Data Article

Equine diet during protohistoric times in the Northeast of the Iberian Peninsula: Stable isotope data (C, N) from bone collagen

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Abstract

The analysis of stable isotopes in bone collagen allows us to infer the diet of the animals studied. This dataset consists of isotopic signatures (δ13C and δ15N) obtained by isotope ratio mass spectrometry from the skeletal remains of 42 equines (horse, ass and their hybrids) from the Can Roqueta site (Sabadell, Northeast Iberian Peninsula). Their chronology spans from Late Bronze Age to Late Roman Period, with particular emphasis on the Early Iron Age. These animals were found in storage silos and graves and were probably sacrificed as ritual offerings. The isotopic values are accompanied by data to assess the quality of the collagen analyzed. This fills a gap in equine isotopic values for this region and chronology, which may be of use to archaeologists interested in the study of livestock management or palaeodiet.

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Keywords: Late bronze age Early iron age Equidae Palaeodiet Stable isotopes Iberian Peninsula
Specifications Table

| Subject | Social Sciences, Archaeology |
|---------|-----------------------------|
| Specific subject area | Application of isotopic biogeochemistry to equine bone remains from an archaeological site, from the Late Bronze Age to the Late Roman period, in the Northeast of the Iberian Peninsula. |
| Type of data | Table |
| How data were acquired | Stable carbon and nitrogen analysis of bone collagen samples. Instruments: Elemental analyzer FlashEA1112 (ThermoFinnigan) coupled via a ConfloIII interface (ThermoFinnigan) to an isotope ratio mass spectrometer MAT253 (ThermoFinnigan). Laboratory: Molecular Palaeontology lab of the University Institute of Geology and Research Support Services, University of A Coruña, Spain. Raw Data format |
| Parameters for data collection | Equine bone remains in grain storage silos and graves from Early Bronze Age to Roman Period, with particular emphasis on the Early Iron Age in Northeast of the Iberian Peninsula. |
| Description of data collection | Isotope analysis ($\delta^{13}$C and $\delta^{15}$N) on bone collagen of protohistoric domestic equines, including horses, asses and a mule. |
| Data source location | City/Town/Region: Can Roqueta, Sabadell, Barcelona Country: Spain Geographic coordinates (latitude, longitude): 41.5380556, 2.137777778 (wgs84) Data accessibility Repository: IsoArch [1] Data identification number: 10.48530/isoarch.2021.008 Direct URL: 10.48530/isoarch.2021.008 Data is available under the Creative commons BY-NC-SA 4.0 license. Related research article | Albizuri Cahadell, S., Grandal-d'Anglade, A., López-Cachero, F. J. Pastures and forages for feeding equids 3000 years ago. Can Roqueta site (Barcelona, Spain) as a model of equine herd management. Forthcoming. |

Value of the Data

- The dataset consists of isotopic signatures ($\delta^{13}$C and $\delta^{15}$N) of 42 bone samples of domestic equines (horse, ass, and their hybrid) from Late Bronze Age to Late Roman Period, with particular emphasis on the Early Iron Age of the Northeast Iberian Peninsula, filling a gap in the knowledge of the isotopic biogeochemistry of equines from that region and chronology [2,3].
- Isotopic analyses of first domesticated horses are scarce [4]. Normally these studies focus of animal mobility without analyzing their paleodiet [5–7]. Can Roqueta data provide an approximation to the diet, as a model of equine herd management in the Early Iron Age in the western Mediterranean.
- These data may be useful for archaeologists interested in the management of domestic livestock in the past, specifically equines.
- The data can also serve as an isotopic baseline for further studies to reconstruct human behaviour during Iron Age by examining their subsistence strategies and the management for agricultural and pasture use.

1. Data Description

In total, 42 bone samples of domestic equines (being 39 Equus caballus, 2 Equus asinus and one hybrid) were analyzed. All these samples are from Can Roqueta. This site is located in Sabadell, in a natural corridor running parallel to the coast of Barcelona, in the Northeast of the Iberian Peninsula (Fig. 1).

The samples come from 35 structures, mostly grain storage silos and graves [2]. Most of the structures date back to the Early Iron Age (24 structures, 30 samples), followed by 6 Late Bronze Age structures (7 samples), 2 samples from the Early–Middle Iron Age/Iberian period and 3 of
Late Roman age (Table 1). The chronological sequence of the samples provides a diachronic view of the diet of these animals [3].

Bone collagen was extracted for stable carbon and nitrogen isotope analysis. The quality of the extracted collagen was measured according to commonly accepted quality criteria, based on the collagen yield, the carbon and nitrogen contents and the atomic C:N ratio.

All samples met the minimum thresholds: collagen yield higher than 1.0% [8], %C >13%, %N >5% [9], atomic C:N between 2.9 and 3.6 [10]. In addition, 30 of them (71.4%) yielded a collagen percentage higher than 3.5%, a more restrictive criterion pointed out by Van Klinken [8], and 26 (61.9%) have carbon and nitrogen contents in collagen higher than 30 and 11, respectively, as suggested by Ambrose [9]. There is no relationship between the carbon and nitrogen contents with their respective isotopic signatures ($R^2 = 0.029$ for C, $R^2 = 0.003$ for N), neither between the collagen yield and the isotopic values ($R^2 = 0.001$ for C, $R^2 = 0.018$ for N). This indicates that the isotopic values of samples with low content of both elements, or low collagen yield (but always above the accepted thresholds) are not altered by this circumstance. We consider that, taken together, all the samples showed good collagen preservation and therefore their isotopic signatures are reliable.

2. Experimental Design, Materials and Methods

To extract the collagen from the bone samples, compact bone fragments were subjected to abrasive cleaning of all surfaces, followed by up to 10 alternating washes in milliQ water and acetone in a sonicator. The acetone washes were applied even though the bones had not been treated with consolidants, as they serve to remove possible contaminants present on the surface of bones subjected to intensive handling by zooarchaeologists. Acetone pre-treatment has not been found to affect the isotopic values of collagen [11 and references therein].

After allowing the samples to dry for 24–48 h at room temperature, the bone fragments were manually crushed with an agate pestle and mortar. Collagen extraction was based on the
method proposed by Longin [12], with modifications described in detail in [13]. Briefly, it consists of performing digestion of the bone powder in hydrochloric acid 1 M to dissolve the bone apatite during 20 min at room temperature, followed by a digestion in sodium chloride 0.125 M during 20 h in order to dissolve contaminating organic matter such as humic acids acquired during diagenesis. Each digestion was followed by microfiltration (Sartorius cellulose nitrate filter, 5 μm pore size), which removes smaller molecules, including degraded collagen. The final product was gelatinized in mild hydrochloric acid (0.01 M) during 17 h at 90 °C and freeze-dried for subsequent analysis.
The determination of $\delta^{15}N$ and $\delta^{13}C$ was made in the Instrumental Analysis Techniques Unit (UTIA) of the Research Support Services of the University of A Coruña. It is carried out by combustion in a FlashEA1112 elemental analyzer (ThermoFinnigan) linked via a ConfloIII interface to a MAT253 isotope ratio mass spectrometer (ThermoFinnigan). Samples are weighed in tin capsules using a UMx-2 balance (Mettler Toledo). Two aliquots of approximately 0.5 mg were measured from each sample. The result reported is the mean value of both aliquots.

For the conversion of total nitrogen and carbon to N$_2$ and CO$_2$ gas, the sample is analyzed by instantaneous combustion in a quartz tube with oxidant maintained at 1020 °C in an oxygen-enriched helium atmosphere. The combustion products are transported to a reduction reactor maintained at 650 °C, where excess oxygen is removed, and the nitrogen oxides are converted to N$_2$. Through this process CO$_2$, N$_2$ and H$_2$O are formed. After the water is retained in a filter, the chromatographic separation of the two gases generated takes place, which will be introduced into the mass spectrometer through an interface. Once in the mass spectrometer, the gas molecules are ionised by electron impact and separated under the action of a magnetic field according to the masses of the constituent isotopes.

The $\delta^{15}N$ and $\delta^{13}C$ results are expressed in ‰ for Atmospheric Air Reservoir (AIR) and Vienna Pee Dee Belemmite (VPDB), respectively. In each analytical sequence, USGS 40 (−4.52‰), USGS41a (+47.55‰) (IAEA-N-1 (+0.4‰)), IAEA-N-2 (+20.3‰) and USGS-25 (−30.4‰) are used as international standards for $\delta^{15}N$. For $\delta^{13}C$: USGS 40 (−26.39‰), USGS41a (+36.55‰) NBS 22 (−30.031‰) and USGS 24 (−16.049‰) are used. Analytical precision was calculated from 10 duplicates of the in-house standard acetanilide, resulting in an error of ±0.15 for both C and N.

**Ethics Statement**

Not applicable.

**Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships which have or could be perceived to have influenced the work reported in this article.

**CRediT Author Statement**

*Aurora Grandal-d’Anglade*: Conceptualization, Methodology, Investigation, Writing – original draft, Writing – review & editing; *Silvia Albizuri*: Conceptualization, Investigation, Writing – original draft, Writing – review & editing; *F. Javier López-Cachero*: Investigation, Writing – review & editing.

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**References**

[1] K. Salesse, R. Fernandes, X. de Rochefort, J. Brůžek, D. Castex, É. Dufour, IsoArch.eu: an open-access and collaborative isotope database for bioarchaeological samples from the Graeco-Roman world and its margins, J. Archaeol. Sci. Rep. 19 (2018) 1050–1055.

[2] S. Albizuri, A. Grandal-d’Anglade, J. Maroto, M. Oliva, A. Rodríguez, N. Terrats, A. Palomo, F.J. López-Cachero, Dogs that ate plants: changes in the canine diet during the late bronze age and the first iron age in the northeast Iberian Peninsula, J. World Prehist. 34 (2021) 75–119, doi:10.1007/s10963-021-09153-9.
[3] S. Albizuri, A. Grandal-d’Anglade, N. Terrats, N.M. Oliva, T. Maj, F. Antolin & F.J. López-Cachero, Continuity and innovation in human diets through bioarchaeological analysis: The community of Can Roqueta in the pre-coastal of Barcelona and its Mediterranean contacts, 8th–6th centuries BC. In: Mixed Tastes: Diets, Cuisines and Consumption Practices in Ancient Western Mediterranean. Babesch. Annual Papers of Mediterranean Archaeology (in press).

[4] L.S. Fleming, R.J. Losey, T. Nomokonova, S. Garvie-Lok, A.A. Kharinskii, E.V. Kovychev, Medieval animal management practices at Proezzaia I: insights from dietary stable isotope analysis, J. Archaeol. Sci. Rep. 22 (2018) 45–57, doi:10.1016/j.jasrep.2018.09.001.

[5] S. Albizuri, S. Valenzuela-Lamas, D. Bosch, M. Fernández, F.J. López-Cachero, Equid use and provision during the early iron age in Can Roqueta (NE Iberian Peninsula). Zooarchaeological study and first strontium isotope results (87Sr/86Sr), J. Archaeol. Sci. Rep. 26 (2019) 101907, doi:10.1016/j.jasrep.2019.101907.

[6] W.D. Hamilton, K.L. Sayle, M.O.E. Boyd, C.C. Haselgrove, G. Cook, Celtic cowboys reborn: application of multi-isotopic analysis (δ13C, δ15N, and δ34S) to examine mobility and movement of animals within an iron age British society, J. Archaeol. Sci. 101 (2019) 189–198, doi:10.1016/j.jas.2018.04.006.

[7] C.A. Makarewicz, C. Winter-Schuh, H. Byerly, J.L. Houle, Isotopic evidence for ceremonial provisioning of Late Bronze age khirigusurs with horses from diverse geographic locales, Quat. Int. 476 (2018) 70–81, doi:10.1016/j.quaint.2018.02.030.

[8] G.J. Van Klinken, Bone collagen quality indicators for palaeodietary and radiocarbon measurements, J. Archaeol. Sci. 26 (1999) 687–695, doi:10.1006/jasc.1998.0385.

[9] S.H. Ambrose, Preparation and characterization of bone and tooth collagen for isotopic analysis, J. Archaeol. Sci. 17 (1990) 431–451, doi:10.1016/0305-4403(90)90007-R.

[10] M.J. De Niro, Post-mortem preservation and alteration of in vivo bone collagen isotope ratios in relation to palaeodietary reconstruction, Nature 317 (1985) 806–809, doi:10.1038/317806a0.

[11] C.A.M. France, A. Epitropou, G.M. Kavich, The effects of cellulose nitrate treatment and organic solvent removal on δ13C, δ15N, and δ18O values of collagen and bioapatite in modern mammal bone, Archaeol. Anthrop. Sci. 11 (2019) 1421–1429, doi:10.1007/s12520-018-0616-8.

[12] R. Longin, New method of collagen extraction for radiocarbon dating, Nature 230 (1971) 241–242, doi:10.1038/230241a0.

[13] A. Grandal-d’Anglade, S. Albizuri, A. Nieto, J. López Melció, T. Majó, A. Palomo, A. Rodríguez, B. Agustí Farjas, A. Moya i Garra, Dogs and foxes in early-middle bronze age funerary structures in the Northeast of the Iberian Peninsula. Human control of canid dier at the sites of Can Roqueta (Barcelona) and Minferri (Lleida), Archaeol. Anthropol. Sci. 11 (2019) 3949–3978 11, doi:10.1007/s12520-019-00781-z.