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RESEARCH ARTICLE

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Absence of bones in archaeological sites from the southeast of Uruguay: Taphonomy or human behavior?

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Abstract

The rework of daily refuse, including large quantities of faunal remains, is a common explanation for earthen mound construction in the Uruguayan lowlands, which started about 5000 years ago. While some earthen mounds contain human and animal bones in high abundance, several others contain only a few fragments. Thousands of years later (17th to 18th centuries), stone structures known as cairns were used in the same region and are believed to have served as the burial ground for local chiefs. However, no bone remains were ever found during excavations. The acidity of local soils has been the common explanation for the low frequency and/or complete absence of bone remains in earthen mounds and cairns. To investigate the absence of bones possibly induced by a corrosive environment, we applied Fourier transform infrared spectroscopy (FTIR), X-ray powder diffraction (XRPD), and micromorphology to study the sediments at three sites: (1) an earthen mound rich in macroscopic bone fragments (CH2D01 site—CH); (2) an earthen mound with only a few macroscopic bone fragments (Las Palmas—LP); and (3) sediments from beneath a cairn with no macroscopic bone remains (Mario Chafalote cairn—MC). FTIR and XRPD showed the existence of burnt bones at the CH mound and a complete absence of bone mineral at LP and MC. Micromorphology revealed that, though invisible in the FTIR spectra, the LP mound contains micro-bone fragments, but in extremely low frequency. Analyses indicate that taphonomy did not play a major role in the low frequency or absence of bones at the LP and MC sites, located in a similar environmental context, and that differences in site use and mound technology explain the contrasting composition of the CH mound.

KEYWORDS
cerritos, FTIR, geoarchaeology, micromorphology, XRPD

1 | INTRODUCTION

Animal bones are indispensable sources of data for inferring past subsistence and foraging activities, and their taphonomic study reveals fundamental information about the weathering processes affecting archaeological deposits. Determining their presence or absence has great implications for the interpretation of site formation processes and the human activities at a specific site. The absence of bones in an archaeological site leads to two kinds of interpretations: one related to human behavior (i.e., bones were never deposited) and

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another related to diagenesis (i.e., bones were present in the past, but dissolved due to local burial conditions).

The type and the intensity of postdepositional processes that affect bone remains in archaeological sediments depend on the degree of burning, and local burial and environmental factors (e.g., the sedimentary context, rain regime, pH, groundwater, microbial attack, etc.) (Berna et al., 2004; Collins et al., 2002; Dal Sasso et al., 2016; Hedges, 2002; Jans, 2004; Karkanas, 2010; Nielsen-Marsh & Hedges, 2000; Nielsen-Marsh et al., 2007; Smith et al., 2007; Trueman et al., 2004; Weiner, 2010). Collagen loss, dissolution, and recrystallization of the bone mineral, commonly defined as bioapatite or carbonate hydroxyapatite [$Ca_5(PO_4)_3(OH)$] (LeGeros & LeGeros, 1984; Skinner, 2005), are the most usual diagenetic pathways.

In the southeast of Uruguay, indigenous constructions, such as earthen mounds and cairns, are the most prominent features of the archaeological landscape. Earthen mounds, locally known as *Cerritos de Indios*, are widespread in the north and southeast of Uruguay and the south of Brazil from ca. 5000 B.P. to the 17th–18th centuries (Bracco, 2006; Bracco et al., 2015; Cabrera, 2005; Gianotti, 2005; Lopez-Mazz, 2001). Earthen mounds are formed by a complex combination of uses and activities, including intentional construction episodes, unintentional sediment accumulation produced by domestic settlements, phases of abandonment and reoccupation, human burials, and maize cultivation. (Bracco, 2006; Cabrera, 2005; Femenias et al., 1990; Gianotti & Lopez-Mazz, 2009; Iriarte, 2006; Moreno, 2017; Villagran & Gianotti, 2013). As part of these dynamics, domestic waste, mostly consisting of bone remains, charcoal, lithic debris, and pottery sherds, may become integrated to the sedimentary matrices.

Cairns are human-made stone arrangements in the shape of small circles or piled up to create small mounds. They are commonly located on the top of prominent hills, in places with good visibility. They are described in chronicles from the 17th to 19th centuries as points of observation or territorial control, as the location of hearths that produce fire signals or as markers signaling the location of indigenous tombs (Femenias, 1983; Figueira, 1958; Sotelo, 2018). Both earthen mounds and cairns are believed to contain bones, and their absence has been commonly attributed to soil acidity (Capdepont et al., 2005; Capdepont & Pintos, 2006; Pintos & Capdepont, 2001).

In open-air contexts, the direct exposure to environmental factors and, often, low pH increase the alteration and final destruction of bones. Berna et al. (2004) showed that bone mineral is well preserved at pH above 8, dissolves and recrystallizes at pH between 8.1 and 7.4, and dissolves and bonds with Al or Mg at pH below 7. Thus, in the absence of alkaline material, the expected situation in open-air contexts is that of intense dissolution of the bone mineral. In our study area, soil pH varies from 4.8 to 6.9, favoring the dissolution and bonding of bone mineral with Al or Mg. However, if bones were present in the soil, traces of their existence could be detected by the identification of carbonate hydroxyapatite or other phosphate minerals associated with bone dissolution and recrystallization, such as montgomeryite or francolite (also termed carbonate fluorapatite). These minerals have been identified in archaeological sediments mostly by Fourier transform infrared spectroscopy (FTIR), sometimes combined with other techniques such as X-ray fluorescence and X-ray diffractometry (Friesem et al., 2016; Karkanas, 2000; C. E. Miller et al., 2013; Nriagu et al., 1976; Schiegl, 1996; Stiner et al., 2001; Weiner, 2010; Weiner et al., 1995, 2002).

FTIR is frequently used in archaeological science to study the mineral replacement and recrystallization processes that take place in both weathered and heated bones (Berna et al., 2004; Dal Sasso et al., 2016; Lebon et al., 2010; Shahack-Gross et al., 1997; Snoeck et al., 2014; Squires et al., 2011; Stiner, Khun, Bar-Yosef, et al., 1995; Stiner et al., 2001; Stiner, Khun, Weiner, et al., 1995; Thompson et al., 2009; Toffolo et al., 2015; Trueman et al., 2004; Weiner & Bar-Yosef, 1990). FTIR is an inexpensive, easy to use technique that allows rapid analysis of large sets of samples (Monnier, 2018; Weiner, 2010; Weiner & Bar-Yosef, 1990).

Here, we report the results of a geoarchaeological study of two earthen mounds and a cairn from southeastern Uruguay using FTIR spectroscopy, X-ray powder diffraction (XRPD), and micromorphology. The FTIR spectra were analyzed using the second derivative for better identification of the phosphate bands. We focused on the spectral range between 700 and 500 cm^{-1}, where phosphate bands can be readily isolated from clay using the second derivative. To validate the FTIR results, XRPD measurements were performed in selected samples and the results were contrasted with micromorphological observations. To group all sets of samples according to their FTIR spectra, and extract statistically significant information from high-dimensional data, both the absorbance and second derivative spectra were processed by multiple component analyses (principal component analysis [PCA]).

2 | MATERIALS AND METHODS

2.1 | Archaeological samples

Samples for this study came from three indigenous constructions located in the southeast of Uruguay (Figure 1a): two earthen mounds (Figure 1b,c) and one cairn (Figure 1d). The CH2D01 site (CH) is an earthen mound site formed by two mounds (A and B) about 1.3 m high and 35 m long. Both mounds were built between 2755 and 2426 cal B.P. (93.8% probability; URU022) and 232–124 cal B.P. (37.5% probability; URU014) (Bracco et al., 2000; Curbelo et al., 1990) (Figures 1b and 2). Human remains were recovered from both mounds, with 21 individuals in mound A, and six individuals in mound B (Moreno et al., 2014; Portas & Sans, 1995; Sans & Femenias, 2000). Bones are present in almost every layer of the site, and zooarchaeological studies indicated that the wide majority corresponds to fish, followed by deer (*Ozotoceros bezoarticus*) and guinea-pigs (*Cavia apereá*), among other species (Bica, 2020; Moreno, 2014, 2016; Pintos, 2000; Pintos & Gianotti, 1995).

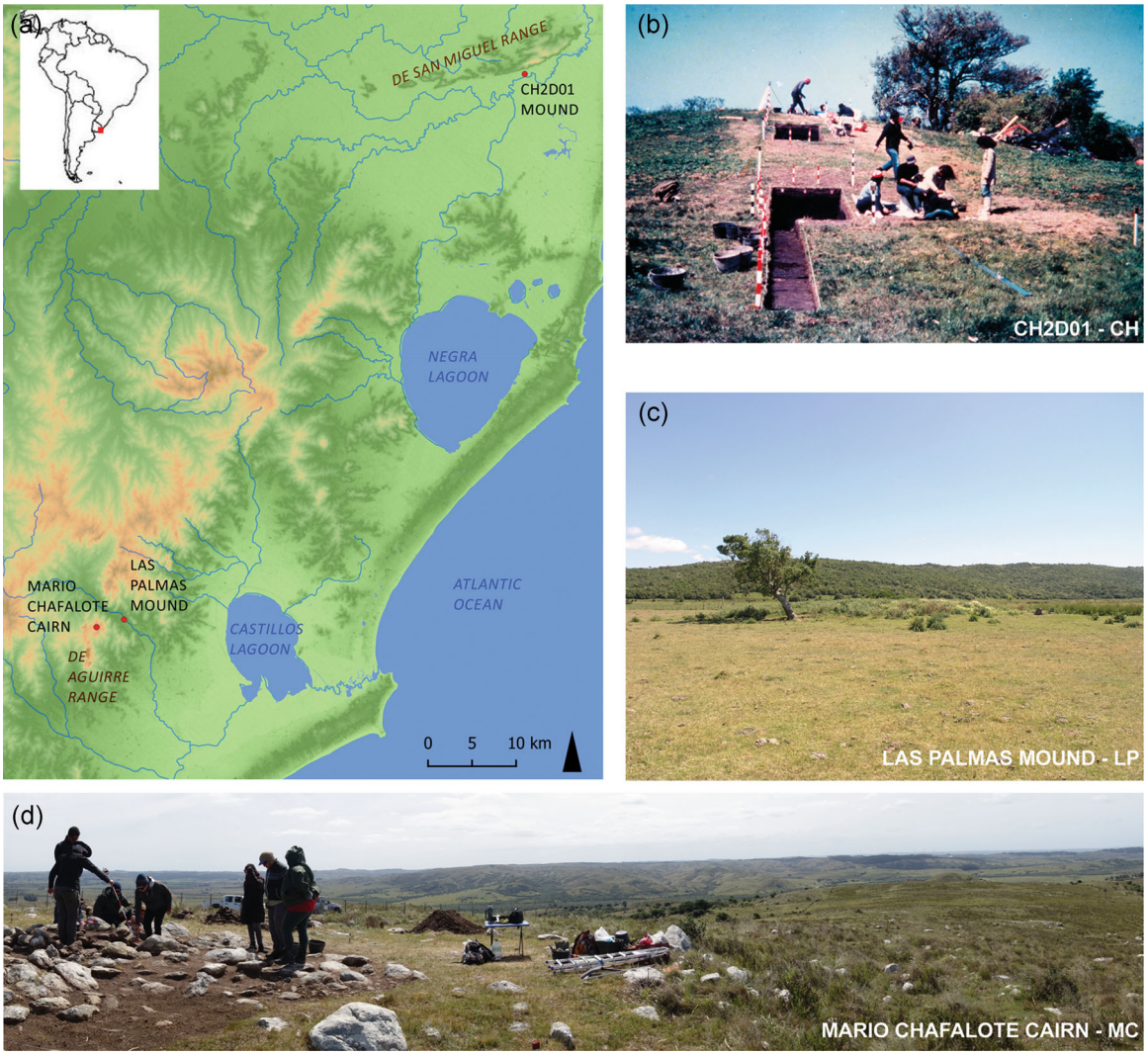

FIGURE 1 (a) Map of eastern Uruguay with the location of the three archaeological sites analyzed in this study: CH2D01 mound (CH), Las Palmas mound (LP), and Mario Chafalote cairn (MC); (b) CH site excavations in the 1990s (photograph by José López-Mazz); (c) View of the LP mound with de Aguirre mountain range in the background; and (d) view of the MC cairn located on top of the Aguirre range [Color figure can be viewed at wileyonlinelibrary.com]

The Las Palmas site (LP) is an earthen mound also consisting of two mounds (1 and 2) (Figures 1c and 2). Mound 1 is 35 m long and 1 m high, and mound 2 is 28 m long and 0.5 m high. Few heated and calcined bone fragments were recovered after wet sieving of the archaeological sediments. The first episode of mound construction took place at 2965–2859 cal B.P. (95% probability; Beta458464) and the second at 2745–2680 cal B.P. (95% probability; Beta442080) (the third and last episode has not yet been radiocarbon dated).

The Mario Chafalote (MC) cairn (Figures 1d and 2) was abandoned by the time of the European colonization (540–497 cal B.P., 95.4% probability, CNA4079-1; 315–267 cal B.P., 44.7% probability; Beta442081) and provided the first radiocarbon chronology for the Uruguayan cairns. The MC cairn is a structure 7 m in length, with a maximum height of 0.68 m. Besides numerous quartz flakes, only a polished lithic artifact (locally known as *boleadora*) was recovered, a type of artifact described in the chronicles as being commonly found next to indigenous human interments (Figueira, 1965). However, no human remains were recovered from the site, weakening the hypothesis of its use as burial ground.

The archaeological samples from CH and LP were collected from the stratigraphic profiles at 5 cm intervals. The samples from CH were taken from mound B, sector IB (excavation III). Samples from LP were taken from the stratigraphic profile of mound 1 (east profile). Sediment samples from MC were collected from different locations at shallow depths beneath the stone pile at the center of the site (Figure 2). All archaeological samples were analyzed by FTIR and a selection was also measured by XRPD. For micromorphological analyses, two undisturbed block samples were collected from MC (Figures 2 and 6a) and four blocks were collected from LP (Figures 2 and 5a). No samples for micromorphology are available for CH because excavations at this site were concluded in the 1990s.

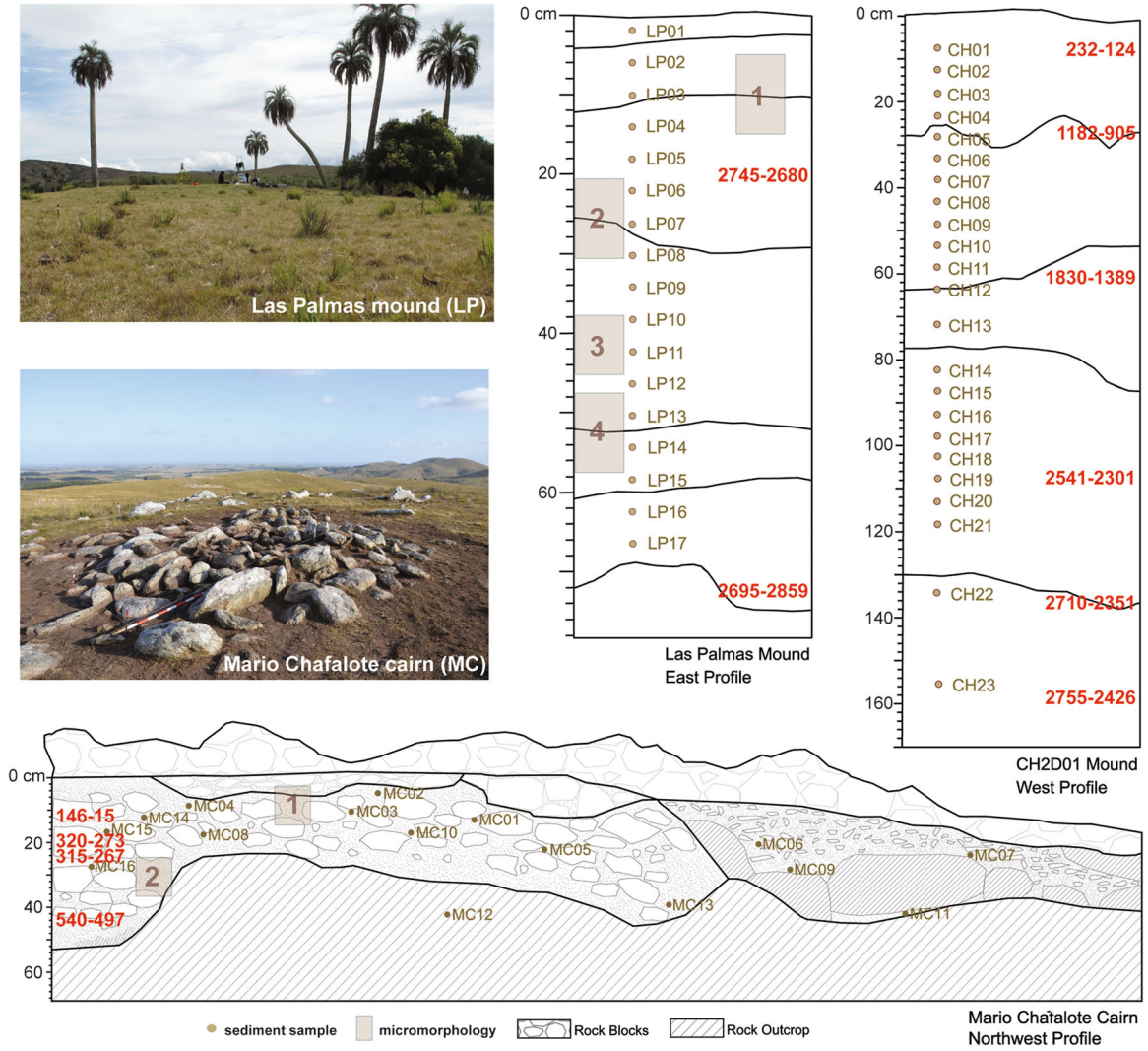

FIGURE 2 Archaeological sites used for this study. Las Palmas mound (photo); CH2D01 mound; and Mario Chafalote cairn (photo). Schematic view of the stratigraphic profiles with the location of the sediment and micromorphology samples used for this study. Calibrated ages are in years B.P. (red captions) [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 1 List of archaeological and reference samples used in this study

	Quantity	Description
Archaeological samples		
CH	23	CH2D01-B mound, profile samples
LP	17	Las Palmas mound, profile samples
MC	16	Mario Chafalote cairn, excavation samples
Reference samples		
SOIL1	1	Local soil material from the A horizon near MC site
SOIL2	1	Local soil material from the A horizon near MC site
HB-A	1	Human humerus from the India Muerta site (Rocha, Uruguay)
HB-B	1	Human maxilla from the India Muerta site (Rocha, Uruguay)

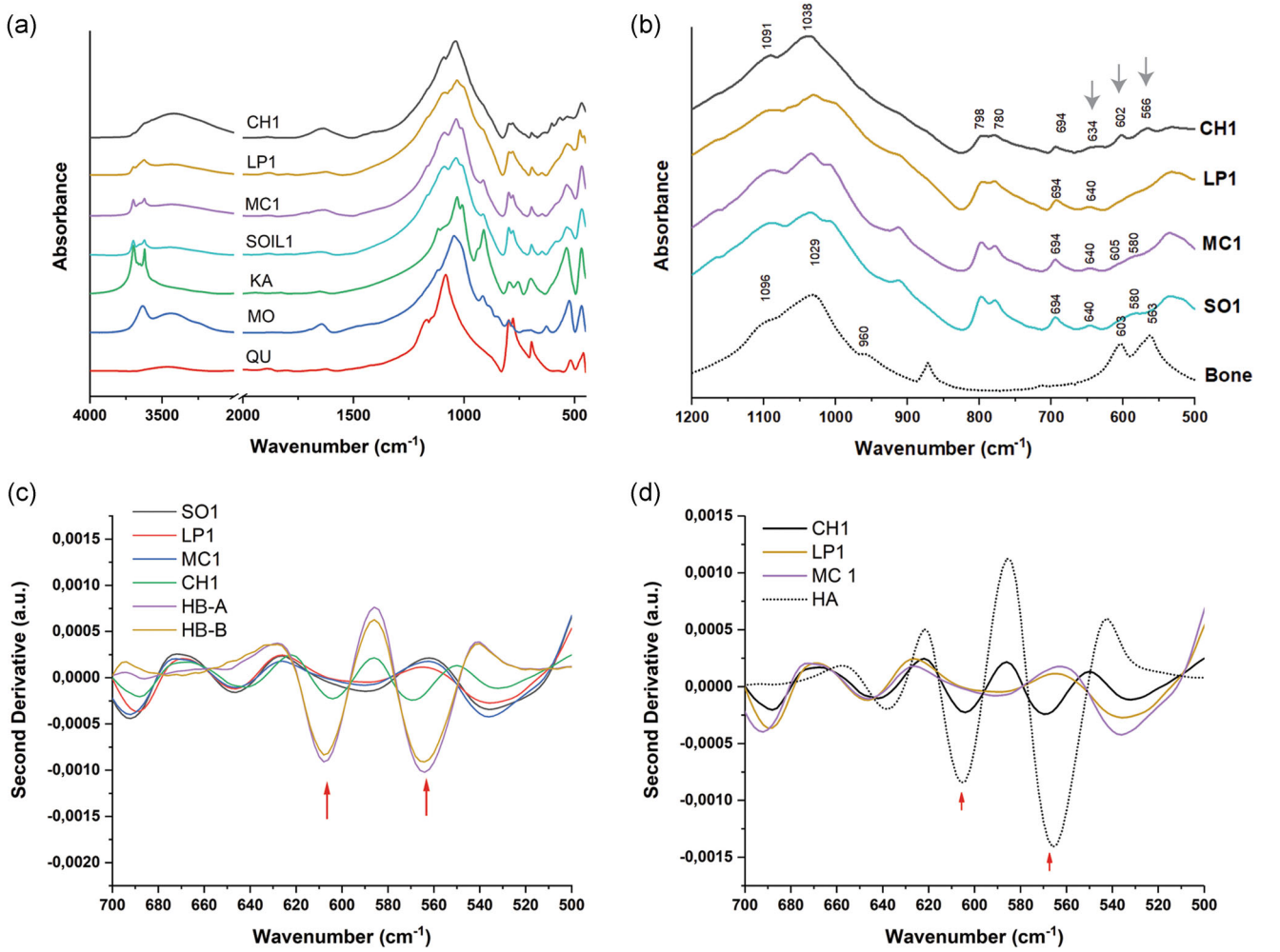

FIGURE 3 (a) Fourier transform infrared spectroscopy (FTIR) spectra of representative samples from CH, LP, and MC, with reference spectra from local soils (SOIL1) and several reference minerals present in the samples (QU = quartz; KA = kaolinite; MO = montmorillonite) (the complete spectra from CH, LP, MC, and reference soils are available as the Supporting Information Material). (b) Details of the FTIR spectra between 1200 and 500 cm^{-1} of archaeological and reference samples from local soils and bone with an indication of peak values. Only samples from CH show the 602 and 566 cm^{-1} v_4PO_4 phosphate components in the FTIR absorbance spectra. (c) Second derivative spectra of three representative archaeological samples from CH, LP, and MC, one sample of the reference soil, and two samples of human bone (HB). The two arrows indicate the locations of the v_4PO_4 phosphate components (at 604 and 565 cm^{-1}) of carbonate hydroxyapatite in the bones. See the almost perfect match between LP, MC and the reference soil and the two phospahte componentes in the CH sample. (d) Second derivative spectra of three representative archaeological samples from CH, LP, MC, and one sample of hydroxyapatite from Sigma-Aldrich. Only the sample from CH demonstrates the presence of hydroxyapatite. Two minima are also observable in the MC and LP samples at lower wavenumbers, which cannot be assigned to the v_4PO_4 vibrational modes. CH, CH2D01 mound; LP, Las Palmas mound; MC, Mario Chafalote cairn [Color figure can be viewed at wileyonlinelibrary.com]

Off-site reference samples from the surface horizon near the MC site were measured to serve as control spectra for the archaeological samples (Table 1).

2.2 | FTIR

Archaeological and reference samples were measured by FTIR spectroscopy using a Perkin Elmer Frontier spectrophotometer. Measurements were performed at room temperature between 4000 and 450 cm^{-1} with a resolution of 2 cm^{-1} (60 scans per sample). Samples were ground using a ball mill (Across international) to micro-size particles, homogenized in an agate mortar, and left to dry overnight. FTIR analyses were performed using KBr pellets. Diluted samples (0.25% wt) were homogenized in an agate mortar for 1 min. Samples mixed with KBr were pressed using vacuum pressure at 10 Ton. The transparent pellets (13 mm) obtained from this process were immediately measured in triplicate. All spectra were collected in

absorbance mode, and background correction was performed before measurements using Perkin Elmer software.

The second derivative of the FTIR spectra is commonly applied to analyze complex spectra with overlapping bands and to identify the position of weak peaks that appear as shoulders in the raw spectra (Aragão & Messaddeq, 2008; Lebon et al., 2008). We used the second derivative to highlight the underlying bands in the spectral region between 700 and 500 cm^{-1}, where the two antisymmetric bending phosphate bands (v_4PO_4) at 656 and 605 cm^{-1} can be present in sediments containing carbonate hydroxyapatite. This is a complex region in our samples, given the multiple overlapping peaks corresponding to clay minerals, quartz, and bone mineral (the stretching vibrations of the carbonate and phosphate groups). Second derivative spectra were calculated from the absorbance spectra and after smoothing using 10-point Savitzky-Golay algorithms. All calculations were performed using Origin Pro software (OriginLab).

2.3 | XRPD

A selection of archaeological samples from CH (samples 2, 3, 4, 7, 9, 11, 16, and 20), LP (samples 1, 4, 8, 12, and 15), MC (samples 1, 4, 8, 12, and 16), and reference bone samples (HB) were analyzed by XRPD. Samples were grounded in an agate mortar to less than 25 µm and analyzed with PANalytical X'Pert equipment using Bragg Brentano configuration and a linear detector. Data were collected between the range 5 and 90° with a 0.02° step for all samples. The precision of the instrument was confirmed using NIST standard reference material 640d (< 1% precision). Highscore software (PANalytical) was used to compare the data with the PDF-2 (ICDD) and the COD databases.

2.4 | Micromorphology

Samples for micromorphological analyses were collected only from the stratigraphic profiles of LP and MC sites (Figures 2, 5a, and 6a). No samples from CH sites were available for this study because the site no longer exists. Blocks were impregnated with a mixture of polyester resin, styrene, and a catalyst and sliced into 7.5 × 5 cm and 30-µm-thick thin sections. Samples were analyzed using a Leica S9i stereomicroscope and a Leica 2700P petrographic microscope under plane-polarized light and cross-polarized light at magnifications ranging from ×25 to ×200. Micromorphological descriptions followed the guidelines of Stoops (2003).

2.5 | Statistical analyses

The results of the FTIR measurements were grouped and classified by PCA using Origin Pro Software (OriginLab). The correlation matrix of a spectrum contains hundreds to thousands of values, depending on

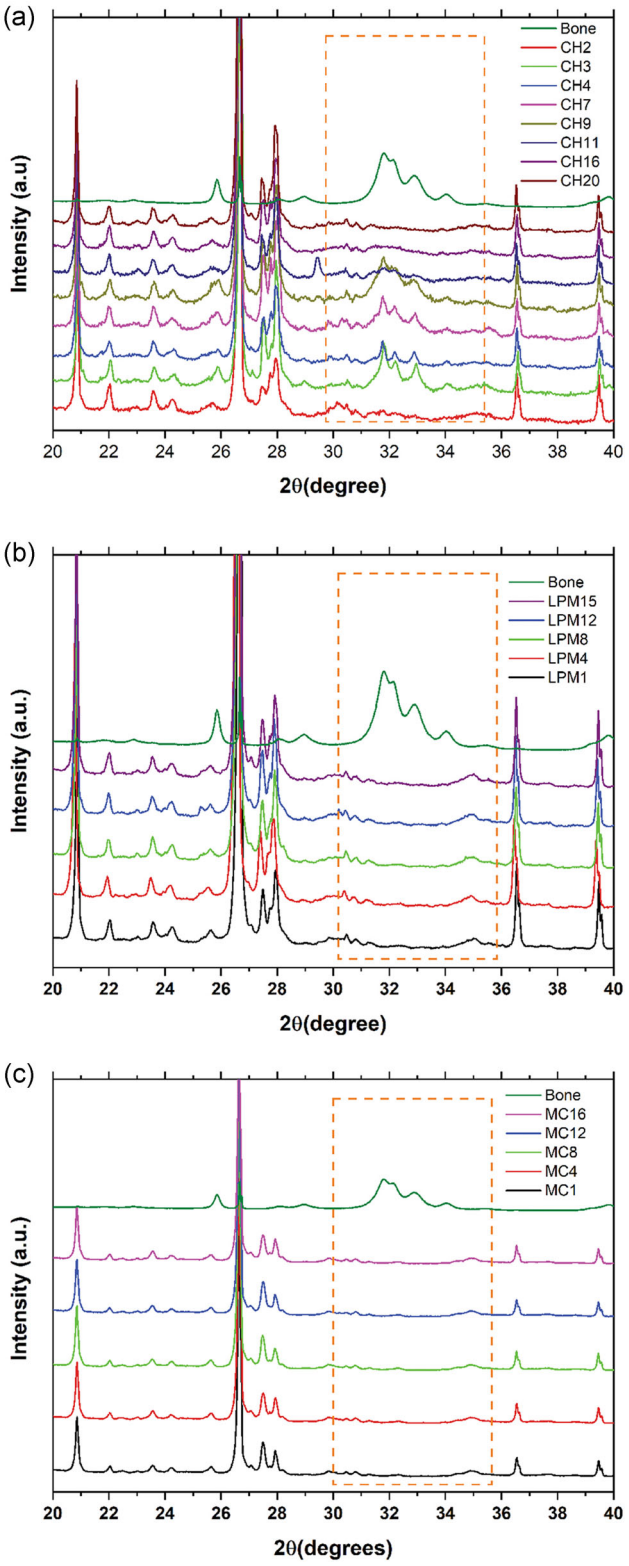

FIGURE 4 (a) Diffractograms of eight samples from the CH site and bone mineral; (b) diffractograms of five samples from the LP site and bone mineral; and (c) diffractograms of five samples from the MC site and bone mineral. The intensity of the plots was adjusted for better visualization. CH, CH2D01; LP, Las Palmas; MC, Mario Chafalote [Color figure can be viewed at wileyonlinelibrary.com]

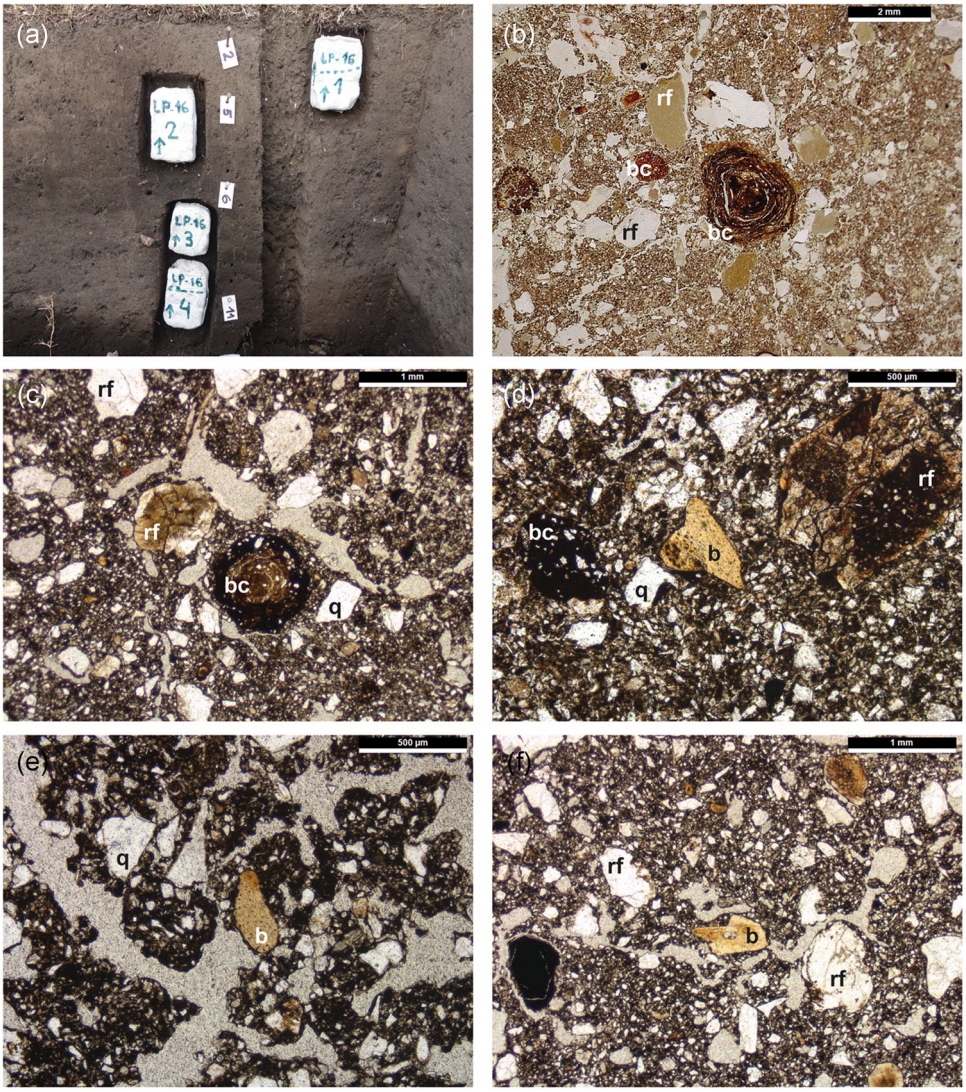

FIGURE 5 Micromorphology from the LP site. (a) Profile of the Las Palmas mound (LP) with the location of the four blocks for micromorphology; (b) sample LP-15-1 with burnt clay aggregates (bc) and rock fragments (rf) (PPL); (c) photomicrograph of sample LP-15-4 with burnt clay aggregate (bc) in the dark brown organomineral matrix (PPL); (d) photomicrograph of sample LP-15-1 with micro-bone fragment (b), burnt clay aggregate (bc), and rock fragment (rf) (PPL); (e) photomicrograph of sample LP-15-2 with micro-bone fragment (b) in the spongy microstructure (PPL); (f) sample LP-15-3 with micro-bone fragment (b) and rock fragments (rf) (PPL). PPL, plane-polarized light [Color figure can be viewed at wileyonlinelibrary.com]

the resolution (all spectra in our study contain 1800 intensity measurements). PCA is a mathematical model of data reduction that classifies multidimensional data sets into a coordinate system (based on the principal components) and may potentially reveal clusters (J. N. Miller & Miller, 2010). It is a powerful tool to infer patterns and relationships, and to explain the variations within complex series of data. In this study, PCA was used to explain the variations within the three sets of archaeological samples, and to determine if there is any relationship between spectral variance and the presence or absence of carbonate hydroxyapatite in the sediments. Although this evaluation could be done visually, PCA allows for the rapid classification of large quantities of samples (e.g., the more than 80 infrared spectra processed in this study).

3 | RESULTS AND DISCUSSION

3.1 | FTIR and second derivative spectra of archaeological samples

Three main mineral components can be identified in the FTIR spectra between 4000 and 450 cm^{-1} produced by all archaeological samples, also present in the natural soil near the MC site (SOIL1): quartz; kaolinite; and montmorillonite (Figure 3a) (see the Supporting Information Material for the raw data of each spectra from CH, LP, MC, and the soil references). All archaeological samples show peaks at ~1090, ~1035, ~914, ~796, ~781, ~695, ~645, ~535, and ~512 cm^{-1}. These peaks correspond to clay

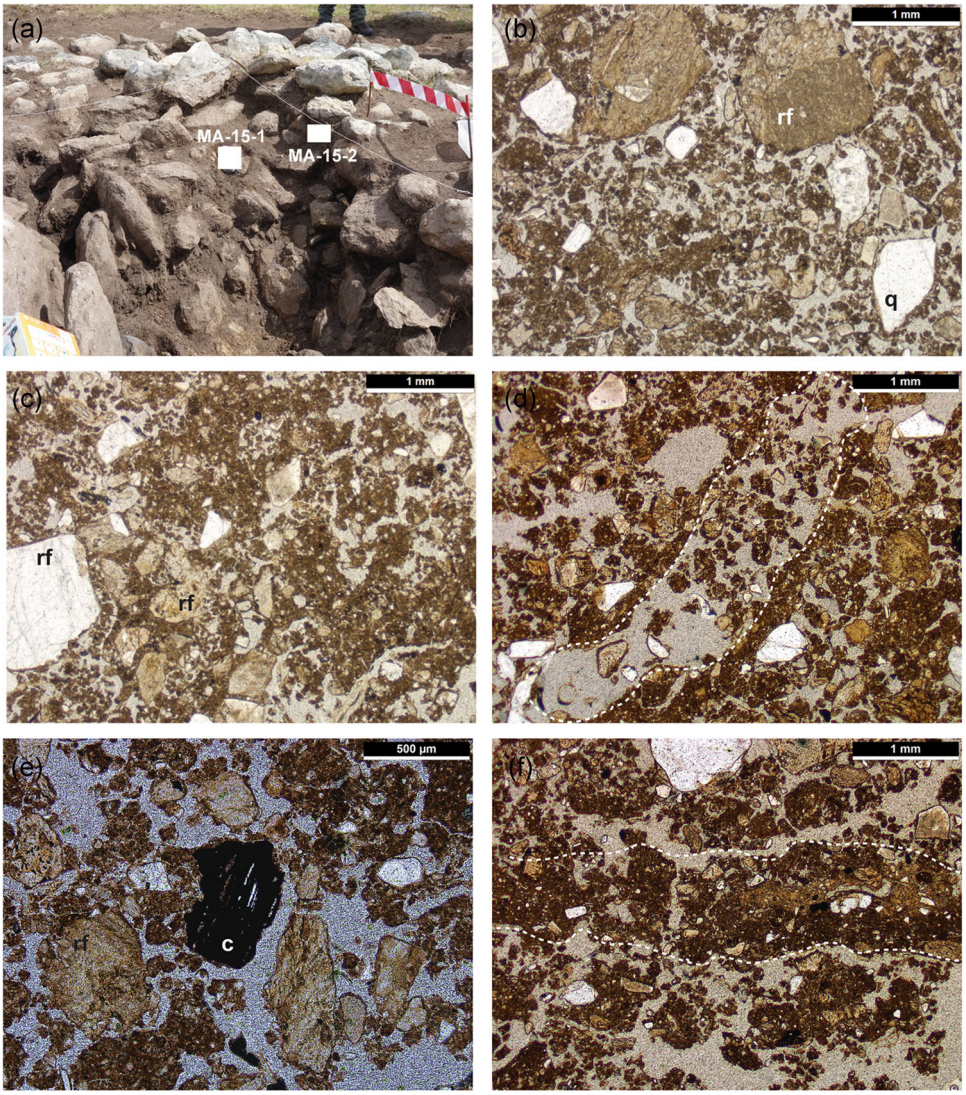

FIGURE 6 Micromorphology from the Mario Chafalote (MC) cairn. (a) Profile of M with the location of two micromorphology blocks; (b) photomicrograph of sample MA-15-1 with rock fragments (rf) and quartz (q) (PPL); (c) photomicrograph of sample MA-15-2 with rock fragments (rf) (PPL); (d) photomicrograph of sample MA-15-1 with fauna channel with excrements; (e) sample MA-15-1 with charcoal (c); and (f) surface crust in sample MA-15-1. PPL, plane-polarized light [Color figure can be viewed at wileyonlinelibrary.com]

minerals, quartz, and plagioclase. Clay minerals are identified by the peaks at the OH stretching area of the spectra (between ~3700 and 3600 cm^{-1}), a dominant peak at ~1035, corresponding to the Si–O–Si stretching band, and the secondary peaks at ~914 cm^{-1} (Al–O–H) and ~535 cm^{-1} (Si–O–Al) (Beauvais & Bertaux, 2002; Madejova & Bujd, 1996; Ritz et al., 2010). The doublet at ~796, ~781 could correspond to clay and/or quartz; the latter is also identified by the shoulder at ~1090 cm^{-1}, and the single peak at ~695 cm^{-1} (Lippincott et al., 1958; Razva et al., 2014). The shoulder at ~580 cm^{-1} is assigned to plagioclase.

Figure 3b shows selected spectra from each site for comparison. The CH1 sample shows the v_4PO_4 peaks at 602 and 566 cm^{-1} of carbonate hydroxyapatite, which are absent from the LP1 and MC1 samples that show the closest resemblance to the soil (SO1). Measurements of pH at the CH site indicated an alkaline environment (8.3) favoring bone preservation, with acidic pH only at the topmost layer (6.3). LP has pH values between 6.5 and 5.3, and one sample from MC showed an acidic pH of 4.9, favoring bone dissolution.

Only in samples from CH (CH1, CH3, CH4, CH7, CH9) do the v_4PO_4 peaks at ~604 and ~565 cm^{-1} appear together with a peak at ~634 cm^{-1}, corresponding to the hydroxyl peak of hydroxyapatite (Figure 3b). This means that hydroxyapatite is present in the sediments from CH, despite soil acitidy (pH ranges from 6.3 to 8.1 in samples CH1 to CH9). One sample from CH also showed a calcite peak at ~710 cm^{-1}.

In the second derivative spectra in which the underlying bands become visible, samples from CH also reveal the v_4PO_4 bands and the OH peak of hydroxyapatite, while samples from LP and MC do not (Figure 3c,d). This could be due to the complete absence of bone mineral in samples from LP and MC.

Other phosphate minerals that could have recrystallized from the combination of leached clay iron and/or aluminum and dissolving bone mineral in low pH, such as crandallite, montgomeryte, or taranakite, were not detected in the FTIR-KBr spectra. These minerals have diagnostic peaks in the spectral range between 700 and 500 cm^{-1} (Chukanov, 2014; C. E. Miller et al., 2016).

The consistent presence of carbonate hydroxyapatite with the peak at ~634 cm^{-1} in the sediments from CH indicates that bones burnt at high temperatures (above 500°C) are in the sediments (Berna et al., 2012; Snoeck et al., 2014; Stiner et al., 1995; Thompson et al., 2009, 2013). This has been reported by previous zooarchaeological studies in mound A, excavation IA. Moreno (2014) reported thermal alteration in about 30% of the deer bones analyzed at the CH site (among which, ca. 50% were calcined), and also in fish bones, guinea-pigs, rodents, armadillos, birds, reptiles, and ñandu egg fragments (*Rhea americana*). About 50% of undetermined bone remains showed signs of heating, most of them calcined (Moreno, 2014). Bica (2020) showed that about 7.5% of the fish bones analyzed from the CH site show various degrees of heating.

The presence of calcite in the sediments from CH is also consistent with observations made by Moreno (2014, 2017), who identified the precipitation of calcium carbonates in bones from the CH site, sometimes reaching almost 50% of the faunal assemblage in some stratigraphic layers (about 25% of the 4576 bones analyzed at the site). Physical fragmentation of the animal bones at CH is also a common phenomenon, affecting 94% of the remains analyzed by Moreno (2014, 2017). Highly fragmented and heated bones could be contributing to the presence of hydroxyapatite in the CH sediments.

3.2 | XRPD

The variable concentrations of hydroxyapatite described for CH samples by FTIR were also identified in XRDP analyses (e.g., CH3, CH4, CH7, and CH9) (Figure 4a). XRPD analyses in selected samples from MC and LP indicate the absence of bone mineral and hydroxyapatite in the sediments within the experimental error of the technique (Figure 4b,c). Only the MC samples have weak signals in the region 30–35° that do not match the reflections of bone mineral (intense reflection at 31.8° (100), 32.18° (72), and others between 31 and 34°). LP samples have no signal in this region. Thus, XRPD measurements confirm the absence of bone mineral in the samples from MC and LP, as indicated in the FTIR data.

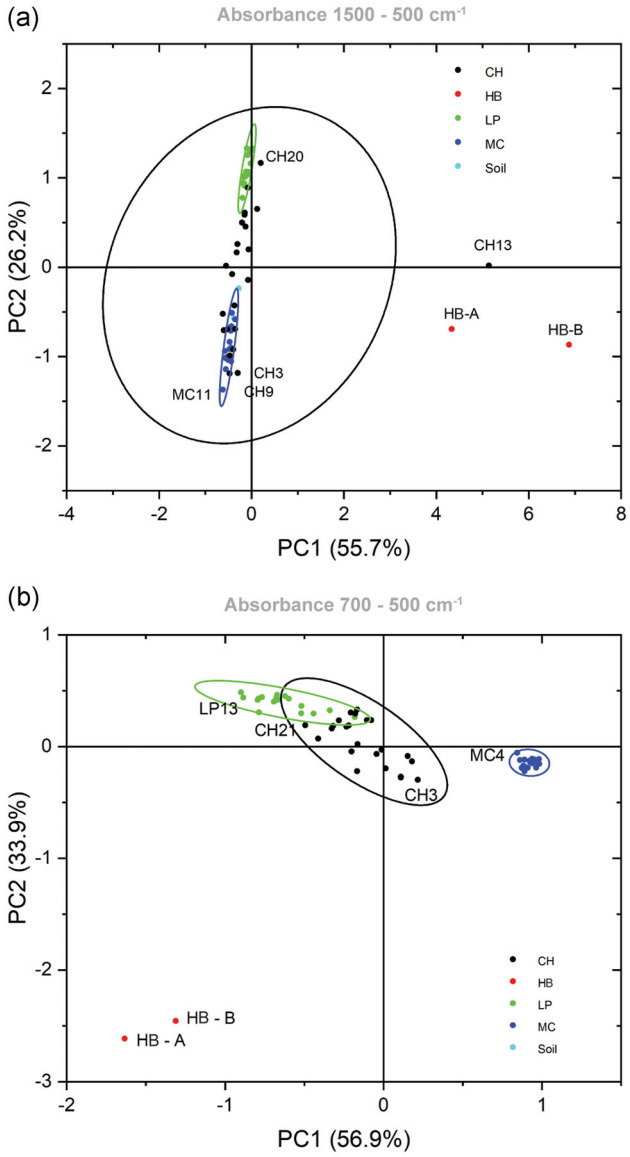

FIGURE 7 Multivariate statistics of the absorbance spectra from archaeological and reference samples (soil and bone—HB). (a) Principal component analysis (PCA) score plot for the first two principal components of the normalized absorbance spectra between 1500 and 500 cm^{-1} of archaeological samples (from sites CH, LP, and MC), natural soil, and bone mineral (HA-A and HA-B). Archaeological samples form overlapping clusters in the center of the score plot. (b) PCA score plot for the first two principal components of the normalized absorbance spectra between 700 and 500 cm^{-1}. Note how the same samples form three distinct groups. CH, CH2D01; LP, Las Palmas mound; MC, Mario Chafalote cairn [Color figure can be viewed at wileyonlinelibrary.com]

3.3 | Micromorphology

Micromorphological analyses of MC and LP showed typical characteristics of A horizons, such as granular, channel-vughy, or spongy microstructures; numerous channel and chamber voids; and passage features (Figure 6d). In LP, the fine fraction is dark brown, with undifferentiated b-fabric made of clay and organic matter (Figure 5b–f). In MC, the fine fraction is brown, containing more clay than organic matter (Figure 6b–f). MC sediments contain a millimetric fragment of a dislocated surface crust (Figure 6f). In both sites, the coarse fraction contains angular to subangular quartz grains and local rock fragments. Rock fragments are more abundant in the sediments from MC (Figure 6b,c), while LP has a higher frequency of quartz grains (Figure 5b,c).

A single fragment of charcoal (500 µm) was identified in one sample from MC (Figure 6e), but no other anthropogenic

TABLE 2 Cumulative percentages of the correlation matrix for principal components 1 to 10 (PC1–PC10) of archaeological samples from CH, LP, and MC

	Absorbance spectra		Second derivative	
	1500–500 cm^{-1}	700–500 cm^{-1}	1500–500 cm^{-1}	700–500 cm^{-1}
	7A	*7B*	*7C*	*7D*
PC1	55.7	56.8	69.6	80.8
PC2	81.9	90.8	86.5	92.9
PC3	92.6	98.0	93.6	95.1
PC4	96.4	98.8	95.7	96.8
PC5	98.8	99.2	97.2	98.3
PC6	98.9	99.5	97.9	98.8
PC7	99.5	99.6	98.4	99.1
PC8	99.6	99.7	98.7	99.4
PC9	99.7	99.8	99.0	99.6
PC10	99.8	99.9	99.2	99.8

Note: Cumulative percentages are given both for the absorbance and second derivative spectra in the spectral ranges between 1500–500 cm^{-1} and 700–500 cm^{-1}.

Abbreviations: CH, CH2D01; LP, Las Palmas; MC, Mario Chafalote.

components were described in the thin sections. Sediments from LP contain very few, 1–2 mm rounded aggregates of heated clay in the coarse fraction (<1%) (Figure 5b,c). The identification of heating in these aggregates was confirmed by previous FTIR-Attenuated Total Reflectance analyses. Only sample LP-16-2 (~20–30 cm deep) contains a micro-flake and a pottery fragment. Very few dispersed micro-fragments of charcoal (≤500 µm) and bone (≤300 µm) are described in the four samples from LP, but in concentrations below 1% in the thin sections (Figure 5d–f). All the micro-bone fragments display sharp edges and show no signs of dissolution at high magnifications.

Micromorphology revealed that bone fragments in LP are in such low concentrations that they were not detected in the FTIR and XRPD analyses (between 1 and 5 micro-fragments per slide). In MC, no micro-bones were described and this, coupled with the FTIR results showing no traces of bone mineral, confirms the absence of bones in the deposit. Unfortunately, no micromorphology samples are available CH, the only site with macroscopic bone fragments and hydroxyapatite in the FTIR measurements. No other macroscopic sources of phosphates have been reported for the analyzed sites (e.g., coprolites or other fecal material, plant remains) or identified in the thin sections.

3.4 | Multivariate analysis

To investigate the relation between our three sets of archaeological samples (CH, LP, and MC), we applied a PCA model to the normalized absorbance spectra in the 1500–500 cm^{-1} range. We plotted the archaeological samples together with our references for natural soil and bone mineral. The bone samples correspond to fragments from a human humerus and maxilla recovered from an archaeological site nearby (Table 1). The maxilla (HB-B) shows diagenetic precipitation of calcite in the FTIR spectra (peak at ~710 cm^{-1}—v_4CO_3). For the first two components, no major differences were revealed between the samples from CH, LP, and MC, which formed one overlapped cluster in the center of the score plot with two outliers corresponding to our references for bone mineral (HB-A and HB-B), and a sample from CH containing calcite (Figure 7a).

To remove areas of irrelevant variation, we applied the PCA model to the spectra between 700 and 500 cm^{-1}, which comprise the v_4PO_4 bending vibration bands of phosphates. Here, three discrete groups were revealed for the first and second components, explaining 90.8% of the variance (Figure 7b; Table 2). The three well-differentiated groups are as follows: (1) samples from CH at the center of the score plot, with higher proximity to bone, corroborating the presence of bone mineral in some of the samples, as indicated by FTIR and XRPD; (2) samples from LP, with higher proximity to samples from CH; and (3) samples from MC, which group with the natural soil and deviate from the CH, LP, and bone mineral samples (Figure 7b).

When using the PCA model in the second derivative spectra between 1500 and 500 cm^{-1}, the same three discrete clusters are revealed for the first and second components, which explain 86% of the variance (Figure 8a; Table 2). The soil samples cluster with the sediments from MC and samples from CH group closer to the bone references (sample CH13 is closest to the bone references because of its calcite content). This means that samples can be grouped and classified when using the PCA model in a wider spectral range of the second derivative.

The PCA model of the second derivative spectra between 700 and 500 cm^{-1} shows the same three clusters, and here, the first two components explain 92.9% of the variance (Figure 8b; Table 2). Samples from the MC cluster with the soil samples and separate from

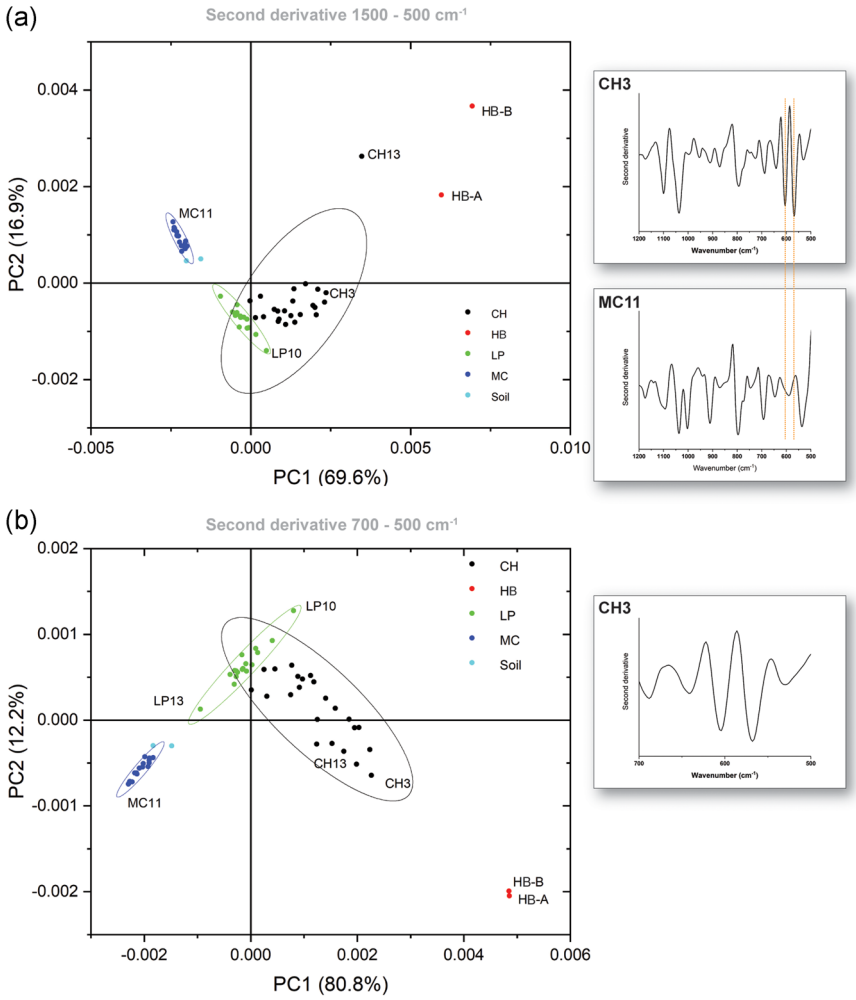

FIGURE 8 Multivariate statistics of the second derivative spectra from archaeological and reference samples. (a) Principal component analysis (PCA) score plot for the first two principal components of the second derivative spectra between 1500 and 500 cm^{-1} of archaeological samples (CH, LP, and MC), natural soil, and the bone references (HB). The archaeological samples form three discrete clusters, with partial overlapping between the CH and LP samples. Details of the second derivative spectra between 1200 and 500 cm^{-1} for sample CH3, with the two minima corresponding to the 604 and 565 cm^{-1} phosphate vibration bands, and sample MC11, without any indication of phosphates. (b) PCA score plot for the first two principal components of the second derivative spectra between 700 and 500 cm^{-1}. Details of the second derivative spectra between 700 and 500 cm^{-1} for sample CH3, containing hydroxyapatite. CH, CH2D01; LP, Las Palmas mound; MC, Mario Chafalote cairn [Color figure can be viewed at wileyonlinelibrary.com]

LP and CH. CH displays a wider dispersion, with some samples closer to the bone reference, due to the presence of the v_4PO_4 bending vibration bands of phosphates.

The PCA model suggests that, while some of the samples from CH display the v_4PO_4 bending vibration bands of phosphates, others do not and group with LP and MC. The narrower the spectral range for the second derivative (Figure 8b), the more separated the clusters become, pointing to a variance explained by the v_4PO_4 peaks, for the CH samples, and by other bands in the spectrum, for LP and MC. The PCA results confirm that neither the LP nor MC samples have detectable amounts of bone mineral and that differences between them, at least in the 700–500 cm^{-1} range, are due to other minerals.

4 | CONCLUSION

Earthen mounds containing low amounts of bones have challenged the traditional model for earthen mound construction in the Uruguayan lowlands, which refers to the reworking of faunal remains and other domestic refuse. Bone dissolution in acidic soils has been the most common explanation to account for the absence of bones in several earthen mounds. In this study, we applied for the first time the inexpensive and rapid technique of FTIR spectrometry, coupled with XRPD and micromorphology, to investigate the presence of bone remains in three archaeological sites of Eastern Uruguay located in similar environmental conditions. Our premise is that phosphates may still be present in the soil after dissolution of the bone mineral and be detectable by FTIR, XRPD, and micromorphology.

FTIR analyses show hydroxyapatite exclusively in samples from the CH mound, indicating the presence of burnt bones in the sediments, as has been shown by previous zooarchaeological studies (Bica, 2020; Moreno, 2014). The samples containing hydroxyapatite come from the upper layers of the site, dated between 1830–1389 and 232–124 cal B.P. At the LP site, hardly any macroscopic bones were recovered in the excavation, and neither FTIR nor XRPD revealed traces of bone mineral or other phosphates. The same situation was reported for the MC site, indicating that bones were never deposited at the sites.

Micromorphology confirmed that bone mineral or other recrystallized phosphates are completely absent from MC, but revealed very few (less than 1%) micro-bone fragments in LP whose concentration is below the detection limit of FTIR (and XRPD). Unfortunately, no micromorphology samples were available for the CH

site, which was excavated in the 1990s and no longer exists. The PCA model once again confirmed that some CH samples show the v_4PO_4 bending vibration bands of phosphates, grouping them closer to the bone reference, while samples from LP and MC do not, and group with natural soils.

Our study shows that samples from the LP mound and the MC cairn are intrinsically different from the CH mound, and do not contain any detectable traces of bone mineral or burnt bone. The compositional differences between the CH and LP earthen mounds do not result from taphonomic processes, such as intense bone dissolution in corrosive environments. Macroscopic bone fragments are abundant in the CH site and this, together with the noticeable presence of burnt bones in the sediments (see Bica, 2020; Moreno, 2014), confirms the use of domestic waste as the primary material for mound construction.

The very few bone fragments seen in the thin sections from LP were not detected by FTIR and do not derive from large amounts of bones that ultimately dissolved, despite the low pH reported for local soils. This points to different uses and building processes for both anthropogenic structures (e.g., CH and LP mounds), despite their proximity, close chronology, and cultural affinity. The ubiquitous heated clay aggregates described in the thin sections from LP could be signaling a different history for this site. Heated clay aggregates are present from the base to the top of the mound, while pottery only appears in the upper layers of LP, about 500 years ago (thermoluminescence age). Heated clay aggregates have also been described in the sediments from CH and La Tapera sites (del Puerto et al. 2021), indicating the potential use of these hard, coarse aggregates as building material for the mounds.

Earthen mounds with very low amounts of bone remains are common in the Uruguayan lowlands (e.g., Pago Lindo, Lemos PR14D01, Rubio, Cañada de los Caponcitos) (Cabrera & Marozzi, 2001; Gianotti, 2005; Gianotti et al., 2013; Villagran & Gianotti, 2013), and could be the result of mound technologies where coarse-sized domestic residues (e.g., bones, charcoal) are not used as prime material for mound building. Mounds could result from the preparation of special deposits for horticulture (e.g., elevated gardens), with high porosity and granular textures produced after reworking of surface A horizons, rich in organic matter (Gianotti, 2015, 2021). Another possibility could be related to the construction of mounds for specific activities using local soils, whose surfaces were regularly cleaned out of organic waste. The regular cleaning of a surface removes most macro and even microscopic components from the sediments (Milek, 2012). At the Los Ajos site, Iriarte (2003) interpreted the regular cleaning of specific occupation surfaces inside the excavated mounds. This was also suggested for the Pago Lindo earthen mound complex (Gazzán, 2018; Villagran & Gianotti, 2013), located in a similar environment in central-eastern Uruguay, where microscopic bones are absent and macroscopic bones are rare in sedimentary matrices made of reworked A horizons. Several lines of evidence point to multiple uses and life histories for the earthen mounds, through the active management of domestic waste and natural soils, which are possible within the context of community-based villages that built hundreds of earthworks in the Uruguayan lowlands (Gianotti, 2015; Iriarte, 2003).

In the case of the MC cairn, the absence of any indication of macroscopic and/or microscopic bone remains, plus no detectable bone mineral, hydroxyapatite, or other secondary phosphates, contradicts historic sources suggesting that cairns were used to bury tribal chiefs. Other uses, such as points of observation or a location to make fire signals, may be attributed to this specific site, and more excavations are needed to test the hypothesis raised by ethnographic sources.

Future studies should focus on understanding the chemistry of local soils and its effects on the preservation of archaeological materials. The revelation of micro-bone fragments at LP, whose concentration was not detected by FTIR or XRDP analyses, points to the need to establish the detection limit of bone mineral in FTIR measurements of complex spectra. Quantifying the detection limit of bone mineral in FTIR measurements could potentially be used to estimate the amount of weathered bone that can contribute to detectable bone mineral in archaeological sediments and soils.

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