Quaternary International 683-684 (2024) 92–103

Contents lists available at ScienceDirect

Quaternary International

journal homepage: www.elsevier.com/locate/quaint

Archaeomagnetic analyses on fumiers burned under controlled experimental conditions

Ángel Carrancho[a,*], Balász Bradák[b], Ángela Herrejón-Lagunilla[c,d], Josep María Vergès[e,f]

[a] Área de Prehistoria, Departamento de Historia, Geografía y Comunicación. Universidad de Burgos, Edificio I+D+I, Plaza Misael Bañuelos S/n 09001, Burgos, Spain
[b] Faculty of Oceanology, Kobe University, 5-1-1 Fukaeminami-machi, Higashinada-ku, Kobe, 658-0022, Japan
[c] Dpto. Física, Universidad de Burgos. Escuela Politécnica Superior, Avda. Cantabria S/N 09006, Burgos, Spain
[d] Departamento de Física de La Tierra y Astrofísica, Facultad de Ciencias Físicas, Universidad Complutense de Madrid, Spain
[e] Institut Català de Paleoecologia Humana I Evolució Social (IPHES-CERCA), Zona Educacional 4, Campus Sescelades URV (Edifici W3), 43007, Tarragona, Spain
[f] Universitat Rovira I Virgili, Departament D'Història I Història de L'Art, Avinguda de Catalunya 35, 43002, Tarragona, Spain

ARTICLE INFO

Keywords:
Fumier
Taphonomy
Archaeological fire
Experimental archaeology
Paleotemperatures
Secular variation

ABSTRACT

The improvement of the archaeomagnetic dating method requires compiling new and older data of the Earth's magnetic field (EMF) variations for the last millennia. Combustion events from *fumier* sequences have been proposed as good directional EMF recorders. However, they are subjected to diverse taphonomical processes and how these affect the archaeomagnetic record has not yet been studied. In order to evaluate it, here we report the first archaeomagnetic and rock-magnetic results on samples from experimentally recreated *fumiers* since 2014 under controlled conditions. A facies description with unprecedent resolution was used to study the variation of magnetic properties in depth. Rock-magnetic analyses indicate a homogenous magnetic mineralogy dominated by pseudo-single domain magnetite as main carrier in all facies, with not very high and similar contribution of the finest (superparamagnetic) grains. The low values of anisotropy of magnetic susceptibility (AMS) indicate that the studied sample set is mainly isotropic. The directional behaviour in well-preserved burned facies (here described as G, LM and DGB), are jointly characterized by highly reversible thermomagnetic curves, high Koenigberger (Q_n) ratio values and intense, univectorial and normal polarity orthogonal NRM demagnetization diagrams. On the contrary, specimens affected by mechanical alteration processes are less magnetic and show anomalous directional behaviours. The high thermomagnetic reversibility of ashes indicates that they reached *ca.* 600–700 °C, in line with the thermocouples' data. Temperatures of 460 °C were obtained for the DGB facies (subyacent black carbonaceous facies). Sampling of ashes located on the top of these combustion events should be avoided for archaeomagnetism. Being just beneath the last stabling episode they are the most prone to undergo mechanical alterations and do not preserve well the Earth's magnetic field direction. Despite their unlithified nature and the multiple taphonomic processes that *fumier* sequences may undergo, under certain quality criteria, they are valid geomagnetic field recorders providing both information of archaeological and geophysical interest.

1. Introduction

One of the main changes that entailed the adoption of agropastoral practices since the Neolithic was the occupation of caves and rock shelters as livestock-pens. Throughout all the Mediterranean Europe, the holocene stratigraphies of many caves contain evidences of dometic animal stabling (e.g.: Angelucci et al., 2009 and references therein). These contexts are known in the archaeological literature as *fumiers* (Brochier, 1983). They are interpreted as sedimentary sequences generated by the intense and recurring combustion of vegetal remains and animal dung (e.g.: Brochier et al., 1992; Boschian, 1997; Burguet-Coca et al., 2022; Macphail et al., 1997; Vergès et al., 2016a). The main objective of these combustion practices was to eliminate the parasites of the site and reduce the enormous volume of excrements generated by the herd (Boschian, 1997; Karkanas, 2006; Vergès, 2011; Vergès et al. 2016a, 2016b).

Fumier-like sequences exhibit a high variability of sedimentary facies (some burned intercalated with others unburned), which are

* Corresponding author.
E-mail address: acarrancho@ubu.es (Á. Carrancho).

https://doi.org/10.1016/j.quaint.2023.09.005
Received 10 January 2023; Received in revised form 5 September 2023; Accepted 11 September 2023
Available online 22 September 2023

recurrently repeated in the stratigraphy. There is no full consensus to classify these sequences, although their sedimentological description has been addressed through a relatively uniform facies system (e.g.: Angelucci et al., 2009). Traditionally, the study of fumier sequences has been approached from a paleobotanical and geoarchaeological perspective (e.g.: Brochier et al., 1992; Canti, 1999; Macphail et al., 1997). In addition to the more classic studies of lithic, ceramic or faunal remains, other techniques have been applied on bones, sediments or paleobotanical remains to characterize the formation and use of these sites. For instance, soil micromorphology analyses allow a high-resolution sedimentary evaluation to reconstruct the formation and alteration conditions (e.g.: Angelucci et al., 2009; Polo-Díaz et al., 2016; Macphail et al., 1997). Studies of lipids and faecal biomarkers have shown their utility in identifying faunal taxa (e.g.: Gea et al., 2017; Vallejo et al., 2022), complementing the more traditional zooarchaeological studies (Martín-Rodríguez and Vergés, 2016a, 2016b). Another source of data are isotope studies both on faunal remains to infer the diet and seasonality of the stabled livestock (e.g.: Martín-Rodríguez et al., 2021) and on the sediments and plant remains (ej.: Égüez et al., 2022). All this information has proven to be very useful for reconstructing the dynamics of occupation and use of caves, as well as their formation and diagenesis processes (e.g.: Cabanes et al., 2009; Expósito and Burjachs, 2016; Expósito et al., 2022; Portillo and Albert, 2011; Shahack-Gross et al., 2003; Albert et al., 2008).

Archaeomagnetism is one of the techniques that have been recently applied to the study of these contexts. Studies carried out in several European sites propose that, under adequate preservation conditions, the combustion events contained in these sequences can record the directional changes of the Earth's magnetic field through time (Carrancho et al., 2013, Carrancho and Villalaín, 2012, Herrejón-Lagunilla et al., 2022; Kapper et al. 2014a, 2014b). This is of geochronological interest since it allows to improve the secular variation curves and geomagnetic field models used for archaeomagnetic dating. This is possible for several reasons. First, the combustion events present in these stratigraphies are generally well dated by radiometric techniques (mainly ^{14}C), so their chronological control is adequate and precise. Second, these deposits usually present high sedimentation rates, which favours their good preservation. For example, Angelucci et al. (2009) report peak sedimentation rates of 15 mm/year in certain Neolithic levels of the El Mirador cave (Spain). If properly preserved (e.g.: ashes over carbonaceous facies showing lateral continuity, etc.), each combustion episode is stratigraphically sealed by the rapid burial of the upper level, preventing alteration processes. Third, the burned facies from these combustion events can preserve a good record of the direction of the Earth's magnetic field at the time of its last heating and subsequent cooling (Carrancho et al., 2009, 2013, 2016; Carrancho and Villalaín, 2012; Herrejón-Lagunilla et al., 2022; Kapper et al. 2014a, 2014b). Thus, the archaeomagnetic study of multiple well-preserved and independently well-dated combustion episodes has made possible to elaborate the first directional European paleosecular variation (SV) curve for the Earth's magnetic field up to the Neolithic (Carrancho et al., 2013). This curve can be used for dating and for certain chronologies, it reaches precisions comparable to radiocarbon ranging between ±30 and ± 200 yr (Carrancho et al., 2013).

Nonetheless, this type of sediments are subjected to diverse syn/post-depositional processes, with the implications that it has for the reliability of the archaeomagnetic record. Although the acceptance of archaeomagnetic directional data are filtered under strict quality selection criteria (Carrancho et al., 2013; Herrejón-Lagunilla et al., 2022; Kapper et al., 2014b), being able to recognize whether a specific combustion event is affected or not by taphonomic alteration processes is relevant. The most common post-depositional processes include partial or total removal of ashes to clean specific areas of the caves, trampling, bio-cryoturbation, etc. Diagenetic processes such as dissolution, fluid migration or precipitation may also take place, promoting the redistribution, alteration and even disappearance of minerals, including ferromagnetic (*s.l.*) phases. Some of these processes are visible on a macroscopic scale, but this is not always the case. Tradicional techniques such as soil micromorphology (e.g.: Polo-Díaz et al., 2014), zooarchaeology (Martín-Rodríguez and Vergés, 2016a) or paleobotany (Burguet-Coca et al., 2020, 2022; Cabanes et al., 2009), along with others less standard such as archaeomagnetism (Carrancho et al., 2012) or molecular analyses of bones through ATR-FTIR (Del Valle et al., 2022) have been successfully applied to distinguish alteration processes involved in these sequences. Although the taphonomic processes that can affect the archaeomagnetic record are diverse, to our knowledge, there is not still any archaeomagnetic work that has studied it in detail under controlled experimental conditions.

Here we report the archaeomagnetic and rock-magnetic results of a program of livestock's residues burned by means of experimental archaeology performed since 2014. Thanks to the control of multiple variables (number of heatings, temperatures, type and amount of fuel, etc.), the objectives of this research are to study how the *in situ* burning of manure heaps occurs under controlled conditions, to characterize magnetically in detail the variability of facies generated, and to evaluate whether the archaeomagnetic record is affected by post-depositional processes.

2. Materials and methods

2.1. Experimental recreation

This experimental burning program of livestock residues began in 2014 and so far, 4 burning sessions were carried out (2014, 2015, 2016 and 2018). Each year a herd of 400 heads of livestock (350 goats and 50 sheeps) has been stabled in the Mas del Pepet pen (Rojals, Tarragona, NE Spain), located at 880 m a.s.l. The main objective of this experimental program seeks to characterize and interpret how these deposits are formed, what formation and alteration processes they undergo and how human activity conditions these contexts in relation to the management of domestic livestock.

The pen is a closed and roofed enclosure that reproduces quite well what can happen in a similar archaeological cave sequence. The livestock stabling usually lasts about 6 months, approximately from April/May to October/November. The animals spend the night in the pen and during the day they are grazing. During this half year, the herd generates a significant amount of excrements which after its stabling period, are piled up and burned under controlled conditions. Various variables such as the temperatures reached, humidity, location or type and amount of fuel are carefully monitored. The realization of 3D models allows the exact location of each combustion event and the conditions in which they took place. A detailed description of the specific methodology followed in this experimentation can be found in Vergès et al. (2016b).

2.2. Sampling and facies description

In this study, we sampled Heap 5 which was located at the rear of the pen (Fig. 1a). That heap includes at least 3 different burnings because the first one (2014) was artificially extinguished with water by firefighters, so it cannot be considered a complete cycle. Subsequent burnings were extinguished naturally. Heap 5 is an accumulation of manure and vegetal remains that in their successive burnings exceeded 600–700 °C. On its ashes and prior to the annual stabling of the herd, a thick bed of straw was placed. This action is relatively frequent in current livestock practices to favour rest and/or feeding of the herd and has been inferred in archaeological contexts from paleobotanical analyses (Rasmussen, 1993; Alonso-Eguíluz et al., 2017). That heap was selected because it is interesting to assess whether these beds of straw really have a damping effect on the trampling generated by the herd during its stabling in terms of the archaeomagnetic record.

With the aid of a chainsaw, three hand-blocks were collected and magnetically oriented with a Brunton magnetic compass (Fig. 1a–f). The

Fig. 1. (A–f). Photos illustrating the main steps of archaeomagnetic sampling. (a) General view of the pen after the 2015 burning season (from Vergès et al., 2016b). (b) Sampling with an aid of a chainsaw. (c) View of two blocks before being plastered. Note the excrement facies on top (facies BB). (d) After pouring Plaster of Paris on top, the surface was leveled horizontally with two bubble levels. (e–f) Magnetic north marked on the samples before extracting them. Note the facies variations. Sample 3 is shown in panel e and sample 2 in panel f. Dashed rectangle in panel an indicates the sampled area (heap 5).

upper part of each block corresponds to a facies of between 1 and 2 cm of unburned excrement (from the last unburned stabling episode; Fig. 1c). Plaster of Paris was poured on it, leveled horizontally with a methacrylate plate and two spirit levels (Fig. 1d) and finally, the magnetic north was marked before extracting the samples (Fig. 1e and f). In previous archaeomagnetic studies of burning events in archaeological fumier sequences, a facies of white and/or grey ash of variable thickness (from a few millimeters to several centimeters) and a burned carbonaceous facies underlying the ash and of dark colour (~2 cm) were mainly distinguished (Carrancho et al., 2009, 2012, 2013, Carrancho y Villalaín, 2012; Herrejón-Lagunilla et al., 2022, Kapper et al., 2014a,b). In this case study, we distinguished a facies sequence according to the variation of texture, colour and thermoalteration degree, as is illustrated in Fig. 4, which corresponds to a microprofile from block 1. From top to bottom it can be distinguished.

(i) **Facies BB**: a facies of unburned excrement (between 1 and 2 cm) of dark colour.
(ii) **Facies UM**: it contains mixture of unburned excrement (facies BB) and a high ash component (facies G).
(iii) **Facies G**: Fine grained, light to medium grey colour and relatively loose burned sediment. It is basically composed of ash.
(iv) **Facies LYB**: Only identified in block 3. It is fine grained, has light yellow/brown colour and also contains ashes.
(v) **Facies LM** (lower mix): mixture of facies G and DGB. Normally compact and with mixture of colours (light-grey-dark component). In block 3 is a mixture of facies LYB and DGB.
(vi) **Facies DGB** (dark grey/black): Compact and fine-grained burned facies, dark colour and always subjacent to ashes. It would correspond to the carbonaceous facies underlying the ashes identified in similar studies.

In the G, LYB and LM facies (essentially ash facies), 3–5 mm size rock fragments have also occasionally been observed. The contacts between facies are in general clear and distinguishable. However, in block 3 the ashes have irregular boundaries and this block is the only one where the LYB facies was identified. This along with the lack of lateral continuity in the facies suggests a poor preservation or some kind of mechanic alteration in this block. During sampling, a partially charred fragment of wood cutting its facies was observed. For this reason, although some specimens of this block were demagnetized to analyse the signal resulting from this presumable poor preservation, they were finally not considered for the calculation of the mean direction as explained later.

The reason to perform such a detailed facies description was to verify if actually, there is variation in the magnetic properties and if the traditional description of "*ashes over underlying carbonaceous facies*" (Carrancho et al., 2013) is sufficiently diagnostic and descriptive. Each hand block was consolidated in the laboratory in a solution of sodium silicate with water (75:25). Subsequently, each block was subsampled in cubes (10 cm^3) respecting the orientation marks drawn in the field and noting the depth and type of facies to which they correspond.

2.3. Magnetic methods

All magnetic analyses reported here were carried out in the laboratory of Palaeomagnetism of Burgos University (Spain). The measurement of the natural remanent magnetization (NRM) was performed with a 2G-755 superconducting cryogenic magnetometer (noise level $\sim 5 \times 10^{-12}$ Am^2). The magnetic susceptibility and its anisotropy (AMS) were analysed at room temperature with a kappabridge KLY-4 (AGICO, noise level 3×10^{-8} S.I.). The AMS values and directions of principal axes were determined to characterize the magnetic fabric (MF) calculated using the principal susceptibility values (Anisoft 4.2 software; Chadima and Jelinek, 2009). The analysis of the directional stability of the NRM was carried out by means of progressive thermal demagnetization and decreasing alternating fields (AF). AF demagnetization included 15 steps up to a peak field of 100 mT using the demagnetization unit attached to the cryogenic magnetometer. Thermal demagnetization of the NRM was done in 18 steps from room temperature up to 625 °C through a TD48-SC (ASC) shielded thermal demagnetiser. The analysis of the structure of orthogonal NRM demagnetization diagrams and the determination of the characteristic remanent magnetization (ChRM) direction was carried out by principal component analyses (Kirschvink, 1980) using the Remasoft v.3 software (Chadima and Hrouda, 2006). The mean archaeomagnetic direction was calculated using Fisher (1953) statistics.

The magnetic properties of the materials were analysed through various experiments in order to identify the composition, concentration and granulometric variations of the ferromagnetic mineralogy (*s.l.*). This is important to assess how stable is the remanent magnetization. Using bulk sample (~ 450 mg) of each facies identified the following experiments were performed with a variable field translation balance (MM_VFTB): progressive IRM (isothermal remanent magnetization) acquisition curves up to 1 T, hysteresis cycles (±1 T), backfield coercivity curves and thermomagnetic (magnetization *vs.* temperature) curves up to 700 °C in air. These experiments were carried out on subsamples extracted at different depths from block 1 in order to evaluate how the thermal impact affected the magnetic properties. The determination of the Curie/Néel temperatures was performed using the two-tangent method of Grommé et al. (1969). After correcting the hysteresis loops for their dia/paramagnetic fraction, the parameters *Ms* (saturation magnetization), *Mrs* (saturation remanent magnetization) and *Bc* (coercive field) were determined. The remanent coercive field or *Bcr* was calculated from the backfield curves. All these parameters were used to represent the so-called "Day plot" (Day et al., 1977; Dunlop, 2002), which allows to infer granulometric differences (Mrs/Ms *vs.* Bcr/Bc). Addititional experiments included the measurement of the dual frequency susceptibility (Xfd%) with a Bartington MSE2 meter (maximum nominal resolution 2×10^{-6} S.I.) to assess the relative contribution of ultrafine superparamagnetic (SP) particles. Normalized absolute frequency dependence (Xfs) and normalized relative frequency dependence (Xfn; expressed as percentage) were calculated. The percentages of susceptibility at two frequencies were calculated as: $\chi FD\% = [(\chi 0.46\ kHz - \chi 4.60\ kHz)/\chi 0.46\ kHz] \times 100$ (Dearing, 1999).

3. Results

3.1. Magnetic properties

Initial NRM intensity values of the studied collection oscillates between 1.36×10^{-5} and 3.18×10^{-4} Am^2kg^{-1}. NRM values are similar among facies G, LYB, LM and DGB (basically those facies with a higher ash component), while facies UM and BB display slightly lower values (Figs. 2 and 3a). Magnetic susceptibility values range from 4.70×10^{-7} and 1.62×10^{-6} m^3kg^{-1} (Figs. 2 and 3b). Again, the most burned facies exhibit the highest values and facies BB (excrement layer), the lowest ones. The Koenisgberger (Q_n) ratio (*cf.* Stacey, 1967), a parameter frequently used in archaeomagnetic studies to evaluate the stability of the remanence, oscillates between 0.47 and 6.86 (mean = 3.57; median = 3.89). Only one specimen from block 3 exhibits $Q_n < 1$. The highest values correspond to facies LM, G and DGB. Slightly lower values but also over unity are observed in the BB, UM and LYB facies (Fig. 2a and b). The trend towards lower values in LYB facies is influenced by one specimen with very low NRM values (Fig. 2a). Moreover, only 4 specimens were available for LYB facies. As explained later, LYB facies was only identified in block 3, the one with most orthogonal NRM demagnetization diagrams rejected due to mechanical alterations. Most likely, some taphonomic (mechanical) alteration disordered the ferromagnetic

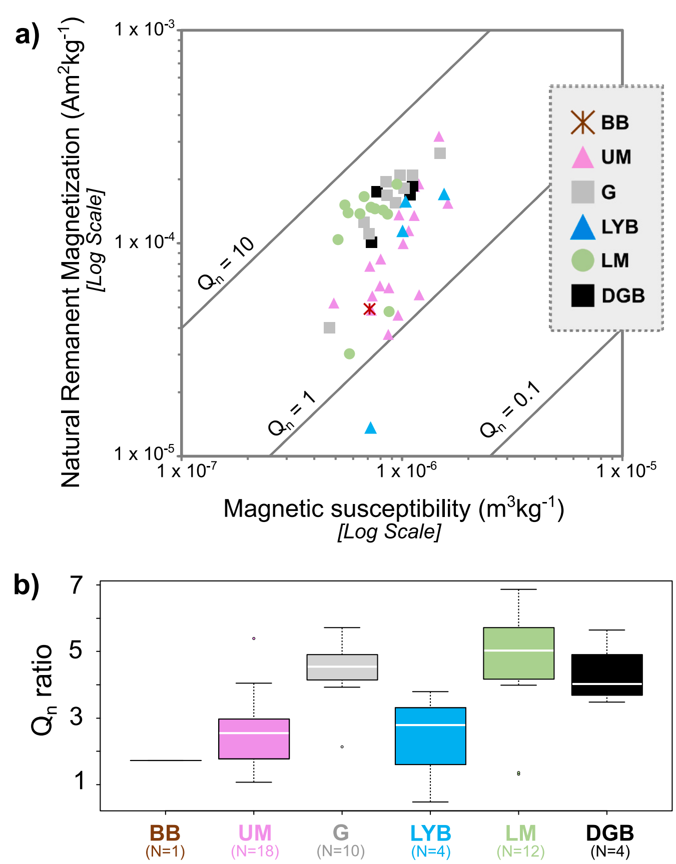


Fig. 2. Koenigsberger (Q_n) values by facies. (a) Intensity of the natural remanent magnetization (NRM) vs. magnetic susceptibility on a mass-specific basis. Isolines indicate constant Q_n values from 0.1 to 10. Facies are denoted according to the legend. (b) Box-plot distinguishing the Q_n values by facies indicating the number of specimens analysed.

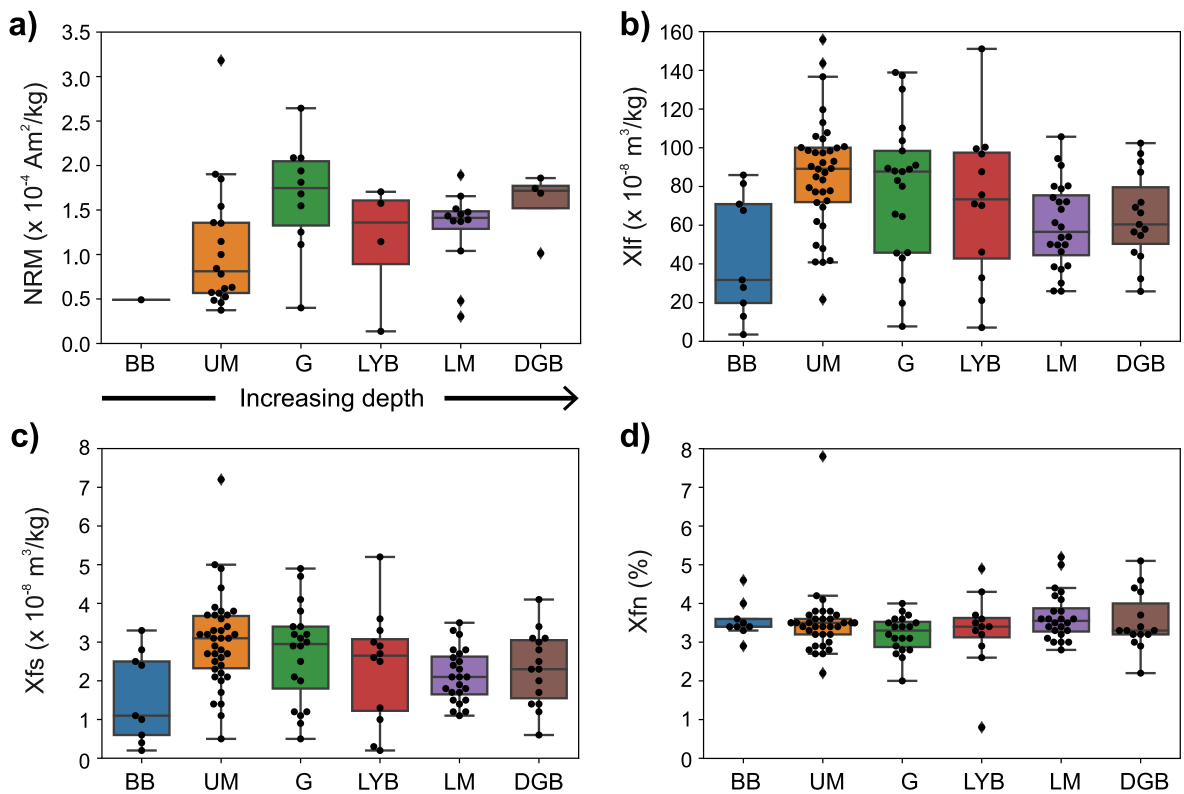


Fig. 3. Box-plots for (a) initial NRM intensities; (b) low-field magnetic susceptibility; (c) normalized absolute frequency dependence; (d) normalized relative frequency dependence (expressed as percentage). Results are shown by facies and as a function of depth from left to right.

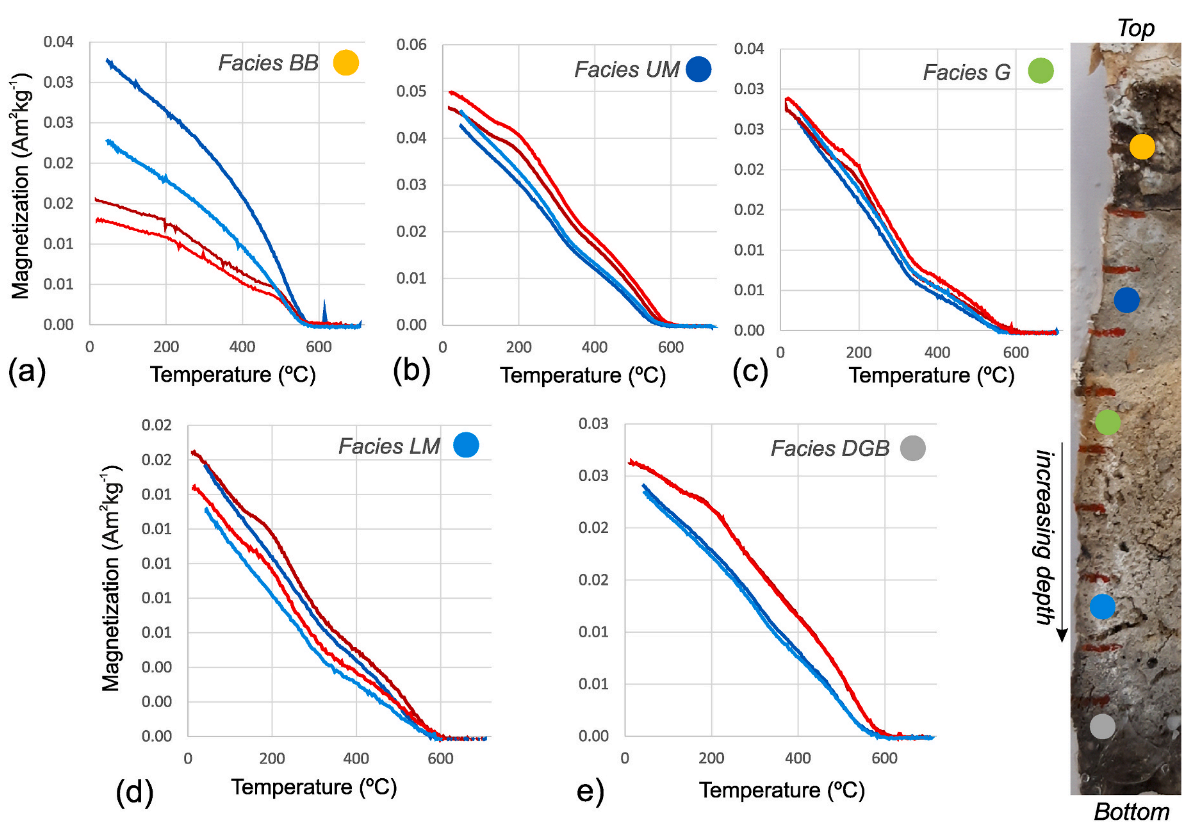


Fig. 4. Thermomagnetic curves (magnetization *vs.* temperature) on representative samples collected in depth along the profile of block 1. Colours indicate the sampling points from the microsection corresponding to every facies distinguished. Heating (cooling) cycles are indicated in red (blue). Two curves were carried out for each facies.

grains and explain such a low value. Despite their variability, these Q_n results indicate that the magnetization in these facies can be considered of thermal origin.

3.1.1. Dual frequency susceptibility

The box-plots of Fig. 3a-d illustrate the results of the NRM, magnetic susceptibility and the susceptibility at two frequencies distinguished by facies and as a function of depth. Low-field magnetic susceptibility (Xlf) results are shown in Fig. 3b. From top to bottom of each block (from left to right of each panel) a decreasing pattern in depth is observed, except in the BB facies, which has significantly lower MS than the other facies. Facies BB corresponds to the uppermost 2 cm of the blocks and is a facies of unburned excrements resulting from the last stabling (Fig. 1c). According to its relatively low susceptibility values, the concentration of ferromagnetic (*s.l.*) minerals in this facies is lower compared to the rest of the burned facies, especially the ashes (UM, G, LYB and LM facies) and to a lesser extent, the underlying carbonaceous facies (DGB). Fig. 3c shows the Xfs (normalized absolute frequency dependence) whose results are similar to those explained for Xlf. Again, a decreasing trend in depth is observed following the same facies pattern described for Fig. 3b. Finally, Fig. 3d illustrates the Xfn or normalized relative frequency dependence (expressed as percentage). Interestingly, no significant differences among facies are observed here with mean values around 3–4%. This means that the relative content of the finest superparamagnetic (SP) magnetite particles (~0.01–0.03 μm) is not very high in comparison with the values of between 6 and 9% reported for archaeological ashes from *fumiers* (Carrancho et al., 2009; Kapper et al., 2014a).

3.1.2. Thermomagnetic curves

Thermomagnetic curves (magnetization *vs.* temperature) were performed in depth with a subsampling resolution of between 0.5 and 1 cm for block 1, trying to characterize each facies identified (Fig. 4). Regardless of the type of facies analysed, the dominant presence of magnetite is observed with Curie temperatures around 580 °C (Dunlop y Özdemir, 1997), although other phases are also present. Curves of sample BB are clearly irreversible (Fig. 4a). That is, heating and cooling cycles differ, generating in this case secondary magnetite in the cooling. Considering that it is the facies of unburned excrements with a high content of organic matter, it is an expected result. Between 2 and 5 cm approximately, various facies of variable grey colour are observed that we could generically characterize as ashes (Fig. 4b–d). In addition to magnetite, in all of them a slight increase is observed at low temperatures in the heating cycles (*ca.* 180–200 °C) and an inflection at intermediate temperatures (*ca.* 300–350 °C) in this case, in both cycles. This last inflection may be a spinel phase with a high content of substituent cations or, more likely, stable maghemite. However, the phase around 200 °C is more difficult to explain, although it has been observed in other burned archaeological materials such as bricks from ceramic kilns (Herrejón-Lagunilla et al., 2021) or palaeolithic hearths (e.g.: Bradák et al., 2021). One candidate might be the recently described low Curie temperature high coercivity stable phase (HCSLT) or Epsilon iron oxide (ε-Fe_2O_3), which generally requires high firing temperatures, above 800–900 °C (López-Sánchez et al., 2017; McIntosh et al., 2007). This are above those achieved here at least for the DGB facies. However, in our opinion, it is unlikely to be the HCSLT phase because that same inflection should be seen around 200–250 °C in the cooling cycle and it is not observed. The high degree of reversibility exhibited by these ashen samples is interesting (Fig. 4b,c,d) and even to a lesser extent Fig. 4e, indicating that these facies reached high temperatures, of the order of 600–700 °C (the same as those used in these experiments). The deepest sample (Fig. 4e) shows a slightly lower cooling cycle than its heating cycle, indicating that hematite has been neoformed. This sample corresponds to a carbonaceous facies (DGB), but still incorporates a certain amount of ash.

3.1.3. Magnetic granulometry (Day plot)

The biparametric ratio Mrs/Ms *vs.* Bcr/Bc, colloquially known as "Day et al. (1977) plot", is represented in Fig. 5. This diagram has traditionally been used to infer the domain state (granulometry) of ferromagnetic particles in materials dominated by Ti-low titanomagnetites. Recently, Roberts et al. (2018) have questioned its interpretative validity. However, in assemblages where the magnetic mineralogy is relatively homogeneous and accompanied by other mineralogical experiments, it is an additional informative tool. In this case, it is remarkable the good grouping exhibited by all the studied specimens around the PSD or vortex state area (Roberts et al., 2017), near the mixing curves for SD and MD grains described by Dunlop (2002). The contribution of fine SP grains does not seem to be very significant, in accordance with the not particularly high percentages (3–4%) mentioned above for the dual-frequency (Fig. 3d).

3.2. NRM directional stability

Paleomagnetic analyses allowed to calculate the mean characteristic remanent magnetization (ChRM) direction, which is the direction of the Earth's magnetic field recorded in the last burning. With some exceptions described below, the orthogonal NRM demagnetization plots of most burned specimens are defined by highly magnetic, univectorial normal polarity plots (Fig. 8b and c and e-h). Demagnetization steps below 10–15 mT (AF demagnetization) and 200–250 °C (thermal demagnetization) were excluded for the calculation of the mean ChRM direction in order to avoid any potential viscous influence. The ChRM direction was isolated between 200/250 °C and 460/600 °C in thermally demagnetized specimens. In the case of AF demagnetized specimens, the ChRM direction was calculated from 10-15 mT to 80–100 mT.

In contrast to the good paleomagnetic properties described above, some specimens from specific facies and blocks exhibited anomalous behaviour which we relate with mechanical alterations. Only one specimen of facies BB was demagnetized (not shown here). It corresponds to an AF demagnetized specimen with strongly overlapped components and anomalous directions (e.g.: negative inclination). It should be noted that facies BB is the uppermost facies (*ca.* 1–2 cm), with a fibrous texture and recognizable excrement fragments. It is basically

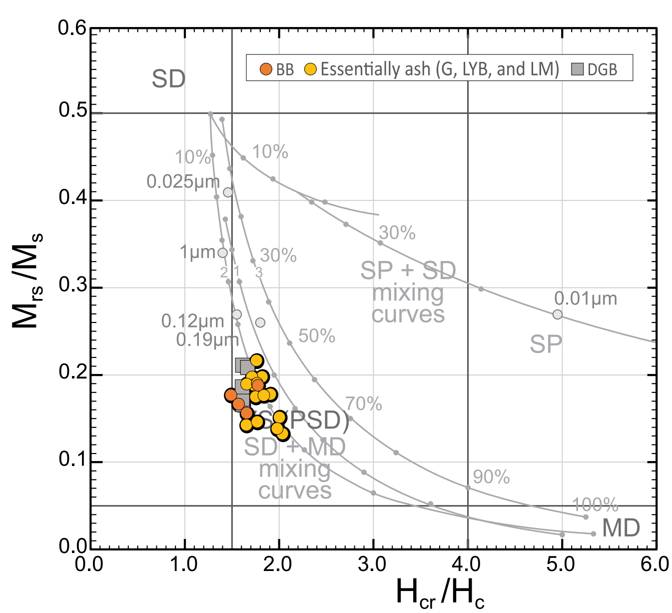


Fig. 5. "Day plot" (Day et al. 1977) for the studied sample set. The grey lines represent mixing curves taken from Dunlop (2002) for mixtures of single-domain (SD) with multi-domain (MD) or superparamagnetic (SP) magnetite particles. All samples are well grouped in the PSD or SD + MD mixture with *ca.* 40–50% SD contribution.

unburned manure which corresponds to the last episode of livestock stabling after the last burning (Fig. 1c). Moreover, the analysed specimen comes from block 3, characterized by irregular contacts among facies indicative of some sort of mechanical alteration. Anyway, being not burned, facies BB was considered of no archaeomagnetic interest. Another interesting behaviour that deserves a comment is that of facies UM. This facies, just below the BB facies, consists of a mixture of BB (excrements) and G facies (grey ashes). Due to its BB content, its

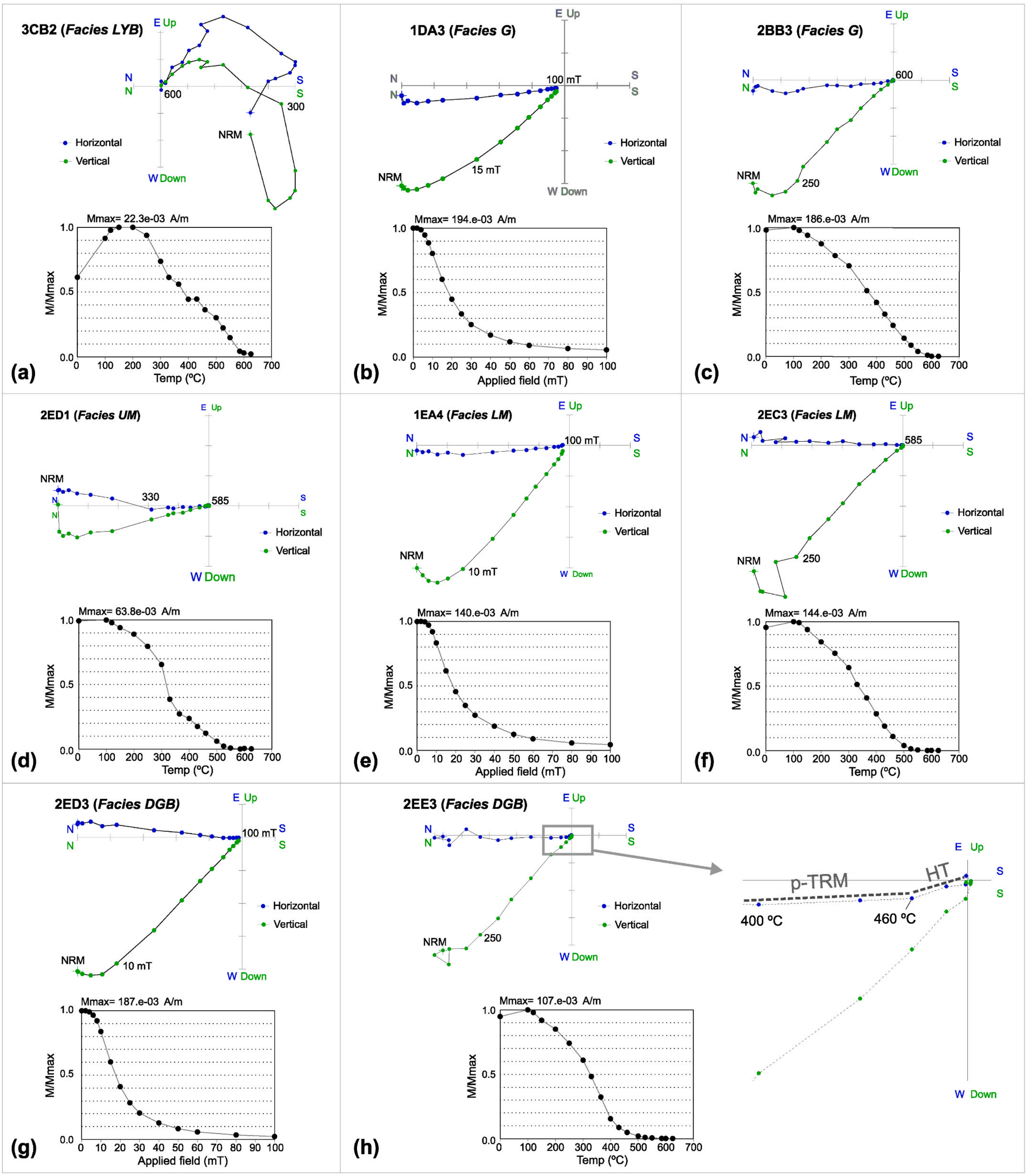


Fig. 6. (A–h). Orthogonal NRM demagnetization plots of representative specimens from different facies. Blue (green) circles show projections of vector endpoints onto the horizontal (vertical) plane. The specimen code, facies, intensity (NRM_0) and normalized intensity decay curves are shown for each specimen. In Fig. 6h, the final steps of the diagram are blown up to show the presence of a high-temperature (HT) component. p-TRM = partial thermoremanence.

magnetic record is rather variable. A representative example of the UM facies is illustrated in Fig. 6d. As they incorporate unburned excrement in different percentages, they are less magnetic than pure ashes and, in addition, they have a multicomponent and directionally anomalous NRM structure. Their intensity decay diagrams also show a characteristic drop around 300–330 °C which, in the absence of additional mineralogical experiments, we suggest it may be some ferromagnetic sulphide (e.g.: pyrrhotite). If the ash content in specimens of UM facies is higher, thereby they are magnetically more intense. However, in general, they are less magnetic than the G, LM and DGB facies (Fig. 3a). Most of them display very low inclinations, probably because it is trampled excrement that incorporates more or less ashes (Fig. 6d), and also, they do not have directional coherence (even some with negative inclinations). These results indicate some syn/post-burning process of mechanical distortion.

The specimens of block 3 deserve a particular comment. All demagnetized specimens from this block, regardless of their facies and depth, exhibit unstable orthogonal NRM demagnetization diagrams, strongly overlapped components (mainly those demagnetized by AF) and/or highly scattered directions (e.g.: not northward). Fig. 6a shows a representative example. As mentioned before, the contacts between facies in block 3 were discontinuous and irregular and also, much of this block was cut by a partially charred fragment of wood. Consequently, they were not considered for the calculation of the mean archaeomagnetic direction. Most likely, again, this indicates some kind of syn/post-burning mechanical alteration which affected this block.

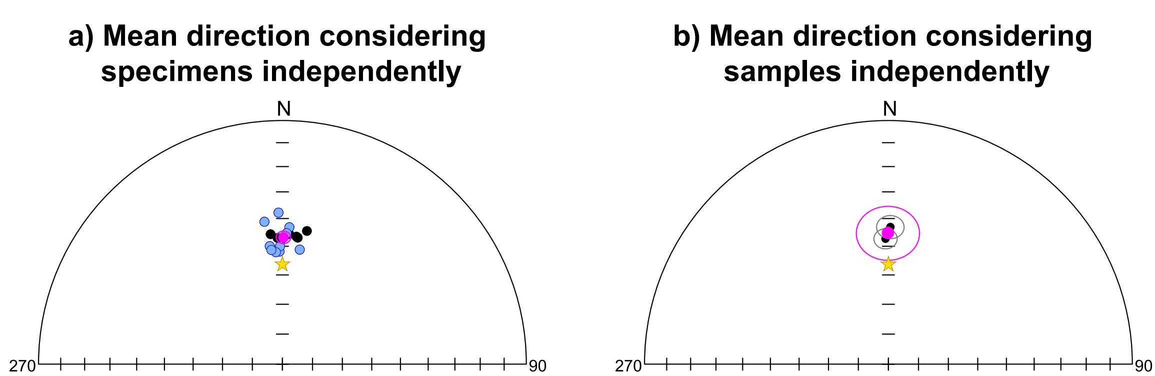
Regarding facies G (Fig. 6b and c), LM (Fig. 6e and f) and DGB (Fig. 6g and h), essentially those with the highest ash content, the orthogonal NRM demagnetization plots are magnetically intense, stable and unidirectional. That is what one would expect in a material burned at high temperature and well preserved. In facies DGB, the maximum unblocking temperatures (max T_{UB}) of the ChRM direction varies between 460 °C up to 600 °C. Fig. 6h illustrates one specimen where the ChRM direction has been identified as a p-TRM with a max T_{UB} of 460 °C. The high temperature component (*ca.* 460–600 °C), also of normal polarity, presumably would correspond to a previous heating event.

The obtained mean direction and its associated statistics were calculated both at specimen at sample levels (Fig. 7a and b, respectively). Following similar criteria as those used by Kapper et al. (2014a, b) and Herrejón Lagunilla et al. (2022) in the same type of materials, all specimens with an angular deviation > $\alpha95 \times 3$ respect the mean were disregarded. In Fig. 7a (specimen level), this filter was applied considering all the specimens together (not per blocks). In Fig. 7b (sample level), it was applied calculating the mean direction of each block.

4. Discussion

This study is the first time that archaeomagnetic and rock-magnetic analyses have been performed on experimental recreations of livestock's residues burned under controlled conditions. Combustion events in fumier sequences are not at all standard materials in archaeomagnetic studies. Fumiers sequences have the advantage of being widely distributed throughout the circum-Mediterranean region since the Neolithic (Angelucci et al., 2009 and references therein), being remarkably old and generally well dated by radiometric methods. These characteristics make them theoretically suitable materials to obtain data on the directional variations of the Earth's magnetic field (EMF) in the past. However, as they are soft (unlithified) materials where the thickness of the burned facies is usually thin, they are difficult to sample. The accurate collection of oriented samples is essential in archaeomagnetic analyses. Our team uses a cylindrical corer with a built-in orientation system which is pressed against vertical profiles where the burned facies of the combustion episodes are exposed (Carrancho et al., 2009, 2012, 2013; Carrancho and Villalaín, 2012; Herrejón-Lagunilla et al., 2022). It is a minimally invasive technique, allows the collection of multiple specimens from each facies and has high precision. Other authors (Kapper et al. 2014a, 2014b) have also collected hand samples on horizontal surfaces using the classic Plaster of Paris tecnique (e.g.: Tarling, 1975). Beyond the fact that sampling very thin facies is not straightforward, the main problem is collecting oriented hand samples with ashes on the top because the plaster of Paris percolates through the ash remobilizing the grains and hence, distorting the direction. Here we used the Plaster of Paris tecnique with the advantage of having on the top of the blocks a dung facies (BB facies) of the last stabling episode. This not only allowed to accurately sample the blocks but also to study in detail the variation of the magnetization by facies and in depth as well as carrying out thermal demagnetization experiments.

The results of magnetic properties indicate that the main remanence carrier is PSD magnetite with not very high and relatively similar contributions of SP grains depending on facies (Fig. 3d). The dominant presence of magnetite indicates that the combustion takes place under predominantly reducing conditions. From the mineralogical point of view, an interesting aspect is the possible presence of ferromagnetic sulphides in the facies few or nothing burned. The drop in intensity around 330 °C in the UM facies suggests this (Fig. 6d), although additional experiments are necessary to corroborate it. If confirmed, it would be the first time that its presence in this type of materials has been



N_{sp}	N_{sp}'	Dec. (°)	Inc. (°)	α_{95} (°)	*k*
18	37	0.5	46.8	2.2	238.11

N_s	N_s'	Dec. (°)	Inc. (°)	α_{95} (°)	*k*
2	2	359.8	45.2	9.7	622.45

Fig. 7. Equal area projections showing the mean direction (pink circle) calculated at (a) specimen and (b) sample level, respectively. The 95% circle of confidence or α_{95} is shown in pink. Yellow star indicates the expected field direction. In panel a, specimens from block 1 and 2 are shown in black and blue, respectively. [N_{sp}/N_s = number of specimens/samples considered to calculate the mean direction; N_{sp}'/N_s' = number of specimens/samples analysed; Dec. = declination; Inc. = inclination; α_{95} = 95 per-cent confidence cone of mean direction and *k*, precision parameter according to Fisher (1953)].

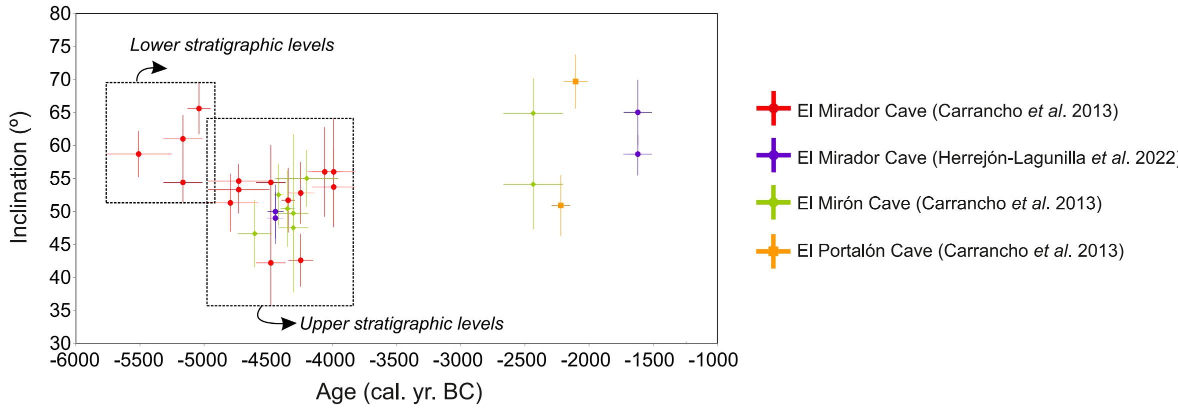


Fig. 8. Variation of magnetic inclination through time (from 6000 to 1000 cal. Yr. BCE), exclusively obtained from burning events of fumiers' cave sequences. Data shown is according to the legend. The boxes shown with dashes lines indicate different stratigraphic parts (lower and upper) from the same stratigraphic sequence, El Mirador cave. See text por explanation.

documented.

An interesting source of information provided by magnetic analysis is to determine the heating temperatures. With the exception of facies BB (unburned excrement), and to a lesser extent facies DGB, the other facies show high reversibility in their thermomagnetic curves (Fig. 4b–d). This indicates that they reached temperatures of the order of 600–700 °C, which also fits well with the temperature records of the thermocouples. With regard to DGB facies, hematite neoformation is inferred by its slightly less magnetic cooling cycle than its heating cycle, but also displaying a high degree of reversibility (Fig. 4e). Interestingly, the identification of p-TRMs with a max. T_{UB} of 460 °C in this facies (Fig. 6h), reveals the maximum heating temperature reached by these DGB specimens. Actually, the DGB facies would be equivalent to the dark carbonaceous facies underlying the ashes identified in similar studies. This datum, in addition to being archaeologically interesting, coincides very well with determinations of similar temperatures obtained in burned facies from fumiers in caves (Carrancho et al., 2009, 2016; Herrejón-Lagunilla et al., 2022; Kapper et al., 2014a).

Following the suggestion of a reviewer, the anisotropy of magnetic susceptibility (AMS) results performed on 72 specimens are not shown as they cannot be interpreted in terms of anisotropy. Only 6 specimens exhibited F values > 5, which means that this sample set is essentially isotropic. Regarding the NRM directional results, specimens from the most superficial and/or mechanically reworked facies (BB, UM and LYB; the latter one only identified in block 3), exhibit less stable NRM demagnetization plots than those from the most burned and deepest facies (G, LM and DGB) defined by magnetically intense, stable and normal polarity NRM demagnetization plots (Fig. 6). This can be explained by the mechanical reworking generated by trampling, which affects the directional record of most superficial facies.

Whether the directional behaviour is the expected one depends on the material not having undergone severe reworking or mechanical alteration processes. Otherwise, the ferromagnetic grains would lose the record of the EMF direction acquired at the time of last heating and subsequent cooling. There is a direct correlation between anomalous directional behaviour and lower NRM intensity (e.g.: UM facies) with mechanically altered (not *in situ*) specimens. In addition to being magnetically less intense than the well preseved G, LM and DGB burned facies (Fig. 3a), the orthogonal NRM demagnetization plots of facies BB and UM are anomalous and usually show inconsistent directions (e.g.: negative inclinations). In a sense this is not strange since facies UM is just below the BB facies (unburned dung). Therefore, they are more prone to be affected by trampling. This anomalous behaviour is reflecting what happens on the top of the ashes, which are not always pure because they appear mixed in variable percentages with excrements from the next stabling episode (BB facies). The directional behaviour of the UM facies is interesting because it indicates that sampling the top of these combustion events for archaeomagnetic purposes should be avoided. Moreover, despite the Q_n ratio values of the UM facies are slightly over unity (between 1.2 and 5.4), they are the lowest of the burned facies (Fig. 2a and b). This indicates that the efficiency of the magnetization mechanism of UM facies is lower than that of the other ashes. Specimens of facies BB (excrements), UM (trampled ashes mixed with excrements) and LYB (reworked ashes from block 3) show lower Q_n ratio values than those from facies G, LM and DGB (Fig. 2b). In other words, a correlation among specimens exhibiting consistent and stable directional results (if preserved *in situ*) and high Q_n ratio values is observed. In this sense, the usefulness of Q_n ratio is out of doubt. Indeed, only specimens from facies G, LM and DGB were selected for the calculation of the mean ChRM direction, all with the highest Q_n ratio values. As proposed in previous studies on fumiers' sequences (Carrancho et al., 2009, 2012, 2013, 2016; Herrejón-Lagunilla et al., 2022; Kapper et al., 2014a), our results support that Q_n ratio is a useful parameter to assess that the material is carrying a stable TRM (or pTRM).

It has been demonstrated how vector analysis of archaeomagnetic samples from fumier sequences can determine which specific parts of these combustion events are actually altered (not *in situ*), beyond macroscopic observations (Carrancho et al., 2012, 2016). That is, if the burned facies have lateral continuity and are not truncated, if their geometry is not irregular, or if the ashes are not mixed. Magnetic methods help to determine to what extent these alterations have occurred and a good example is the anomalous behaviour of block 3, whose specimens were excluded for calculating the mean direction for the given reasons.

As far as the calculation of the mean archaeomagnetic direction is concerned, the ratio of accepted *vs*. analysed specimens (N_{sp}/N_{sp}') in Fig. 7a is 48.6%, which does not seem to be particularly high. It is, however, similar to those reported in other studies in this type of contexts (e.g.: Carrancho et al., 2013; Kapper et al., 2014a). The semi-angle of 95% confidence (α95) varies significantly depending on whether the direction is calculated at the specimen level (α95 = 2.2°; Fig. 7a) or at sample level (α95 = 9.7°; Fig. 7b). This is logical because the α95 is dependent on the number of samples considered. That is, α95 is smaller as more samples are included. As explained, the nature and dimension of this heap only allowed us to collect three magnetically oriented hand-block samples using Plaster to be as accurate as possile. Anyway, from the statistical point of view, the precision parameter (k) is more informative than the α95. The quality of the obtained mean direction is noteworthy with a very high precision parameter (k = 238 at specimen level or k = 622 at sample level; Fig. 7a and b). This is a remarkable result considering the non-lithified nature of these materials, as well as the diverse post-burning processes affecting them (e.g.: animal trampling).

In order to assess the reliability of the obtained direction it was compared to the expected one for the site and date (2018) of the last burning. This was calculated by means of the Magnetic Field Calculator of the National Centers For Environmental Information (NCEI) of the National Oceanic and Atmospheric Administration (NOAA) of the United States of America, using the 13th generation IGRF model (Alken et al., 2021). The expected direction is declination = 0°, inclination = 56° (yellow star in Fig. 7a and b). Although the declination matches the expected one, the inclination is around 10° lower. It must be noted that IGRF does not provide an error associated with the expected mean direction and only considerates the main field (not taking into account the crustal influence). The ideal scenario for a more accurate estimation of the expected direction at the time of last burning would have been the use of a magnetometer in the field. Unfortunately, that was not possible. Nevertheless, it would not explain that difference of ~10° in inclination, which probably is an effect of the compactation and/or animal trampling.

There are very few archaeomagnetic studies on cave sequences with archaeological fumiers to compare with. Carrancho et al. (2013) studied 26 prehistoric burning events from three fumiers' cave sequences in northern Spain. Most of the data came from El Mirador cave, specially for the VI and V millennia BC. More recently, Herrejón-Lagunilla et al. (2022) reported additional archaeomagnetic data from this site. Fig. 8 illustrates the variation in magnetic inclination through time obtained only from fumiers' sequences of the Iberian Peninula. We have selected these data because if we want to check if the inclination is underestimated due to compaction or analogous processes, this must be mainly evaluated by comparing data from the same sequence. El Mirador Cave is without doubt where there is more archaeomagnetic data. Considering the magnetic and chronological errors (boxes with dashed lines), for the time interval between *ca.* 5000–3800 BCE, the inclination values oscillate between 36° and 62° (Fig. 8). Interestingly, these are lower inclinations than those of stratigraphically lower levels from the same sequence (El Mirador Cave), whose values oscillate between 52° and 69° for the VI millennium BC. Even just only considering the mean inclination values (excluding the magnetic errors) that pattern of variation in inclination would be almost the same for those intervals. This is striking and unexpected because theoretically, the lithostatic weight should be higher in the lower stratigraphic levels (thus recording lower inclinations values) than in the upper ones of the same sequence. However, that is not the case looking at El Mirador data shown in Fig. 8. It should be noted that all data from El Mirador cave shown in red in Fig. 8 come from the same part of the sequence (a 6 m^2 survey-pit). The two data shown in purple for the V^{th} millennium BC come from other part of the site (Herrejón-Lagunilla et al., 2022). However, they reproduce quite well the inclination values of the other data for that period, which demonstrates that the directional record of the Earth's magnetic field in these burning events is reliable. Kapper et al. (2014a,b) also reported archaeomagnetic data from fumiers in Northern Italy but their data are still rather sparse and irregularly distributed in time as to determine a clear pattern. What seems obvious is that the variations in magnetic inclination in cave sequences with fumiers are inhomogeneous through time.

This lends weitght to the argument that compaction by lithostatic charges does not generate a systematic reduction in inclination in fumiers' sequences. Inclination flattening is a well-known process in any sedimentary environment such as lake, marine or karstic sequences, but paleomagnetic data continue to be reported from them. Here we are dealing with burned sediments (the Earth's magnetic field record is instantaneous). However, as the material is not lithified, it is susceptible to undergo alterations (e.g.: a burrow can also rework the sediment). Fig. 8 demonstrates that inclinations vary unevenly through time. Our archaeomagnetic results correspond to a specific case of trampling shortly after burning and for that reason we interpret them as the likely result of taphonomic processes. Nevertheless, that does not necessarily make these burned facies invalid as directional recorders of the Earth's magnetic field. Considering the experimental conditions explained, it is not without surprise that most specimens are strongly magnetic exhibiting stable, normal polarity and rather reproducible diretions. If the alteration had been severe affecting the whole burning feature, the directional result would be totally chaotic and that is not observed. It should not be forgotten that the directions must be of normal polarity and fall within the range of secular variation as Fig. 7 shows. Even with their limitations (as any other material), burned facies from fumiers can be considered as valid records of the directional variations of the Earth's magnetic field in the past. Although there are numerous archaeological sites in Eastern Europe and SouthwestAsia (e.g.: hundres of multilayered sites are known in Bulgaria, Turkey, Romania or Greece), well-dated and even archaeomagnetically investigated (e.g.: Gallet et al., 2014; Shaar et al., 2015), they are not present throughout the world. Lava flows are also good geomagnetic field recorders but they are temporarily discontinuous and also restricted to certain regions. In many regions as the Iberian Peninsula, materials of archaeomagnetic interest carrying TRM of such ancient chronologies (from Neolithic to Iron Age), exposed in continuous sequences and independently well dated, are scarce. The compilation of new archaeomagnetic data for the last 6–7 millennia in fumiers' sequences, will make possible to improve geomagnetic field models which, in turn, can already be used for dating.

5. Conclusions

This research represents the first archaeomagnetic and rock-magnetic study on an experimental archaeology program of livestock's residues burned almost annually since 2014 under controlled conditions. The main conclusions are.

- The description of facies used here has made possible to differentiate various types of ashes by their texture and colour with unprecedented resolution. Overall, their composition, concentration and granumoletry of ferromagnetic minerals is quite homogeneus. It is shown that the traditional description of "*ashes over underlying carbonaceous facies*" is equally valid to perfom magnetic analysis on these burning episodes.
- The magnetization is dominated by PSD or vortex-state magnetite with not very high and relatively similar contributions of SP grains among facies. The presence of stable maghemite has also been observed is ashes. Likely, dung facies few or not burned might contain ferromagnetic sulphides (e.g.: pyrrothite), although this remains to be confirmed.
- With the exception of facies BB (unburned excrement) and to a lesser extent facies DGB (carbonaceous facies), the burned facies show high reversibility in their thermomagnetic curves. This indicates that they reached high temperatures (*ca.* 600–700 °C), in agreement with the thermocouples' records. The thermal NRM demagnetization of some specimens from facies DGB has revealed that they reached maximum heating temperature of 460 °C. This also agrees well with data about this facies from other studies.
- Remarkably low AMS values have been observed in the sample set studied, showing the magnetic behaviour is mainly isotropic.
- Well preserved burned facies (here described as G, LM and DGB facies) show together a high reversibility in their thermomagnetic curves, high Koenigberger (Q_n) ratio values and intense, univectorial and normal polarity orthogonal NRM demagnetization diagrams. By contrast, specimens affected by mechanical alteration processes such those from UM facies (trampled ashes mixed with dung) and block 3 are less magnetic and exhibit anomalous directional behaviours. It is also confirmed that the Q_n ratio is a useful parameter to evaluate the NRM stability.
- There is a direct correlation between anomalous directional behaviour, lower NRM intensities and Q_n values (e.g.: UM facies) with mechanically altered specimens.

- It should be avoided the sampling of the ashes located on top of these combustion events (UM facies). Being just beneath the last stabling episode (BB facies), they are the most prone to undergo mechanical disturbances and do not preserve well the direction of the Earth's magnetic field. Additionally, it includes unburned remains from BB facies, which can bias the magnetic record.
- A quantitative outlier rejection approach was followed to calculate the archaeomagnetic mean direction both at specimen and sample level, yielding indistinguishable results. The mean direction obtained (Dec.: 359.8°, Inc.: 45.2°) exhibits an underestimation of ~ 10° the inclination with respect to the expected one (Dec.: 0°, Inc.: 56°). A comparison of archaeomagnetic data from cave sequences with fumiers do not detect inclination flattening induced by compactation or analogous processes. We interpret our result as a specific effect of the animal trampling in this experiment. Despite their unconsolidated nature and the taphonomic processes that these materials may undergo, under certain quality criteria, burning events from fumiers' sequences can be considered as valid recorders of the Earth's magnetic field direction in the past. These data are useful not only to reconstruct ancient geomagnetic field variations (geomagnetic interest) but also to improve and extend back in time geomagnetic field models, which are used for archaeomagnetic dating.

Author contributions

Ángel Carrancho: Conceptualization, funding acquisition, sample collection, data interpretation, Writing, review and editing. Balász Bradák: sample collection, analyses, data interpretation, writing, review and editing. Ángela Herrejón-Lagunilla: Data interpretation, writing, review and editing. Josep María Vergès: Experimental field recreation, sampling, writing, review and editing.

Data availability

All necessary data are included in the manuscript.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This research was funded by project PID 2019-105796 GB-I00 of the Agencia Estatal de Investigación (AEI/10.13039/501100011033). Á.H. L. thanks the financial support of a Margarita Salas postdoctoral contract (2022–2024), funded by the Spanish Ministry of University & European Union-NextGenerationEU. The Institut Català de Paleoecologia Humana i Evolució Social (IPHES-CERCA) has received financial support from the Spanish Ministry of Science and Innovation through the "María de Maeztu" program for Units of Excellence (CEX 2019-000945-M). We are very grateful to Pere Domenech, for the facilities given to carry out the experimental program with his livestock and pen, as well as for his help and advice. We also thank Anton Vallvey and Ester Trullols, PNIN Poblet's directors, for their help and collaboration. Thanks are also due to the members of the brigades of maintenance of Poblet PNIN, for their help and support with the fieldwork and also to Aitor Burguet for his assistance with the sampling. The experimental program has been funded by MICINN project (PID 2021-122355NB-C32), the Generalitat de Catalunya (SGR 2017 1040) and the Municipality of Montblanc. The editors of this volume are kindly acknowledged for considering us for this contribution. We are also grateful to two anonymous reviewers for their useful and thoughtful comments which improved the manuscript.

References

Albert, R.M., Shahack-Gross, R., Cabanes, D., Gilboa, A., Lev-Yadun, S., Portillo, M., Sharon, I., Boaretto, E., Weiner, S., 2008. Phytolith-rich layers from the late bronze and iron ages at tel dor (Israel): mode of formation and archaeological significance. J. Archaeol. Sci. 35, 57–75. https://doi.org/10.1016/j.jas.2007.02.015.

Alken, P., Thébault, E., Beggan, C.D., et al., 2021. International geomagnetic reference field: the thirteenth generation. Earth Planets Space 73, 49. https://doi.org/10.1186/s40623-020-01288-x.

Alonso-Eguíluz, M., Fernández-Eraso, J., Albert, R.M., 2017. The first herders in the upper Ebro basin at Los Husos II (Álava, Spain): microarchaeology applied to fumier deposits. Veg. Hist. Archaeobotany 26, 143–157. https://doi.org/10.1007/s00334-016-0590-y.

Angelucci, D.E., Boschian, G., Fontanals, M., Pedrotti, A., Vergès, J.M., 2009. Shepherds and karst: the use of caves and rock-shelters in the Mediterranean región during the Neolithic. World Archaeol. 41 (2), 191–214. https://doi.org/10.1080/00438240902843659.

Boschian, G., 1997. Sedimentology and Soil Micromorphology of the Late Pleistocene and Early.

Bradák, B., Carrancho, Á., Herrejón-Lagunilla, Á., Villalaín, J.J., Monnier, G.F., Tostevin, G., Mallol, C., Pajović, G., Baković, M., Borovinić, N., 2021. Magnetic fabric and archaeomagnetic analyses of anthropogenic ash horizons in a cave sediment succession (Crvena Stijena site, Montenegro). Geophys. J. Int. 224 (2), 795–812. https://doi.org/10.1093/gji/ggaa461.

Brochier, J.E., 1983. Combustion et parcage des herbivores domestiques. Le point de vue du sédimentologue. Bull. Soc. Prehist. Fr. 80 (5), 143–145.

Brochier, J.E., Villa, P., Giacomarra, M., Tagliacozzo, A., 1992. Shepherds and sediments: geo-ethnoarchaeology of pastoral sites. J. Anthropol. Archaeol. 11, 47–102. https://doi.org/10.1016/0278-4165(92)90010-9.

Burguet-Coca, A., Polo-Díaz, A., Martínez-Moreno, J., Benito-Calvo, A., Allué, E., Mora, R., Cabanes, D., 2020. Pen management and livestock activities based on phytoliths, dung spherulites, and minerals from Cova Gran de Santa Linya (Southeastern pre-Pyrenees). Archaeological and Anthropological Sciences 12. https://doi.org/10.1007/s12520-020-01101-6.

Burguet-Coca, A., Del Valle, H., Expósito, I., Herrejón-Lagunilla, Á., Buitkute, E., Cabanes, D., Cáceres, I., Carrancho, Á., Villalaín, J.J., 2022. The fumier sequences of el mirador: an approach to fire as a sociocultural practice and taphonomic agent. In: Allué, E., Martín, P., Vergès, J.M. (Eds.), *Prehistoric Herders And Farmers. A Transdisciplinary Overview To the Archaeological Record From* El Mirador *Cave*. Springer, pp. 89–110. https://doi.org/10.1007/978-3-031-12278-1.5.

Cabanes, D., Burjachs, F., Expósito, I., Rodríguez, A., Allué, E., Euba, I., Vergès, J.M., 2009. Formation processes through archaeobotanical remains: the case of the Bronze Age levels in El Mirador cave, Sierra de Atapuerca, Spain. Quat. Int. 193, 160–173. https://doi.org/10.1016/j.quaint.2007.08.002.

Canti, M.G., 1999. The production and preservation of fecal Spherulites: animals, environment and Taphonomy. J. Archaeol. Sci. 26 (3), 251–258. https://doi.org/10.1006/jasc.1998.0322.

Carrancho, Á., Villalaín, J.J., 2012. Preliminary archaeomagnetic and rock-magnetic results from the holocene fire lenses in el Mirón cave. In: Straus, L.G., González-Morales, M.R. (Eds.), El Mirón Cave, Cantabrian Spain: the Site and its Holocene Archaeological Record. University of New Mexico Press, pp. 103–118.

Carrancho, Á., Villalaín, J.J., Angelucci, D.E., Dekkers, M.J., Vallverdú, J., Vergès, J.M., 2009. Rock-magnetic analyses as a tool to investigate archaeological fired sediments: a case study of Mirador cave (Sierra de Atapuerca, Spain). Geophys. J. Int. 179, 79–96. https://doi.org/10.1111/j.1365-246X.2009.04276.x.

Carrancho, Á., Villalaín, J.J., Vergès, J.M., Vallverdú, J., 2012. Assessing postdepositional processes in archaeological cave fires through the analysis of archaeomagnetic vectors. Quat. Int. 275, 14–22. https://doi.org/10.1016/j.quaint.2012.01.010.

Carrancho, Á., Villalaín, J.J., Pavón-Carrasco, F.J., Osete, M.L., Straus, L.G., Vergès, J.M., Carretero, J.M., Angelucci, D.E., González Morales, M.R., Arsuaga, J.L., Bermúdez de Castro, J.M., Carbonell, E., 2013. First directional European palaeosecular variation curve for the Neolithic based on archaeomagnetic data. Earth Planet Sci. Lett. 380, 124–137. https://doi.org/10.1016/j.epsl.2013.08.031.

Carrancho, Á., Herrejón-Lagunilla, Á., Vergès, J.M., 2016. Three archaeomagnetic applications of archaeological interest to the study of burnt anthropogenic cave sediments. Quat. Int. 414, 244–257. https://doi.org/10.1016/J.QUAINT.2015.10.010.

Chadima, M., Hrouda, F., 2006. Remasoft 3.0 a user-friendly palaeomagnetic databrowser and analyzer. Travaux Géophysiques XXVII, 20–21.

Chadima, M., Jelinek, V., 2009. Anisoft 4.2: Anisotropy Data Browser for Windows. Agico, Inc. https://www.agico.com/text/software/anisoft/anisoft.php.

Day, R., Fuller, M., Schmidt, V.A., 1977. Hysteresis properties of titanomagnetites—grain size and compositional dependence. Phys. Earth Planet. In. 13, 260–267. https://doi.org/10.1016/0031-9201(77)90108-X.

Dearing, J.A., 1999. Magnetic susceptibility. In: Walden, J., Oldfield, F., Smith, J. (Eds.), *Methods Of Environmental Magnetism: A Practical Guide*". Technical Guide No. 6. Quaternary Research Association, pp. 35–62.

Del Valle, H., Cáceres, I., Tornero, C., Burguet-Coca, A., Moclán, A., Vergès, J.M., 2022. ATR-FTIR to distinguish Holocene fumier facies. A perspective from bone diagenesis at El Mirador cave (Sierra de Atapuerca, Spain). J. Archaeol. Sci. 141, 105582 https://doi.org/10.1016/j.jas.2022.105582.

Dunlop, D.J., 2002. Theory and application of the Day plot (Mrs/Ms versus Hcr/Hc) 2. Application to data for rocks, sediments, and soils. J. Geophys. Res. Solid Earth 107. https://doi.org/10.1029/2001JB000487.

Dunlop, D.J., Özdemir, Ö., 1997. Rock Magnetism: Fundamentals and Frontiers. Cambridge University Press. https://doi.org/10.1017/CBO9780511612794.

Égüez, N., Mallol, C., Makarewicz, C.A., 2022. n-Alkanes and their carbon isotopes (δ13C) reveal seasonal foddering and long-term corralling of pastoralist livestock in eastern Mongolia. J. Archaeol. Sci. 147, 105666 https://doi.org/10.1016/j.jas.2022.105666.

Expósito, I., Burjachs, F., 2016. Taphonomic approach to the palynological record of burnt and unburnt samples from El Mirador cave (Sierra de Atapuerca, Burgos, Spain). Quat. Int. 414, 258–271. https://doi.org/10.1016/j.quaint.2016.01.051.

Expósito, I., Allué, E., Burjachs, F., 2022. Vegetation and climate at El Mirador cave: exploring the beginning of the cultural landscapes. In: Allué, E., Martín, P., Vergès, J. M. (Eds.), Prehistoric Herders and Farmers – A Transdisciplinary Overview of the Archaeological Record from El Mirador Cave (Interdisciplinary Contributions to Archaeology). Springer, pp. 35–55. https://doi.org/10.1007/978-3-031-12278-1_3.

Fisher, R.A., 1953. Dispersion on a sphere. Proc. Roy. Soc. Lond. 217, 295–305. https://doi.org/10.1098/rspa.1953.0064.

Gallet, Y., D'Andrea, M., Genevey, A., Pinnock, F., Le Goff, M., Matthiae, P., 2014. Archaeomagnetism at ebla (tell mardikh, Syria). New data on geomagnetic field intensity variations in the near east during the bronze age. J. Archaeol. Sci. 42, 295–304. https://doi.org/10.1016/j.epsl.2017.11.013.

Gea, J., Sampedro, M.C., Vallejo, A., et al., 2017. Characterization of ancient lipids in prehistoric organic residues: chemical evidence of livestock-pens in rock-shelters since early neolithic to Bronze Age. J. Separ. Sci. 40 (23), 4549–4562. https://doi.org/10.1002/jssc.201700692.

Grommé, C.S., Wright, T.L., Peck, D.L., 1969. Magnetic properties and oxidation of iron–titanium oxide minerals in Alae and Makaopuhi lava lakes, Hawaii. J. Geophys. Res. 74 (22), 5277–5293. https://doi.org/10.1029/JB074i022p05277.

Herrejón-Lagunilla, Á., Villalaín, J.J., Carrancho, Á., Alonso-Fernández, C., Jiménez-Echevarría, J., Pavón-Carrasco, F.J., 2021. Dating a medieval pottery workshop of the city of Burgos (Spain): archaeomagnetic and archaeological evidences. Phys. Earth Planet. In. 316, 106723 https://doi.org/10.1016/j.pepi.2021.106723.

Herrejón-Lagunilla, Á., Carrancho, Á., Villalaín, J.J., 2022. On the suitability of prehistoric anthropogenic burnt sediments (fumiers) for archaeomagnetic studies at El Mirador cave (Burgos, Spain). In: Allué, E., Martín, P., Vergès, J.M. (Eds.), Prehistoric Herders and Farmers A Transdisciplinary Overview of the Archaeological Record from El Mirador Cave (Interdisciplinary Contributions to Archaeology). Springer, pp. 111–128. https://doi.org/10.1007/978-3-031-12278-1_6.

Kapper, K.L., Anesin, D., Donadini, F., Angelucci, D.E., Cavulli, F., Pedrotti, A., Hirt, A. M., 2014a. Linking site formation processes to magnetic properties. Rockand archeomagnetic analysis of the combustion levels at Riparo Gaban (Italy). J. Archaeol. Sci. 41, 836–855. https://doi.org/10.1016/j.jas.2013.10.015.

Kapper, K.L., Donadini, F., Mauvilly, M., Panovska, S., Hirt, A.M., 2014b. New directional archeomagnetic data of burned cave sediments from Switzerland and geomagnetic field variations in Central Europe. Geophys. J. Int. 198 (2), 1208–1221. https://doi.org/10.1093/gji/ggu184.

Karkanas, P., 2006. Late Neolithic household activities in marginal areas: the micromorphological evidence from the Kouveleiki caves, Peloponnese, Greece. J. Archaeol. Sci. 33 (11), 1628–1641. https://doi.org/10.1016/j.jas.2006.02.017.

Kirschvink, J., 1980. The least-squares line and plane and the analysis of palaeomagnetic data. Geophys. J. Int. 62 (3), 699–718. https://doi.org/10.1111/j.1365-246X.1980.tb02601.x.

López-Sánchez, J., McIntosh, G., Osete, M.L., del Campo, A., Villalaín, J.J., Pérez, L., Kovacheva, M., Rodrı́guez de la Fuente, O., 2017. Epsilon iron oxide: origin of the high coercivity stable low Curie temperature magnetic phase found in heated archeological materials. G-cubed 18 (7), 2646–2656. https://doi.org/10.1002/2017GC006929.

Macphail, R., Courty, M.A., Hather, J., Wattez, J., 1997. The soil micromorphological evidence of domestic occupation and stabling activities. In: Maggi, R. (Ed.), Arene Candide: a Functional and Environmental Assessment of the Holocene Sequence (Excavations Bernarbo' Brea-Cardini 1940-50). Memorie dell'Istituto Italiano di Paleontologia Umana, Il Calamo, pp. 53–88.

Martín-Rodríguez, P., García-González, R., Nadal, J., Vergès, J.M., 2016b. Perinatal ovicaprine remains and evidence of shepherding activities in early holocene enclosure caves: el mirador (sierra de atapuerca, Spain). Quat. Int. 414, 316–329. https://doi.org/10.1016/j.quaint.2015.08.024.

Martín-Rodríguez, P., Tornero, C., Salazar García, D.C., Vergès, J.M., 2021. Early sheep herd management in the inland of the Iberian Peninsula: results of the incremental isotopic analyses of dental remains from El Mirador cave (Sierra de Atapuerca, Spain). Archaeological and Anthropological Sciences 13, 99. https://doi.org/10.1007/s12520-021-01355-8.

Martín-Rodríguez, P., Vergés, J.M., 2016a. Bone alterations in fumiers: experimental approach. Quat. Int. 414, 294–303. https://doi.org/10.1016/j.quaint.2015.12.056.

McIntosh, G., Kovacheva, M., Catanzariti, G., Osete, M.L., Casas, L., 2007. Widespread occurrence of a novel high coercivity, thermally stable, low unblocking temperature magnetic phase in heated archeological material. Geophys. Res. Lett. 34 (21), L21302 https://doi.org/10.1029/2007GL031168.

Polo-Díaz, A., Martínez-Moreno, J., Benito-Calvo, A., Mora, R., 2014. Prehistoric herding facilities: site formation processes and archaeological dynamics in cova gran de Santa linya (southeastern prepyrenees, iberia). J. Archaeol. Sci. 41, 784–800. https://doi.org/10.1016/j.jas.2013.09.013.

Polo-Díaz, A., Alonso-Eguíluz, M., Ruiz, M., Pérez, S., Mujika, J.A., Albert, R.M., Fernández-Eraso, J., 2016. Management of residues and natural resources at San Cristóbal rock-shelter: contribution to the characterisation of chalcolithic agropastoral groups in the Iberian Peninsula. Quat. Int. 414, 202–225. https://doi.org/10.1016/j.quaint.2016.02.013.

Portillo, M., Albert, R.M., 2011. Husbandry practices and livestock dung at the Numidian site of Althiburos (el Médéina, Kef Governorate, northern Tunisia): the phytolith and spherulite evidence. J. Archaeol. Sci. 38 (12), 3224–3233. https://doi.org/10.1016/j.jas.2011.06.027.

Rasmussen, P., 1993. Analysis of goat/sheep faeces from Egolzwil 3, Switzerland: evidence for branch and twig foddering of livestock in the Neolithic. J. Archaeol. Sci. 20 (5), 479–502. https://doi.org/10.1006/jasc.1993.1030.

Roberts, A.P., Almeida, T.P., Church, N.S., Harrison, R.J., Heslop, D., Li, Y., Li, J., Muxworthy, A.R., Williams, W., Zhao, X., 2017. Resolving the origin of pseudo-single domain magnetic behavior. J. Geophys. Res. Solid Earth 122 (12), 9534–9558. https://doi.org/10.1002/2017JB014860.

Roberts, A.P., Tauxe, L., Heslop, D., Zhao, X., Jiang, Z., 2018. A critical appraisal of the "Day" diagram. J. Geophys. Res. Solid Earth 123 (4), 2618–2644. https://doi.org/10.1002/2017JB015247.

Shaar, R., Tauxe, L., Ben-Yosef, E., Kassianidou, V., Lorentzen, B., Feinberg, J.M., Levy, T.E., 2015. Decadal scale variations in geomagnetic field intensity from ancient Cypriot slag mounds. G-cubed 16, 195–214. https://doi.org/10.1002/2014GC005455.

Shahack-Gross, R., Marshall, F., Weiner, S., 2003. Geo-ethnoarchaeology of pastoral sites: the identification of livestock enclosures in abandoned Maasai settlements. J. Archaeol. Sci. 30 (4), 439–459. https://doi.org/10.1006/jasc.2002.0853.

Stacey, F.D., 1967. The koenigsberger ratio and the nature of thermoremanence in igneous rocks. Earth Planet Sci. Lett. 2 (1), 67–68. https://doi.org/10.1016/0012-821X(67)90174-4.

Tarling, D.H., 1975. Archaeomagnetism: the dating of archaeological materials by their magnetic properties. World Archaeol. 7 (2), 185–197.

Vallejo, A., Gea, J., Massó, L., Navarro, B., Gorostizu-Orkaiztegi, A., Vergès, J.M., Sánchez-Ortega, A., Sampedro, M.C., Ribechini, E., Barrio, R., 2022. Lipid biomarkers as a tool for the identification of herder activities in El Mirador cave. In: Allué, E., Martín, P., Vergès, J.M. (Eds.), *Prehistoric Herders and Farmers – A Transdisciplinary Overview of the Archaeological Record from El Mirador Cave (Interdisciplinary* 829 *Contributions to Archaeology)*. Springer, pp. 251–270. https://doi.org/10.1007/978-3-031-12278-1_13.

Vergès, J.M., 2011. La combustión del estiércol: aproximación experimental a la quema en montón de los depósitos de redil. In: Morgado, A., Baena, J., García, D. (Eds.), La investigación Experimental aplicada a la Arqueología. Universidad de Granada, pp. 325–330.

Vergès, J.M., Allué, E., Fontanals, M., Morales, J.I., Martín, P., Carrancho, Á., Expósito, I., Guardiola, M., Lozano, M., Marsal, R., Oms, X., Euba, I., Rodríguez, A., 2016a. El mirador cave (sierra de Atapuerca, Burgos, Spain): a whole perspective. Quat. Int. 414, 236–243. https://doi.org/10.1016/j.quaint.2016.01.044.

Vergès, J.M., Burguet-Coca, A., Allué, E., Expósito, I., Guardiola, M., Martín, P., Morales, J.I., Burjachs, F., Cabanes, D., Carrancho, Á., Vallverdú, J., 2016b. The mas del Pepet experimental programme for the study of prehistoric livestock practices: preliminary data from dung burning. Quat. Int. 414, 304–315. https://doi.org/10.1016/j.quaint.2016.01.032.