



## Buried in ashes: Site formation processes at Lapa do Santo rockshelter, east-central Brazil



Ximena S. Villagran <sup>a, b, \*</sup>, André Strauss <sup>c, d, e, f</sup>, Christopher Miller <sup>b</sup>, Bertrand Ligouis <sup>g</sup>, Rodrigo Oliveira <sup>e</sup>

<sup>a</sup> Museu de Arqueologia e Etnologia – Universidade de São Paulo, Av. Prof. Almeida Prado 1466, 05588070 São Paulo, Brazil

<sup>b</sup> Institute for Archaeological Sciences – University of Tübingen, Rümelinstr. 23, 72070 Tübingen, Germany

<sup>c</sup> Senckenberg Center for Human Evolution and Paleoenvironment – University of Tübingen, Rümelinstr. 23, 72070 Tübingen, Germany

<sup>d</sup> Department of Human Evolution – Max Planck Institute for Evolutionary Anthropology, Deutscher Platz 6, 04103 Leipzig, Germany

<sup>e</sup> Laboratório de Estudos Evolutivos e Ecológicos Humanos – Universidade de São Paulo, Rua do Matão 277, 05508090, Brazil

<sup>f</sup> Centro de Arqueologia Annette Laming-Emperaire, Rua Acadêmico Nilo Figueiredo 62, 33400-000 Lagoa Santa, Brazil

<sup>g</sup> Laboratory for Applied Organic Petrology (LAOP) – Institute for Archaeological Sciences, University of Tübingen, Rümelinstr. 23, 72070 Tübingen, Germany

### ARTICLE INFO

#### Article history:

Received 13 August 2015

Received in revised form

27 June 2016

Accepted 12 July 2016

Available online 26 July 2016

#### Keywords:

Tropical geoarchaeology

Micromorphology

Organic petrology

μFTIR

Lagoa Santa

Experimental hearths

Paleoamerican

### ABSTRACT

Few archaeological sites in the Americas contain high concentrations of human burials dating back to the early Holocene. The tropical karstic region of Lagoa Santa, in central Brazil (state of Minas Gerais) is one of the richest bioanthropological records available to study the behaviors and funerary practices of early Holocene South Americans, with more than 200 skeletons found so far. One of the key locations to examine the history of human settlement in Lagoa Santa is the site of Lapa do Santo, a rockshelter known to contain the oldest rock art and the earliest evidence of funerary complexity in the continent. In this geoarchaeological investigation we focus on the early Holocene settlement at Lapa do Santo (7.9–12.7 cal kyBP) applying high-resolution geoarchaeological techniques, such as micromorphology, organic petrology and μFTIR, on both archaeological, modern reference and experimental samples. This is the first time that a micro-contextual approach integrated with experimental geoarchaeology has been applied to study the formation of rockshelter deposits in a tropical setting. Our results show that the stratigraphic sequence formed under the dual influence of anthropogenic sedimentation—through continuous combustion activities—and geogenic sedimentation in the form of oxisol aggregates which fell from above the limestone cliff into the rockshelter. Intact hearths and remobilized combustion debris, possibly hearth rake-out, are close to the graves suggesting repeated burning activities as part of the ritual behavior of early Holocene South Americans. Large amounts of ash are intermixed with heated and unheated oxisol aggregates. Heated termite mound fragments were also found mixed within the sediments. Post-depositional alteration of the site includes limited bioturbation and localized, low energy surface water and sub-surface concentrations of moisture, leading to precipitation of dense, secondary carbonates. The age inversions can be attributed to the human action of reworking the ashy sediments and not to post-abandonment processes. Despite this, the overall preservation of the sediments is good and most human burials can be considered to be in primary context.

© 2016 Elsevier Ltd. All rights reserved.

### 1. Introduction

The karstic region of Lagoa Santa in east-central Brazil (state of Minas Gerais) is an important location to understand the activities

and behaviors of early Holocene populations in South America. An astonishing amount of well-preserved human remains were found, the oldest being the female skeleton of “Luzia” stratigraphically dated ca. 11.3–15.1 cal kyBP (Araujo et al., 2012; Feathers et al., 2010; Neves and Hubbe, 2005; Neves et al., 1999). Peter Lund, a Danish naturalist, conducted the first research in the area in the 19th century. Lund’s observations on the skeletal remains led to bioanthropological research focused on the study of a skeletal/cranial type named “Paleoamerican”. Morphologically distinct from

\* Corresponding author. Museu de Arqueologia e Etnologia – Universidade de São Paulo, Av. Prof. Almeida Prado 1466, 05588070 São Paulo, Brazil.

E-mail address: [villagran@usp.br](mailto:villagran@usp.br) (X.S. Villagran).

most archaeological and present-day Native Americans, Paleoamericans are characterized by a morphological affinity with modern populations from Africa and the South Pacific (Neves et al., 1999, 2003, 2004, 2007; Powell and Neves, 1999; Neves and Hubbe, 2005; A. Hubbe et al., 2011).

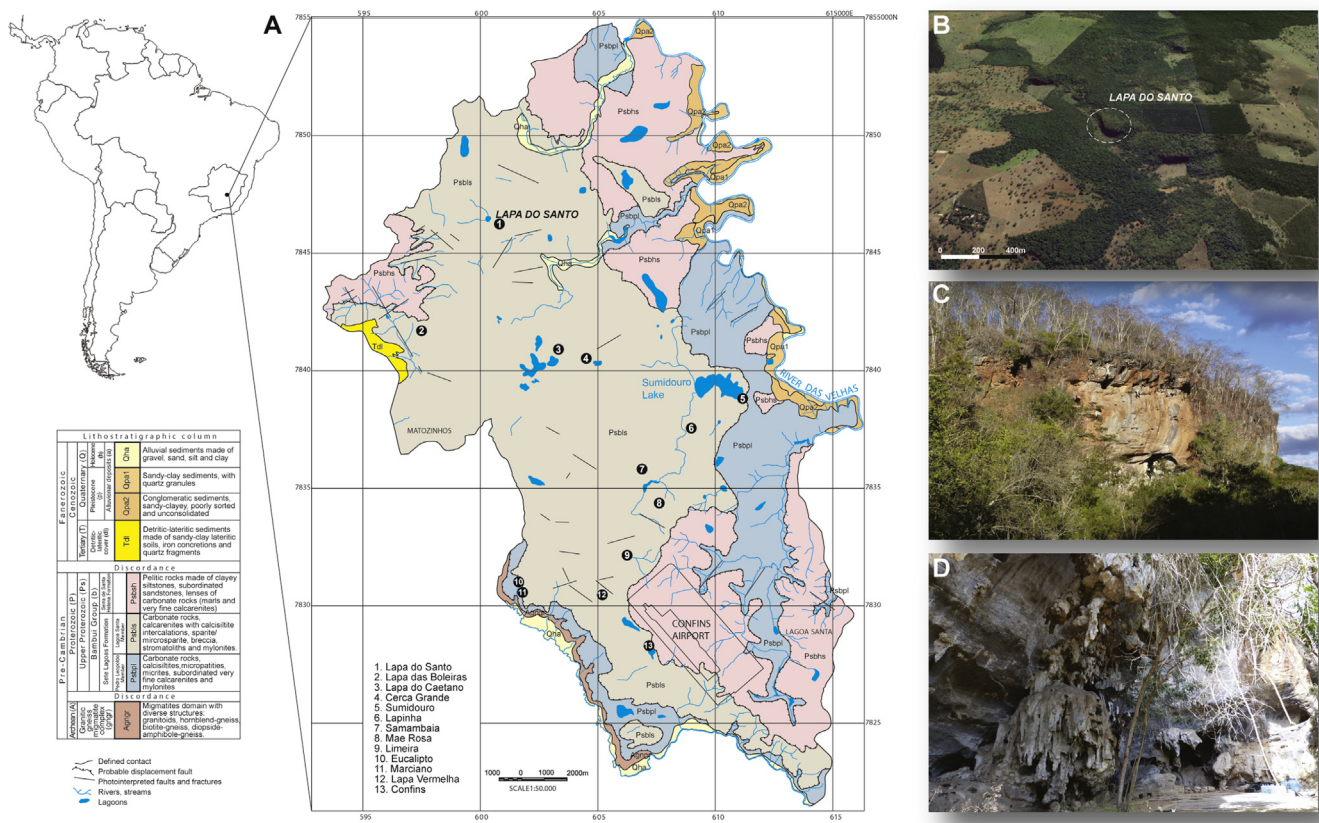
Most of the 200 skeletons recovered from the region date to the early Holocene, when systematic burial inside rockshelters first appears. The burial practices of early Holocene South Americans from Lagoa Santa were traditionally thought to be simple and homogeneous, involving primary deposition of flexed bodies in shallow graves (Araujo et al., 2012; Neves and Hubbe, 2005; Neves et al., 2012, 2002; Prous and Fogaça, 1999). Recent studies demonstrate low mobility among these groups (Da-Gloria, 2012) and novel funerary practices since early Holocene times, including complex rituals involving the *perimortem* reduction of the dead body (Neves et al., 2002; Strauss, 2010, 2016; Strauss et al., 2015).

The settlement of Lagoa Santa dates from the Paleoindian to Archaic periods (Araujo et al., 2012, 2008; Feathers et al., 2010; Laming-Emperaire, 1979; Neves et al., 2004, 1999; Prous and Fogaça, 1999; Prous, 1992). The stone tool technology typically consists of small flakes and informal tools made of local quartz and quartzite, with rare bone tools and bifacial points (Araujo and Pugliese, 2009; Araujo et al., 2012; Moreno de Sousa, 2014; Pugliese, 2008). Despite the technological differences between Lagoa Santa and other sites in central Brazil (Bueno, 2005; Bueno et al., 2013a, 2013b; Dias, 2004; Kipnis, 1998; Prous and Fogaça,

1999; Prous, 1992), the subsistence in the earliest settlements is persistently characterized by broad-spectrum strategies, including plants (roots and fruits) and small and medium-sized game (Bissaro Jr., 2008; Bueno et al., 2013a; Dias, 2004; Hermenegildo, 2009; Kipnis, 1998; Strauss et al., in press).

Adding to the long history of archaeological and bio-anthropological research in Lagoa Santa, geoaerchaeological studies have provided essential information on local sedimentation processes inside several local caves and rockshelters (e.g. Piló et al., 2005; Araujo et al., 2008, 2013; Feathers et al., 2010; M. Hubbe et al., 2011). The site of Lapa do Santo, a rockshelter with deposits dating to the Pleistocene/Holocene transition, offers one of the most complete stratigraphic sequences available to study the chronology, subsistence, funerary ritual and artistic expressions of early South Americans. The site is located in the north of the Lagoa Santa karstic region (Fig. 1A), where 33 human interments have been recovered so far. Field observations suggested that the almost 5 m of powdery grey sediments could be ash from anthropogenic fires, pointing to rapid accumulation and intensive occupation of the site. However, the possible human origin for the sediments at Lapa do Santo has until now been unconfirmed. Few data and analyses exist that explicitly study the relation between the ashy sediments and the multiple human burials.

High-resolution geoaerchaeological analysis, including micro-morphology, FTIR analysis (Fourier Transform Infrared Spectrometry) and FTIR microspectroscopy ( $\mu$ FTIR) have proven their



**Fig. 1.** Map with location of the site of Lapa do Santo in the state of Minas Gerais, central Brazil: A) Geological map of the karst of Lagoa Santa with location of Lapa do Santo and other archaeological sites in rock shelters (geological map adapted from IBAMA-CPRM, 1998); B) Satellite image of the limestone massifs where Lapa do Santo is located (image from GoogleEarth); C) Photograph of the limestone wall above the site; D) Photograph inside the rock shelter.

potential to study archaeological sites in caves and rockshelters (e.g., Berna et al., 2004, 2012; Goldberg and Sherwood, 2006; Schiegl and Conard, 2006; Karkanas and Goldberg, 2007; Goldberg et al., 2009; Mallol et al., 2009; Feathers et al., 2010; Goldberg and Berna, 2010; Wadley et al., 2011; Miller et al., 2013). Microstratigraphic analyses are especially suitable to evaluate the relative input of geogenic vs. anthropogenic sediments in archaeological deposits. The use of high-resolution techniques also allows us to identify past human activities that are only recorded in the sediments themselves.

In this paper, we focus on the early Holocene occupation of the site where most of the human remains are found. We conducted high-resolution geoarchaeological analyses with the goal of understanding the nature and mode of accumulation of the sediments that contain the interments and archaeological remains. We present data from micromorphology,  $\mu$ FTIR and organic petrology, testing the utility of a micro-contextual approach for the study of rockshelters in tropical settings. The data are combined with a complete set of reference samples of local soils and sediments and with experimental heating studies for a comprehensive interpretation of the archaeological sediments. The wide-scale sampling strategy aims at covering all the possible components involved in site formation, from natural to anthropogenic ones to investigate the occupation and ritual use of the rockshelter.

### 1.1. Lapa do Santo and the karst of Lagoa Santa

Lapa do Santo consists of a wide sheltered area of approximately 1300 m<sup>2</sup> at the base of a ca. 30 m-high limestone massif that rises from a doline valley (Fig. 1A–D). The karst is formed in Upper Pre-Cambrian metasedimentary rocks of the Bambuí Group, with a basal metacalcareous body corresponding to the Sete Lagoas Formation (dated 740 ± 22 Ma, Babinski et al., 2006) covered by metapellitic rocks (siltstones and claystones) of the Serra de Santa Helena Formation (IBAMA-CPRM, 1998) (Fig. 1A). Dissolution of the Sete Lagoas limestone resulted in several karstic features on the landscape, such as caves and doline lakes. The soil cover is dominated by clayey, hematite-rich, red Oxisols (*latossolos* in the Brazilian Soil System) over yellow, goethite-rich oxisols developed on the Serra de Santa Helena metapellites (Araujo et al., 2013; IBAMA-CPRM, 1998; Piló, 1998). Oxisols are highly weathered soils, rich in Fe-(hydr)oxides (e.g. hematite, goethite), Al-(hydr)oxides (e.g. gibbsite) and resistant clay minerals (e.g. kaolinite) (Embrapa, 2006; Schaetzl and Anderson, 2005; Soil Survey Staff, 2003).

The modern climate is tropical semi-humid, with mean annual temperature of 22° C, rainy summers and dry winters. The vegetation cover is dominated by *Cerrado* (woody savanna) and semi-deciduous forests, although in most flat areas the natural vegetation has been replaced by crops and pastures (CPRM, 2010).

Lapa do Santo was excavated between 2001 and 2009 as part of a project entitled “Origins and microevolution of man in America: a paleoanthropological approach” coordinated by Prof. Walter Neves (FAPESP 99/0670-7). The site is currently protected and no visitors are allowed. The work exposed an area of 30.5 m<sup>2</sup> in the southern portion of the site containing 26 well-preserved human burials (Fig. 2A). A total of 52 radiocarbon ages were determined on charcoal fragments and ten on human bones (Araujo et al., 2012; Strauss et al., in press). The radiocarbon ages cluster in three distinct and non-overlapping chronological periods corresponding to the early, mid and late Holocene: 1) 7.9–12.7 cal kyBP; 2) 3.9–5.4 cal kyBP; 3) 0.0–2.1 cal kyBP (intervals of 95.4%) (Strauss et al., in press).

The human burials are dated between ca. 8.2–10.6 cal kyBP and are stratigraphically restricted to the uppermost 1 m of sediment. They include primary and secondary burials, some of them with evidence for decapitation, defleshing and tooth removal (Strauss, 2010, 2016; Strauss et al., 2015, in press). A 30 × 20 cm petroglyph consisting of an anthropomorphic figure occurs at the base of the sedimentary succession, reaching 4 m below the surface. The petroglyph was dated by radiocarbon and OSL between ca. 9200 and 12,500 BP, making it the oldest evidence of rock art in the Americas (Neves et al., 2012).

Excavations at the site resumed in 2011 through the project “The mortuary rituals of the first Americans”, coordinated by André Strauss (Department of Human Evolution, MPI-EVA). Two new excavations were opened: a main area of 10,5 m<sup>2</sup> to the east of the 2001–2009 excavation and a second area of 2 m<sup>2</sup> (Fig. 2A and D, Fig. 4A). During three field seasons (2011, 2012 and 2014) seven human burials were exhumed from the main area (Fig. 4A). The chronology of the archaeological record in the areas of the new excavation is based on 14 radiocarbon ages, 13 of which are between 9.4 and 10.1 cal kyBP (Fig. 4B) (see supplementary material online 1 for the complete list of dates).

An understanding of the sedimentary dynamics inside the rockshelter is essential for reconstructing the formation processes of Lapa do Santo. The topography of the sheltered area shows a south-north slope of approximately 10° that dips into a sinkhole (Fig. 2A–C). Watermarks on the limestone wall about 4 m above the modern surface suggest an area of intermittently ponded water (see Fig. 2A and B). A colluvial fan in the northwest part of the site dips into the sinkhole and consists of soil material derived from the oxisol on top of the limestone (Fig. 2C.1). The rockshelter shows an east-west slope of approximately 45° near the edges that flattens to the east.

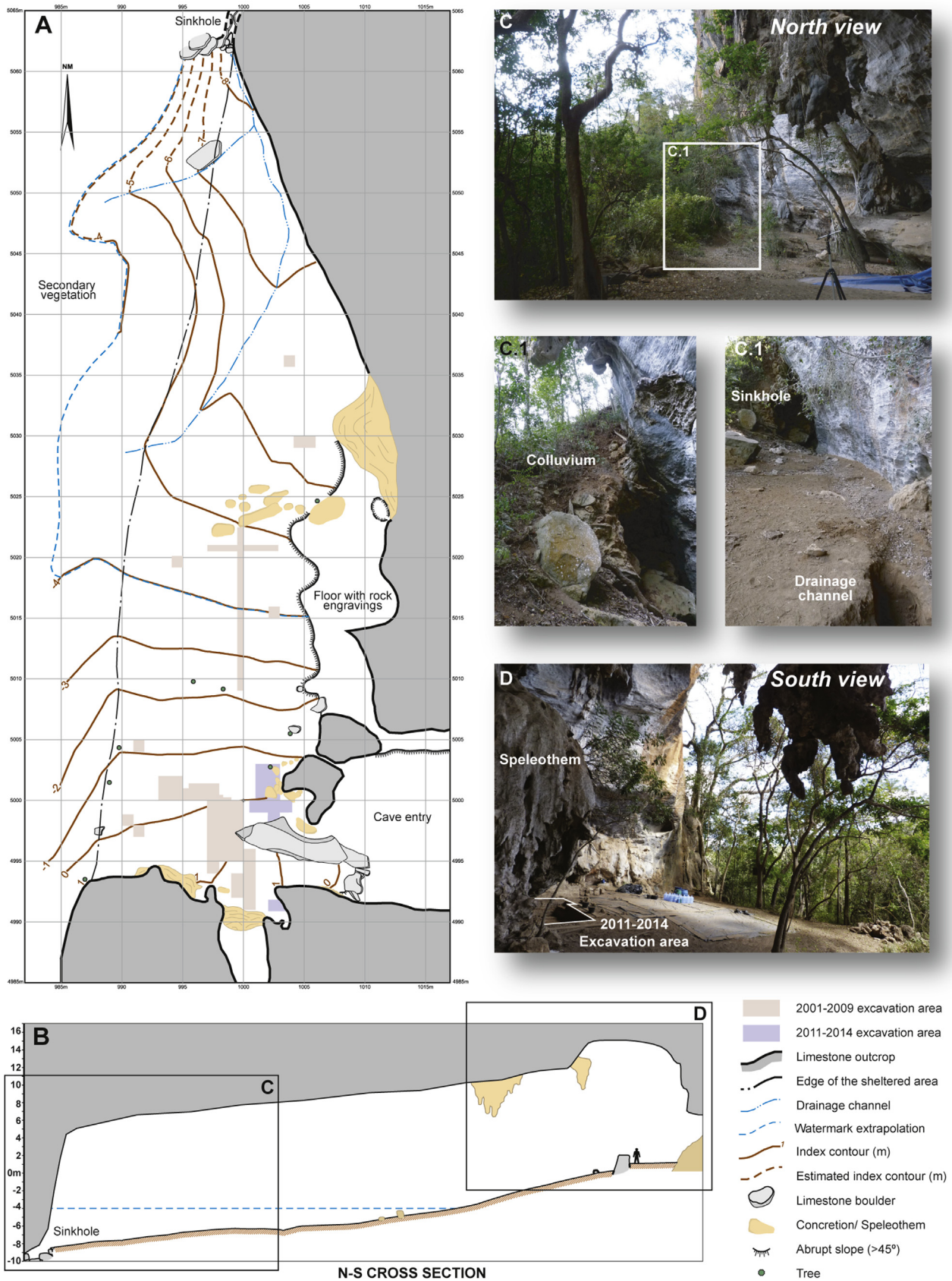
Most excavations are located to the south of the rockshelter, on its highest part. Neither colluvial fans, nor chimneys were observed in this location (Fig. 2D). About 6 m south of the 2011–2014 excavation, an outcrop of reddish, loamy breccia consisting of limestone fragments fills a dissolution pipe (Fig. 3D and F). Large speleothems occur over the excavation area and several speleothem fragments have fallen to the ground and are now contained within the archaeological sediments.

## 2. Materials and methods

In this work we focus on the 2011–2014 excavation area, using a micro-contextual approach (Goldberg and Berna, 2010), integrating micromorphology,  $\mu$ FTIR and organic petrology to better understand the sediments in Lapa do Santo and study the context of the archaeological findings (artifacts and human interments). Micromorphology is the study of intact blocks and thin sections of sediment and soil, often under magnification (Courty, 2001; Courty et al., 1989). This approach allows us to identify the components of deposits and soils, and also study their spatial and stratigraphic relationship to one another. This approach is especially useful at archaeological sites found in caves and rockshelters, where it can provide high-resolution data that can help resolve complex stratigraphic issues and formation processes (Goldberg and Arpin, 1999; Goldberg and Sherwood, 2006; Goldberg, 2000; Homsey and Capo, 2006; Karkanas, 2000; Mallol et al., 2010; Schiegl, 1996; Weiner, 2009).

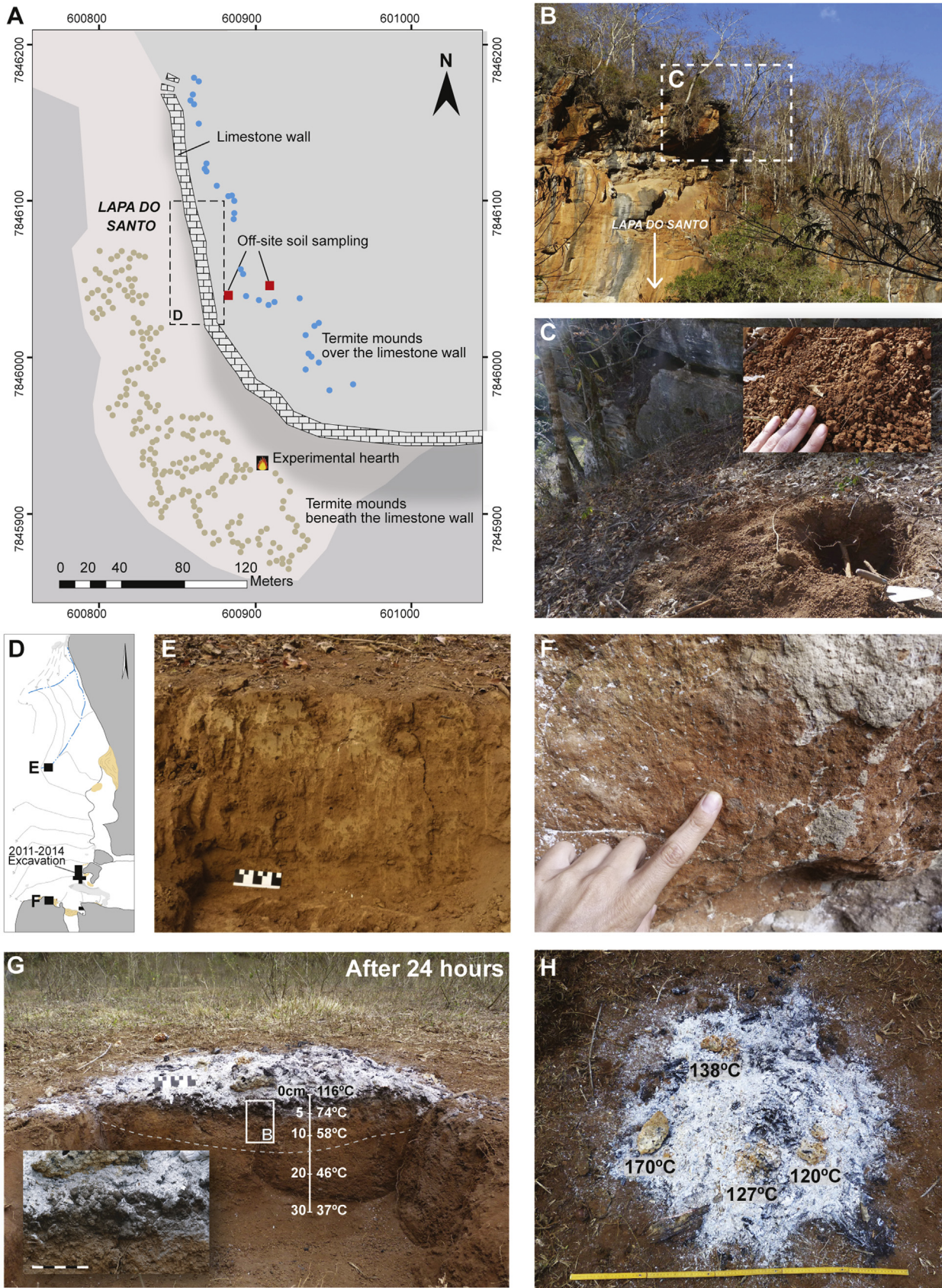
In archaeological contexts, FTIR analyses have proven especially useful for the identification of diagenetic minerals and heated materials (Weiner et al., 2002; Berna et al., 2007; Mallol et al., 2009; Miller et al., 2013; Stahlschmidt et al., 2015a, 2015b). Recently,  $\mu$ FTIR has become an ideal, complementary





**Fig. 2.** Topography and photographs of Lapa do Santo rockshelter: A) Topography of the site with location of the two main excavation areas; B) Schematic cross-section of the rock shelter from north to south; C) North view of the rock shelter with detail of the red colluvium and sinkhole (C.1); D) South view of the rock shelter with indication of the 2011–2014 excavation area. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)





**Fig. 3.** Off-site sampling for reference and experimental studies: A) Schematic view of the limestone massif from above with location of the site (D), sampling locations for the red oxisol, the experimental hearth and the termite mounds; B) View of the topmost part of the limestone wall above the site, where the oxisol samples were collected; C) Photograph



technique for micromorphological studies, as it allows for the identification of minerals and heated materials directly in the thin section (Berna and Goldberg, 2007; Berna et al., 2012; Goldberg and Berna, 2010).

Organic petrology is also a useful, complementary technique to soil micromorphology. It allows not only the precise identification of burned organic material within the sediments, but also the classification of the remains according to the type of tissue, degree of burning (from slightly heated to completely carbonized), weathering and permineralization (Goldberg et al., 2009; Ligouis, 2006; Villagran et al., 2013; Stahlschmidt et al., 2015a, 2015b).

For this work, two sets of samples were collected for analysis, in order to cover all the possible sedimentary agents involved in the formation of the deposit: 1) archaeological samples, taken from the excavation surfaces and stratigraphic profiles. 2) Reference and experimental samples from local sediments, soils, termite mounds and an experimental hearth to identify the effects of surface heating on tropical oxisols.

### 2.1. Reference and experimental sampling

To assess the input of local geogenic sediments and soils, bulk samples and undisturbed blocks for micromorphological and  $\mu$ FTIR analysis were collected from: 1) the red oxisol developed over the limestone outcrop (Fig. 3A–C); 2) the colluvial deposits in the northern portion of the site (Fig. 3D–E); 3) the reddish breccia south of the 2011–2014 excavation area (Fig. 3D and F).

To serve as reference for identifying the thermal alteration of local clay, red oxisol aggregates were heated in a porcelain crucible for 4 h using a muffle furnace at temperatures of 200 °C, 300 °C, 400 °C, 500 °C, 600 °C, 700 °C, 800 °C, 900 °C and 1000 °C, following the protocol of Berna et al. (2007). The heated aggregates were impregnated, cut into 30  $\mu$ m-thick thin sections and analyzed with a petrographic microscope and  $\mu$ FTIR following Miller et al. (in prep.).

Another set of samples was taken from an experimental hearth lit over the red oxisol, about 80 m southeast of the site (Fig. 3A and G). The experiment was made to observe the properties of thermally altered oxisols and the extremely abundant termite mounds in simulated field conditions. Logs, branches and dry grasses from the local vegetation were used as fuel, as well as nuts from palm trees and fragments of termite mounds (Fig. 3A). The fire was kept for 2 h and the temperature of the flames, the ground surface and the substrate (ca. 2–5 cm below the surface) was measured using a thermocouple and a digital thermometer. The temperature of the remaining ashes and the substrate was measured for 2 h after the fire was extinguished and 24 h later during sampling (Fig. 3G). The temperature inside the termite mound fragments was measured during the fire and after it extinguished (Fig. 3H).

### 2.2. Archaeological sampling

Undisturbed blocks of sediment were taken at depths between 10 and 90 cm (Figs. 4C and 5). Sample collection focused on sedimentary changes seen in the excavation surfaces, as opposed to profile sampling ( $n = 30$ ). This was done to identify

potential differences in sedimentation or weathering related with activity areas and/or taphonomic processes, respectively. However, the horizontal collection indirectly favored sampling of compacted layers over friable sediments. Therefore, another three undisturbed blocks were collected from an excavation profile to serve as stratigraphic reference (Fig. 4D). Fig. 5 shows the location of the micromorphology samples at different depths.

### 2.3. Analysis – micromorphology, organic petrology and $\mu$ FTIR

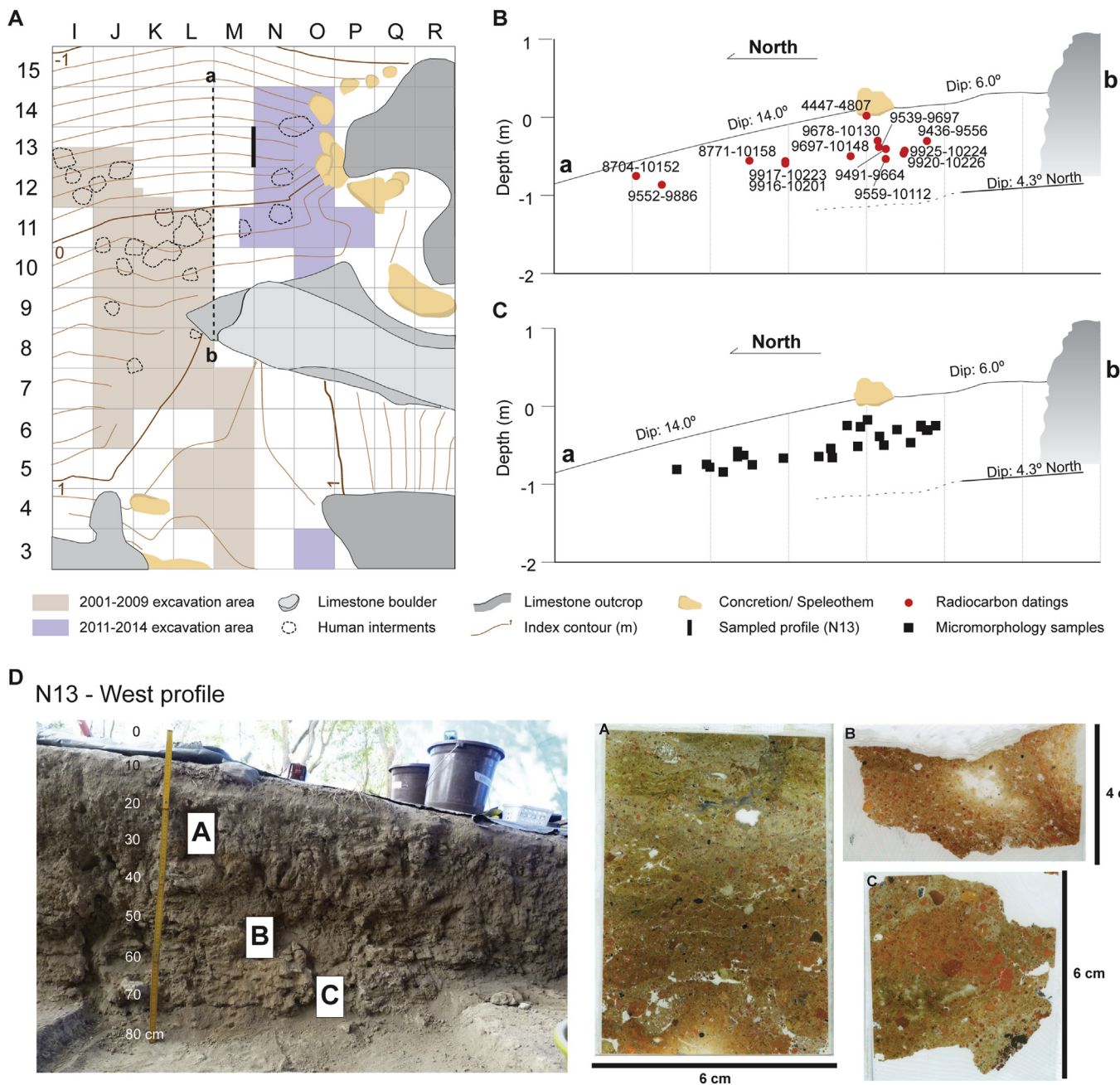
All micromorphology blocks were oven-dried and impregnated with a mixture of polyester resin (Viscovoss N5), diluent (Styrene) and catalyst (MKEP). Thin sections of 9 × 6 cm and 30  $\mu$ m thick were made out of the impregnated blocks and analyzed with a Stemi 2000-C stereomicroscope and Zeiss Axio Imager A2 petrographic microscope under plane polarized light (PPL), cross-polarized light (XPL) and fluorescent light (blue light) at magnifications ranging from ×8.0 to ×500. Micromorphological descriptions followed the guidelines of Stoops (2003).

All the thin sections from Lapa do Santo were used for  $\mu$ FTIR analyses. Analyses were done with a Cary 610 Series FTIR microscope with Resolutions Pro software (Agilent Technologies). Spectra were collected both in transmission (64 scans), and attenuated total reflectance using a germanium crystal (ATR – 32 scans), with wavenumbers ranging between 4000 and 400  $\text{cm}^{-1}$  at 4  $\text{cm}^{-1}$  resolution. Transmission measurements have high resolution at the hydroxyl (OH) region of clays (wavenumbers between 3550 and 3750  $\text{cm}^{-1}$ ), allowing for a more precise identification of changes in the spectra due to heating. In this region of the spectrum the glass slides do not interfere with the measurements, since amorphous silicates are transparent to the IR radiation at wavenumbers above 2500  $\text{cm}^{-1}$  (Beauvais and Bertaux, 2002). For transmission measurements, the background was collected on a portion of the slide with spectra resulting exclusively from the absorption of the glass and the resin used for impregnation. For ATR spectra background measurements were taken on air.

Of the 33 undisturbed archaeological samples nine were selected for analysis with organic petrology. Five of the impregnated blocks were dry-polished and analyzed with reflected white light (RLo) and incident ultra violet light (RVLo) under oil immersion (Taylor et al., 1998) using a Leica DMRX/MPV-SP microscope photometer (50 × to 500 × magnifications). Complementary analyses were done with PPL and XPL. Four thin sections were dry polished for the same analysis. The description and classification of organic micro-components followed the nomenclature for brown coal and coal (ICCP, 2001, 1998; Sykorová et al., 2005; Taylor et al., 1998). Reflectance measurements were done on plant tissues identified in the polished blocks and one polished thin section to determine the degree of humification and/or carbonization (Borrego et al., 2006; Guo and Bustin, 1998; Jacob, 1980; Jones et al., 1991; Schwaar et al., 1990). The random reflectance in oil (mean % Rr) of the organic micro-components was measured following the procedure in Taylor et al. (1998).

of the trench dug to collect the oxisol sample, with details of characteristic granular structure; D) Topography of the rock shelter with indication of the locations where samples from the colluvium (E) and red breccia (F) were collected; E) Detail of the colluvium deposits north of the site; F) Detail of the red breccia south of the 2011–2014 excavation; G) Experimental hearth lit on red oxisol substrate, with indication of the temperature of the substrate at various depths; H) The experimental hearth with the heated termite mound fragments and their temperatures after the fire extinguished. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)





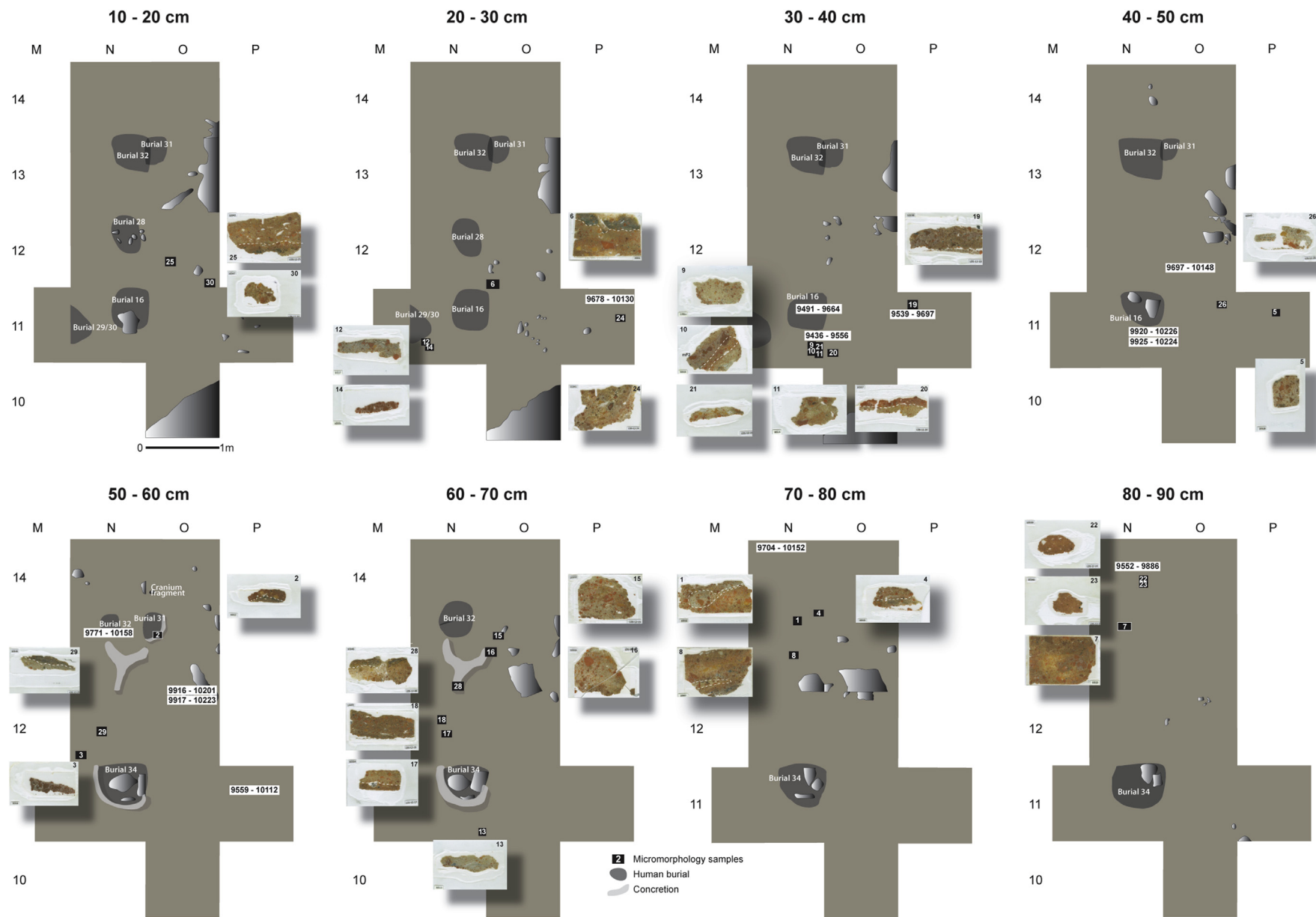
**Fig. 4.** Sampling for micromorphology and radiocarbon dating: A) Close-up of the topography with the two main excavations at Lapa do Santo (2001–2009 and 2011–2014) showing the location of human burials; B) Cross section of the 2011–2014 excavation with vertical location of samples for radiocarbon dating (age intervals of 95.4% in cal yrsBP); C) Same as B with vertical location of micromorphology samples; C) Profile sampled for micromorphology with location of sampling boxes and scanned view of the thin sections.

### 3. Reference and experimental results

#### 3.1. Local oxisol, red breccia and colluvium

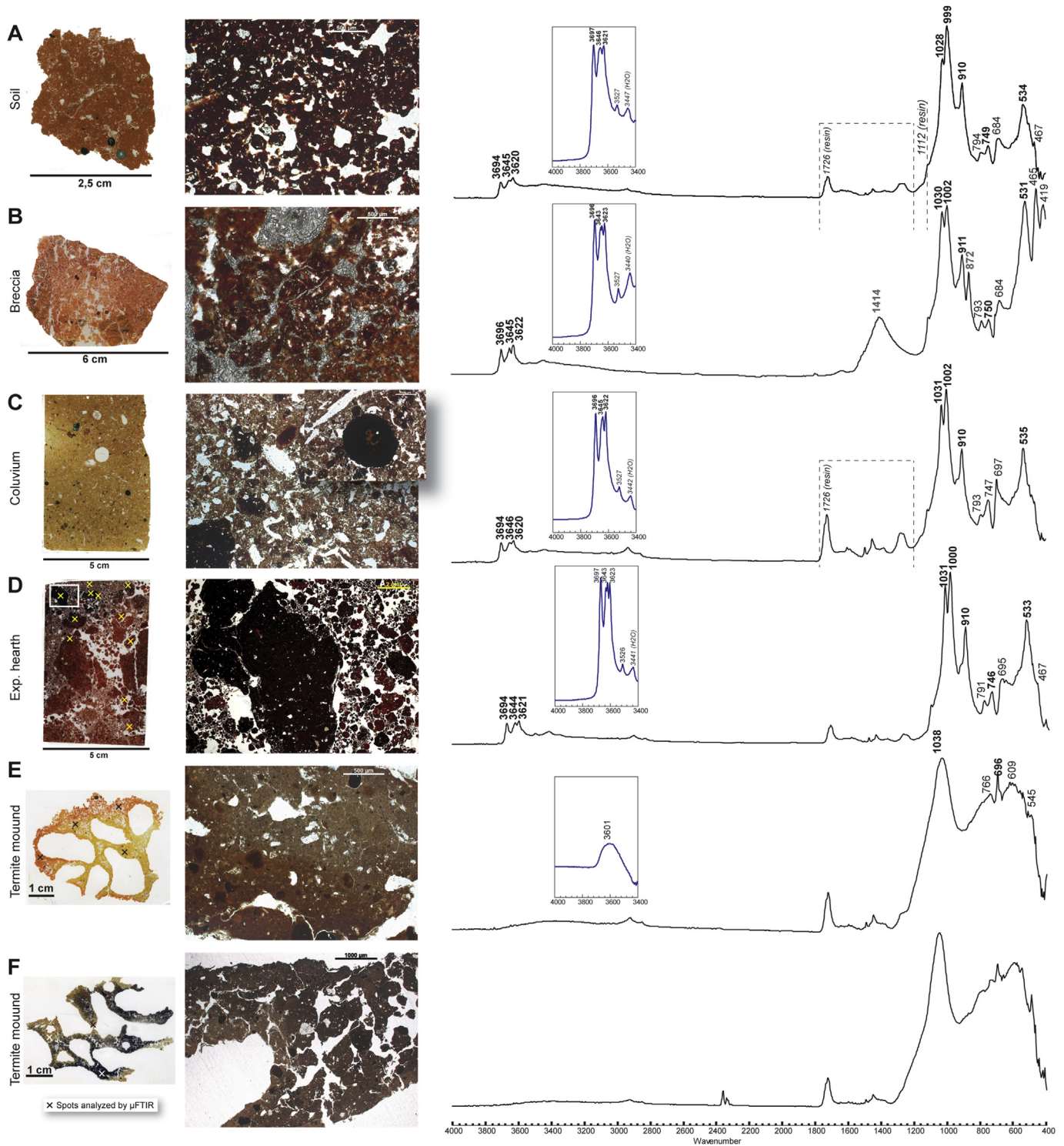
Red oxisols near Lapa do Santo are made of homogeneous, red colored (PPL), coalesced granules, with mammillated chamber and channel voids, and undifferentiated b-fabric due to high Fe-oxide content: all typical micromorphological attributes of tropical oxisols (see Marcelino et al., 2010) (Fig. 6A). Fine to very fine sand-sized grains of quartz are frequent, large Fe(hydr)-oxide nodules, rock fragments and opaque grains are rare. The ATR spectrum of the oxisol shows the presence of kaolinite in the clay fraction

(absorption bands at 3694, 3645, 3620, 1028, 999, 910, 749, 534  $\text{cm}^{-1}$ ), with small amounts of quartz (794 and 684  $\text{cm}^{-1}$ ). The kaolinite bands at the OH region are also readily seen in the transmission spectrum (3697, 3646, 3621  $\text{cm}^{-1}$  and shoulder at 3666  $\text{cm}^{-1}$ ) (see Beauvais and Bertaux, 2002), together with an absorption band corresponding to gibbsite (3527  $\text{cm}^{-1}$ ) (Fig. 6A). The main interference of the resin is seen between 1200 and 1800  $\text{cm}^{-1}$ , with only minor interference between 800 and 700  $\text{cm}^{-1}$ . In general, the low porosity of the material prevents the resin to completely mask the main clay peaks in the spectra, although slight shifts in the main clay peaks are sometimes observed.



**Fig. 5.** Schematic view of the excavation surface at the different depths where micromorphology samples were collected, including scanned view of the thin sections and location of the radiocarbon datings (age intervals of 95,4% in cal yrsBP).





**Fig. 6.** Reference and experimental data: Scanned thin sections from the red oxisol (A), red breccia (B), colluvium deposit (C), experimental hearth on oxisol substrate (D), and heated termite mound fragment with orange (E) and black (F) colors, with photomicrograph and ATR- $\mu$ FTIR spectra, including detail of transmission spectra at the hydroxyl region of clays (blue line). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

The red breccia that outcrops south of the 2011–2014 excavation area shows a clayey red matrix (PPL), with undifferentiated b-fabric and dense sparite infillings.  $\mu$ FTIR analysis shows kaolinite and calcite in its composition, with small amount of quartz and gibbsite (Fig. 6B). The colluvial deposit north of the excavation area has a heterogeneous composition made of speckled to cloudy clay with

weak stipple-speckled b-fabric, coarse Fe-(hydr)oxide nodules, plant remains, rock fragments and charcoal fragments. Channel and chamber voids are dominant, indicating intense bioturbation. Surface crusts are frequent, suggesting reworked surfaces by colluvial transport. As in the oxisol and red breccia, kaolinite is the predominant clay mineral with some gibbsite and quartz.  $\mu$ FTIR

analysis also showed that several of the Fe(hydr)-oxide nodules are goethite (Fig. 6C).

### 3.2. Heated clay from local oxisols

A distinct change in color is the most evident macroscopic alteration of the heated oxisol aggregates (Fig. 7). Kaolinite is identified in the soil aggregates heated from 200° C to 500° C, both in the ATR and transmission spectra, at the main Si–O–Si peak ( $\sim 1030\text{ cm}^{-1}$ ) and the OH region, respectively (Fig. 7). The interference of the resin is mainly seen in the absorption band at  $1045\text{ cm}^{-1}$  in the sample heated at 200° C, and in the broad peak at  $1038\text{ cm}^{-1}$  for the sample heated at 300° C; both caused by a main resin peak at  $\sim 1066\text{ cm}^{-1}$ . The spectra for the aggregates heated above 600° C are consistent with previous FTIR studies on heated kaolinite (Berna et al., 2007; Friesem et al., 2013; Karkanas and Koumouzelis, 2004; Shoval and Beck, 2005; Shoval, 1994; Shoval et al., 2011) which show temperature-induced changes in the OH region and the main Si–O–Si peak (Fig. 7). Changes in the OH region are caused by the dehydroxylation of the clay minerals and loss of structurally bound water. The changes in the main Si–O–Si band are associated to the destruction of the clay minerals, formation of an amorphous phase (named metakaolinite) and crystallization of new minerals at higher temperatures (Berna et al., 2007; Friesem et al., 2013; Karkanas and Koumouzelis, 2004; Shoval and Beck, 2005; Shoval, 1994; Shoval et al., 2011).

The transmission spectra show a broad absorbance peak in the OH region above 600° C, which progressively decreases in intensity at higher temperatures and shifts to higher wavenumbers (Fig. 7). Similar abrupt changes at 600° C are also seen in the ATR spectra at the main Si–O–Si peak and the Al–O–H absorbance peak ( $\sim 910\text{ cm}^{-1}$ ): the Si–O–Si peak broadens and the Al–O–H peak disappears. The shift to lower wavenumbers seen at the main Si–O–Si peak is caused by interference of the resin. The shift to higher wavenumbers that characterizes heated kaolinite (see Shoval, 1994; Shoval and Beck, 2005; Shoval et al., 2011; Friesem et al., 2013) is only seen above 800° C.

The results of the  $\mu$ FTIR analysis of heated oxisol aggregates indicate that clear alterations of the clay due to heating (at the OH region, main Si–O–Si and Al–O–H bands) will only be identifiable after exposure to temperatures above 500–600° C.

### 3.3. Hearth over red oxisol substrate and heated termite mounds

The maximum temperature reached by the experimental fire was 875° C, with a mean temperature of  $\sim 650$ ° C in the flames. The maximum temperature in the substrate (2–5 cm deep) reached 236° C, with a mean of  $\sim 110$ ° C (see supplementary material online 2 for further information on the experimental hearth).

The first 5 cm of heated soil beneath the ashes showed dark brown to black color with coarse granular structure, turning gradually red with increasing depth (Fig. 3G). No rubefication occurred, since the soil is naturally rich in red iron oxides. Despite the color changes underneath the fire,  $\mu$ FTIR analysis shows no signs of alteration of the clays (Fig. 6D). Even the clay at the topmost part of the blackened lens was not affected. This is consistent with the temperatures reached by the substrate, below the threshold of heat-induced alteration of kaolinite (500–600° C).

The heating of termite mound fragments in the fire proved their capacity to attain high temperatures and preserve heat for long periods of time. During the experiment the temperature inside the termite mound fragments varied from 609 to 430° C, according to the proximity to the flames. After the fire extinguished, the temperature inside the fragments varied from 120° C to 170° C (Fig. 3H). The next day all fragments had temperatures around 30° C. Most

termite mound fragments turned bright orange after heating, showing a color gradient from yellow to orange when sectioned (Fig. 6E). ATR- $\mu$ FTIR and transmission spectra are consistent with the transformation of kaolinite above 600° C (Fig. 8E). Other aggregates showed a dark brown core and yellow rims, with spectra also showing heat-altered kaolinite (Fig. 8F).

## 4. Archaeological results

The stratigraphic profiles from the 2011–2014 excavation at Lapa do Santo show little variation and are mostly composed of tabular, grey, centimetric layers (5 YR 6.1) of powdery carbonate-rich sediments, with common sand grains, frequent clay aggregates (20–40%) and dispersed charcoal. Some red centimetric lenses (5 YR 5.6) of indurated clay are also present, as well as black, millimetric and centimetric lenses of black silty-clay sediments. Some areas of grey, cemented sediments were discovered during excavation (see Fig. 5). Besides this, sediments are mostly loose and dusty, showing slight variations in color and texture more visible in the excavation surfaces than in the profiles.

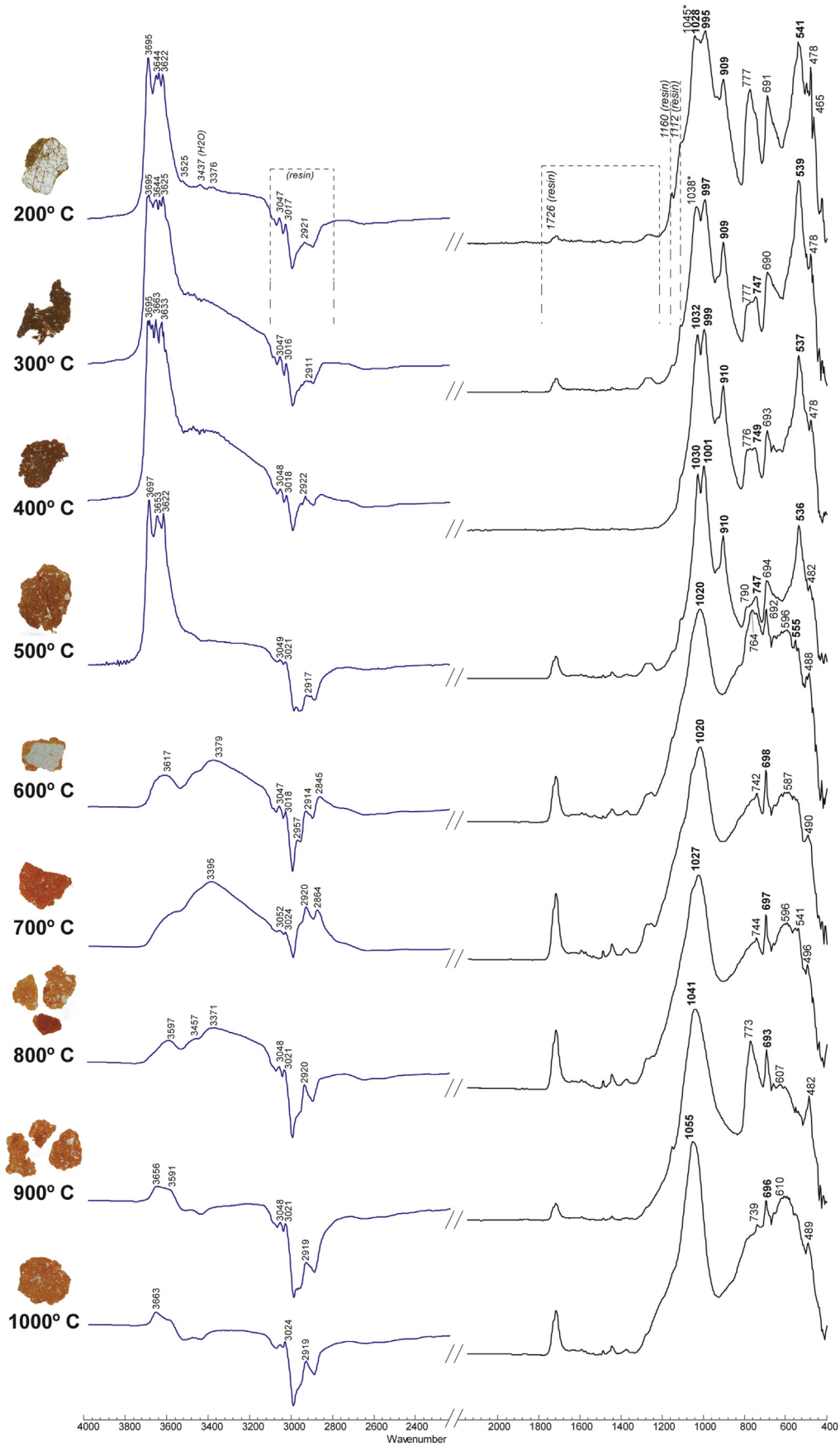
### 4.1. Micromorphology

More than 90% of the thin sections analyzed show similarities in coarse fraction and micromass: the coarse fraction is always made of clay aggregates with random distribution and poorly sorted (sizes from 30  $\mu\text{m}$  to 1 cm); and the micromass consistently includes well preserved ash rhombs (see supplementary material online 3 for tables with micromorphological descriptions) (Fig. 8A and B). Besides clay aggregates and ashes, the sediments at Lapa do Santo include minor concentrations (below 5%) of other components such as: charcoal (unsorted); limestone fragments from the cave walls; quartzite micro-artifacts (stone flakes); opaque minerals and quartz grains detached from the clay aggregates; fine bone (mostly fish bones) and shell fragments, frequently burned; articulated ashes; partially carbonized tissue; tissue residues; and loose and articulated phytoliths. The low amount of charcoal in the samples may result from a sample bias, since high quantities of charcoal were recovered during excavation.

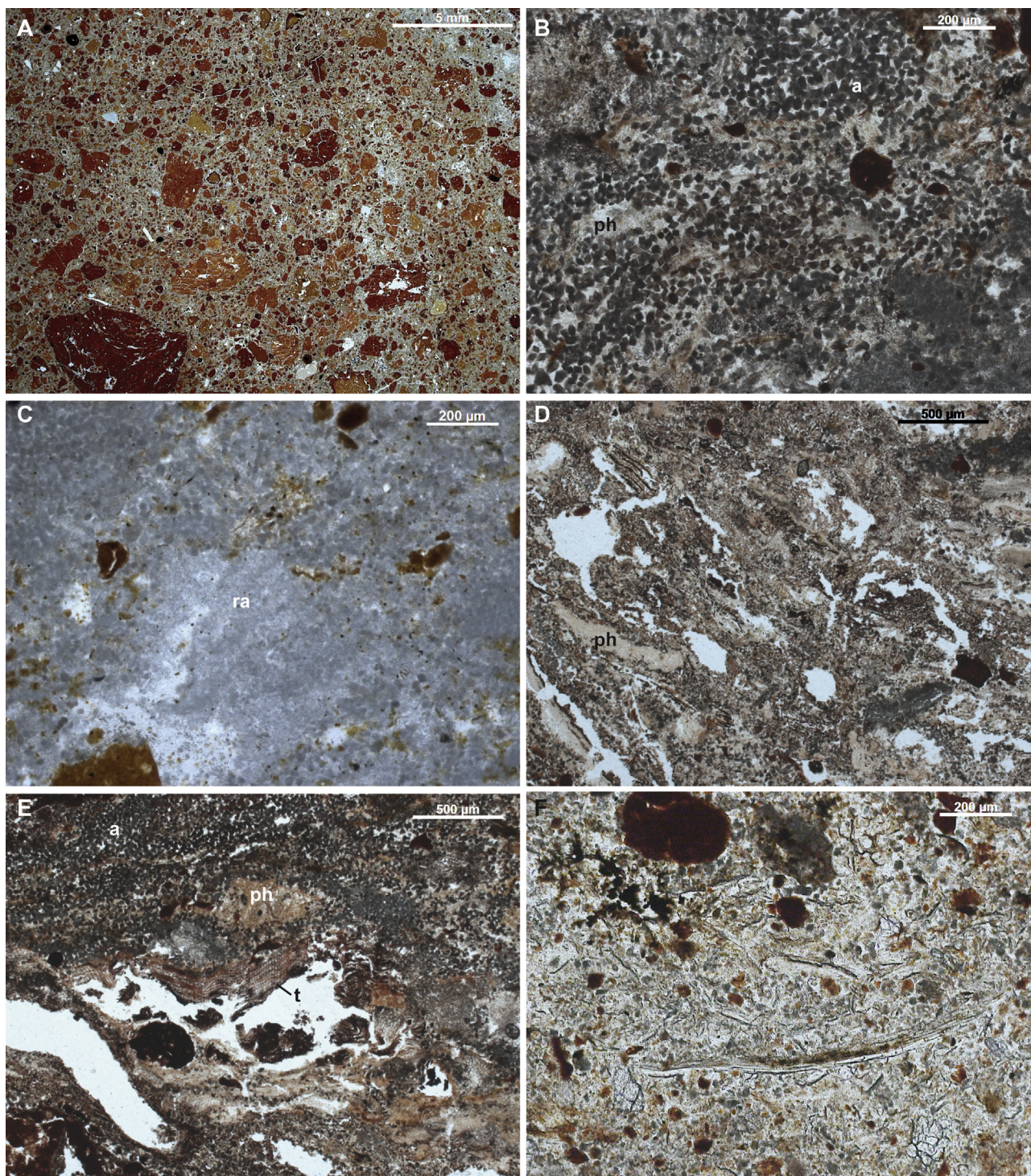
Ashes are certainly the predominant component at the site. Random accumulations of ashes make up the fine fraction of all the analyzed samples. Ashes form when calcium oxalate crystals within the plant tissue decompose through heating. At lower temperatures ( $< 500$ °C) calcite forms spontaneously in solid state, whereas at higher temperatures (above 740° C), the oxalates first transform to calcium oxide (CaO) which, after cooling, can transform to calcite through re-carbonization (Regev et al., 2010; Shahack-Gross and Ayalon, 2013; Shahack-Gross et al., 2008). Under the microscope ashes frequently appear as rhombs (10–30  $\mu\text{m}$ ) of micro-crystalline calcite. Because they often preserve the original form of the oxalate druses, they are called pseudomorphs of calcite after calcium oxalates (POCC) (Brochier, 1983; Canti, 2003; Courty et al., 1989). The ashes in Lapa do Santo are generally well preserved, though areas of cemented ashes are also present (Fig. 8C).

In almost all the samples the ashes are embedded in a pale yellow (PPL) undifferentiated (XPL) phosphatic micromass (Fig. 8D). Phosphate concentrations and nodules are frequently associated with tissue residues, phytoliths and plant pseudo-voids (Fig. 8E and F). This association indicates that phosphates could derive from the ashes and/or the decay of plant tissue (Karkanas et al., 2002; Weiner et al., 2002). Another possible source may be bat guano, a common input in caves and rockshelters (Goldberg and Nathan, 1975; Karkanas, 2000; Shahack-Gross et al., 2004).





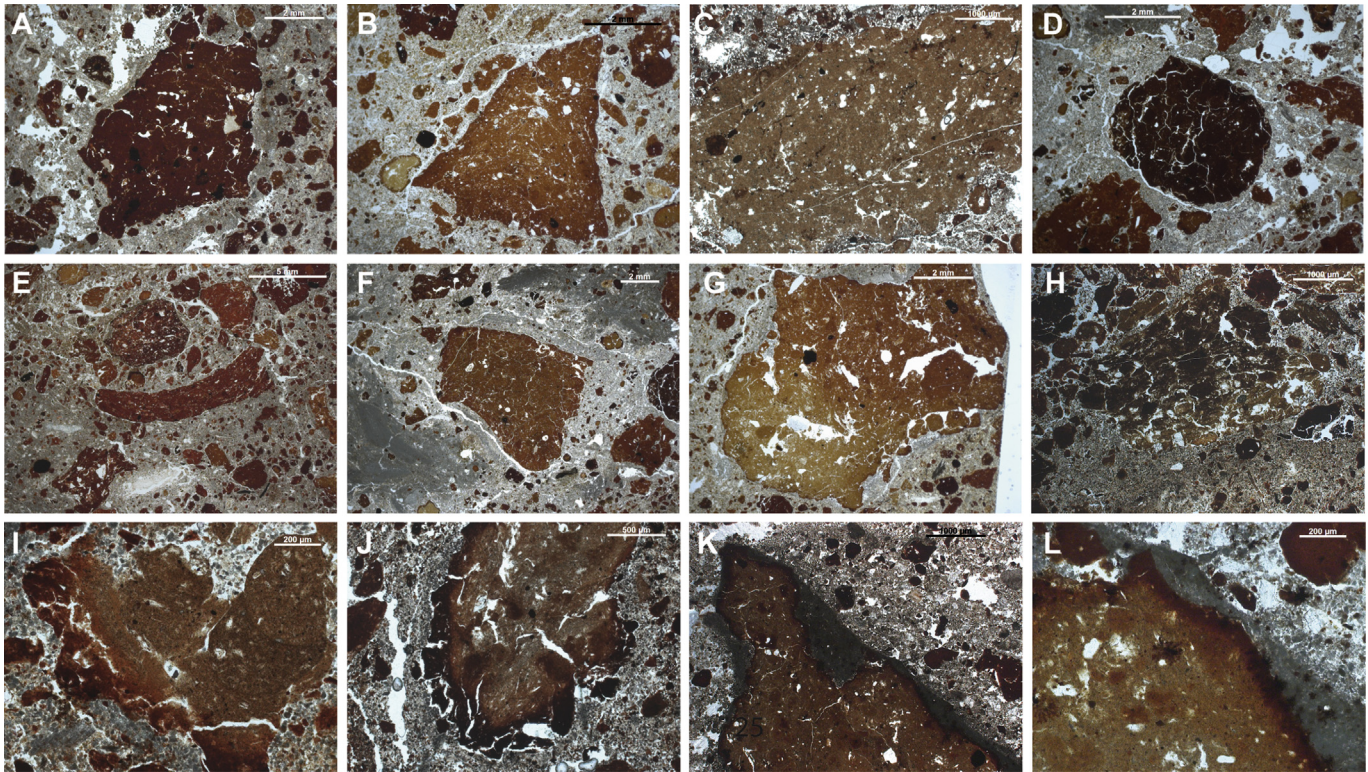




**Fig. 8.** Photomicrographs of microfacies (mF) from Lapa do Santo, with phosphates and plant remains (PPL): A) Predominant microfacies (mF I) with clay aggregates in between ashy micromass, sample A from profile; B) Detailed view of well-preserved ash crystals (a) in between a phosphatic matrix (ph), sample 21; C) Recrystallized ash crystals (ra), sample 1; D) Ash crystals, dense phosphates, tissue residues and chamber voids, sample 21; E) Phosphatic nodule (ph) in association with a plant tissue fragment (t) partially removed during sectioning, in an ashy matrix (a), sample 21; F) Articulated and dispersed phytoliths in between ashes and phosphates, sample A.

**Fig. 7.** Heated oxisol aggregates from 200 to 1000 °C with ATR- $\mu$ FTIR spectra (black line) and transmission spectra at the hydroxyl region of clays (blue line). The peak numbers in bold are diagnostic of kaolinite. According to Beauvais and Bertaux (2002), the absence of the  $3668\text{ cm}^{-1}$  band and the good resolution of the  $3695$ ,  $3654$  and  $3622\text{ cm}^{-1}$  bands are indicative of small-size, poorly ordered kaolinite. As also described by Friesem et al. (2013), the Si–O–Al band at  $\sim 540\text{ cm}^{-1}$  shifts to  $550\text{ cm}^{-1}$  at  $600\text{ }^{\circ}\text{C}$ . The Kaolinite band at  $\sim 1110\text{ cm}^{-1}$  is masked by a resin band at  $1112\text{ cm}^{-1}$ . Absorption bands at  $3525$  and  $3376\text{ cm}^{-1}$  indicate gibbsite. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)





**Fig. 9.** Photomicrographs of different types of clay aggregates identified at Lapa do Santo (PPL): A) Red clay aggregate, sample 5; B) Orange clay aggregate, sample 1; C) Yellow clay aggregate, sample 8; D) Brown clay aggregate, sample 1; E) Orange clay with smooth edges resembling termite mound fragments, sample 15; F) Same as E, sample 1; G) Clay aggregate with transition from yellow to orange color, sample 1; H) Clay aggregate with transition from yellow to dark brown color, sample 25; I) Yellow clay aggregate with red rim, sample 7; J) Yellow clay aggregate with dark red rim, sample 30; K) Clay aggregate with dark rim and coated with recrystallized ashes, sample A; L) Closer view of K showing sharp edge of the micrite coating. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

However, the overall good preservation of the ash crystals indicates low action of acidic waters, like the ones produced after passing through guano (Karkanias et al., 2002). The few bones in the sediments, combined with the pH conditions favorable for hydroxyapatite preservation (alkaline pH), preclude bone dissolution as a source for the authigenic phosphates. Though plant residues and ashes seem to be the main source of phosphates, the fact that the excavation contains at least seven human burials suggests another likely input of phosphates: the byproducts of the decomposing human bodies.

Besides ashes, clay aggregates are the most conspicuous and frequent component in the sediments. The aggregates are mostly blocky, varying from angular to sub-rounded, with undifferentiated b-fabrics (XPL) due to high iron content. They show four distinct colors in PPL: red (Fig. 9A), orange (Fig. 9B), yellow (Fig. 9C) and, less frequently, dark brown (Fig. 9D). All clay aggregates show coalesced granular to massive microstructures, with star-shaped voids, chamber voids and fissures. Some aggregates contain limpid and laminated clay coatings and infillings suggesting they derive from a Bt horizon of a soil outside the shelter. The yellow clay aggregates have weak interference color and massive or striated microstructures and sometimes show a color gradient from yellow to bright orange (Fig. 9G) or from dark brown to yellow (Fig. 9H); Other yellow aggregates have a distinctive 100–200  $\mu\text{m}$  red or dark red rim (Fig. 9I–L). Some orange aggregates show a distinct morphology, with smooth curved edges that differ from the predominant blocky shape (Fig. 9E and F) and resemble the termite mound fragments (see Fig. 6E and F). Clay aggregates may show a dense rim of recrystallized ashes (Fig. 9K and L), whose sharp edges and

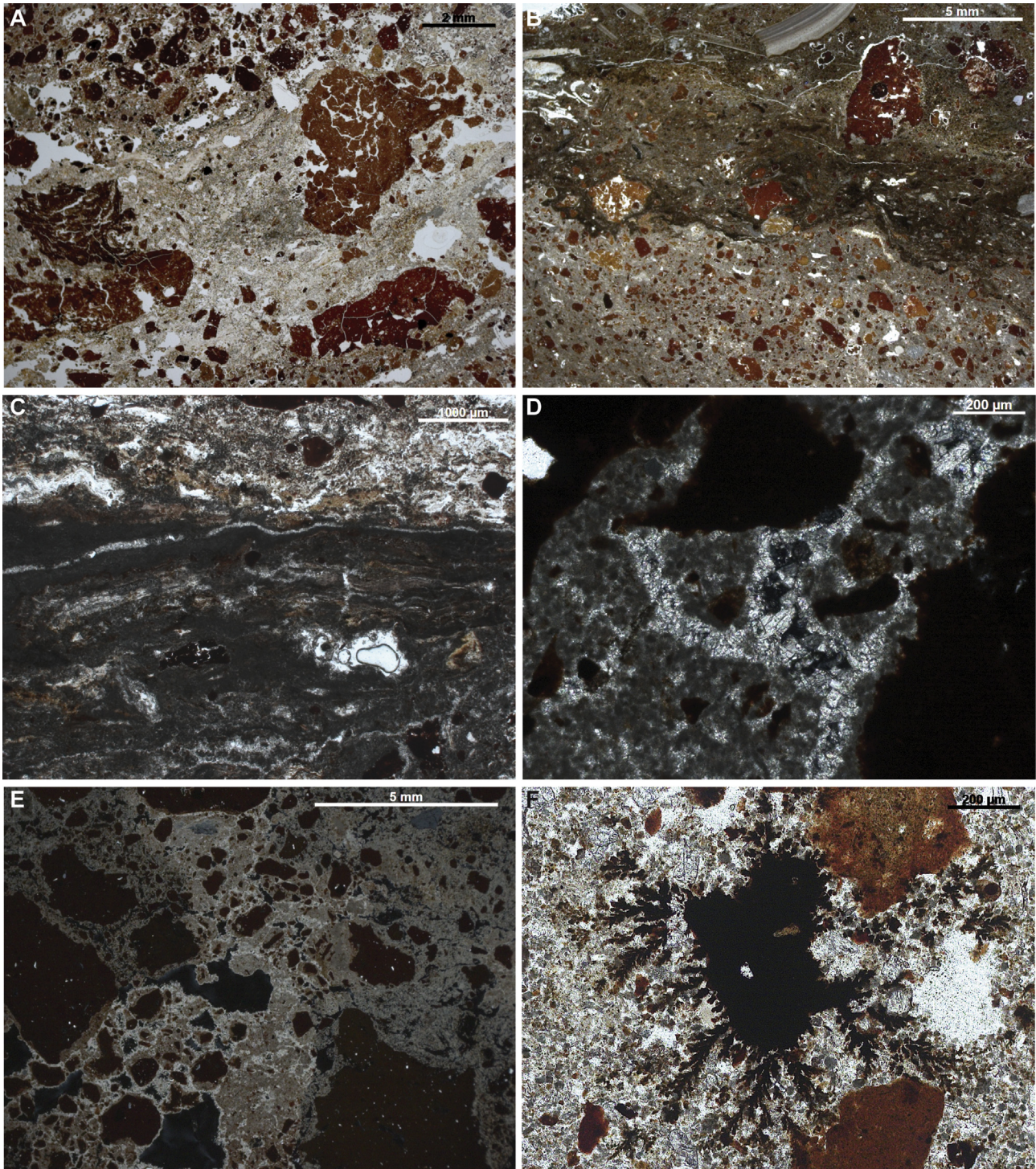
disconnection with the surrounding powdery matrix suggest remobilization of the ash-cemented aggregates.

Changes in porosity and incidence of pedofeatures cementing the sediments reflect post-depositional alterations. Pedofeatures include mostly dense sparitic coatings and infillings (Fig. 10D and E). Secondary sparite implies slow water passage through the sediments and/or episodes of water saturation and drying, also suggested by the few iron (hydroxide) hypocoatings and nodules and manganese dendrites (Fig. 10F) in the micromass. A few channel and chamber voids suggest that some bioturbation influenced the deposits.

#### 4.2. Microfacies identification

A total of 44 microfacies units (mF) was described in the 30 samples collected from the excavation surfaces. Slight changes in the relative frequency of clay aggregates vs. ash crystals (i.e. variations in the c/f ratio), and the presence/absence of organic elements (charcoal, articulated ashes, partially carbonized tissue, tissue residues and phytoliths) express variations between microfacies. Three main microfacies types characterize the deposit: mF I (~85% of the microfacies), containing ashes (randomly distributed and articulated), clay aggregates and organic remains (charcoal, tissue, bone etc) (Fig. 10A); mF II (~10%), made of a massive, highly compacted dark grey micromass (PPL) with ashes and phosphates (Fig. 10B); and mF III (~5%), with only red oxisol material.





**Fig. 10.** Photomicrographs of pedofeatures and microfacies from Lapa do Santo (PPL): A) Detail of typical mf I with clay aggregates embedded in a phosphatic matrix with ash crystals, sample 8; B) mf II (top) made of massive, highly compacted dark grey micromass consisting of permineralized plant remains with ashes and phosphates, sample 6 (note sharp boundary with underlying sediments); C) Laminated organic tissue remains cemented with ashes, phosphates and sparitic infillings, sample 10; D) Detail view of sparitic infillings in XPL, sample 3; E) Area of sparitic cementation (XPL), sample 16; F) Manganese dendrites, frequently found in the groundmass, in the clay aggregates and even on bone fragments, sample A.

#### 4.3. $\mu$ FTIR analysis of clay aggregates

Clay aggregates in the thin sections were analyzed for the identification of heated clay in the site by comparing the spectra with the results of the experimental heating of oxisol aggregates. A total of 30 thin sections were studied: 28 from the excavation

surfaces, and one from the profiles. Table 1 summarizes the results and shows that the majority of the analyzed clay aggregates are in fact heated above 500–600° C. In the non-heated aggregates the kaolinite peaks in the OH region are clearly visible, as well as the main Si–O–Si peak at  $\sim 1030\text{ cm}^{-1}$  and the Al–O–H peak at  $\sim 910\text{ cm}^{-1}$ . In the heated samples, the OH region shows no peaks,



**Table 1**  
Clay aggregates (discriminated by color) analyzed in the thin sections from Lapa do Santo with  $\mu$ FTIR (ATR and transmission). The aggregates were chosen trying to sample at least one of the different colors seen in each slide. The black dots showed no signs of heating at above 500–600 °C, the red dots are heated above this temperature and the grey dots gave ambiguous results (spectra that does not allow a clear differentiation between heating and non-heating). The slides where fewer aggregates were studied are those that contain clay of only one color.

Sample	Color of clay aggregates									Total
	Red		Orange		Orange-yellow	Yellow		Brown		
	non- heated	heated	non- heated	heated	heated	non- heated	heated	heated		
LDS-01	•				••					3
LDS-02	•							•	•	3
LDS-03	•									1
LDS-04	•			•						2
LDS-05		•		•						2
LDS-06			••		••		••••	•		9
LDS-07		•			•		•			3
LDS-08	•••		••	•		•				9
LDS-10	••							•		3
LDS-11	••							•		3
LDS-12		•						•		2
LDS-14	•									1
LDS-15	•			•••				•		5
LDS-16				•	•			•		3
LDS-17		•		•				•		3
LDS-18	•	•			•					3
LDS-19	•••			•						4
LDS-20	••			•	•					4
LDS-21				•				••		3
LDS-22	•••									3
LDS-23	•									1
LDS-24	••	••	•	•			•••			9
LDS-25	•••	••		•	••		•	••••		13
LDS-26				•			•			2
LDS-28	•	••••						••		7
LDS-29				••						2
LDS-30	••	•	•		•					5
LDS-C	•••	•		•			•			6
Total	34	15	6	17	11	1	22	8	114	

the main Si–O–Si peak becomes broad and the Al–O–H peak disappears. The shift to lower wavenumbers in the main Si–O–Si peak was also described in the experimental test as being produced by interference of a main resin peak. Only at above 800 °C does the main Si–O–Si peak shift to higher wavenumbers, indicating that clay aggregates at the site were heated above 500–600 °C, but below 800 °C.

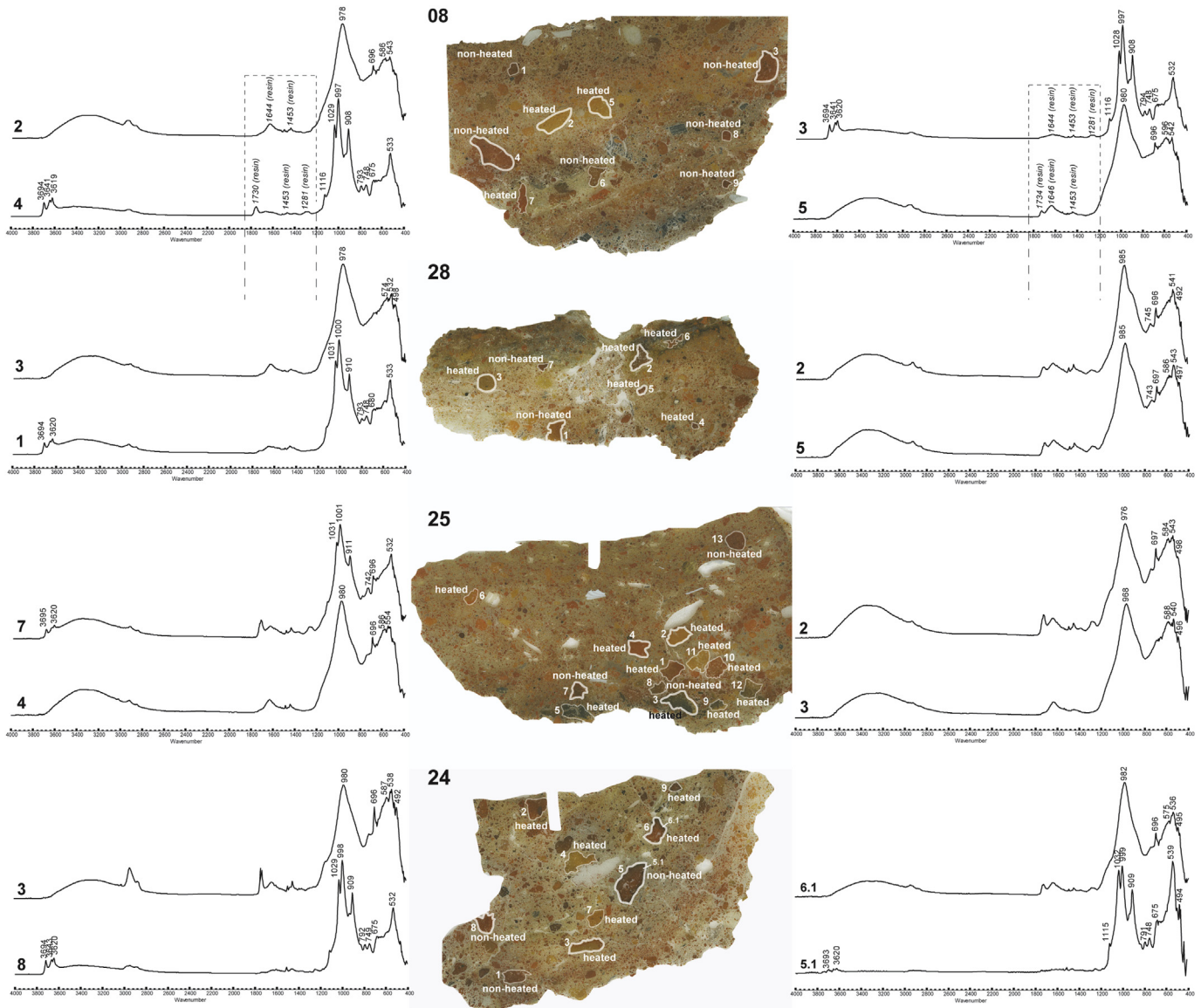
Also noticeable is the fact that most red clay aggregates are non-heated or heated at temperatures below 500 °C, while orange, orange-yellow, yellow and brown aggregates are always heated, with only few exceptions (seven aggregates, mostly orange). Fig. 11 presents four examples of thin sections with the analyzed clay aggregates and selected spectra. The thin sections show the mix of non-heated and heated aggregates that characterize the sediments at Lapa do Santo, with most samples containing aggregates heated above 500–600 °C.

#### 4.4. Organic petrology

Petrographic analysis confirmed that plant tissue remains are only a minor component of the sediments, randomly embedded in the ashy matrix with sizes that vary from 1  $\mu$ m to 200  $\mu$ m. Woody tissue prevail over herbaceous tissue. Plant remains are mostly permineralized (Fig. 12A), especially with phosphate replacement

(Fig. 12C and F), Mn-oxide replacement and silicification (Fig. 12D). Small plant remains described as cell detritus were also found inside the clay aggregates, confirming the association of the aggregates with soil material.

As described in the micromorphology section, secondary phosphates are frequent in the matrix (Fig. 12B, E and F). The dense, dark grey (PPL) micromass described for samples 06, 28 and 29 is made of dense metal oxides (possibly Fe/Mn-oxides) (Fig. 12G) phosphates and secondary carbonates embedding the ash rhombs and the tissue remains. Charcoal fragments of white color in RLo (fusinite and inertodetrinite) show high reflectance values (0.8–1.746%Rr) (Table 2) (Fig. 13A) and are sometimes complete or partially permineralized by Mn-oxides (Fig. 13B). However, most plant detritus seems oxidized and/or degraded by fungi (see supplementary material online 4 for the complete list of reflectance measurements). Despite their low reflectance (0.3–0.7%Rr) indicative of humified plant tissue (Jacob, 1974; Teichmüller, 1961, 1950), they show no fluorescence, have the same morphology as the charcoal particles and occur together with charcoal and ashes. Also, some detritus show domains of variable reflectance (ranging from 0.45 to 1.10%Rr) indicating different degrees of thermal alteration (Fig. 13C). Thus, the data suggest that most plant remains in the sediments are in fact low reflecting charcoal particles (semifusinite) (Fig. 13D) produced



**Fig. 11.** Selected thin sections with ATR- $\mu$ FTIR spectra of analyzed clay aggregates proving the presence of a mixture of unheated and heated clay in the archaeological sediments.

after the incomplete combustion of wood.

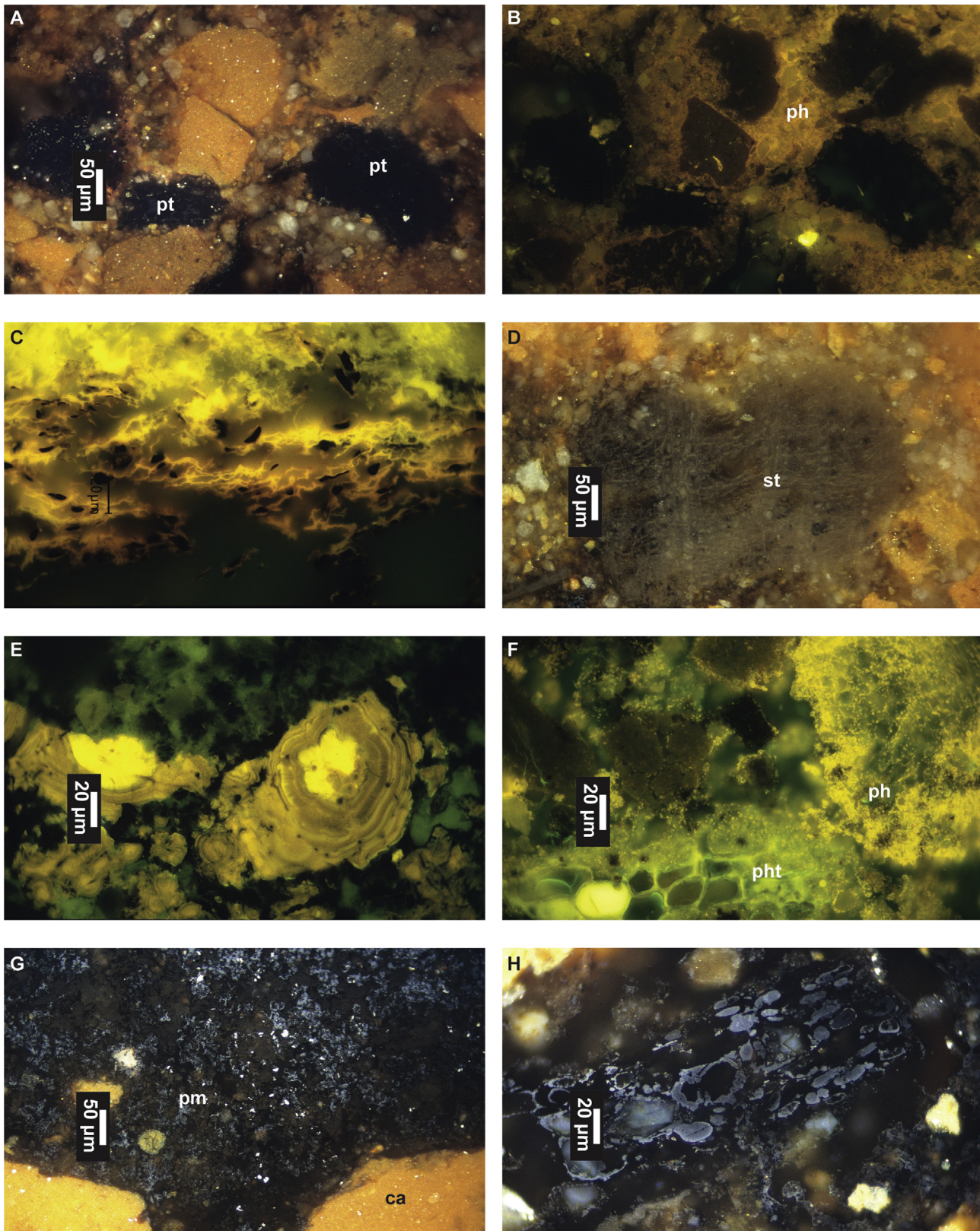
Moreover, a frequent phenomenon is the presence of well-preserved cell fillings (secretinite: organic gels that naturally fill cell voids in plant tissue), both isolated in the groundmass or inside the charcoal (semifusinites) and permineralized tissue (Fig. 13E). Since wood charcoal does not contain secretinite (Braadbaart and Poole, 2008; Braadbaart et al., 2012; Guo and Bustin, 1998; Jones and Lim, 2000; Scott et al., 2000; Taylor et al., 1998), the preservation of organic gels in the charcoal indicate that the tissue underwent advanced humification prior to charring. This is also suggested by the swollen appearance of the cell walls (Diessel, 1992) showing shrinkage cracks and corroded edges (Fig. 13F). Both the cell walls and cell fillings have fungal borings (Fig. 13G), also suggesting biological weathering of wood prior to burning (Guo and Bustin, 1998; Jones and Lim, 2000). These results imply that mostly decayed wood (e.g. forest litter), was used to light the fires at Lapa do Santo (Fig. 13H).

## 5. Discussion

### 5.1. Geogenic sedimentation

The results of the micromorphological and  $\mu$ FTIR analyses show there is no similarity between the clay aggregates in the archaeological sediments and both the two possible geogenic sources near the excavation area: the red breccia and the colluvial deposits. Both the composition and groundmass of the red clay aggregates are similar to the oxisol samples analyzed in this study and described in previous works (see Piló, 1998; Piló et al., 2005; Araujo et al., 2008, 2013). The yellow clay aggregates, which are much less in number and show different micromorphological features (massive microstructure and weak interference color), may derive from the yellow, goethite-rich oxisol that underlies the red oxisols, as suggested for other sites in the region (Araujo et al., 2008; Piló et al., 2005). However, both orange or dark brown clay aggregates are not observed in the natural soils. Neither are soil aggregates with color





**Fig. 12.** Organic petrology of samples from Lapa do Santo: A) Completely permineralized plant tissue (pt) (RLo), sample 18; B) Same as A with phosphates (ph) visible under RVLo; C) Bright fluorescing phosphatized tissue (RVLo), sample 21; D) Silicified tissue (st) (RLo), sample 18; E) Colloform texture in phosphates (RVLo), sample 28; F) Mineral micromass rich in phosphates (ph) with phosphatized tissue fragments (pht) (RVLo), sample 8; G) Contact between red clay aggregates (ca) and permineralized ashy matrix (pm) (RLo), sample 28; H) Permineralized tissue with Mn-oxides (RLo), sample 28. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



**Table 2**  
Plant tissue remains analyzed for organic petrography in samples LDS-11, 25, 28, 18 and 08. Mean reflectance (%Rr) values, types of organic particles (cell walls, cell fillings) and preservation of the tissue remains. Temperatures of charcoal formation, calculated after Jones and Lim (2000). Temperatures range from 225 to 332 °C for production of the charcoal (semifusinite and fusinite).

Sample	%Rr (mean)	Interval of reflectance (%Rr)	Standard deviation	Number of measurements	Temperature (°C) calculated from Rr mean	Temperature range (°C)	Particle size (µm)	Type of organic particles	Tissue preservation	Observations
LDS-11 polished block	0.828	0.371–1.256	0.185	46	281	227–332	5–280	Tissues with cell fillings, rare isolated cell fillings	Poor: strongly altered swollen cell walls with holes and cell fillings with holes	Low reflecting fusinite and low reflecting secretinite; Very rare medium reflecting fusinite
LDS-25 polished block	0.876	0.355–1.189	0.139	55	287	225–324	5–320	Tissues with cell fillings > isolated cell fillings	Poor: strongly altered swollen cell walls with holes and cell fillings with holes	Low reflecting fusinite and low reflecting secretinite
LDS-28 polished block	0.873	0.664–0.991	0.081	19	287	262–300	10–200	Isolated cell fillings > tissues	Poor: strongly altered swollen cell walls, holes in cell fillings	Low reflecting secretinite and low reflecting fusinite
LDS-18 polished block	0.823	0.558–1.142	0.130	35	281	249–318	5–350	Tissues with cell fillings > isolated cell fillings	Poor: strongly altered swollen cell walls with holes and few cell fillings with holes	Low reflecting fusinite and low reflecting secretinite
LDS-8 polished thin section	0.970	0.735–1.746	0.153	57	298	270–390	5–450	Tissues with cell fillings, rare isolated cell fillings	Poor: strongly altered swollen cell walls with holes and few cell fillings with holes	Low reflecting fusinite and low reflecting secretinite; Very rare medium reflecting fusinite

gradients or colored rims, as described for many of the yellow clay aggregates. Bright orange clay could only be observed in the heated clay experiment, while a dark brown color was only seen in the burned termite mound fragments, as will be discussed in the following sections.

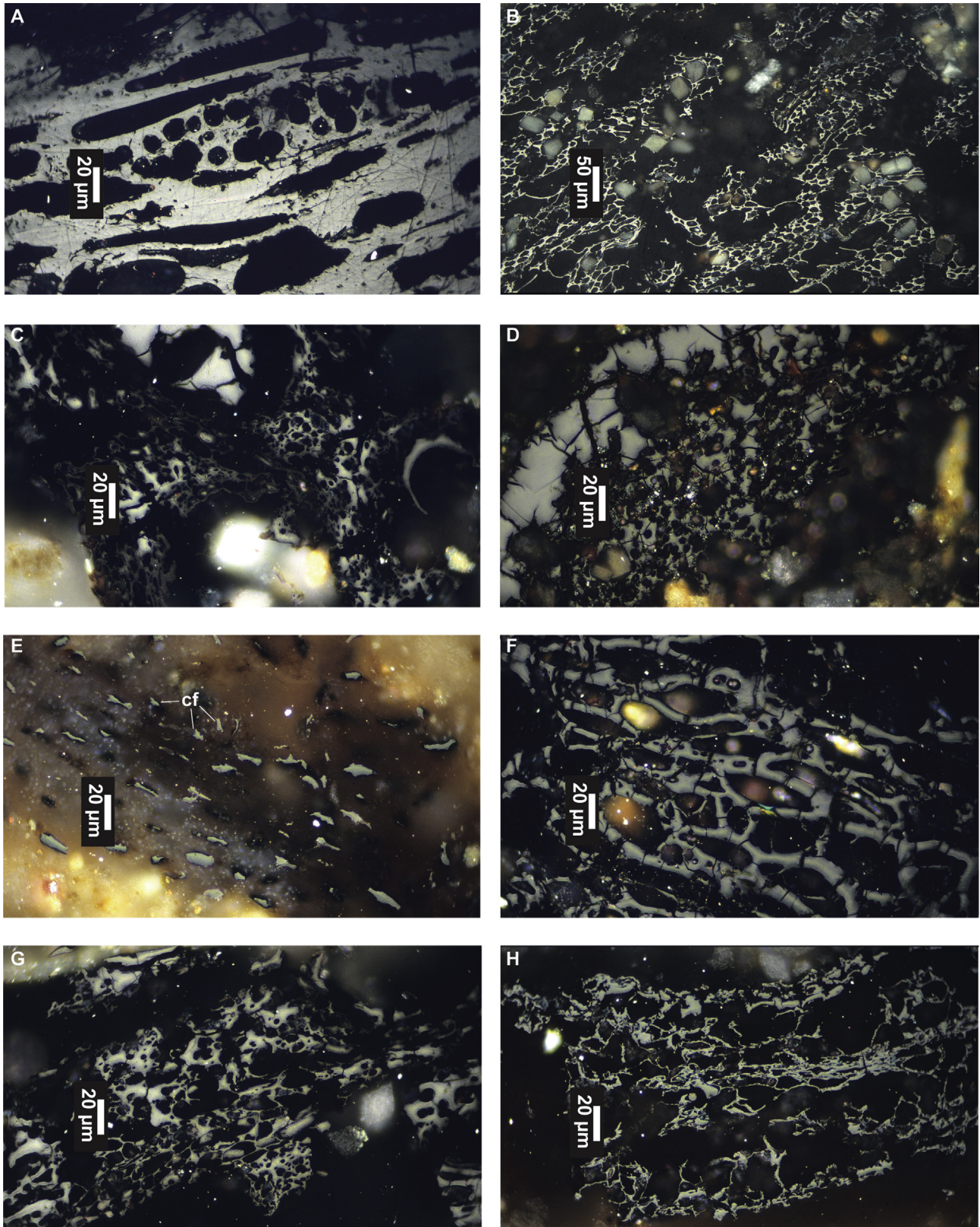
The absence of cracks, conduits or chimneys bringing soil material to the rockshelter, and the similarity in composition, color and structure with the local oxisols suggests that clay aggregates fell from the red oxisol developed directly over the limestone massif. The steep slope on top of the limestone promotes the downhill creep of eroded and loosened soil aggregates. In fact, observations during fieldwork attest to the continuous fall of soil material into the rockshelter, especially in the dry season, when vegetation cover is minimal and soil erosion is naturally enhanced.

## 5.2. Anthropogenic sedimentation and recurrent burning activities

Our study demonstrated that human-made fires and combustion activities were key sedimentary processes at the early Holocene burial area in Lapa do Santo. Anthropogenic burning promoted the accumulation of ashes that mixed with soil continuously falling from above the limestone cliff (Fig. 15). By extrapolating the results of this study to the whole site, we could expect this mixing process to be responsible for the accumulation of almost 5 m of sediments during a time span of almost 5000 years. Decayed wood, possibly coming from forest litter, was used as fuel to light the fires. This observation could serve as indirect evidence of seasonality of site occupation. Forest litter in the *Cerrado* decays at a higher rate during the rainy season (from October to March) (Peres et al., 1983) or the transition from the dry to rainy seasons (September to November) (Sanchez et al., 2008). However, careful analysis of the charcoal remains are needed in order to confirm this, since dry wood may have burned completely, thereby fully converting to the ashes found in the deposit.

Thick concentrations of ashes have been identified at least in another rockshelter in Lagoa Santa, at the site of Lapa das Boleiras (Araujo et al., 2008), and recently at Lapa Grande de Taquaraçu (Villagran et al., 2013). The sediments at Lapa das Boleiras were interpreted as reworked hearths, mobilized by humans as part of site maintenance activities/trampling, or as wind blown particles transported by local air currents (Araujo et al., 2008). At Lapa do Santo the ashes and byproducts of combustion activities seem to include both intact and reworked combustion structures. On the one hand, the random distribution of unsorted coarse fraction components (clay aggregates, charcoal, tissue residues etc.) and the absence of microstratification and/or clear boundaries between the ash layers are indicative of continuous fire-building at a location (see Karkanas et al., 2007; Mallo et al., 2013). A similar arrangement of components has been interpreted as resulting from dumped, burned materials or hearth rake-outs (Aldeias et al., 2012; Goldberg, 2003; Goldberg et al., 2009; Meignen et al., 2007; Mentzer, 2011; Miller et al., 2013; Vallverdu, 2002).

However, some of the microfacies units with high frequency of partially carbonized tissue and articulated ashes at Lapa do Santo (see Fig. 14) indicate low reworking of parts of the deposit and possible ash lenses from intact combustion features (see Mentzer, 2014). Thin ash lenses (~1 cm) containing articulated ashes and partially carbonized tissue can also correspond to intact hearths, as described by Mentzer (2011) for the Uçağızlı I site. Both components are very fragile structures and minimal disturbance, or only short-distance transport, is necessary for their preservation. They have been described in ethnographic



**Fig. 13.** Organic petrology of samples from Lapa do Santo (RLo): A) Wood-derived fusinite tissue, sample 8; B) Partially permineralized fusinite tissue in ash matrix (ash crystals seen as grey rombs), sample 18; C) Semifusinite with heterogeneous reflectance, sample 11; D) Poorly preserved semifusinite tissue, sample 8; E) Relicts of cell fillings (cf) in a permineralized tissue, sample 25; F) Semifusinite tissue with swollen cell wall, sample 11; G) Semifusinite tissue with holes in cell fillings from fungal attack, sample 11; H) Fusinitized humified tissue with droplets of humic colloids droplets attached to cell walls, sample 21.



and experimental hearths (Mallol et al., 2007; Villagran et al., 2011) and also in archaeological contexts where they are interpreted as intact combustion structures (Homsey and Capo, 2006; Karkanas, 2010; Karkanas et al., 2007; Mentzer, 2011; Shahack-Gross et al., 2008).

Articulated ashes and partially carbonized tissue exist at Lapa do Santo and are especially abundant in the samples that contain higher amount of heated clay aggregates. This association allowed the mapping of intact ash layers in the excavation area, which frequently coincide with areas of higher concentration of plant tissue remains (see Fig. 14, samples 24, 25). One exception is sample 06, which contains a concentration of plant tissue but no articulated ashes. However, the sample was taken from underneath the area covered by a large speleothem fragment (square O12, see Fig. 4).

Traditionally, a tripartite sequence of rubified sediment, charcoal and ash is used to interpret intact combustion structures (see Mentzer, 2014, for a complete review of the micromorphological characteristics of combustion features). The absence of this sequence at Lapa do Santo, or at least in the 2011–2014 excavation area, can be explained in different manners: 1) the lack of rubified sediments may be due to the fact that hearths are stacked and successively lit over each other (i.e. over the ash lenses of previous hearths) lacking a clayey sedimentary substrate to be rubified in the first place; 2) the low amount of charcoal in the micromorphology samples is a product of sampling bias, since thin and sparse charcoal lenses are visible during the excavation.

Thus, the sediments at Lapa do Santo include few stacked combustion features, containing superimposed ash lenses, and dumped burned materials or hearth rake-out. The geo-archaeological evidence points to the recurrent use of the site for combustion activities, with some combustion features maintained intact while others were remobilized (see Fig. 14). The dual presence of intact and remobilized combustion features explains the inversions in the radiocarbon ages from the 2011–2014 excavation area (see supplementary material online 1).

The high concentration of human burials at the site, both surrounded and covered by combustion residues, suggests that the lighting of fires and the dumping of hearths was done close to the interments. A possible explanation for the dumped hearths could be to accommodate the human interments, since highly manipulated human bodies were buried in the ashy sediments and covered by them.

### 5.3. Human-made fires and their effects on geogenic sediments

The  $\mu$ FTIR analysis showed that more than half of the clay aggregates analyzed in 28 thin sections was heated at temperatures above 500–600° C (68 aggregates of 119). Heating also explains the different colors described in the aggregates during the micromorphological study. Orange, yellow and brown aggregates consistently show signs of heating as opposed to red colored aggregates, which are mostly non-heated. As shown by the experimental heating of oxisol material, the clay in the local soils will only present orange color and signs of heat-induced alterations when exposed to temperatures above 500–600° C (see Fig. 7). However, the experimental hearth on oxisol substrate demonstrated that such high temperatures are not attained in the substrate of the fire, even close to the surface with flames reaching temperatures above 800° C. This means that clay aggregates must be either in direct contact with the flames to be altered at temperatures above 500–600° C.

This is consistent with the provenance of geogenic sediments, eroding from above the rockshelter and partially falling inside the human-made fires. The clay aggregates may have been also heated already after deposition, by being beneath the fires lit on the ashy substrate. Low-density ashy sediments may transfer heat more readily than solid soil, thus heating the clay aggregate already contained within the ashes. Whatever the possibilities, our study shows that heated soil aggregates make up more than half of the clay aggregates at the site and they likely do not come from fires lit outside the rockshelter on natural soils. Hearths appear to have only been built inside the rockshelter.

### 5.4. The presence of termite mound fragments in the sediments

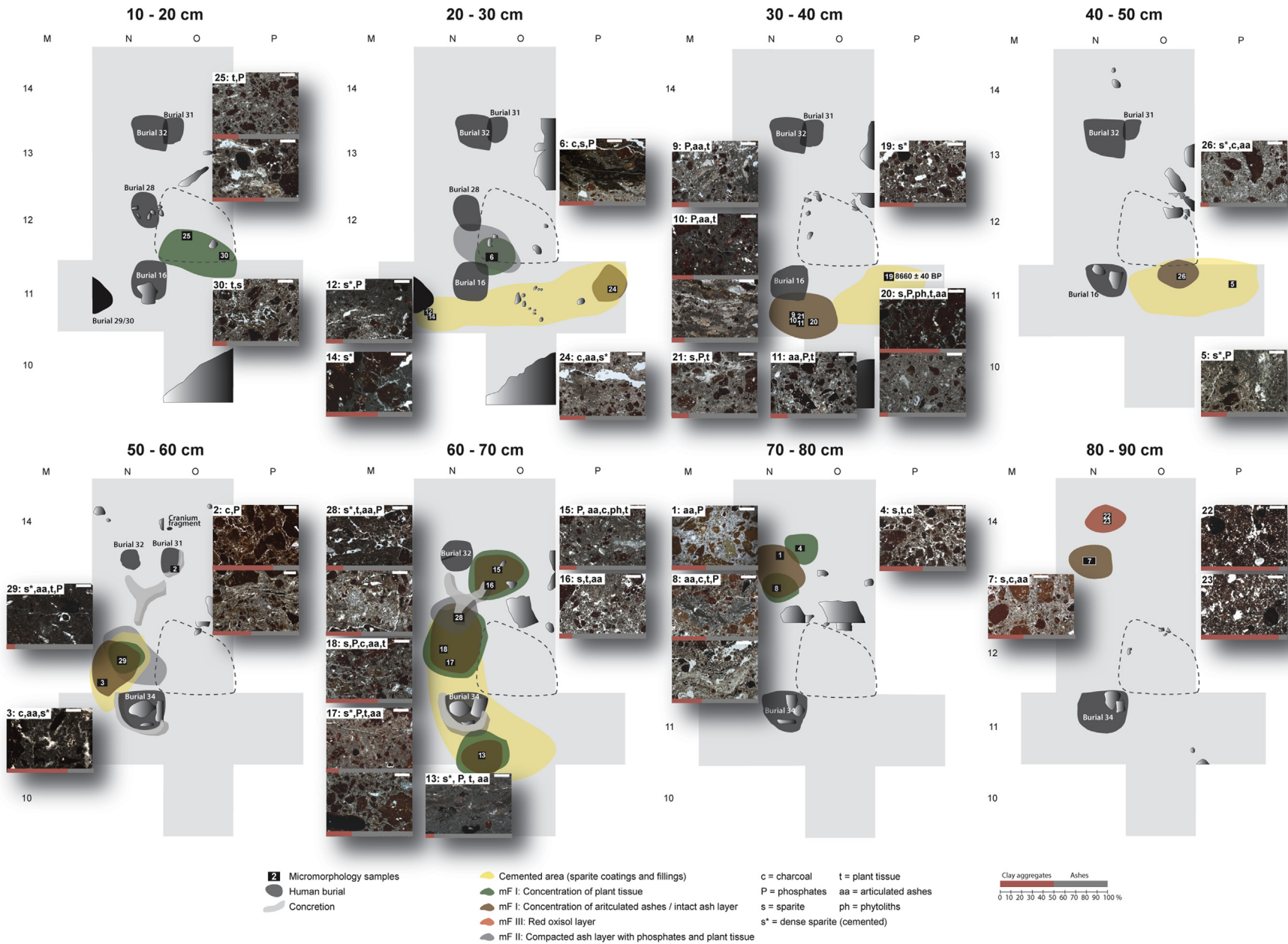
Human selection of materials may also account for the presence of some of the heated aggregates within the site. For instance, several clay aggregates whose heating was demonstrated by  $\mu$ FTIR are similar to the heated termite mound fragments. Similarities include: yellow and dark brown-colored aggregates with signs of heating; the color gradient seen in some heated aggregates; the orange and red rims that do not exist in the natural soils; and the massive microstructure of the aggregates with weak interference color. Termite mound fragments would not fall naturally into the site like the soil aggregates, since termites do not built their nests on shallow loose soil. Above the site, the termite mounds appear on the limestone massif only over flat terrain and far from the cliff (Fig. 3A).

The use of termite mounds by the prehistoric inhabitants of Lagoa Santa should not come as a surprise. Termite mounds are extremely frequent in the region: near the site we noted the presence of 256 nests identified at the elevation of the site and 41 above the limestone massif. Termites have existed for millions of years before the human settlement in the area and some authors believe that termite activity since the Paleogene/Neogene (former Tertiary) is responsible for the characteristic granular microstructure of Brazilian oxisols (Sarcinelli et al., 2009; Schaefer, 2001).

Termite mounds are dense and compact (Cosarinsky and Roces, 2007) and our experimental heating studies show they can retain heat for long periods of time. Because of its characteristics, the ancient and modern populations of Minas Gerais used termite mounds as a type of natural clay oven for food preparation (Nunes and Nunes, 2001). It is also known that ethnographic Xavante groups from central Brazil use termite mounds to build small ovens (Prous, 1992) and for the fires used to cook their traditional maize cake (Lewis, 1967). The termite mound fragments at Lapa do Santo are possible evidence for the use of such resources by early Holocene human groups in South America. However, our analyses cannot determine at this point whether the termite mounds were in fact carried to the site or unintentionally brought as attached fragments to firewood.

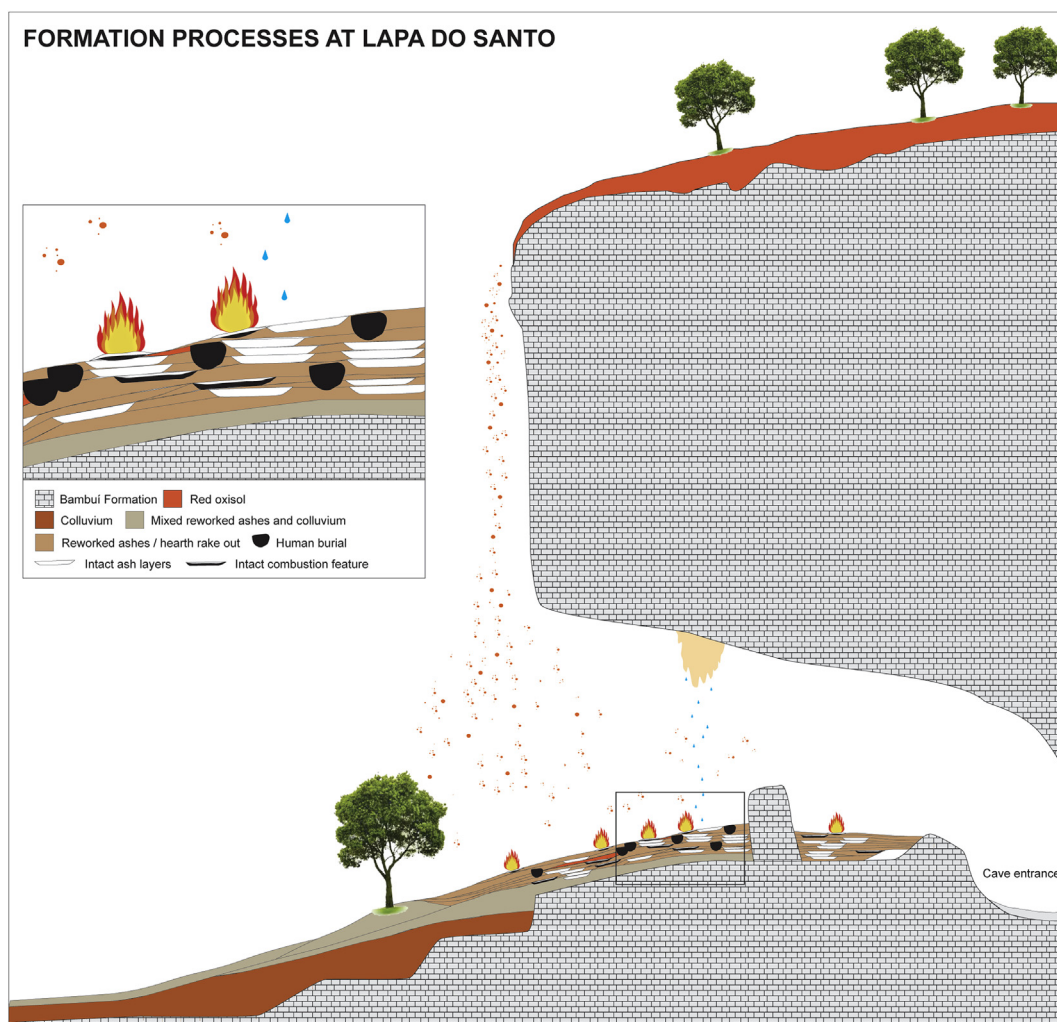
### 5.5. The post-depositional alteration of the site

The horizontal strategy of sampling for micromorphology revealed that spatial differences within the excavation area are mostly due to post-depositional processes, caused by water passage through the sediments. Dense sparite infillings are mostly concentrated around the large speleothem fragment that existed in the excavation area (see Figs. 4 and 14) (see supplementary material online 5 for 3D view of the excavation area with the speleothem). These pedofeatures attest to water accumulation and slow drainage in this area of the site.



**Fig. 14.** Schematic view of the excavation surfaces at different depths where micromorphology samples were taken, with representative photomicrographs. In the photomicrographs it is indicated the percentage of clay aggregates vs. ashes and the presence of phosphates and organic remains in the samples. Micromorphological analysis allowed mapping of the approximate location of: 1) cemented areas within the excavation, surrounding the large speleothem fragment that covered a portion of the excavation surface (indicated with a dashed line), where sparitic coatings and infillings are prevalent; 2) concentrations of plant tissue remains and articulated ashes in mF type I; 3) the location of mF type III, composed of pure red oxisol at about 80–90 cm depths; 4) and the location of an ancient depression within the site where Mn-water dripping from the roof cemented an area rich in plant tissue remains (described as mF type II). Scale bar in samples 4, 8-top, 9, 10, 11, 15, 16, 17-top, 18, 19, 20-bottom, 21, 22, 23, 24, 25-top, 28-top and 29 is 1000  $\mu$ m. Scale bar in samples 1, 3, 5, 7, 8-bottom, 10-bottom, 12, 13, 14, 17-bottom, 20-top, 25-bottom, 26, 28-bottom and 30 is 500  $\mu$ m. Scale bar in sample 2 (top and bottom) is 200  $\mu$ m and in sample 6 is 2 mm. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)





**Fig. 15.** Formation processes at Lapa do Santo. The combined action of geogenic and anthropogenic sedimentation produced the archaeological sediments described in this study, which contain a mixture of soil aggregates eroding and falling from the top of the limestone massif into the rock shelter, with the reworked residues of hearths and other burning activities. Sediments mostly contain reworked ashes and successive layers of hearth rake-out that mixed with the soil falling from above. Thin layers of intact ashes are also described, indicating the presence of in situ synchronic and diachronic hearths throughout the studied time-span of human settlement. Ash layers combined with charcoal lenses have also been described during excavation. Human interments are frequently close to the fires; the graves were dug in the ashy matrix.

## 6. Conclusions

The site of Lapa do Santo contains one of the thickest archaeological deposits of the rockshelters in Lagoa Santa, a region well-known for its human remains dating back to the early Holocene. The site has gained recognition for its earliest evidence of rock art in the Americas and the complexity of the funerary practices (Araujo et al., 2012; Neves et al., 2012; Strauss, 2016; Strauss et al., 2015, in press), both opposing the traditional expectations of cultural “simplicity” for the early Holocene populations of the continent. By studying the stratigraphic sequence at Lapa do Santo using a micro-contextual approach (Goldberg and Berna, 2010)—integrating micromorphology with  $\mu$ FTIR analysis, organic petrology and experimental studies—our study provides key information on 1) the human activities at the site; 2) the influence of natural processes in site formation; 3) the potential use of local resources (e.g. termite mounds); 4) and intensity of occupation.

The hearths that people lit during occupation of the site contributed significantly to the formation of the archaeological deposit. The ashes and other combustion remains are spread across the southern portion of the rockshelter, which was used as multi-

functional space including a burial ground. Micromorphological evidence points to a combination of intact hearths and remobilized hearths through site maintenance activities. The remobilization and lateral reworking of sediments, which explains the age inversions reported for the 2011–2014 excavation, may be related to the funerary practices of the site inhabitants, since all the interments were dug into the ashy sediments and later covered by them. This opens a venue for future studies, focusing on understanding the causal or intentional relation between the anthropogenic sediments and the funerary practices of the site inhabitants.

Micromorphology showed a dual influence in sediment formation that is not evident in the field. There is a noticeable input of geogenic sediments in the form of clay aggregates derived from soil eroding from a steep slope over the limestone cliff and falling into the rockshelter. The clay aggregates are unsorted and vary from rounded to angular. They show different colors which  $\mu$ FTIR studies proved to be related with their thermal alteration: red clay aggregates are mostly non-heated fragments of oxisols, whereas orange aggregates are mostly heated at temperatures above 500–600° C.

This type of anthropogenic infilling is documented around the world, in areas as distant as the Levant (Mentzer, 2011) and South

Africa (Miller, personal observation). However, this work is the first micro-contextual approach applied to fully understand the natural and anthropogenic sedimentary dynamics behind the formation of thick ash deposits containing oxisol aggregates. The results of this work indicate that the techniques and approaches here should be applied at other sites to unravel the full set of information contained in mixed ash/oxisol deposits in rockshelters, despite their geographic and/or climatic context. Besides micromorphology, which has long proved its efficacy in site formation studies, complementary techniques such as  $\mu$ FTIR and organic petrology should be included as sources of data not easily obtained through standard, optical microscopic observations, such as: the heat-induced alteration of sediments; temperatures attained by human-made fires; and fuel sources.

Other components identified in the sediments at Lapa do Santo have a less straightforward association with local soils. These aggregates are heated and show resemblance in shape, color and micromorphology to heated termite mound fragments. This observation suggests the use of termite mounds by the early inhabitants of Lagoa Santa, which appear in dense concentrations around this and other sites in the region. Their presence at the site may be the first potential evidence for the use of this local resource by early South Americans, possibly for heating and/or cooking. However, further experimental studies are needed to refute a possible natural cause for the presence of this resource at the site.

Despite the high amount of ashes and charcoal, fresh plant remains are scarce and, when present, are persistently permineralized with Mn-oxides, silicified or phosphatized. The high concentration of secondary phosphates in the sediments seems to derive from the ashes and the charred plant remains, as indicated by the association of concentrated plant residues and phosphate nodules and the ash crystals persistently embedded in a phosphatic micromass. However, the possible relation of secondary phosphates with decaying human bodies must be further investigated. The charred plant remains in the sediments derived from decomposed wood (degraded by fungi) point at the use of decayed forest litter as fuel.

Understanding the dual composition of the sediments at Lapa do Santo (i.e. ashes and oxisol aggregates) is essential when discussing the intensity of occupation. The thick archaeological deposit resulted from the mixed input of anthropogenic sediments from intact and remobilized hearths, and from the constant fall of soil aggregates into the rockshelter. This indicates that thickness in the archaeological deposit does not necessarily correlate with a more intensive occupation of the site, and that local sedimentation processes must be taken into consideration for comparison. However, the recurrent use of Lapa do Santo and other sites for almost 5000 years certainly reflects the central position of the region in the cultural landscape of the early Holocene.

## Acknowledgements

Authors would like to thank the Alexander von Humboldt Foundation and CAPES/CNPq Science Without Borders (224577/2012-2) for supporting X. Villagran post-doctoral fellowships at the Institute for Archaeological Sciences between 2014–2015 and 2013–2014, respectively. Between 2001 and 2009 excavations at Lapa do Santo were funded by the São Paulo Research Foundation (FAPESP, proc. 99/0670-7 and 04/01321-6). The 2011–2014 excavations were funded by the Department of Human Evolution, Max Planck Institute for Evolutionary Anthropology. Analyses were done at the laboratory of micromorphology and the laboratory for applied organic petrology, Institute of Archaeological Sciences, University of Tübingen. The FTIR equipment was funded through the Deutsche Forschungsgemeinschaft by a grant to Christopher

Miller (MI 1748/1-1). Special thanks to: Leandro Vieira da Silva, Andersen Lyrio, Claudio Dias, Alberto Barioni, Max Ernani, Marcony Alves, Peter Kloos, Prof. Danilo Bernardo, Prof. Philipp Nigst, Prof. Bence Viola, Dr. Marjolein Boesch, Jose Hein, Rogerio Tavares and Joao Barba Filho. Thanks to Dr. Susan Mentzer and Dr. David Friesem for their assistance with the  $\mu$ FTIR analysis and interpretations. Special thanks to Prof. Walter Neves, Prof. Astolfo Araujo and Dr. Renato Kipnis for initiating the study of Lapa do Santo that made this research possible.

## Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.jas.2016.07.008>.

## References

- Aldeias, V., Goldberg, P., Sandgathe, D., Berna, F., Dibble, H.L., McPherron, S.P., Turk, A., Rezek, Z., 2012. Evidence for Neandertal use of fire at Roc de Marsal (France). *J. Archaeol. Sci.* 39, 2414–2423.
- Araujo, A.G.M., Feathers, J.K., Arroyo-Kalin, M., Tizuka, M.M., 2008. Lapa das boleiras rockshelter: stratigraphy and formation processes at a paleoamerican site in Central Brazil. *J. Archaeol. Sci.* 35, 3186–3202.
- Araujo, A.G.M., Neves, W.A., Kipnis, R., 2012. Lagoa Santa revisited: an overview of the chronology, subsistence and material culture of paleoindian sites in eastern central Brazil. *Lat. Am. Antiq.* 24, 533–550.
- Araujo, A.G.M., Pugliese, F., 2009. The use of non-flintraw materials by paleoindians in eastern south America (Araujo & Pugliese 2009).pdf. In: Sternke, F., Eigeland, L., J. C.L. (Eds.), *Non-Flint Raw Material Use in Prehistory: Old Prejudices and New Directions*. Archaeopress, Oxford, pp. 169–173.
- Araujo, A.G.M., Strauss, A.M., Feathers, J.K., Paisani, J.C., Schrage, T.J., 2013. Paleoindian open-air sites in tropical settings: a case study in formation processes, dating methods, and paleoenvironmental models in Central Brazil. *Geoarchaeology* 28, 195–220.
- Babinski, M., Trindade, R.I.F., Alvarenga, C.J.S., Boggiano, P.C., Liu, D., Santos, R., Brito Neves, B.B., 2006. Chronology of Neoproterozoic ice ages in Central Brazil. In: *V South American Symposium on Isotope Geology*. Punta del Este, pp. 223–226.
- Beauvais, A., Bertaux, J., 2002. In situ characterization and differentiation of kaolinites in lateritic weathering profiles using infrared microspectroscopy. *Clays Clay Min.* 50, 314–330.
- Berna, F., Behar, A., Shahack-Gross, R., Berg, J., Boaretto, E., Gilboa, A., Sharon, I., Shalev, S., Shilstein, S., Yahalom-Mack, N., 2007. Sediments exposed to high temperatures: reconstructing pyrotechnological processes in late bronze and iron age strata at tel dor (Israel). *J. Archaeol. Sci.* 34, 358–373.
- Berna, F., Goldberg, P., 2007. Assessing Paleolithic pyrotechnology and associated hominin behavior in Israel. *Isr. J. Earth Sci.* 56, 107–121.
- Berna, F., Goldberg, P., Horwitz, L.K., Brink, J., Holt, S., Bamford, M., Chazan, M., 2012. Microstratigraphic evidence of in situ fire in the acheulean strata of wonderwerk cave, northern cape province, South Africa. *Proc. Natl. Acad. Sci. U. S. A.* 109, E1215–E1220.
- Berna, F., Matthews, A., Weiner, S., 2004. Solubilities of bone mineral from archaeological sites: the recrystallization window. *J. Archaeol. Sci.* 31, 867–882.
- Bissaro Jr., M.C., 2008. *Tafonomia como ferramenta zooarqueológica de interpretação: viés de representatividade óssea em sítios arqueológicos, paleontológico e etnográfico*. University of São Paulo.
- Borrego, A.G., Araujo, C.V., Balke, A., Cardott, B., Cook, A.C., David, P., Flores, D., Hámor Vidó, M., Hiltmann, W., Kalkreuth, W., Koch, J., Kommeren, C.J., Kus, J., Ligouis, B., Marques, M., Mendonça Filho, J.G., Misz, M., Oliveira, L., Pickel, W., Reimer, K., Ranasinghe, P., Suárez-Ruiz, I., Vieth, A., 2006. Influence of particle and surface quality on the vitrinite reflectance of dispersed organic matter. *Int. J. Coal Geol.* 68, 151–170.
- Braadbaart, F., Poole, I., 2008. Morphological, chemical and physical changes during charcoalification of wood and its relevance to archaeological contexts. *J. Archaeol. Sci.* 35, 2434–2445.
- Braadbaart, F., Poole, I., Huisman, H.D.J., van Os, B., 2012. Fuel, Fire and Heat: an experimental approach to highlight the potential of studying ash and char remains from archaeological contexts. *J. Archaeol. Sci.* 39, 836–847.
- Bueno, L., 2005. As indústrias líticas da região do Lajeado e sua inserção no contexto do Brasil Central. *Rev. do Mus. Arqueol. Etnol* 15–16, 37–58.
- Bueno, L., Dias, A.S., Steele, J., 2013a. The Late Pleistocene/Early Holocene archaeological record in Brazil: a geo-referenced database. *Quat. Int.* 301, 74–93.
- Bueno, L., Feathers, J., De Blasis, P., 2013b. The formation process of a paleoindian open-air site in Central Brazil: integrating lithic analysis, radiocarbon and luminescence dating. *J. Archaeol. Sci.* 40, 190–203.
- Cosarinsky, M.I., Roces, F., 2007. Neighbor leaf-cutting ants and mound-building termites: comparative nest micromorphology. *Geoderma* 141, 224–234.
- CPRM, 2010. Programa Geologia do Brasil - Projeto Sete Lagoas, Abaeté, Estado de Minas Gerais. Ministério de Minas e Energia - CPRM, Belo Horizonte.
- Da-Gloria, P., 2012. Health and Lifestyles in the Paleoamericans: Early Holocene Biocultural Adaptation at Lagoa Santa. University of Ohio.



- Dias, A.S., 2004. Diversificar para poblar: el contexto arqueológico brasileño en la transición Pleistoceno-Holoceno. *Complutum* 15, 249–263.
- Diessel, C.F.K., 1992. Coal-bearing Depositional Systems. Springer-Verlag, Berlin.
- Embrapa, 2006. Sistema brasileiro de classificação de solos. Embrapa-SPI, Rio de Janeiro.
- Feathers, J., Kipnis, R., Piló, L., Arroyo-Kalin, M., Coblentz, D., 2010. How old is luzia? luminescence dating and stratigraphic integrity at Lapa Vermelha, Lagoa Santa, Brazil. *Geoarchaeology* 25, 395–436.
- Friesem, D.E., Tsartsidou, G., Karkanas, P., Shahack-Gross, R., 2013. Where are the roofs? A geo-ethnoarchaeological study of mud brick structures and their collapse processes, focusing on the identification of roofs. *Archaeol. Anthropol. Sci.* 6, 73–92.
- Goldberg, P., 2000. Micromorphology and site formation at die kelders cave 1, South Africa. *J. Hum. Evol.* 38, 43–90.
- Goldberg, P., 2003. Some observations on Middle and Upper Paleolithic ashy cave and rockshelter deposits in the Near East. In: Goring-Morris, A.N., Belfer-Cohen, A. (Eds.), *More than Meets the Eye: Studies on Upper Paleolithic Diversity in the Near East*. Oxbow Books, Oxford, pp. 19–32.
- Goldberg, P., Arpin, T.L., 1999. Micromorphological analysis of sediments from meadowcroft rockshelter, Pennsylvania: implications for radiocarbon dating. *J. F. Archaeol.* 26, 325–342.
- Goldberg, P., Berna, F., 2010. Micromorphology and context. *Quat. Int.* 214, 56–62.
- Goldberg, P., Miller, C.E., Schiegl, S., Ligouis, B., Berna, F., Conard, N.J., Wadley, L., 2009. Bedding, hearths, and site maintenance in the middle stone age of sibudu cave, kwazulu-natal, South Africa. *Archaeol. Anthropol. Sci.* 1, 95–122.
- Goldberg, P., Nathan, Y., 1975. The phosphate mineralogy of et Tabun cave, Mount Carmel, Israel. *Mineral. Mag.* 40, 253–258.
- Goldberg, P., Sherwood, S.C., 2006. Deciphering human prehistory through the geochronological study of cave sediments. *Evol. Anthropol. Issues, News, Rev.* 15, 20–36.
- Guo, Y., Bustin, R., 1998. FTIR spectroscopy and reflectance of modern charcoals and fungal decayed woods: implications for studies of inertinite in coals. *Int. J. Coal Geol.* 37, 29–53.
- Hermenegildo, T., 2009. Reconstituição da dieta e dos padrões de subsistência das populações pré-históricas de caçadores-coletores do Brasil Central através da ecologia isotópica. University of São Paulo.
- Homsey, L.K., Capo, R.C., 2006. Integrating Geochemistry and micromorphology to interpret feature use at dust cave, a paleo-Indian through middle-archaic site in northwest Alabama. *Geoarchaeology* 21, 237–269.
- Hubbe, A., Haddad-Martim, P.M., Hubbe, M., Mayer, E.L., Strauss, A., Auler, A.S., Piló, L.B., Neves, W. a., 2011. Identification and importance of critical depositional gaps in pitfall cave environments: the fossiliferous deposit of Cuvier Cave, eastern Brazil. *Palaeogeogr. Palaeoclimatol. Palaeoecol.* 312, 66–78.
- Hubbe, M., Harvati, K., Neves, W., 2011. Paleoamerican morphology in the context of European and East Asian late Pleistocene variation: implications for human dispersion into the New World. *Am. J. Phys. Anthropol.* 144, 442–453.
- IBAMA-CPRM, 1998. APA Carste de Lagoa Santa - Meio Físico. Instituto Brasileiro do Meio Ambiente e dos Recursos Minerais Renováveis - Serviço Geológico do Brasil, Belo Horizonte.
- ICCP, 1998. The new vitrinite classification (ICCP system 1994). *Fuel* 77, 349–358.
- ICCP, 2001. The new inertinite classification (ICCP system 1994). *Fuel* 80, 459–471.
- Jacob, H., 1974. Mikroskop-photometrie der organischen Stoffe von Böden, Reflexions un Fluoreszenz Photometrie der organischen Substanz von zwei Schwarzwälder. *Bodenkultur* 25, 1–9.
- Jacob, H., 1980. Die Anwendung der Mikrophotometrie in der Organische Petrologie. *Leitz Mittl. für Wiss. Tech* 7, 209–216.
- Jones, T.P., Lim, B., 2000. Extraterrestrial impact and wildfires. *Palaeogeogr. Palaeoclimatol. Palaeoecol.* 164, 57–66.
- Jones, T.P., Scott, A.C., Cope, M., 1991. Reflectance measurements and the temperature of formation of modern charcoals and implications for studies of fusain. *Bull. la Société Géologique Fr.* 162, 193–200.
- Karkanas, P., 2000. Diagenesis in prehistoric caves: the use of minerals that form in situ to assess the completeness of the archaeological record. *J. Archaeol. Sci.* 27, 915–929.
- Karkanas, P., 2010. Geology, stratigraphy and site formation processes in the Upper Paleolithic and later sequence in Kissonura Cave 1, Eurasian Prehistory 7, 15–36.
- Karkanas, P., Goldberg, P., 2007. Micromorphology of sediments: deciphering archaeological context. *Isr. J. Earth Sci.* 56, 63–71.
- Karkanas, P., Koumouzelis, M., 2004. The earliest evidence for clay hearths: auri-gnacian features in Klisoura Cave 1, southern Greece. *Antiquity* 1, 513–525.
- Karkanas, P., Rigaud, J.-P., Simek, J.F., Albert, R.M., Weiner, S., 2002. Ash bones and guano: a study of the minerals and phytoliths in the sediments of Grotte XVI, dordogne, France. *J. Archaeol. Sci.* 29, 721–732.
- Karkanas, P., Shahack-Gross, R., Ayalon, A., Bar-Matthews, M., Barkai, R., Frumkin, A., Gopher, A., Stiner, M.C., 2007. Evidence for habitual use of fire at the end of the Lower Paleolithic: site-formation processes at Qesem Cave. *Isr. J. Hum. Evol.* 53, 197–212.
- Kipnis, R., 1998. Early hunter-gatherers in the Americas: perspectives from Central Brazil. *Antiquity* 72, 581–592.
- Laming-Emperaire, A., 1979. Missions archéologiques franco-brésiliennes de Lagoa Santa, Minas Gerais, Brésil. *Grand-Abri Lapa Vermelha. Rev. Pré-História* 1, 53–89.
- Ligouis, B., 2006. Jais, lignite, charbon et autres matières organiques fossiles: application de la pétrologie organique à l'étude des éléments de parure et des fragments bruts. In: Bullinger, J., Leesch, D., Plummetaz, N. (Eds.), *Le Site* Magdalénien de Monruz, 1. Premiers Éléments Pour L'analyse D'un Habitat de Plein Air. Neuchâtel.
- Mallol, C., Cabanes, D., Baena, J., 2010. Microstratigraphy and diagenesis at the upper pleistocene site of esquilieu cave. *Quat. Int.* 214, 70–81.
- Mallol, C., Marlowe, F., Wood, B., Porter, C., 2007. Earth, wind, and fire: ethno-archaeological signals of Hadza fires. *J. Archaeol. Sci.* 34, 2035–2052.
- Mallol, C., Mentzer, S.M., Wrinn, P.J., 2009. A micromorphological and mineralogical study of site formation processes at the late Pleistocene site of Obi-Rakhmat, Uzbekistan. *Geoarchaeology* 24, 548–575.
- Mallol, C., Hernandez, C.M., Cabanes, D., Machado, J., Sistiaga, A., Perez, L., Galvan, B., 2013. Human actions performed on simple combustion structures: an experimental approach to the study of Middle Paleolithic fire. *Quat. Int.* 315, 3–15.
- Marcelino, V., Stoops, G., Schaefer, C.E.G.R., 2010. Oxidation and related materials. In: Stoops, G., Marcelino, V., Mees, F. (Eds.), *Interpretation of Micromorphological Features of Soils and Regoliths*. Elsevier, Amsterdam, pp. 305–327.
- Meignen, L., Goldberg, P., Bar-Yosef, O., 2007. The hearths at Kebara cave and their role in site formation processes. In: Bar-Yosef, O., Meignen, L. (Eds.), *Kebara Cave, Mt. Carmel, Israel: the Middle and Upper Paleolithic Archaeology Part I*. Peabody Museum of Archaeology and Ethnology, Cambridge.
- Mentzer, S., 2011. Macro- and Micro-scale Geoarchaeology of Üçağızlı Caves I and II. University of Arizona, Hatay, Turkey.
- Mentzer, S.M., 2014. Microarchaeological approaches to the identification and interpretation of combustion features in prehistoric archaeological sites. *J. Archaeol. Method Theory* 21, 616–668.
- Miller, C.E., Goldberg, P., Berna, F., 2013. Geoarchaeological investigations at diepkloof rock shelter, western cape, South Africa. *J. Archaeol. Sci.* 40, 3432–3452.
- Moreno de Sousa, J.C., 2014. Cognition e cultura no mundo material: os Itaparicas, os Umbus e os 'Lagoasantenses'. University of São Paulo.
- Neves, W.A., Araujo, A.G.M., Bernardo, D.V., Kipnis, R., Feathers, J.K., 2012. Rock art at the pleistocene/holocene boundary in Eastern South America. *PLoS One* 7, e32228.
- Neves, W.A., Gonzalez Jose, R., Hubbe, M., Kipnis, R., Araujo, A., Blasi, O., 2004. Early holocene human skeletal remains from cerca Grande, Lagoa Santa, Central Brazil, and the origins of the first Americans. *World Archaeol.* 36, 479–501.
- Neves, W.A., Hubbe, M., 2005. Cranial morphology of early Americans from Lagoa Santa, Brazil: implications for the settlement of the new world. *Proc. Natl. Acad. Sci. U. S. A.* 102, 18309–18314.
- Neves, W.A., Hubbe, M., Araujo, A.G.M., 2002. A late-paleoindian secondary burial from Lagoa Santa, Minas Gerais, Brazil. *Current research in the pleis- tocene. Curr. Res. Pleistocene* 19, 83–85.
- Neves, W.A., Hubbe, M., Piló, L.B., 2007. Early Holocene human skeletal remains from Sumidouro Cave, Lagoa Santa, Brazil: history of discoveries, geological and chronological context, and comparative cranial morphology. *J. Hum. Evol.* 52, 16–30.
- Neves, W.A., Powell, J.F., Prous, A., Ozolins, E.G., Blum, M., 1999. Lapa Vermelha IV Hominid 1: morphological affinities of the earliest known american. *Genet. Mol. Biol.* 22, 461–469.
- Neves, W.A., Prous, A., Gonzalez Jose, R., Kipnis, R., Powell, J., 2003. Early Holocene human skeletal remains from Santana do Riacho, Brazil: implications for the settlement of the New World. *J. Hum. Evol.* 45, 19–42.
- Peres, J.R.R., Suhett, a. R., Vargas, M. a. T., Drozdowicz, a., 1983. Litter production in areas of brazilian "cerrados." *pesqui. Agropecuária Bras.* 18, 1037–1043.
- Piló, L.B., 1998. Morfologia cársica e materiais constituintes: dinâmica e evolução da depressão poligonal Macacos-Baú. Universidade de Sao Paulo, Carste de Lagoa Santa, MG.
- Piló, L.B., Auler, A.S., Neves, W. a., Wang, X., Cheng, H., Edwards, R.L., 2005. Geochronology, sediment provenance, and fossil emplacement at Sumidouro Cave, a classic late Pleistocene/early Holocene Paleoanthropological site in eastern Brazil. *Geoarchaeology* 20, 751–764.
- Powell, J.F., Neves, W.A., 1999. Craniofacial morphology of the first Americans: pattern and process in the peopling of the new world. *Yearb. Phys. Anthropol.* 42, 153–188.
- Prous, A., 1992. *Arqueologia Brasileira*. Universidade de Brasília, Brasília.
- Prous, A., Fogaça, E., 1999. Archaeology of the pleistocene-holocene boundary in Brazil. *Quat. Int.* 53–54, 21–41.
- Pugliese, F., 2008. Líticos de Lagoa Santa: um estudo sobre organização tecnológica de caçadores-coletores do Brasil Central. University of São Paulo.
- Regev, L., Poduska, K.M., Addadi, L., Weiner, S., Boaretto, E., 2010. Distinguishing between calcites formed by different mechanisms using infrared spectrometry: archaeological applications. *J. Archaeol. Sci.* 37, 3022–3029.
- Sanches, L., Valentini, C.M.A., Júnior, O.B.P., Nogueira, J.D.S., Vourlitis, G.L., Biudes, M.S., da Silva, C.J., Bambi, P., Lobo, F.D.A., 2008. Seasonal and interannual litter dynamics of a tropical semideciduous forest of the southern Amazon Basin. *Braz. J. Geophys. Res.* 113, 1–9.
- Sarcinelli, T., Schaefer, C., Lynch, L., Arato, H., Viana, J., Filho, M., Goncalves, T., 2009. Chemical, physical and micromorphological properties of termite mounds and adjacent soils along a toposequence in Zona da Mata, Minas Gerais State, Brazil. *Catena* 76, 107–113.
- Schaefer, C.E., 2001. Brazilian latosols and their B horizon microstructure as long-term biotic constructs. *Aust. J. Soil Res.* 39, 909–926.
- Schaetzl, R., Anderson, S., 2005. *Soils: Genesis and Geomorphology*. Cambridge University Press, Cambridge.
- Schiegl, S., 1996. Ash deposits in Hayonim and Kebara Caves, Israel: macroscopic, microscopic and mineralogical observations, and their archaeological

- implications. *J. Archaeol. Sci.* 23, 763–781.
- Schiegl, S., Conard, N.J., 2006. The Middle Stone Age sediments at Sibudu: results from FTIR spectroscopy and microscopic analyses. *South African Humanit.* 18, 149–172.
- Schwaar, J., Jacob, H., Hufnagel, H., 1990. Zur Bewertung von Bodenverbesserungsmitteln durch Bestimmung von Zersetzungsgrad und Rotte der oragischen Substanz. *Geol. Jahrb.* 3–160.
- Scott, A.C., Cripps, J.A., Collinson, M.E., Nichols, G.J., 2000. The taphonomy of charcoal following a recent heathland fire and some implications for the interpretation of fossil charcoal deposits. *Palaeogeogr. Palaeoclimatol. Palaeoecol.* 164, 1–31.
- Shahack-Gross, R., Ayalon, A., 2013. Stable carbon and oxygen isotopic compositions of wood ash: an experimental study with archaeological implications. *J. Archaeol. Sci.* 40, 570–578.
- Shahack-Gross, R., Ayalon, A., Goldberg, P., Ofek, B., Rabinovich, R., Hovers, E., 2008. Formation processes of cemented features in karstic cave sites revealed using stable oxygen and carbon isotopic Analyses: a case study at middle paleolithic amud cave, Israel. *Geoarchaeology* 23, 43–62.
- Shahack-Gross, R., Berna, F., Karkanas, P., Weiner, S., 2004. Bat guano and preservation of archaeological remains in cave sites. *J. Archaeol. Sci.* 31, 1259–1272.
- Shoval, S., 1994. The firing temperature of a persian-period pottery kiln at Tel Michal, Israel, estimated from the composition of its pottery. *J. Therm. Anal.* 42, 175–185.
- Shoval, S., Beck, P., 2005. Thermo-FTIR spectroscopy analysis as a method of characterizing ancient ceramic technology. *J. Therm. Anal. Calorim.* 82, 609–616.
- Shoval, S., Yadin, E., Panczer, G., 2011. Analysis of thermal phases in calcareous Iron Age pottery using FT-IR and Raman spectroscopy. *J. Therm. Anal. Calorim.* 104, 515–525.
- Soil Survey Staff, 2003. *Keys to Soil Taxonomy*, World. USDA-NRSC, Washington.
- Stahlschmidt, M.C., Miller, C.E., Ligouis, B., Goldberg, P., Berna, F., Urban, B., Conard, N.J., 2015a. The depositional environments of Schöningen 13 II-4 and their archaeological implications. *J. Hum. Evol.* 89, 71–91.
- Stahlschmidt, M.C., Miller, C.E., Ligouis, B., Hambach, U., Goldberg, P., Berna, F., Richter, D., Urban, B., Serangeli, J., Conard, N.J., 2015b. On the evidence for human use and control of fire at Schöningen. *J. Hum. Evol.* 89, 1–21.
- Stoops, G., 2003. *Guidelines for Analysis and Description of Soil and Regolith Thin Sections*. Soil Science Society of America, Madison.
- Strauss, A., 2010. *As Práticas Mortuárias dos Caçadores-Coletores Pré-Históricos da Região de Lagoa Santa (MG): Um Estudo de Caso do Sítio Arqueológico “Lapa do Santo.”* University of Sao Paulo.
- Strauss, A., 2016. Os padroes de sepultamento do sitio arqueologico Lapa do Santo (Holoceno Inicial, Brasil). *Bol. do Mus. Para. Emílio Goeldi* 11, 243–276.
- Strauss, A., Oliveira, R.E., Bernardo, D.V., Salazar-García, D.C., Talamo, S., Jaouen, K., Hubbe, M., Black, S., Wilkinson, C., Richards, M.P., Araujo, A.G.M., Kipnis, R., Neves, W.A., 2015. The oldest case of decapitation in the new world (Lapa do Santo, East-Central Brazil). *PLoS One* 10, e0137456.
- Strauss, A., Oliveira, R.E., Villagran, X.S., Bernardo, D.V., Salazar-García, D.C., Bissaro, M.C., Pugliese, F., Hermenegildo, T., Santos, R., Barioni, A., de Oliveira, E.C., De Sousa, J.C., Jaouen, K., Ernani, M., Hubbe, M., Inglez, M., Gratao, M., Rockwell, H.M., Machado, M.C., Souza, G.N., Chemale, F., Kawashita, K., O’Connell, T., Israde, I., Black, S., Feathers, J., Castro, C., Richards, M.P., Wahl, J., Kipnis, R., Araujo, A., Neves, W.A., 2016. Early Holocene Funerary Complexity in South America: the Archaeological Record of Lapa Do Santo (East-central Brazil). *Antiquity* (in press).
- Sykorová, I., Pickel, W., Christanis, K., Wolf, M., Taylor, G., Flores, D., 2005. Classification of huminite (ICCP sistem 1994). *Int. J. Coal Geol.* 62, 85–106.
- Taylor, G.H., Teichmüller, M., Davis, A., Diessel, C.F.K., Littke, R., Robert, P., 1998. *Organic Petrology*. Gerbrüder Borntraeger, Berlin.
- Teichmüller, M., 1950. Zum petrographischen Aufbau und Werdegang der Weichbraunkohle. *Geol. Jahrb.* 64, 429–488.
- Teichmüller, M., 1961. Beobachtungen bei einem Torfbrand. *Geol. Jahrb.* 78, 653–660.
- Vallverdu, J., 2002. Micromorfología de las facies sedimentarias de la Sierra de Atapuerca y del nivel J del Abric Romaní. Implicaciones geoarqueológicas e paleoetnográficas. *Universitat Rovira i Virgili*.
- Villagran, X.S., Balbo, A.L., Madella, M., Vila, A., Estevez, J., 2011. Experimental micromorphology in Tierra del Fuego (Argentina): building a reference collection for the study of shell middens in cold climates. *J. Archaeol. Sci.* 38, 588–604.
- Villagran, X.S., Schaefer, C.E.G.R., Ligouis, B., 2013. Living in the cold: geoarchaeology of sealing sites from byers peninsula (livingston Island, Antarctica). *Quat. Int.* 315, 184–199.
- Wadley, L., Sievers, C., Bamford, M., Goldberg, P., Berna, F., Miller, C., 2011. Middle stone age bedding construction and settlement patterns at sibudu, South Africa. *Science* 334 (80), 1388–1391.
- Weiner, S., 2009. Diagenetic transformations in cave sites: deciphering the archaeological record of prehistoric caves. In: Shea, J.J., Lieberman, D.E. (Eds.), *Transitions in Prehistory: Essays in Honour of Ofer Bar-yosef*. Oxbow Books, Cambridge, pp. 419–430.
- Weiner, S., Goldberg, P., Bar-Yosef, O., 2002. Three-dimensional distribution of minerals in the sediments of hayonim cave, Israel: diagenetic processes and archaeological implications. *J. Archaeol. Sci.* 29, 1289–1308.