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JUNTA DE ANDALUCIA
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Menga es una publicación anual del Conjunto Arqueológico Dólmenes de Antequera (Consejería de Cultura de la Junta de Andalucía). Su objetivo es la difusión internacional de trabajos de investigación científicos de calidad relativos a la Prehistoria de Andalucía.

Menga se organiza en cuatro secciones: Dossier, Estudios, Crónica y Recensiones. La sección de Dossier aborda de forma monográfica un tema de investigación de actualidad. La segunda sección tiene un propósito más general y está integrada por trabajos de temática más heterogénea. La tercera sección denominada como Crónica recogerá las actuaciones realizadas por el Conjunto Arqueológico Dólmenes de Antequera en la anualidad anterior. La última sección incluye reseñas de libros y otros eventos (tales como exposiciones científicas, seminarios, congresos, etc.).

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Menga is a yearly journal published by the Dolmens of Antequera Archaeological Site (the Andalusian Regional Government Ministry of Culture). Its aim is the international dissemination of quality scientific research into Andalusian Prehistory.

Menga is organised into four sections: Dossier, Studies, Chronicle and Reviews. The Dossier section is monographic in nature and deals with current research topics. The Studies section has a more general scope and includes papers of a more heterogeneous nature. The Chronicle section presents the activities undertaken by the Dolmens of Antequera Archaeological Site in the previous year. The last section includes reviews of books and events such as scientific exhibitions, conferences, workshops, etc.

Menga is open to original and unpublished papers that have not been submitted for publication to other journals. All original manuscripts will be submitted to an external and anonymous peer-review process before being accepted for publication. In exceptional cases, the editorial board will consider the publication of Spanish and English translations of already published papers on the basis of their interest and/or the difficulty of access to their content.

Ídolo placa procedente del dolmen 40 de la necrópolis de Cabezas Rubias (Huelva).
Foto: Miguel A. Blanco de la Rubia.

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Sampling for OSL dating inside the socket of Menga's first pillar, March 2014. From left to right: Katerina Theodorakopoulou, Constantin Athanassas and Victoria Pérez Nebreda. Photo: Leonardo García Sanjuán.

TESTING THE POTENTIAL OF OPTICALLY STIMULATED LUMINESCENCE (OSL) FOR THE DATING OF THE ANTEQUERA MEGALITHS (MÁLAGA, SPAIN): ASSESSING THE RESULTS OF THE FIRST ROUND OF SAMPLING

Constantin Athanassas[1], Leonardo García Sanjuán[2], Katerina Theodorakopoulou[3], Mayank Jain[4], Reza Sohbati[4], Guillaume Guerin[5] and José Antonio Lozano Rodríguez[6]

Abstract:

The megalithic monuments of Antequera (Málaga, Spain) are among the most significant of Iberia and have become a reference for the study of the megalithic phenomenon worldwide. Despite the long history of their research, dating back to first half of the 19th century, the Antequera megaliths currently lack a robust scientific chronology, which hampers the study of their complex biographies, especially the dates of their construction. Optically Stimulated Luminescence (OSL) is a powerful tool in the toolbox of direct monument dating techniques. It is expected that OSL dating can provide the chronological resolution needed to decipher the chronology of the Antequeran megaliths. Here we present the results of the first round of sampling that aimed at assessing the suitability of the materials involved. Ultimately, results would indicate the most effective sampling strategy for a successful OSL dating attempt at a later stage.

Keywords: Megaliths, Neolithic, Numerical Chronology, Optically Stimulated Luminiscence.

ENSAYO DEL POTENTIAL DE LA LUMINISCENCIA POR ESTIMULACIÓN ÓPTICA (OSL) PARA LA DATACIÓN DE LOS MEGALITOS DE ANTEQUERA (MÁLAGA, ESPAÑA): VALORACIÓN DE LOS RESULTADOS DE LA PRIMERA RONDA DE MUESTREO

Resumen:

Los monumentos megalíticos de Antequera (Málaga, España) se encuentran entre los más importantes de la Península Ibérica, y se han convertido en referencia para el estudio del fenómeno megalítico a escala mundial. A pesar la larga historia de investigación, que se remonta a la primera mitad del siglo XIX, los megalitos antequeranos carecen en la actualidad de una cronología científica robusta, lo cual dificulta el estudio de sus complejas biografías, especialmente las fechas de construcción. La Luminiscencia por Estimulación Óptica (OSL) es una potente herramienta dentro del arsenal de técnicas de datación directas de monumentos. Se espera que la datación por OSL pueda proporcionar la resolución cronológica necesaria para descifrar la cronología de los megalitos antequeranos. En este artículo se presentan los resultados de la primera ronda de muestreo, realizada con el objetivo de valorar la adecuación de los materiales implicados. En última instancia, estos resultados indicarán la estrategia de muestreo más efectiva para un intento de datación por OSL que resulte exitosa, y que se encuentra ya en curso.

Palabras clave: Megalitos, Neolítico, Cronología Numérica, Luminiscencia por Estimulación Óptica.

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1. INTRODUCTION

The megalithic monuments of Antequera (Málaga, Spain), namely Menga, Viera and El Romeral, have a long history of archaeological research. After the early work by Rafael Mitjana y Ardison (1847), and throughout the 19th century, Menga became one of the most internationally-known megalithic monuments of Europe, as a recent review has shown (Sánchez-Cuenca López, 2011). Viera and El Romeral were discovered in 1903-1904, and soon they also became the focus of major scientific interest within the then rising Spanish archaeology (Gómez-Moreno Martínez, 1905; Mergelina, 1922). The research on Menga and Viera intensified in the last quarter of the 20th century, with various excavations being carried out by the University of Málaga (Ferrer Palma, 1997a; 1997b; Ferrer Palma *et al.*, 2004) and in 2005-2006, with excavations undertaken by the university of Granada (Carrión Méndez *et al.*, 2009; 2010). From today's perspective, the fact that most of these excavations were not followed by fully published post-excavation studies makes the analysis of the complex biographies of these monuments a very difficult task –for a recent discussion see García Sanjuán and Lozano Rodríguez, 2016–.

One of the most pressing problems regarding the study of the Antequeran megaliths is precisely their chronology. In fact, up until 2006 only one ^{14}C date had been published for all three monuments. At the time of writing these lines (April 2016), a total of 18 radiocarbon dates have been published for Menga and Viera, all of them resulting from the research project "Societies, Territories and Landscapes in the Prehistory of Antequera (Málaga)" carried out jointly

Megalith	Context	Laboratory	SampleType	Date BP	Date 2σ Cal BC/AD	Reference
Menga	Atrium (pit)	Ua-24582	Charred material	4935 ± 40	3790-3690 BC	García Sanjuán and Lozano Rodríguez, 2016
Menga	Atrium (pit)	Ua-24583	Charred material	4865 ± 40	3760-3530 BC	García Sanjuán and Lozano Rodríguez, 2016
Menga	Mound (base)	Ua-36216	Charred material	4760 ± 30	3639-3384 BC	García Sanjuán and Lozano Rodríguez, 2016
Viera	Mound (base)	GrN-16067	Charred material	4550 ± 140	3631-2916 BC	Ferrer Palma,1997a
Viera	Chamber	Beta-353820	Faunal remains	4090 ± 30	2860-2500 BC	Aranda Jiménez *et al.*, 2013
Viera	Chamber	Beta-353822	Faunal remains	3580 ± 30	2020-1880 BC	Aranda Jiménez *et al.*, 2013
Menga and Viera	Vicinity	Beta-412999	Human bone	1790 ± 30	133-330 AD	Aranda Jiménez *et al.*, 2015
Menga and Viera	Vicinity	Beta-413000	Human bone	1730 ± 30	243-386 AD	Aranda Jiménez *et al.*, 2015
Menga and Viera	Vicinity	Beta-413001	Human bone	1770 ± 30	138-345 AD	Aranda Jiménez *et al.*, 2015
Menga and Viera	Vicinity	Beta-413002	Human bone	1700 ± 30	253-406 AD	Aranda Jiménez *et al.*, 2015
Menga and Viera	Vicinity	Beta-413003	Faunal remains	1500 ± 30	432-639 AD	Aranda Jiménez *et al.*, 2015
Menga and Viera	Vicinity	Beta-413004	Faunal remains	1540 ± 30	426-588 AD	Aranda Jiménez *et al.*, 2015
Menga and Viera	Vicinity	Beta-413005	Faunal remains	1490 ± 30	436-644 AD	Aranda Jiménez *et al.*, 2015
Menga and Viera	Vicinity	Beta-413006	Faunal remains	1520 ± 30	428-609 AD	Aranda Jiménez *et al.*, 2015
Menga	Atrium	CNA-1174	Human bone	1250 ± 35	676-871 AD	Díaz-Zorita Bonilla and García Sanjuán, 2012
Menga	Atrium	CNA-1173	Human bone	1100 ± 45	783-1022 AD	Díaz-Zorita Bonilla and García Sanjuán, 2012
Menga	Shaft	Beta-322311	Faunal remains	120 ± 30	1679-1940 AD	Riquelme Cantal, 2012
Menga	Shaft	Beta-322312	Faunal remains	150 ± 30	1667-1951 AD	Riquelme Cantal, 2012

Tab. 1. Published ^{14}C dates for Menga and Viera.

between 2013 and 2018 by the Universities of Seville, Alcalá de Henares, Granada and Southampton (Tab. 1). Although a significant improvement, this is still a clearly insufficient basis to interpret the biography of monuments that were founded in the Late Neolithic period (Menga and Viera) or the Copper Age (El Romeral), and then went on to be used through the Bronze Age, Iron Age, Antiquity, Middle Ages and Modern History. The available numerical chronology is particularly weak as far as the early time of their use (Neolithic and Copper Age) is concerned, especially for El Romeral *tholos*, for which not a single date has yet been obtained.

In the case of Menga, three ^{14}C dates of Neolithic chronology have been published so far, falling into the period *c.* 3800-3400 cal BC. Neither of them was obtained from the sockets of the orthostats or pillars and therefore they can only provide a *post quem* chronology for the construction of the monument. The building of Menga is of the greatest interest in light of its orientation (and visual connection) towards La Peña de los Enamorados, a prominent landscape feature of the Antequera plain, which suggest that the design of this extraordinary monument was somewhat related to previous activity that took place at La Peña (García Sanjuán and Wheatley, 2009; García Sanjuán *et al.*, 2011; 2015). Establishing the date for the construction of Menga is also important to understand its possible connection with the earthquake recorded at of El Toro cave, distant only a few kilometers to the south (García Sanjuán and Lozano Rodríguez, 2016).

Given the present unavailability of samples datable by radiocarbon, and the importance of advancing towards a reliable chronology of the foundation of all three Antequeran megaliths, in 2013 the Universities of Seville (Spain) and Aix-en-Provence (France), together with the Conjunto Arqueológico Dólmenes de Antequera agreed to collaborate in order to explore the suitability of the building materials of the megaliths for Optically Stimulated Luminescence (OSL) dating, and the potential of this method to establish their construction dates. This collaboration is currently funded by two separate research projects: the project *Works of the Old Men: Chronology and Spatial Analysis of Prehistoric Stone-Built Structures in the Peri-Mediterranean*, led by the Centre Européen de Recherche et d'Enseignement des Géosciences de l'Environnement (Aix- Marseille University) (2013-2015) and the project *Nature, Society and Landscape: High-Resolution Archaeological Research of the Antequera Megalithic Landscape*, led by the Department of Prehistory and Archaeology of the University of Seville (2014-2017).

In this paper we present the results of the first round of dating carried out in all three Antequeran megaliths through 2014. Our preliminary experiments highlight the mineralogical suitability of the building materials for OSL dating on the one hand, but on the other hand underline the need for additional work to more accurately constrain factors such as the local environmental dose rate and OSL signal resetting in the rock surfaces toward a reliable numeric chronology of the megaliths. Although essentially negative towards the final end of establishing the construction dates for the megalithic monuments, this first round of sampling has provided crucial clues to undertake the second round of sampling, which is currently under way. This work has been carried out as part of a larger sampling effort that has also included the *tholos* of La Pastora, which is part of the Copper Age settlement of Valencina de la Concepción (Seville), and a megalithic construction of unknown date recently re-discovered in the historical centre of Carmona (Seville)[1].

2. METHODS AND MATERIALS

OSL dating is based on the emission of light, termed luminescence, from irradiated minerals when they are submitted to stimulation with artificial light sources (e.g. lasers and LEDs) (Huntley *et al.*, 1985). Natural ionizing radiation is omnipresent in the environment and involves radioactivity, delivered by the emission of α, β and γ particles from decaying atomic nuclei of ^{235}U, ^{238}U, ^{232}Th (and their daughter products) and ^{40}K, and the cosmic rays. Natural ionizing radiation brings minerals to a long excitation condition. At the excited state electron charge builds up in intrinsic mineral imperfections (traps); the longer the excitation stage, the more the amount of trapped electrons in the crystal defects.

[1] Although this structure was first discovered in the early 20th century and was listed as "dolmen" given its apparent resemblance with some local prehistoric architectures, recent re-excavations by Ricardo Lineros Romero (Museo de la Ciudad de Carmona) date it to a post-Roman period.

Step	Treatment	Measurement	Explanation
1	Dose D_i		Give regenerated dose (no irradiation before measurement of the natural signal)
2	PH (240ºC, 10 sec)		Thermally erase charge from geologically unstable traps
3	OSL 240ºC, 10 sec)	L_{OSL}	Registering the OSL signal (at elevated temperature so as to maintain unstable traps inactive)
4	Test Dose TD		Give the test dose
5	PH (160ºC, 0 sec)		Thermally erase charge from geologically unstable traps
6	OSL (125ºC, 100 sec)	T_{OSL}	Registering the test dose OSL signal
7	Go to Step 1		Repeat sequence with a different regenerated dose

Tab. 2. The single aliquot regenerated (SAR) dose protocol and measurement settings for conventional quartz-OSL dating (Murray & Wintle, 2000).

An OSL age is the ratio of the received radiation dose over the rate of exposure to the natural ionizing radiation. State-of-the-art devices, known as "luminescence readers", can record the OSL signal by submitting the minerals to stimulations with artificial light. The natural dose, as also called the paleodose, can be evaluated by interpolating the natural OSL signal intensity onto an experimental graph consisting of data points which pair artificial doses of known size and corresponding OSL signal intensities. That graph is termed the "dose response curve".

This method of paleodose estimation is represented by the single aliquot regenerated (SAR) protocol (Murray and Wintle, 2000). SAR protocol (Tab. 2) allows replicate measurements on separate subsamples (aliquots) allowing recording the variation of the paleodose, as well as recording any variations in the sensitivity of quartz taking place as the measurements progress. Along with quartz, feldspars are the most suitable minerals in OSL dating.

Assessment of the dose rate includes estimation of radioelement concentrations either by typical methods used in analytical chemistry (i.e. Neutron Activation Analysis, NAA or Inductively Coupled Plasma Mass Spectrometry, ICP-MS) or particle-emission counting/spectrometry techniques. Radioelemental concentrations are then converted to dose rate units using specific conversion factors (Guerin *et al.*, 2011). An alternative way to determine dose rates is to measure it on the site using "dosimeters". Dosimeters are crystalline pellets of minerals especially sensitive to natural radioactivity.

In a fashion similar to the dating of sediments, OSL also can also be employed for the numeric dating of archaeological monuments (Greilich *et al.*, 2005; Greilich and Wagner, 2006) by assessing the time elapsed since the last exposure of the surface of the building material to sunlight. OSL dating of ancient monuments is based on the hypothesis that the stone surfaces (e.g. masonry, slabs, orthostats and pillars) would be sufficiently exposed to daylight before they are built together to construct the monument (Sohbati *et al.*, 2011; 2012; Liritzis, 2011). Given that a sufficient quantity of quartz grains can be collected from the surface of the slab, standard OSL dating methods can be used to produce a numeric date for the monument.

On Menga, sampling focused on the debris filling up the pillar sockets (Fig. 1a), presuming that its clasts would have received enough daylight during cutting and backfilling, as it has been shown in similar studies in Iberia (Calado *et al.*, 2003; López-Romero, 2011), whereas in the case of Viera and El Romeral mortar was sampled from the slab/orthostat joints and between the slabs respectively. The Carmona underground structure was deemed ideal for surface dating of the calcarenite cobbles in the corridor walls. Dosimeters were left in the sample holes for several weeks to record the environmental dose rate on-site. Preparation of samples and measurement of the paleodose were conducted by the first author at Risø National Laboratory, DTU, Roskilde, Denmark.

Evaluation of the paleodose relied on quartz, isolated following the typical chemical process (c.f. Athanassas *et al.*, 2012). Grain-sizes of 90-125 μm were involved in the measurements. Moisture content was estimated to 5%, and the attenuation factor considered for this grain size was 0.9 (Prescot and Hutton, 1994). The unit used for luminescence measurements was a RISØ TL-DA-15 reader. In order to estimate the intensity of the sensitivity-corrected OSL, SAR (Murray and Wintle, 2000) was used in its original form (Tab. 2).

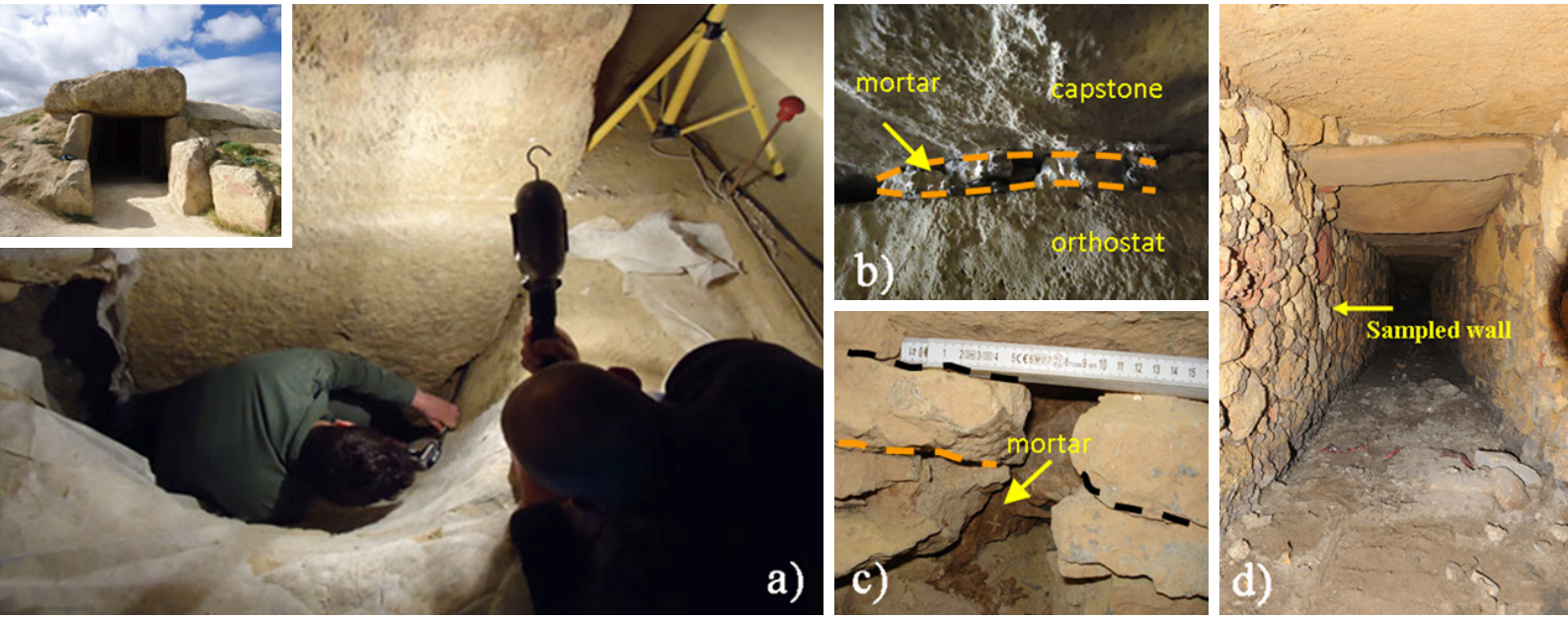

Fig. 1. a) Sampling infill debris in an excavated pillar socket of Menga. b) Clay mortar between slab and orthostat in Viera; c) Clay mortar as binging material of slabs in El Romeral; d) Corridor wall in Carmona megalithic structure consisting of calcarenite cobbles.

3. DISCUSSION AND CONCLUSIONS

The paleodose of several quartz aliquots from Menga infill were widely distributed (Fig. 2a). Noticeably, this broad range of paleodoses suggests either that the debris clasts were not sufficiently exposed to daylight during cutting, transport and trickling of the debris into the ditch, with most of the grains preserving a significant residual dose or that the dose rate is not homogeneous in the deposit. It is therefore difficult to precisely set the OSL chronology of the erection of Menga exclusively relying on the socket infill. Mortars of Viera and El Romeral demonstrated a similarly broad range of paleodoses indicating that any quartz particles in the clayey mortar were not uniformly bleached during the manufacturing process. Therefore numeric dating of the megaliths based on mortars taken from the fabric of their walls might not be an effectual way either.

The above results oblige reconsideration of the sampling strategy on the Antequeran dolmens, avoiding pillar-socket infill and mortars due to poor OSL signal resetting of the containing grains. Thus, the sampling campaign carried out in 2015 aimed for original rock surfaces carved on the orthostats buried under the infill in the case of Menga and Viera, and for masonry slabs in the case of El Romeral. It is more likely that direct OSL dating of the rock and slab surfaces will lead to greater precision as it is expected that the buried surfaces may have received sufficient sunlight exposure during quarrying, transportation and before emplacement.

A similar test was run on calcarenite blocks constituting the corridor walls of the Carmona underground structure. OSL measurements on quartz grains extracted from the surface of the cobbles making up the walls of the structure brought forth quite reproducible paleodoses, suggesting that the cobble surfaces had received sufficient sunlight before they were stacked one on another to build the corridor walls.

In addition, reproducibility in the paleodose would also suggest that possible spoliation of the signal by grains from deeper layers of the stone having geologically old paleodoses owes to be unimportant, as no skew is seen from the probability density diagrams (Fig. 2, b). Nevertheless, if the dose rate (Tab. 3) is taken into the calculations the monument's age becomes prehistoric, a fact that stands in direct contradiction with the current archaeological assumptions.

That deviation necessitates thoroughly probing into the characteristic form of the OSL signal resetting profile (Sohbati *et al.*, 2011) into the rock surface for all of the structures concerned here during the forthcoming re-examination of their OSL chro-

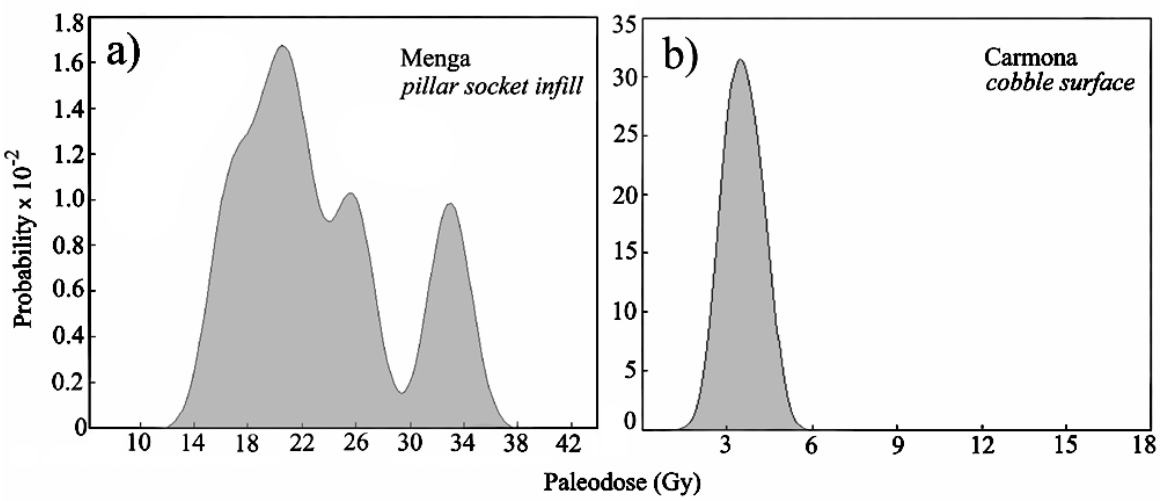

Fig. 2. a) OSL paleodose distribution for the orthostat socket infill from Menga; b) OSL age distribution of quartz extracts from the surface of cobbles making up the corridor walls of Carmona megalithic structure.

nology. Such an analysis would provide additional information on the burial/exposure history of the rock surfaces, in comparison to spotted measurements on the surface, allowing controlling the validity of the expected paleodoses and the forthcoming OSL ages.

In conclusion, the anticipated upcoming sampling campaign at Antequera shall aim for rock surfaces of orthostats buried directly under the infill. Nevertheless, measurement of a substantially larger number of sub samples per mortar sample may allow the engagement of age models (Galbraith *et al.*, 1999) which can be exploited as to identify different populations of paleodoses that comprise the paleodose distribution. More to the point, single-grain OSL dating (Duller, 1996) would perhaps constitute the ultimate tool to the dating of the mortars. The method involves direct stimulation of individual grains so that measurement of the OSL signal of individual grains is possible. In that manner grains fully reset during the manufacturing procedure can be separated from grains with residual paleodoses, allowing in that way the establishment of an OSL age for the mortars as well.

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Material	U(μgxg^{-1})	Th(μgxg^{-1})	K(wt %)	"γ+cosmic" dose rate* (Gyxka^{-1})	Doserate (Gyxka^{-1})	Paleodose (Gy)
Menga infill	0.20±0.02	0.4±0.04	0.05±0.01	0.37±0.02	0.59±0.03	23±20
Carmona wall	0.30±0.04	0.8±0.08	0.06±0.01	0.38±0.02	0.61±0.06	3.7±8

Tab. 3. Indicative (average) radioelemental concentrations, dose rates and paleodoses for the monuments of Menga and Carmona. U, Th & K measured by ICP-MS (ACME Laboratories, BC, Canada). * estimated by dosimeters (IRAMAT, Bordeaux, France).

at the Nordic Laboratory for Luminescence Dating (DLL), RISØ National Laboratory, DTU, Roskilde, Denmark. Dosimetric measurements were obtained by Guillaume Guerin, Institut de Recherche sur les Archéomatériaux, Université Bordeaux Montaigne, Maison de l'Archéologie, Pessac, France.

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Modelado 3D del tercer pilar del dolmen de Menga.
Ortofoto de Victor Baceidero Rodríguez.