite records from Western Europe (Lechleitner et al., 2018). During 61 glaciations, glacial melting often affects karst aquifers favoring coarse grain sedimentation or erosion 62 processes according to glacial evolution. Pioneering works by Ford (1971, 1979) and Glover (1977) 63 already established that main endokarst detrital aggradation episodes are coeval with glacial retreats 64 and, occasionally, with the onset of glacial advances. Glaciers can inject sediments in cave passages 65 located at more than 1 km depth in glaciated areas (Audra et al., 2002), as well as in shallow caves 66 influenced by glacial outwash and located at distances of over 1000 km from the icefield (Mylroie, 67 1984). Since these sediments result from the glacial erosion of limestone, they are mostly formed of 68 carbonate-rich silt, the so-called "glacial rock-flour" or "glacial milk" (Bočić et al., 2012; Weremeichik 69 and Mylroie, 2014). In addition, cave detrital aggradation may also include allochthonous cobbles and 70 gravels derived from the erosion of any lithology outcropping in the catchment area (Audra et al., 71 2002; Ballesteros et al., 2017). In temperate settings, glacial valley incision dominantly occurs during 72 glaciations (Häuselmann et al., 2007). Therefore, the increase of hydraulic gradients in the endokarst 73 favours the development of torrential water flow that broadly erodes and evacuates cave sediments, 74 simultaneously to the general enlargement of previous vadose canyons (Plan et al., 2009; Adamson et 75 al., 2014). However, if glaciers reach the bottom of fluvial valleys, karst springs can be blocked by till, 76 thus triggering inundations in the endokarst (Skoglund et al., 2010). During these floods, glacial 77 carbonate silt decants forming rhythmic slackwater deposits related to sedimentary changes in 78 underground lakes (Weremeichik and Mylroie, 2014; Ballesteros et al., 2017).

79 In the NW of Spain, Picos de Europa is an outstanding location worldwide to study glaciokarst 80 processes and more deeply explore the potential of cave archives as records for paleoenvironmental 81 changes, since Picos de Europa combines outstanding glaciokarst landforms (Smart, 1986) with the 82 largest cluster of deep caves worldwide (Ballesteros et al., 2019a). This study focuses on the 83 Covadonga Lakes and their surroundings, located in the NW of Picos de Europa, a reference location 84 for glacial and karst research (e.g. Rodríguez-Rodríguez et al., 2014; Serrano et al., 2017; Telbisz et 85 al., 2019). Gale and Hoare (1997) and Alonso (1998) elaborated glacial reconstruction studies and Jiménez-Sánchez and Farias (2002) concluded that the local Glacial Maximum (local GM) took place 86 87 ca. 20 ka before the global LGM, proposing a subsequent phase of generalized glacier retreat. Later, 88 Moreno et al. (2010) established the regional paleoenvironmental evolution since 38 ka based on the Enol Lake record and geomorphological evidence, and Jiménez-Sánchez et al. (2013) refined the age
of the local GM at ca. 45 ka. Likewise, Nieuwendam et al. (2016) studied periglacial processes during
late MIS 3 and Ruiz-Fernández et al. (2016) reconstructed the timing of the last deglaciation.
Regarding to karst studies, Ballesteros et al. (2015, 2017, 2019b) defined the Quaternary landscape
evolution of the area based on cave geomorphology and geochronology.

Nevertheless, the environmental evolution of the Covadonga Lakes area before 45 ka remains mostly unknown, especially regarding to ancient fauna that remains virtually unexplored. The present study is focused on the karst cave named Hayéu l'Osu with the aim to establish the paleoenvironmental evolution of the Western Massif of Picos de Europa during MIS 5c–3. To achieve this goal, we carried out a multidisciplinary work combining glacial reconstruction based on field evidence, speleothem U-Th dating, cave detrital sediment analyses and OSL dating, and the identification of micro- and macromammal remains dated by AMS radiocarbon.

101 2. SETTING

102 Picos de Europa National Park (NW of Spain) is a high mountainous area up to 2650 m above sea 103 level (asl), located in the northern slope of the Cantabrian Mountains (Figure 1). Picos de Europa is 104 divided in three massifs (Western, Central and Eastern) by deep fluvial gorges. The main rocks that 105 crop out are Carboniferous limestone and, secondly, Ordovician, Carboniferous and Permian-Triassic 106 sandstone and shale (Merino-Tomé et al., 2013). The climate of Picos de Europa is humid and 107 temperate with fresh summers and without a dry season (Dfb and Dfc following the Köppen 108 classification). The annual precipitation ranges from 1000 to 1800 mm and is distributed throughout 109 the year, whilst the average air temperature ranges between -3 and 17°C. Snow is present around 6-9 110 months per year above 1500 m altitude.

111 Picos de Europa is one of the most relevant karst areas worldwide, since it contains 14% of the 112 deepest caves (>1 km deep) in the world (Ballesteros et al., 2019a). The karst evolution was 113 controlled by river incision and past glaciations (Smart, 1986). Fluvial incision created narrow gorges 114 up to 2 km in depth, conditioning the development of at least 3700 caves summing a total documented 115 length of 420 km, which were most likely formed during the Pliocene and Quaternary (Figure 1C; Ballesteros et al., 2015, 2019b). Cave detrital infill came from the erosion of the above-mentioned 116 117 nearby bedrock outcrops (Fernández-Gibert et al., 2000; Smart 1986). During the Upper Pleistocene, 118 glaciers reached their local GM extent, occupying ca. 190 km² and with their fronts descending down 119 to 600-900 m altitude (Figure 1C; Serrano et al., 2017). In the study area (Figure 2) glaciers flowed 120 below 1300 m altitude in the surroundings of Covadonga Lakes around 109–95 ka (MIS 5d–c) leading 121 to slackwater sedimentation in caves (Ballesteros et al., 2017). Later, glacial fronts reached at least 122 900 m altitude (Gale and Hoare, 1997; Alonso 1998; Moreno et al., 2010), defining a local maximum at 123 45 ± 3 ka (Jiménez-Sánchez et al., 2013). Glacier fronts retreated since ca. 45 ka, being already 124 located at ca. 1000 m elevation at 39 ka, coeval to the development of the Covadonga Lakes (Moreno 125 et al., 2010; Nieuwendam et al., 2016). According to these authors, this retreat continued during MIS 126 3, taking place under cold, dry and open conditions until 18 ka, coeval with the occurrence of 127 periglacial processes recorded by the Belbin kame deposit (Figure 2; Ruiz-Fernández et al., 2016). 128 Nevertheless, sporadic forest surrounded Picos de Europe during MIS 3 (Uzguiano et al., 2016). 129 Finally, an advance of the glacier front up to similar altitudes than the local maximum is reported at ca. 130 23–19 ka, which is ascribed to the global LGM (Ruiz-Fernández et al., 2016; Serrano et al., 2017).

131 3. METHODOLOGY

The methodology combines geomorphological, paleontological and geochronological analyses, including: (1) paleo-glacier reconstruction; (2) cave geomorphology; (3) paleontological identification of the remains, (4) speleothem U-Th dating, (5) detrital cave sediment OSL dating, and (4) radiocarbon dating of macromammal remains. All the spatial information was managed using the Geographical Information System ArcGIS (ESRI), involving topographic contours, orthophotographies and topographic digital models from the Spanish National Institute of Geography (www.cnig.es; last access on April 2019).

139 3.1 Paleo-glacier reconstruction

140 Paleo-glacier topography was reconstructed in the study area (Figure 2) for the local GM stage based 141 on geomorphological evidence compiled in Ballesteros et al. (2015, 2019b) and bed topography 142 information. Terminal moraines, glacial arêtes and cirques were used to infer the location of former 143 glacier margins during the local GM stage. Glacier topography was interpolated from 25 theoretical ice 144 surface profiles reconstructed comprising the routing pathways followed by glacier tongues along the 145 valleys and inferred from geomorphological indicators of glacier flow direction. Theoretical ice surface 146 profiles were adjusted following the methods explained in Benn and Hulton (2010), using the GlaRe 147 toolbox for ArcGIS (Pellitero et al., 2016). Basal shear stress values up to 80-120 KPa were needed to 148 adjust the theoretical ice surface profiles to existing geomorphological evidence, which are consistent with those observed in modern glaciers (50 to 150 KPa; Pellitero et al., 2016). The shape factor of the 149 valley was calculated for 62 cross sections to account for the topographic controls of glacial valley 150 151 shape on the basal shear stress. Once reconstructed, the digital elevation model of the former glacier 152 topography was used to estimate the Equilibrium Line Altitude (ELA) in steady-state conditions 153 through the Area Altitude Ratio Balance Ratio (AABR) method of Osmaston (2005) using the ELA 154 toolbox (Pellitero et al., 2015). We consider a range of balance ratio values of 1.9 ± 0.8 representative 155 for maritime glacier datasets (Rea, 2009).

156 **3.2 Cave geomorphology**

157 We studied the geomorphology of Hayéu l'Osu Cave in order to establish its development and its 158 sedimentary infill. To achieve this, we carried out the topographic survey in collaboration with 159 speleological groups and the geomorphological mapping of the cavity at 1:100 scale. The cave survey 160 and geomorphological map were developed in ArcGIS following the procedure described in 161 Ballesteros et al. (2015). The cave survey was delineated by means of 569 stations distributed along 162 the cave conduits and 608 measurements of distance, direction and inclination between stations using 163 a DistoX rangefinder, including the vertical and horizontal diameter of the conduit at each station. 164 These measurements were analyzed using the Compass software. Later, the geomorphological map 165 was drawn projecting cave forms and deposits on the cave survey.

166 **3.3 Paleontological analyses**

167 To search for micromammal remains, four samples of detrital sediments (named HAY-07, HAY-08, 168 HAY-09 and HAY-10) summing up to ~90 kg were collected from alluvial sequences of Hayéu l'Osu 169 Cave.. Samples were water-screened using two superimposed sieves of 2- and 0.5-mm mesh size. 170 Sieves were not previously used preventing hypothetic contamination from previous samples in the 171 laboratory. Fine sieved samples was examined using a parallel-optics type stereo microscope Nikon 172 SMZ800N under 10x magnification. Photographs were taken with a 5 Mpx resolution digital camera, 173 and image orientation correction was done with Adobe Photoshop CC software. We used teeth 174 samples of current fauna as a comparison material to identify the micromammal remains found in the 175 study cave. The reference material used belongs to Arvicola monticola, Microtus lavernedii, Microtus 176 lusitanicus and Clethrionomys glareolus, all of them collected from Tyto alba pellets in the Cantabrian 177 Region, as well as MIS 3 fossil material of *Pliomys coronensis (=P. lenki*) recovered from the nearby
178 archeological site of La Güelga Cave (Figure 1C).

Macromammals were discovered by speleologists from Grupo Espeléologico Polifemo (Oviedo) and collaborators. Macro remains were recovered using the archaeological methodology, mapping their occurrence on the geomorphological map (section 3.2). These remains were prepared for taxonomic and taphonomic analyses by removing physically the silt and clay attached to the bones and consolidating the samples with Paraloid B-72. Finally, we calculated the Minimum Number of Individuals (MNI) of each species taking into account each skeletal element. The fossils are stored in the Department of Geology at the University of Oviedo (Spain).

186 3.4 U-Th dating

187 Three flowstone samples from the study cave were dated by U decay series. Supplementary Data 188 (Figure S1) includes the geomorphological sections of the sampled flowstones. All samples were 189 collected in-situ with the aid of a hammer and a chisel, and drilled in the laboratory using a hand drill to 190 extract 0.1-0.2 g of carbonate powder, which was treated and analyzed at the Xi'an Jiaotong 191 University (China), following the procedures described in Edwards et al. (1987). Each sample was dissolved with HNO₃ and spiked with a ²²⁹Th - ²³³U - ²³⁶U carrier solution. Once the Fe solution and 192 193 the ammonium hydroxide is added, the separation of U and Th was carried out using column 194 chromatography with an anion exchange resin. Finally, U and Th fractions were analyzed in a 195 multicollector-inductively coupled plasma mass spectrometer (MC-ICP-MS) ThermoFisher Neptune 196 Plus using U decay constants reported in Cheng et al. (2013). The ages are expressed in years before 197 present (BP) with their 2σ uncertainty.

198 3.5 OSL dating

Optically stimulated luminescence (OSL) dating was applied to date endokarst detrital sediments. We collected five samples from sandy layers recognized in the cave stratigraphic sections detailed in Supplementary Data (Figure S2). Samples were taken driving opaque PVC tubes (550 mm diameter, 4 mm wall) in sandy material and then carefully covered with aluminum foil immediately after extraction. Samples were processed in the luminescence laboratory of the Institute of Geology Isidro Parga Pondal, University of A Coruña (Spain). Under red light conditions, sand materials stored within the tubes were dried and sieved. The grain fraction 180–250 µm was dried at 45°C and treated with

HCI and H₂O₂ in order to remove carbonates and organic matter, respectively, which constituted 65– 90% of the samples. Furthermore, a centrifugation step adding a high-density solution was needed to separate quartz from feldspar and heavy minerals. Later, a HF dilution was used on the quartz-rich fraction to obtain pure quartz. Infrared Stimulated Luminescence (IRSL) was used to verify quartz purity at the end of sample treatment.

211 OSL signals were recorded using an automated RISØ TL/OSL-DA-15 reader equipped with a photomultiplier EMI 9635 QA (PMT) and a ⁹⁰Sr/⁹⁰Y source (dose of 0.120 ± 0.003 Gy·s⁻¹). An optical 6 212 213 mm-thick Hoya U-340 filter was placed between the aliquots and the PMT to measure the UV range 214 emission. The Single Aliquot Regeneration (SAR) protocol (Murray and Wintle, 2000) was applied on 215 multigrain aliquots to estimate the equivalent Dose (De), considering the Central Age Model (Galbraith 216 et al., 1999) to date each sample. Frequency histograms of De of samples HAY-07, 09, 10 and 11 are 217 included in Supplementary Data (Figure S3). Sample HAY-08 is not included because of its low 218 reliability (only 4 aliquots) but its age is consistent with other OSL datings. Preheat tests were 219 previously performed and dose recovery tests used on bleached aliquots (Murray and Wintle, 2003).

The activity concentration of radioisotopes (⁴⁰K, and ²³⁸U, ²³⁵U and ²³²Th decay chains) was estimated using low background gamma-ray spectrometry. Measurements of calcined and grinded sediments were performed in a coaxial Canberra XTRA gamma detector (Ge Intrinsic) model GR6022 within a 10 cm-thick lead shield. The alpha dose-rate was neglected due to the HF etching step, and the beta dose-rate corrected (Brennan, 2003). The cosmic dose-rate was estimated following Prescott and Hutton (1994) and using the conversion factors proposed by Guerin et al. (2011). Resultant OSL ages are expressed in years before present (BP) with their corresponding 2σ uncertainty.

227 3.6 Radiocarbon dating

Two samples from macromammal bones were sampled for radiocarbon dating through accelerator mass spectrometry (AMS). Radiocarbon measurements (reference order 13274/18) were performed at the Poznan Radiocarbon Laboratory, Poland. The obtained ages were calibrated using the software OxCal v.4.3.2 (Bronk Ramsey, 2017) against the INTCAL 13 curve (Reimer et al., 2013), considering the 2σ standard deviation (95.4% probability). The calibrations are included in Supplementary Data (Figure S4) and calibrated dates are expressed in cal ka BP.

234 **4. RESULTS**

235 4.1 Paleo-glacier reconstruction

236 Glaciers formed an ice field in the surroundings of Covadonga Lakes during the local GM, occupying a 237 total surface extent of 36.18 km² and covering the entrance of Hayéu l'Osu Cave (Figure 3). Glacier 238 tongues reached total lengths of ca. 2 km along the valleys of Ordiales, Hunhumia, and Pomperi; up to 239 9.5 km along El Bricial-Vega de Enol valleys; 5.9 km along Ercina valley; and 6.6 km along the former 240 Belbín glacier. Glacier fronts were placed at ca. 1040 m altitude in the Ordiales, Hunhumia and Ercina 241 valleys; 925–1030 m in the El Bricial-Vega de Enol valleys; and 810 m in Belbín (Figure 3). Glacier 242 maximum thickness reached up to 105-220 m along the El Bricial-Vega de Enol valleys, 170 m along 243 the Belbín flow line, 170 m along Ercina valley, and between ca. 114 and 168 m along the valleys 244 Ordiales, Hunhumia, and Pomperi. The steady-state ELA was most likely set at 1524 ± 36 m (Figure 245 3).

246 4.2 Cave geomorphology

247 The Hayéu l'Osu Cave is mainly formed by a 1.6 km-long conduit (named Stream Gallery) slightly 248 tilted towards the SE, and by vertical conduits summing up to 135 m in depth (Figure 4A). Vertical 249 conduits connect the Stream Gallery and the topographic surface as shown in the cave longitudinal 250 profile (Figure 4B). Geomorphological evidence includes alluvial deposits, breakdown deposits, 251 speleothems and slackwater deposits. Alluvial sediments up to 3 m thick are present along the Stream 252 Gallery (Supplementary Data, Figure S2), involving active channel and terrace deposits. Active 253 channel deposits are located along the cave stream (Figure 4C) and resulted from the erosion of 254 terrace deposits placed at the bottom and walls of the NW of the study cave (Figures 4D and 5A-E). 255 Terrace deposits are perched up to 20 m above the streambed and are occasionally covered by 256 flowstones and stalagmites (Figure 4E). Active channel deposits and most terrace deposits are grain-257 supported and contain sub-angular to rounded calcareous pebbles and cobbles, containing no matrix 258 or with 5-20% of sandy matrix (70-85% of carbonate and 15-30% of quartz). Both deposits comprise 259 also sub-rounded allochthonous pebbles and cobbles of sandstone, bauxite and diabase. In addition, 260 alluvial sand (70-95% of carbonate and 5-30% of quartz) form also minor terraces or constitute layers 261 interbedded within the pebble and cobble deposits. These sand deposits may eventually contain 262 micromammal remains, which have been identified and described in detail in section 4.3. Breakdown 263 deposits are common along the study cave, including angular calcareous boulders less than 2 m in 264 diameter formed by rock fall processes. Regarding the speleothems, they commonly appear in cave

passages perched above the cave stream. Flowstone up to 3 m thick and stalagmites less than 2 m height occasionally precipitated on top of alluvial and breakdown deposits, however, flowstones covered by sandy terrace deposits were also recognized. Finally, slackwater deposits show 0.5–3 m of laminated silt and clay (Figure 4F) deposited essentially in the central part of the study cave (Figure 4A). These deposits would be related to cave floods similar to what has been observed in the nearby Torca La Texa shaft (Figure 2; Ballesteros et al., 2017).

271 **4.3 Paleontology of mammal bones**

272 Seven fragments of arvicoline molars and one fragment of a rodent incisor were found in samples from 273 HAY-7, 8 and 9, collected in the Stream Gallery (Figure 4A). These remains show dentine removal by 274 erosion and four of them show also rounded edges. The best-preserved fragment corresponds to a 275 complete enamel prism found in sample HAY-9. When comparing this fragment with the upper first 276 molars of extinct and living arvicolines from the Cantabrian Region (Figure 5), its size and angle in 277 occlusal view (Figure 5a') matches with the morphology of an upper first or second molar (Figure 5b'-278 e'). The curvature of the anterior side of the triangle at the tooth's base indicates that it belongs to a 279 rooted molar (Figure 5a"), clearly different respect to ever-growing molars (Figure 5D, E and F). All 280 these mentioned morphological features and the absence of curvature at the basal part of the 281 posterior side of the prism (Figure 5a"), match with the morphology of the buccal salient angle 3 282 (BSA3) of the first upper molar (M¹). Among the arvicolines from the Upper Pleistocene of the Iberian 283 Peninsula, only two species bear rooted molars. One of them is *P. coronensis* (Figure 5B), a very common vole in rocky environments during MIS 3 and earlier before its extinction during MIS 2 in the 284 285 Cantabrian Region, territory that constituted the last refuge for this species before its complete 286 disappearance (Cuenca-Bescós et al., 2010). The other rooted-molar species is C. glareolus (Figure 287 5C), which currently inhabits the well-developed forests of the Cantabrian Region and other Eurasian 288 areas, but which is absent in alpine environments. Considering the compared morphology and the 289 chronological context (defined in section 4.4), the enamel fragment of HAY-9 has been ascribed to the 290 species P. coronensis. There is another enamel fragment at HAY-7 whose morphological features at 291 its basal part indicate it belongs to a rooted molar, but no further identification can be done due to its 292 poor preservation.

293

294 A total amount of 81 macromammal remains, all of them corresponding to small-sized bovids, were 295 recovered from two sites named Llamaeyu and Stream galleries (Figure 4). In Llamaeyu Gallery, 296 radius, metacarpals, phalanxes, skull fragments, vertebras, ribs, one molar and other bones were 297 found without anatomic connection on top of a breakdown deposit (Figure 6A) whereas, in the Stream 298 Gallery, a scapula was located on a detrital deposit (Figure 6B). Morphology and morphometry of teeth 299 and skeletal elements are distinctive of chamois (Figure 7), so they are ascribed to Rupicapra 300 pyrenaica (a comprehensive discussion about the specific attribution of Upper Pleistocene chamois 301 from the Iberian Peninsula is provided by Álvarez-Lao, 2014). This fossil assemblage corresponds to a 302 minimum of four individuals (MNI): two young and two adults. The lack of anthropic activity signs 303 excludes humans as responsible for the bone accumulation. In general, no carnivore marks are 304 detected, suggesting that the bone accumulation was related to a natural trap. Only one bone shows 305 teeth marks (Figure 7D) produced by a small carnivore, most likely due to scavenging rather than 306 predation. In addition, only one bone (corresponding to one of the young individuals) shows erosion 307 features due to transportation by water (Figure 7C). From an environmental perspective, occurrence of 308 R. pyrenaica, a typical alpine ungulate, at Hayéu l'Osu Cave is consistent with the current rocky 309 mountain landscape of the area. Moreover, presence of a herbivore mammal in this cave clearly 310 indicates ice-free conditions in the near surroundings during the time they lived (Álvarez-Lao, 2014).

311 4.4 Geochronology

312 Three speleothem samples from the study cave were analyzed using U-Th dating (Table 1). Samples exhibit ²³⁸U content lower than 100 ppb and high initial δ^{234} U (1000-3000 ppm). Standard procedures 313 to correct the ages assume an initial 230 Th/ 232 Th atomic ratio of 4.4 ± 2.2 $\cdot 10^{-6}$ (Edwards et al., 1987; 314 315 Cheng et al., 2013) and the obtained ages are considered reliable.. Sample HAY-12 (263 ± 18 ka) 316 corresponds to the base of an overturned set of flowstones and stalagmites, originally precipitated on 317 top of slackwater deposits (Figure 8A). The age provided for HAY-12 represents a minimum age for 318 the slackwater deposits, which would have been decanted by cave flooding before 263 ± 18 ka, during 319 the Middle Pleistocene. Subsequently, the slackwater deposits were eroded causing the collapse of the overlying flowstones and stalagmites. Similarly, samples HAY-06 (132 \pm 1 ka) and HAY-13 (135 \pm 320 321 7 ka) have precipitated fossilizing fluvial deposits (Figure 8B and C), which were eroded later in the 322 case of HAY-06. Both datings indicate the occurrence of a first alluvial sequence older than 135-132 323 ka, probably during MIS 6.

324 In the study cave, five alluvial deposits described in section 4.2 were dated using OSL from 84 \pm 10 to 325 36 ± 3 ka (Table 2, Figure 8C-G). The estimated dose-rates were very low due to the low radioisotope 326 content, being below 1 Gy ka⁻¹ for most samples. No disequilibrium is observed in the U and Th decay 327 chains. The OSL signals were dim but fast, and provided poor signal to background ratios, causing the 328 rejection of a high number of aliquots, being the number of accepted aliquots between 4 and 35. The 329 De distributions were non-skewed and the over dispersion (Table 2) of the mean of the Central Age 330 Model was low, showing no evidence of incomplete bleaching of the alluvial sand grains before they 331 were introduced into the cave. Thus, the Central Age Model was used for assessing the OSL ages. 332 After bleaching, the quartz was for a short period on the topographical surface before being introduced 333 into the study cave. We consider this time to be short since the study cave is located in a hill that 334 probably acted as a by-pass area to lower zones where widespread glacial and fluvioglacial 335 sedimentation took place (Figures 2 and 3). However, reworking processes are not totally discarded. 336 The chronological framework provided allows us to define two alluvial sequences occurring after the 337 first alluvial sequence identified by U-Th dates. According to this, a second alluvial sequence 338 corresponds to terrace sediments deposited between 84 ± 10 and 65 ± 16 ka, ranging from MIS 5c to 339 MIS 4 (Figure 8C-F), while a third sequence includes a fluvial terrace formed at 36 ± 3 ka during MIS 340 3a (Figure 8G).

The chronology of the second and third alluvial sequences provides the chronological framework of the arvicolinae remains, while the radiocarbon dates represent the timing of the chamois. A fragment of metatarsal from Llamaeyu Gallery has an AMS ¹⁴C age of 36 ± 1 cal ka BP whilst the scapula recovered in Stream Gallery provided an age of 34.5 ± 0.6 cal ka BP (Table 3).

345 5. DISCUSSION: PALEOENVIRONMENTAL EVOLUTION

Cave geomorphology and U-Th ages provide new data about the origin and evolution of the Hayéu 346 347 l'Osu Cave during the Middle Pleistocene (Figure 9). Flowstone ages suggest that the cave was 348 formed prior to 263 ka (Figure 9A), like most karst cavities studied in Picos de Europa (Smart, 1986; 349 Ballesteros et al., 2015, 2019). Since its origin, Hayéu l'Osu Cave has undergone inundations that 350 would be related to Middle Pleistocene glaciations, followed by speleothem precipitation at 263 ± 18 351 ka (MIS 8). Other studies have documented coeval breccia cementation in surficial talus deposits in 352 Puertos de Áliva (Villa et al., 2013), between the Central and Eastern massifs of Picos de Europa 353 (Figure 1C). Later, at the end of MIS 6, flowstone precipitation took place in the cave at 135-132 ka,

covering the first alluvial sequence previously deposited. All these speleothems and detrital sediments
partially filled the Hayéu l'Osu Cave, as occurred in other nearby caves named Pozu Llucia and Torca
La Texa (Figure 2; Ballesteros et al., 2019).

The paleoenvironmental evolution of the surroundings of Covadonga Lakes during MIS 5c-3 is established based on the combination of geomorphological-stratigraphical and paleontological data and kernel density functions (Figure 10) of U-Th, OSL and radiocarbon ages obtained in the cave. This probability analysis considered also previous datings carried out in Enol lacustrine sediments by Moreno et al. (2010) and in Vega de Comeya border polje by Jiménez-Sánchez et al. (2013). Four phases have been distinguished (Table 4 and Figure 11) according to the probabilistic distribution of Figure 10.

364 5.1 Phase 1 (98-60 ka; MIS 5c-4)

365 The first phase described for the study area ranges between ca. 98 and 60 ka (Figure 11A). After 366 glacial advances recorded during MIS 5cd, dated in the Central Cantabrian Mountains at 114 ± 7 ka 367 based on in situ produced cosmogenic nuclide ¹⁰Be (Figure 1B; Rodríguez-Rodríguez et al., 2016), a 368 glacial retreat episode occurred in the study area during MIS 5c-4. In Hayéu l'Osu Cave this episode 369 might correspond to the sedimentation of the second alluvial sequence that includes P. coronensis. 370 Alluvial deposits are dominated by carbonate sand and silt (section 4.2), coming from the glacial 371 erosion of Carboniferous limestone, while pebbles and cobbles of sandstone and diabase were 372 eroded from sandstone and igneous dyke outcrops located in higher areas of the Western Massif of Picos de Europa (Ballesteros et al., 2017, 2019b). Thus, the Ercina glacier that flowed from the 373 374 summit areas elevated more than 2000 m altitude of the Western Massif (Figure 3) eroded the 375 limestone, sandstone and diabase bedrock and transported the resulting sediment load to the glacier 376 fronts located higher than 1400 m altitude (Figure 11A). Sediments were probably transported by 377 glacial meltwater to the surroundings of the study cave, in a fluvioglacial environment inhabited by P. 378 coronensis. The roundness of sediment particles observed in the alluvial sequence (section 4.2) as 379 well as in the enamel remains discovered in the study cave (section 4.3) are both compatible with this 380 interpretation. Finally, the presence of P. coronensis indicates that bedrock was exposed near the 381 cave under relatively cold conditions, and hence, the glacier advance was less extensive than the 382 local GM extent.

The sediments of the second alluvial sequence might have formed during MIS 5d glaciation identified directly in the central Cantabrian Mountains (Figure 1B; Rodríguez-Rodríguez et al., 2016) and indirectly in the nearby Torca La Texa shaft (Figure 2; Ballesteros et al., 2017). The decantation of glacial origin rhythmite in this shaft (located at 1350 m elevation) at 109-95 ka suggests that the Ercina glacier front was at around 1350-1400 m at the beginning of phase 1.

388 5.2 Phase 2 (48–42 ka; MIS 3c)

389 The second phase (Figure 11B) corresponds to the local GM advance of glaciers in the study area. 390 The Vega de Comeya border polje was occupied by a proglacial lake that was being filled by 391 calcareous alluvial deposits from the south-facing slopes of the Western Massif and siliceous alluvial 392 sediments from the north-facing slopes (Jiménez-Sánchez and Farias, 2002). OSL dating yielded a 393 minimum age of 45 ± 3 ka for the onset of glacio-lacustrine sedimentation in the border polje while 394 organic remains from a sand and gravel unit placed immediately on top yielded a radiocarbon age of 395 44.1 ± 0.9 cal ka BP, constraining the age of the local GM (Jiménez-Sánchez et al., 2013). Similarly, 396 the nearby Belbín kame terrace deposit (Figure 2) yielded a minimum reference age of 36.5 ± 0.5 cal 397 ka BP for the base of its sequence and, hence, for the Belbín moraine deposition (Ruiz-Fernández et 398 al., 2016). The reconstruction of the local GM extent (section 4.1) suggests that glaciers were less 399 extensive compared to previous reconstructions proposed by Gale and Hoare (1997), but it is in agreement with those developed in Alonso (1998), Serrano et al. (2017), and Ruiz-Fernández et al. 400 401 (2016). The reconstruction also indicates that glacier fronts descended down to -1040-810 m asl, 402 covering the calcareous bedrock where the Hayéu l'Osu Cave develops (Figure 11B), which was 403 located below the estimated ELA (1524 ± 36 m). The altitude of the steady-state ELA in our work is 404 consistent with previous regional ELA reconstructions made by Santos-González et al. (2013), but ca. 405 100 m lower than the mean ELA estimated in Serrano et al. (2017).

406 **5.3 Phase 3 (42-38 ka; MIS 3b)**

407 Phase 3 represents the onset of glacier retreat in the surroundings of Covadonga Lakes (Jiménez-408 Sánchez and Farias, 2002). Glacier retreat was recorded by proglacial lacustrine sedimentation at 409 Enol Lake at 38 ± 2 cal ka BP, exposing the glacial depression now occupied by Enol Lake but with 410 the glacial front still located close to the lake and supplying ablation water and coarse inorganic 411 sediments (Moreno et al., 2010). During this phase (Figure 11C), the evolution of the Ercina glacier 412 front would have been consistent with that of El Bricial-Vega de Enol glacier recording a new episode 413 of detrital sediment infill at 39 ± 3 ka. Hence, the Ercina glacier was likely shorter at ca. 38 ka than 414 previously assumed (Moreno et al., 2010). Glacier retreat prior to 36–39 ka has also been reported in 415 Puertos de Áliva, a site located between the Central and Eastern massifs (Figure 1C; Serrano et al., 416 2012).

417 5.4 Phase 4 (37–33 ka; MIS 3a)

418 Glaciers continued receding during phase 4 (Figure 11D). The Ercina glacier front had already 419 ascended well above 1400 m by 37 ka, while the third alluvial sequence was being deposited within 420 the Hayéu l'Osu Cave. The third detrital sequence is thinner and less extensive than the second 421 detrital sequence, suggesting that the volume of surface sediment was higher during MIS 5d glaciation 422 than during the local GM of MIS 3c. MIS 5d glaciation occurred after the Eemian interglacial substage, 423 when weathering processes would have been more intense than over MIS 4. Besides, the third alluvial 424 sequence deposited at 36 ± 3 ka within the study cave also involved micromammal remains. 425 Consequently, this sequence should come from fluvio-glacial sediments sourcing from the retreating 426 glacier fronts similar to the interpretation for the second alluvial sequence (section 5.1) even if minor 427 reworking processes would take place.

428 Subsequently, at least three chamois accidentally fell inside the cave. The current position of these 429 remains at 50 and 100 m depth (from the topographic surface) and the absence of evidence produced 430 by carnivores, scavengers or humans support the role of the cave as a natural trap. Only one bone 431 shows minor bite marks caused by a small carnivore (e.g. weasel, beech marten). In the Llamaeyu 432 Gallery, bone remains most likely arrived by gravity processes and minor water transport, as the fauna 433 assemblage is not in anatomic connection and is related to a breakdown deposit (section 4.2). In the 434 Stream Gallery, the chamois bone is located in a subhorizontal conduit and have been transported by 435 an underground stream after its accidental fall in the cave. The lack of transport marks recognized in 436 the bone suggests that it may have arrived protected by the soft parts of the animal.

The existence of chamois bones points to the presence of an alpine environment with exposure of rocky areas developed under relatively cold climate at 37–33 ka. This time range coincides with a relative cold period according to the NGRIP δ^{18} O ice core record (Figure 12; NGRIP, 2004). Nevertheless, at a regional scale, the presence of chamois is coeval with a period of less cold conditions marked by an increase in organic matter and coarse sediments in the paleolake of Puertos

de Áliva (1400 m altitude) between 36 and 32 ka (Serrano et al., 2012). In lower areas, the pollen record of El Esquilleu Cave (Figure 1C; 350 m asl) revealed the occurrence of *Pinus* forest with minor deciduous and evergreen oaks, but also heathland shrubs in the SW of Picos de Europa at around 38–34 ka (Uzquiano et al., 2016) that would have been part of the chamois diet.

446 During the late MIS 3, chamois constituted one of the main food sources of the Mousterian-447 Aurignacian humans who lived in areas below 400 m elevation in the Cantabrian Mountains (Yravedra 448 and Cobo-Sánchez et al., 2015). Around Picos de Europa, archeological excavations discovered 449 abundant remains of hunted chamois in Sopeña (Pinto-Llona et al., 2012), El Esquilleu (Uzquiano et 450 al., 2012; Yravedra et al., 2014), La Güelga (Menéndez et al., 2018) and other cavities suggesting that 451 the hunting zones were located in lower areas (Figure 1C). However, the chamois remains found in 452 Hayéu l'Osu Cave indicate that the populations of this caprine could also have occupied areas of 453 Picos de Europa up to, at least, 1400 m elevation, during 37-33 ka.

454 6. CONCLUSIONS

We propose a new paleoenvironmental evolution model during MIS 5c–3 for Picos de Europa based on a multidisciplinary work combining glacial and cave geomorphology, Quaternary paleontology and geochronology on a glaciokarst area. The study of sediment records in Hayéu l'Osu Cave have allowed us to constrain the timing of glacier retreat, and thus complement previous reconstructions on paleo-glacier evolution in Picos de Europa. In addition, mammal remains preserved in caves are exceptional indicators of glacial ice-free areas in the past, pointing out to a paleoenvironment developed over successive pulses of the glacial evolution.

462 Three alluvial stratigraphic sequences have been identified in Hayéu l'Osu Cave. The first sequence 463 was deposited before 135-131 ka (MIS 6), while the second and third sequence, with micrommamal 464 remains, were sedimented at 98-60 ka and ca. 36 ka respectively. Additionally, chamois remains (37-465 33 ka) were found on top of the third sequence. This stratigraphic record suggests the development of 466 alpine ice-free areas at ca. 1400 m altitude during 98-60 ka (MIS 5c-4), when glaciers were probably 467 located at higher altitudes. Later, glaciers with a length of 9.5 km and up to 100-220 m in thickness, 468 descended down to 810-1030 m elevation during the local GM stage, establishing a proglacial lake environment in the Comeya border polie at 48-42 ka (MIS 3c). Over this period, the study cave was 469 470 located in the glacial ablation zone and covered by glaciers. A general trend of glacier retreat occurred 471 from 42-38 ka, with the glacial fronts located above 1400 m altitude at 37-33 ka. In this setting,

472 chamois inhabited the ice-free rocky steep slopes of Picos de Europa, in agreement with a slight473 increase in local temperature.

474 In addition, the distribution of species in the Cantabrian Region provides further insights on the 475 paleoenvironmetal evolution of the study area. Remains of P. coronensis allow us to extend its 476 altitudinal distribution up to ca. 1400 m in the Cantabrian Region during MIS 4, whereas the chamois 477 bone assemblage of Hayéu l'Osu Cave constitutes the oldest evidence of R. pyrenaica above 1000 m 478 altitude of the Cantabrian Region. Chamois inhabited the highlands of Picos de Europa at 37-33 ka 479 coevally with human occupation in lower areas, who broadly hunted chamois. However, our outcomes 480 suggest that the studied chamois remains fell accidently in Hayéu l'Osu Cave, being accumulated in 481 the stratigraphic record by natural processes without any human contribution.

482 AUTHOR CONTRIBUTION

483 DB, LRR and MJS conducted the research. MMD and LRR elaborated the paleo-glacier 484 reconstruction. DB performed the cave geomorphology and collected all samples in cooperation with 485 PV, IDF and the speleological team. AAV found and identified rodent remains under the guidance of 486 CL, whilst DAL studied the macromammal assemblages. JS carried out the OSL datings, and CPM 487 obtained the U-Th ages under the direction of HC. DB, LRR, DAL, AAV, IDF, PV and CPM wrote the 488 article and designed the figures. All authors have contributed to the discussion.

489 ACKNOWLEDGEMENTS

Picos de Europa National Park founded this work through the Geocancosta (FC-GRUPIN6IDI/2018/000184) and FUO-300-17 projects, receiving the grateful assistance of the National Park staff and the speleologists from Grupo Espeleológico Polifemo (Oviedo), GES Montañeiros Celtas (Vigo), Grupo de Espeleología Diañu Burlón (Corvera de Asturias), and Espéleo Club Aradelas (Vigo). LRR is grant holder of the post-doctoral Clarín-COFUND Program, financed jointly by the 7th WP of the European Union–Marie Curie Actions and Gobierno del Principado de Asturias (Reference ACA-17-19). We appreciate the work of Jo de Waele, Rafael López-Martínez and other reviewers.

497 **REFERENCES**

Adamson, K.R., Woodward, J.C., Hughes, P.D. 2014. Glaciers and rivers: Pleistocene uncoupling in a
 Mediterranean mountain karst. Quaternary Science Reviews, 94, 28–43.

Alonso, V. 1998. Covadonga National Park (Western Massif of Picos de Europa, NW Spain): a
 calcareous deglaciated area. Trabajos de Geología 20, 167–181.

- Álvarez-Lao, D.J. 2014. The Jou Puerta Cave (Asturias, NW Spain): a MIS 3 large mammal
 assemblage with mixture of cold and temperate elements. Palaeogeography,
 Palaeoclimatology, Palaeoecology 393, 1–19.
- Audra, P., Quinif, Y., Rochette, P. 2002. The genesis of Tennengerbirge karst and caves (Salzburg,
 Austria). Journal of Cave and Karst Studies 64, 153–164.
- Audra, P., Bini, A., Gabrovšek, F., Häuselmann, P., Hobléa, F., Jeannin, P.Y., Kunaver, J., Monbaron,
 M., Šušteršič, F., Tognini, P., Trimmel, H., Wildberger, A. 2006. Cave genesis in the Alps
 between the Miocene and today: a review. Zeitschrift für Geomorphologie 50, 153–176.
- Ballesteros, D., Jiménez-Sánchez, M., Giralt, S., García-Sansegundo, J., Meléndez-Asensio, M. 2015.
 A multi-method approach for speleogenetic research on alpine karst caves. Torca La Texa
 shaft, Picos de Europa (Spain). Geomorphology 247, 35–54.
- 513 Ballesteros, D., Jiménez-Sánchez, M., Giralt, S., DeFelipe, I., García-Sansegundo, J. 2017. Glacial
 514 origin for cave rhythmite during MIS 5d–c in a glaciokarst landscape, Picos de Europa (Spain).
 515 Geomorphology 286, 68–77.
- Ballesteros, D., Fernández-Martínez, E., Carcavilla, L., Jiménez-Sánchez, M. 2019a. Karst Cave
 Geoheritage in Protected Areas: Characterisation and Proposals of Management of Deep
 Caves in the Picos de Europa National Park (Spain). Geoheritage 11(4), 1919-1939..
- Ballesteros, D., Giralt, S., García-Sansegundo, J., Jiménez-Sánchez, M. 2019b. Quaternary regional
 evolution based on karst cave geomorphology in Picos de Europa (Atlantic Margin of the
 Iberian Peninsula). Geomorphology 336, 133–151.
- 522 Benn, D.I., Hulton, N.R.J. 2010. An ExcelTM spreadsheet program for reconstructing the surface 523 profile of former mountain glaciers and ice caps. Computers & Geosciences 36, 605–610.
- Bočić, N., Faivre, S., Kovačić, M., Horvatinčić, N. 2012. Cave development under the influence of
 Pleistocene glaciation in the Dinarides an example from Štirovača Ice Cave (Velebit Mt.,
 Croatia). Zeitschrift für Geomorphologie, 56, 409–433.
- 527 Bronk Ramsey, C. 2017. Methods for Summarizing Radiocarbon Datasets. Radiocarbon 59, 1-25.
- 528 Brennan, B.J. 2003. Beta doses to spherical grains. Radiation Measurements 3, 299–303.
- 529 Cheng, H., Edwards, L. R., Shen, C.-C., Polyak, V.J., Asmerom, Y., Woodhead, J.,Hellstrom, J.,
 530 Wang, Y., Kong, X., Spötl, C., Wang, X., Alexander, E. C. 2013. Improvements in 230Th
 531 dating, ²³⁰Th and ²³⁴U half-life values, and U-Th isotopic measurements by multi-collector
 532 inductively coupled plasma mass spectrometry. Earth and Planetary Science Letters, 371-372,
 533 82-91.
- 534 Clark, P.U., Dyke, A.S., Shakun, J.D., Carlson, A.E, Clark, J., Wohlfarth, B., Mitrovica, J.X., Hostetler,
 535 S.W., McCabe, A.M. Science 7, 710-714.
- 536 Colhoun, E.A., Kiernan, K., Barrows, T.T., Goede, A. 2010. Advances in Quaternary studies in 537 Tasmania. Special Publications, 346. Geological Society of London. Londres. 165–183.
- 538 Cuenca-Bescós, G., Straus, L. G., García-Pimienta, J. C., González Morales, M.R., López-García,
 539 J.M. 2010. Late Quaternary small mammal turnover in the Cantabrian Region: The extintion of
 540 *Pliomys lenki* (Rodentia, Mammalia). Quaternary International 212, 129–136.

- Edwards, R.L., Chen, J.H., Wasserburg, G.J. 1987. ²³⁸U, ²³⁴U, ²³⁰Th, ²³²Th systematics and the precise
 measurement of time over the past 500,000 years. Earth and Planetary Science Letters, 81,
 175–192.
- Fernández-Gibert, E., Calaforra, J.M., Rossi, C. 2000. Speleogenesis in the Picos de Europa Massif,
 Northern Spain, in: Klimchouk, A., Ford, D., Palmer, A., Dreybrodt, W. (Eds.), Speleogenesis:
 Evolution of Karst Aguifers, National Speleological Society, Huntsville, Alabama, pp. 352–357.
- Ford, D.C. 1971. Characteristics of Limestone Solution in the Southern Rocky Mountains and Slekirk
 Mountains, Alberta and British Columbia. Canadian Journal of Earth Sciences 8, 585–609.
- 549 Ford, D.C. 1979. A review of alpine karst in the southern Rocky Mountains of Canada. National 550 Speleological Society Bulletin 41, 53–65.
- Galbraith, R.F., Roberts, R.G., Laslett, G.M., Yoshida, H., Olley, J.M. 1999. Optical dating of single
 and multiple grains of quartz from Jinmium Rock Sherlter, Northern Australia: Part 1,
 Experimental design and statistical models. Archaeometry 41, 339–364.
- Gale, S.J., Hoare, P.G. 1997. The glacial history of the northwest Picos de Europa of northern Spain.
 Zeitschrift für Geomorphologie 41, 81–96.
- Glover, R. R. 1977. A conceptual model of cave development in a glaciated region. Proceedings of 7th
 International Speleological Congress. International Union of Speleology, Sheffield, pp. 220–
 221.
- 559 Guerin, G., Mercier, N., Adamiec, G. 2011. Dose-rate conversion factors: update. Ancient TL 29, 5–8.
- Häuselmann, P. 2013. Large epigenic caves in high-relief areas. In: Shroder, J., Frumkin, A. (Eds.)
 Treatise on Geomorphology, Vol 6, Karst Geomorphology. Academic Press, San Diego, 207–
 219.
- Häuselmann, P., Granger, D.E., Jeannin, P.Y., Lauritzen, S.E. 2007. Abrupt glacial valley incision at
 0.8 Ma dated from cave deposits in Switzerland. Geology 35, 143–146.
- Isola, I., Ribolini, A., Zanchetta, G., Bini, M., Regattieri, E., Drysdale, R. N., Hellstrom, J.C., Bajo, P,
 Montagna, P., Pons-Branchu, E. 2019. Speleothem U/Th age constraints for the Last Glacial
 conditions in the Apuan Alps, northwestern Italy. Palaeogeography, Palaeoclimatology,
 Palaeoecology 518, 62–71.
- Jiménez-Sánchez, M., Farias, P. 2002. New radiometric and geomorphologic evidences of a last
 glacial maximum older than 18 ka in SW European mountains: the example of Redes Natural
 Park (Cantabrian Mountains, NW Spain). Geodinamica Acta 15, 93–101.
- Jiménez-Sánchez, M., Rodríguez-Rodríguez, L., García-Ruiz, J.M., Domínguez-Cuesta, M.J., Farias,
 P., Valero-Garcés, B., Moreno, A., Rico, M., Valcárcel, M. 2013. A review of glacial
 geomorphology and chronology in northern Spain: Timing and regional variability during the
 last glacial cycle. Geomorphology 196, 50–64.
- Lechleitner, F.A., Amirnezhad-Mozhdehi, S., Columbu, A., Comas-Bru, L., Labuhn, I., Pérez-Mejías,
 P., Rehfeld, K. 2018. The Potential of Speleothems from Western Europe as Recorders of
 Regional Climate: A Critical Assessment of the SISAL Database. Quaternary 1 (3), 30.

- Menéndez, M., Álvarez-Alonso, D., Andrés-Herrero, M. de, Carral, P., García-Sánchez, E., JordáPardo, J.F., Quesada, J.M., Rojo, J. 2018. The Middle to Upper Paleolithic transition in La
 Güelga cave (Asturias, Northern Spain). Quaternary International 474, 71–84.
- Merino-Tomé, O., Suárez Rodríguez, A., Alonso, J., González Menéndez, L., Heredia, N., Marcos, A.
 2013. Mapa Geológico Digital continuo E. 1:50.000, Principado de Asturias (Zonas: 1100–
 1000–1600) [WWW Document]. GEODE. Mapa Geológico Digital Continuo de España.
 SIGECO-IGME. URL http://cuarzo.igme.es/sigeco/default.htm
- Moreno, A., Valero-Garcés, B.L., Jiménez-Sánchez, M., Domínguez-Cuesta, M.J., Mata, M., Navas,
 A., González-Sampériz, P., Stoll, H., Farias, P., Morello, M., Corella, J., Rico, M. 2010. The
 last deglaciation in the Picos de Europa National Park (Cantabrian Mountains). Journal of
 Quaternary Sciences 25, 1076–1091.
- 590 Murray, A.S., Wintle, A.G. 2000. Luminescence dating of quartz using an improved single-aliquot 591 regenerative-dose protocol. Radiation Measurements 32, 57–73.
- 592 Murray, A.S., Wintle, A.G. 2003. The single aliquot regenerative dose protocol: Potential for 593 improvements in reliability. Radiation Measurements 37, 377–381.
- Mylroie, J.E. 1984. Pleistocene climatic variation and cave development: Norsk Geografisk Tidsskrift
 38, 151–156.
- 596 Mylroie, J.E., Mylroie, J.R. 2004. Glaciated karst: how the Helderberg plateau revised the geological 597 perception. Northeastern Geology and Environmental Sciences 26, 82–92.
- Nieuwendam, A., Ruiz-Fernández, J., Oliva, M., Lopez, V., Cruces, A., Freitas, M. da C. 2015.
 Postglacial landscape changes and cryogenic processes in Picos de Europa (northern Spain)
 reconstructed from geomorphological mapping and microstructures on quartz grains.
 Permafrost and Periglacial Processes 27, 96–108.
- NGRIP members 2004. High-resolution record of Northern Hemisphere climate extending into the last
 interglacial period. Nature 431, 147–151.
- Osmaston H. 2005. Estimates of glacier equilibrium line altitudes by the Area×Altitude, the
 Area×Altitude Balance Ratio and the Area×Altitude Balance Index methods and their
 validation. Quaternary International 138–139, 22–31.
- Pellitero, R., Rea, B.R., Spagnolo, M., Bakke, J., Ivy-Ochs, S., Lukas, S., Ribolini, A. 2015. A GIS tool
 for automatic calculation of glacier equilibrium-line altitudes. Computers & Geosciences 82,
 55–62.
- Pellitero, R., Rea, B.R., Spagnolo, M., Bakke, J., Ivy-Ochs, S., Frew, C.R., Hughes, P., Ribolini, A.,
 Lukas, S., Renssen, H. 2016. GlaRe, a GIS tool to reconstruct the 3D surface of
 palaeoglaciers. Computers & Geoscience 94, 77–85.
- Pinto-Llona, A.C., Clark, G., Karkanas, P., Blackwell, B., Skinner, A.R., Andrews, P., Reed, K., Miller,
 A., Macías-Rosado, R., Vakiparta, J. 2012. The Sopeña Rockshelter, a New Site in Asturias
 (Spain) bearing evidence on the Middle and Early Upper Palaeolithic in Northern Iberia.
 Munibe (Antropologia-Arkeologia) 63, 45–79.

- Plan, L., Filipponi, M., Behm, M., Seebacher, R., Jeutter, P., 2009. Constraints on alpine
 speleogenesis from cave morphology A case study from the eastern Totes Gebirge
 (Northern Calcareous Alps, Austria). Geomorphology 106, 118–129.
- Prescott, J.R., Hutton, J.T. 1994 Cosmic ray contributions to dose rates for luminescence and ESR
 dating: large depths and long term variations. Radiation Measurements 23, 497–500.
- Railsback, L.B., Gibbard, P.L., Head, M.J., Voarintsoa, N.R.G., Toucanne, S. 2015. An optimized
 scheme of lettered marine isotope substages for the last 1.0 million years, and the
 climatostratigraphic nature of isotope stages and substages. Quaternary Science Reviews
 111, 94–106.
- Rea, B.R. 2009. Defining modern day Area-Altitude Balance Ratios (AABRs) and their use in glacier climate reconstructions. Quaternary Science Review 28, 237–248.
- Reimer, P., Bard, E., Bayliss, A., Beck, J.W., Blackwell, P., Ramsey, C.B., Buck, C., Cheng, H.,
 Edwards, R.L., Friedrich, M., Grootes, P.M., Guilderson, T.P., Haflidason, H., Hajdas, I.,
 Hatt_e, C., Heaton, T.J., Hoffmann, D.L., Hogg, A.G., Hughen, K., Kaiser, K.F., Kromer, B.,
 Manning, S.W., Niu, M., Reimer, R.W., Richards, D.A., Scott, E.M., Southon, J., Staff, R.A.,
 Turney, C.S.M., Van der Plicht, J. 2013. Intcal13 and Marine13 radiocarbon age calibration
 curves 0–50,000 years cal BP. Radiocarbon 55, 1869–1887.
- Rodríguez-Rodríguez, L., Jiménez-Sánchez, M., Domínguez-Cuesta, M.J., Aranburu, A. 2014.
 Research history on glacial geomorphology and geochronology of the Cantabrian Mountains,
 north Iberia (43–42°N/7–2°W). Quaternary International 364, 6–21.
- Rodríguez-Rodríguez, L., Jiménez-Sánchez, M., Domínguez-Cuesta, M.J., Rinterknecht, V., Pallàs,
 R., Bourlès, D. 2016. Chronology of glaciations in the Cantabrian Mountains (NW Iberia)
 during the Last Glacial Cycle based on in situ-produced 10Be. Quaternary Science Reviews
 138, 31–48.
- Ruiz-Fernández, J., Oliva, M., Cruces, A., Lopes, V., Freitas, M. da C., Andrade, C., GarcíaHernández, C., López-Sáez, J.A., Geraldes, M. 2016. Environmental evolution in the Picos de
 Europa (Cantabrian Mountains, SW Europe) since the Last Glaciation. Quaternary Science
 Reviews 138, 87–104.
- Santos-González, J., Redondo-Vega, J.M., González-Gutiérrez, R.B., Gómez-Villar, A. 2013. Applying
 the AABR method to reconstruct equilibrium-line altitudes from the last glacial maximum in the
 Cantabrian Mountains (SW Europe). Palaeogeography, Palaeoclimatology, Palaeoecology
 387, 185–199.
- 649 Serrano, E., González-Trueba, J.J., González-García, M. 2012. Mountain glaciation and paleoclimate
 650 reconstruction in the Picos de Europa (Iberian Peninsula, SW Europe). Quaternary Research
 651 78, 303–314.
- Serrano, E., González-Trueba, J.J., Pellitero, R., Gomez-Lende, M. 2017. Quaternary glacial history of
 the Cantabrian Mountains of northern Spain: a new synthesis, in: Hughes, P.D., Woodward,
 J.C. (Eds.), Special Publications, 433: Quaternary Glaciation in the Mediterranean Mountains,
- 655 Geological Society of London, London, pp. 55–85.

- Skoglund, R.Ø., Lauritzen, S.E., Gabrovšek, F. 2010. The impact of glacier ice-contact and subglacial
 hydrochemistry on evolution of maze caves: A modelling approach. Journal of Hydrology 388,
 157–172.
- Smart, P.L. 1986. Origin and development of glacio-karst closed depressions in the Picos de Europa,
 Spain. Zeitschrift für Geomorphologie 30, 423–443.
- Stuiver, M., Reimer, P.J. 1993. Extended 14C data base and revised CALIB 3.0 14C age calibration
 program. Radiocarbon 35, 215–230.
- Telbisz, T., Tóth, G., Ruban, D.A., Gutak, J.M. 2019. Notable glaciokarst of the World. In: Veress, M.
 Telbisz, T., Tóth, G., Lózy, D., Ruban, D.A., Gutak, J.M. (Eds), Glaciokarst. Springer, Cham,
 373–499.
- Tîrlă, L., Drăguşin, V., Bajo, P., Covaliov, S., Cruceru, N., Ersek, V., Hanganu, D., Hellstrom, J.,
 Hoffmann, D., Mirea, I., Sava, T., Sava, G., Şandric, I. In press. Quaternary environmental
 evolution in the South Carpathians reconstructed from glaciokarst geomorphology and
 sedimentary archives. Geomorphology, 354: 107038.
- Uzquiano, P., Yravedra, J., Ruiz Zapata, B., Gil García, M.J., Sesé, C., Baena, J. 2012. Human
 behaviour and adaptations to MIS 3 environmental trends (>53–30 ka BP) at Esquilleu cave
 (Cantabria, northern Spain). Quaternary International 252, 82–89.
- Uzquiano, P., Ruiz-Zapata, M.B., Gil-García, M.J., Fernández, S., Carrion, J.S. 2016. Late Quaternary
 developments of Mediterranean oaks in the Atlantic domain of the Iberian Peninsula: The case
 of the Cantabrian region (N Spain). Quaternary Science Reviews 153, 63–77.
- Villa, E., Stoll, H., Farias, P., Adrados, L., Edwards, R.L., Cheng, H. 2013. Age and significance of the
 Quaternary cemented deposits of the Duje Valley (Picos de Europa, Northern Spain),
 Quaternary Research 79, 1-5.
- Weremeichik, J., Mylroie, J. 2014. Glacial Lake Schoharie: an investigative study of glaciolacustrine
 lithofacies in caves, Helderberg Plateau, Central New York. Journal of Cave and Karst Studies
 76, 127–138.
- Yravedra, J., Gómez-Castanedo, A., Aramendi Picado, J., Baena Preysler, J. 2014. Specialised
 hunting of Iberian ibex during Neanderthal occupation at El Esquilleu Cave, northern Spain.
 Antiquity 88, 1035–1049.
- 685 Yravedra, J., Cobo-Sánchez, L. 2015. Neanderthal exploitation of ibex and chamois in southwestern
 686 Europe. Journal of Human Evolution 78, 12–32.

687 FIGURE CAPTIONS

Figure 1. (A) Map of the SW of Europe with the location of Picos de Europa (northern Spain). (B) DEM of the N of Spain including the local Maximum Ice Extent (local MIE) recorded by former glaciers in the Cantabrian Mountains (Rodríguez-Rodríguez et al., 2014). (C) DEM of the three massifs of Picos de Europa with the position of the study cave (named Hayéu l'Osu) in the surroundings of Covadonga Lakes (Enol and Ercina Iakes), within the NW massif. Local glacial maximum (Local GM) extent according to Alonso (1998), Serrano et al. (2012, 2017) and Rodríguez-Rodríguez et al. (2014).
Documented cave data is courtesy of the speleological groups reported in Ballesteros et al. (2019a).

Figure 2. Main geomorphological features of the study area (which position is shown in Figure 1)
projected on the hillshade digital model. Geomorphology is after Ballesteros et al. (2015, 2017,
2019b), who studied the Pozu Llucia and Torca La Texa shafts.

Figure 3. (A) Aerial photo of the Spanish PNOA 2017 Program projected on a digital elevation model
of the Western Massif of Picos de Europa (www.cnig.es; last accessed on May 2019); the star marks
the location of Hayéu l'Osu Cave. (B) Topographic restitution of glaciers for the local GM stage,
showing the location of the Equilibrium Line Altitude (ELA) and the main ice flow pathways: 1Ordiales, 2-Hunhumia, 3-Pomperi, 4-El Bricial (4'-Vega de Enol), 5-Ercina, and 6-Belbín.

Figure 4. (A) Geomorphological map of Hayéu l'Osu Cave showing the location of the paleontological sites, the position of dating samples and pictures shown in C, D, E and F. (B) Cave longitudinal profile (P indicates the depth of each shaft in meters, from its top to bottom). (C) Vadose canyon of Stream Gallery showing active channel deposits. (D) Camp Chamber dominated by fluvial terrace deposits. (E) Vadose canyon of Stream Gallery showing fluvial terrace deposits cemented by flowstone and perched above the active channel. (F) Slackwater deposits from the central part of the cavity.

Figure 5. Comparison of the enamel fragment from the Stream Gallery of the Hayéu l'Osu Cave (Figure 4A) with the upper first molars of some extinct and living arvicolines from the Cantabrian Region. Squares in occlusal view (a'-f') are highlighting the buccal salient angle 3 (BSA3). Squares in buccal view (a''-f'') are highlighting the morphology at the base of the BSA3. (A) Enamel fragment found in HAY–9 sample from the study cave. (B) Rooted molars of *Pliomys coronensis* from La Güelga Cave (Figure 1C). (C) Rooted molar of *Clethrionomys glareolus*. Evergrowing molars of: (D) *Arvicola monticola*, (E) *Microtus lavernedii*, and (F) *Microtus lusitanicus*.

Figure 6. Chamois (*Rupicapra pyrenaica*) sites from Hayéu l'Osu Cave: (A) Debris deposit with bone
remains (Figure 7A and C-K) in the Llamaeyu Gallery (Figure 4). (B) Location of a scapula (Figure 7B)
placed on alluvial deposits of the Stream Gallery (Figure 4).

Figure 7. Chamois (*Rupicapra pyrenaica*) dental and postcranial remains from Hayéu l'Osu Cave. (A) Left M³ in labial view. (B) Young left scapula in lateral view. (C) Fragment of limb bone diaphysis showing erosion due to transportation by water. (D) Young left radius diaphysis in anterior view showing teeth marks (indicated by white arrows) produced by a small carnivore. (E) Adult right

metacarpal in anterior view. (F) Young left metacarpal in anterior view. (G) Adult proximal phalanx in
anterior view. (H) Adult middle phalanx in anterior view. (I) Adult distal phalanx in lateral view. (J) Adult
right calcaneus in anterior view. (K) Adult right talus in anterior view. Scale bars are in cm.

Figure 8. Selected sites of Hayéu l'Osu Cave for U-Th and OSL dating: (A) Toppled stalagmite and flowstone originally located on top of slackwater deposits which were subsequently eroded after 263 ka. (B) Perched flowstone precipitated on a first alluvial sequence. (C, D) Second alluvial sequence deposited on flowstone. (E) Second alluvial sequence covered by speleothems. (F) Zoom of sandy layers dated through OSL. (G) Third alluvial pebble sequence with a sandy layer interbedded and dated by means of OSL. Picture locations are shown in Figures 4A and 8.

Figure 9. Cross-section of the surroundings of Covadonga Lakes showing the position of Hayéu l'Osu Cave, Enol and Ercina lakes and Vega de Comeya border polje, which sedimentary infill was previously described and dated in Jiménez-Sánchez and Farias (2002). The position of the crosssection is shown in Figure 2. Bedrock geology is based on Ballesteros et al. (2015). Dates performed in Hayéu l'Osu Cave are detailed in Tables 1, 2 and 3.

737 Figure 10. Kernel density functions of the OSL, U-Th and radiocarbon ages obtained in the Hayéu 738 l'Osu Cave record, including previous ages from the Vega de Comeya and Enol lacustrine sediments 739 (Moreno et al., 2009; Jiménez-Sánchez et al., 2013). Flowstone precipitation ascribable to the end of 740 MIS 6 fossilized the first detrital sequence identified inside the study cave. The second important 741 episode of cave sediment infill occurred between ca. 98 and 60 ka (phase 1) as glacio-fluvial 742 sediments from a previous glaciation (most likely MIS 5d glaciation) were washed into the cave. The 743 Vega de Comeya sequence indicates that the local Glacial Maximum (local GM) was attained in this 744 area around 45 ± 3 ka (Jiménez-Sánchez et al., 2013), during MIS 3c (phase 2, 48-42 ka). By ~38 ka 745 BP glaciers had already retreated from the Enol Lake depression, allowing proglacial lacustrine 746 sedimentation (Moreno et al., 2010). The retreat of the Ercina glacier front is indirectly recorded in 747 Hayéu l'Osu Cave by the third sedimentary sequence (phase 3, 42-38 ka), suggesting that the Ercina 748 glacier front had also suffered considerable retreat from its previous GM position. The cave entrance 749 and its neighboring areas remained ice-free (phase 4) after ca. 37 ka until, at least, the glacier 750 advance linked to the global LGM of MIS 2 whose extent remains unknown in Picos de Europa (LGM 751 age interval according to Clark et al., 2009).

752 Figure 11. Paleoenvironmental evolution of the Covadonga Lakes surroundings during MIS 5d-3a 753 illustrated using the cross-section shown in Figure 8 (same legend; see Figure 2 for cross-section 754 location). (A) Phase 1: the study cave recorded detrital sediment input at 98-60 ka, evidencing that 755 the cave setting was an ice-free alpine environment and that eventual glacial fronts were located 756 above 1400 m altitude. (B) Phase 2: glaciers attained their local GM extent at 45 ± 3 ka, causing 757 proglacial lacustrine sedimentation at Vega de Comeya (Jiménez-Sánchez et al., 2013). The Ercina 758 glacier flowed down to 1040 m asl, and La Picota moraine was formed between Ercina and Enol 759 glaciers (Moreno et al., 2010). (C) Phase 3: onset of a general trend of glacial retreat according to 760 Jiménez-Sánchez and Farias (2002). Glaciers receded from the Enol Lake depression at ca. 38 cal ka 761 BP, while Hayéu l'Osu Cave recorded detrital sedimentation since 36 ± 3 ka (HAY-7). (D) Phase 4: 762 occurrence of ice-free temperate environment inhabited by chamois populations.

Figure 12. AMS radiocarbon dates of chamois remains from Llamaeyu and Stream galleries of Hayéu 1'Osu Cave, projected over the Greenland δ^{18} O ice core record (NGRIP, 2004). MIS 3 substages according to Railsback et al. (2015).





Karst features



Glacial features

- ▲ Horn
- –---- Arête
- ---- Cirque
- ° ၊ Till

Other features

- Glaciokarst closed depression
 - Lacustrine, kame and peat and alluvial deposits
 - Anthropic deposit
 - River





 active channel deposit
 active cave stream
 fuvial terrace deposit
 active channel deposit
 active channel deposit













A Phase 1 (98-60 ka; MIS 5d-4)





Table 1.²³⁸U-²³⁰Th dating results of Hayéu l'Osu Cave. The error is 2σ . Sample locations are shown in Figures 4A and 8A-C.

Sample	²³⁸ U	(ppb)	²³² Th	(ppt)	²³⁰ Th (atomi	/ ²³² Th ic ·10⁻6)	δ ²³⁴ (meas	¹ U* ured)	²³⁰ Th / ²³⁸	U (activity)	²³⁰ Th A (uncorr	ge (yr) ected)	δ ²³⁴ U (corre	^{Initial**} cted)	²³⁰ Th Age (corr	ected)
HAY-6	65.4	±0.1	8643	±173	293	±6	1994.3	±3.5	2.3433	±0.0072	132630	±732	2892	±10	131570	±1010
HAY-12	28.2	±0.0	48898	±980	16	±0	644.1	±3.4	1.7072	±0.0068	286442	±4903	1353	±68	262977	±17735
HAY-13	95.4	±0.3	66643	±1342	34	±1	793.4	±5.1	1.4240	±0.0063	144811	±1451	1161	±24	134982	±7103

 $\delta^{234}U = ([^{234}U/^{238}U]_{activity} - 1) \cdot 1000. ** \delta^{234}U_{initial}$ was calculated based on the ²³⁰Th age (T), i.e., $\delta^{234}U_{initial} = \delta^{234}U_{measured} \cdot e^{\lambda_{234}xT}$. Corrected ²³⁰Th ages assume the initial ²³⁰Th/²³²Th atomic ratio of $4.4 \pm 2.2 \cdot 10^{-6}$. Those are the values for a material at secular equilibrium, with the bulk Earth ²³²Th/²³⁸U value of 3.8. The errors are arbitrarily assumed to be 50%. *** B.P. stands for "Before Present" where the "Present" is defined as the year 1950 A.D.

Sample	HAY-07	HAY-08	HAY-09	HAY-10	HAY-11
Aliquots (n)	28	4	35	32	35
Equivalent doses (ED) (Gy)	48.4 ± 3.4	35.2 ± 8.4	47.8 ± 3.4	38.3 ± 2.8	44.3 ± 4.3
⁴⁰ K (Bq kg ⁻¹)	393 ± 13	50 ± 12	79 ± 17	71 ± 14	55 ± 9
²³⁸ U (Bq kg ⁻¹)	17 ± 2	18 ± 1	15.6 ± 0.8	18 ± 2	15 ± 2
²³⁵ U(Bq kg ⁻¹)	0 ± 0	0 ± 0	0 ± 0	0 ± 0	3.3 ± 2
²³² Th (Bq kg ⁻¹)	3.3 ± 0.5	0 ± 0	5.5 ± 0.8	0 ± 0	0 ± 0
Beta radiation dose	0.89 ± 0.04	0.31 ± 0.04	0.35 ± 0.04	0.31 ± 0.04	0.29 ± 0.04
Gamma radiation dose	0.39 ± 0.02	0.17 ± 0.01	0.21 ± 0.02	0.16 ± 0.02	0.17 ± 0.01
Cosmic radiation dose	0.06 ± 0.003	0.06 ± 0.003	0.06 ± 0.003	0.07 ± 0.003	0.07 ± 0.003
Dose rate (DR) (Gy ka ⁻ 1)	1.34 ± 0.4	0.54 ± 0.04	0.62 ± 0.05	0.55 ± 0.06	0.53 ± 0.04
Overdispersion (%)	26.3 ± 5.8	38.9 ± 20.0	24.3 ± 5.5	26.1 ± 6.4	38.8 ± 8.7
Age (ka)	36 ± 3	65 ± 16	77 ± 8	70 ± 9	84 ± 10

Table 2. Optically Stimulated Luminescence (OSL) dates from alluvial sandy sediment samples of Hayéu l'Osu Cave. Figures 4A, 8 and 9C-G show the position of the samples in the study cave.

Table 3. Calibrated AMS radiocarbon dates of chamois (*R. pyrenaica*) assemblage of Hayéu l'Osu Cave. The position and remains of the paleontological sites is shown in Figures 4A, 7 and 8.

Sample Paleontological sites		Conventional age (years BP)	Calendar age (2σ) (years BP)		
HAY-31	Stream Gallery	32220 ± 440	37534–35139		
HAY-36	Llamayeu Gallery	30440 ± 360	35076–33836		

Phase	Age (ka)	MIS	Main event	Paleoenvironment at a reference altitude of 1400 m	Evidence from the study cave	Evidence from previous works
1	98-60	5c–4	Glacier retreat	Area with rocky outcrops and fluvioglacial sediments. Probably glaciers in upper areas. Relative cold conditions	Deposition of the second alluvial sequence in the cave Occurrence of <i>Pliomys coronensis</i>	
2	48-42	3c	Local glacial maximum advance	Area covered by glaciers		Glacio-lacustrine sedimentation in Vega de Comeya border polje at 850 m asl (Jiménez-Sánchez et al., 2013)
3	42-38	3b	Onset of glacier retreat	Probably, area covered by glaciers		Glacio-lacustrine sedimentation at Enol Lake at 1075 m asl (Moreno et al., 2009)
4	37-33	3a	Glacier retreat	Area with rocky outcrops and fluvioglacial sediments; relative cold conditions	Deposition of the third alluvial sequence in the cave Occurrence of <i>Rupicapra pyrenaica</i>	

Table 4. Synthesis of the paleoenvironmental evolution of Picos de Europe during MIS 5c to 3. See Section 5 and Figures 10 and 11 for details.