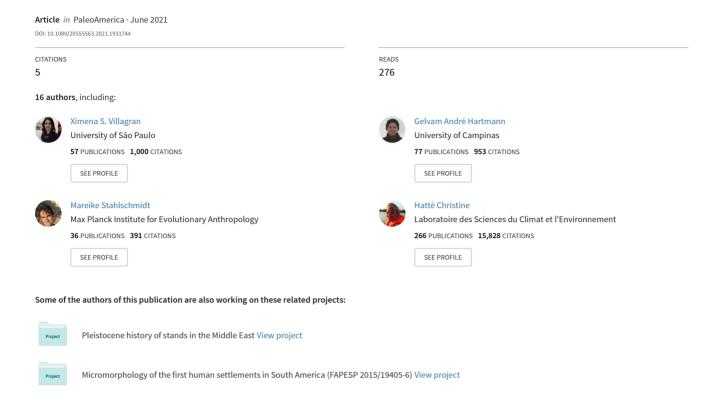
## Formation Processes of the Late Pleistocene Site Toca da Janela da Barra do Antonião – Piauí (Brazil)





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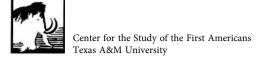
## Formation Processes of the Late Pleistocene Site Toca da Janela da Barra do Antonião – Piauí (Brazil)

Ximena S. Villagran, Gelvam A. Hartmann, Mareike Stahlschmidt, Susan Heinrich, María F. Gluchy, Christine Hatté, Christelle Lahaye, Christophe Griggo, Antonio Pérez, Marcos Paulo de Melo Ramos, Haruan Straioto, Janaina Santos, Ricardo I.F. Trindade, André Strauss, Niède Guidon & Eric Böeda

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#### RESEARCH REPORT



# Formation Processes of the Late Pleistocene Site Toca da Janela da Barra do Antonião – Piauí (Brazil)

Ximena S. Villagran <sup>©</sup><sup>a</sup>, Gelvam A. Hartmann <sup>©</sup><sup>b</sup>, Mareike Stahlschmidt<sup>c</sup>, Susan Heinrich<sup>c</sup>, María F. Gluchy <sup>©</sup><sup>d</sup>, Christine Hatté <sup>©</sup><sup>e</sup>, Christelle Lahaye <sup>©</sup><sup>f</sup>, Christophe Griggo <sup>©</sup><sup>g</sup>, Antonio Pérez <sup>©</sup><sup>h</sup>, Marcos Paulo de Melo Ramos <sup>©</sup><sup>i</sup>, Haruan Straioto<sup>a</sup>, Janaina Santos<sup>j</sup>, Ricardo I.F. Trindade <sup>©</sup><sup>k</sup>, André Strauss <sup>©</sup><sup>a</sup>, Niède Guidon <sup>©</sup><sup>l</sup>, and Eric Böeda <sup>©</sup><sup>m</sup>

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#### **ABSTRACT**

Archaeological research of late Pleistocene sites in northeastern Brazil has rarely analyzed site formation processes from a geoarchaeological perspective. This has contributed to the longheld debate over the reliability of Pleistocene ages and the anthropic origin of stone tools and combustion features. In this work, we combine high-resolution geoarchaeological methods to study the formation processes of the Toca da Janela da Barra do Antonião North site (TJBA-North), located in a surface karst about 12 km northeast of the classical site of Boqueirão da Pedra Furada (Serra da Capivara, Piauí). The site contains stone tools and megafauna remains in sedimentary levels that have been dated by optically stimulated luminescence (OSL) as more than 20,000 years ago up to the early Holocene. Using micromorphology, Fourier transform infrared spectrometry (FTIR), micro-FTIR on the micromorphology thin sections, and magnetic analyses, we carried out a comprehensive study of the climatic conditions during the late and terminal Pleistocene and investigated the depositional history of the site.

#### **KEYWORDS**

Serra da Capivara; micromorphology; FTIR; micro-FTIR; magnetic analyses; paleoenvironment; northeastern Brazil

## 1. Introduction

The Serra da Capivara National Park (Piauí, Brazil) is world-renowned in the debate about the peopling of the Americas. Its archaeological sites contain the oldest evidence of human presence in northeastern Brazil and South America. In addition to the classic site of Boqueirão da Pedra Furada (BPF), which gained notoriety in the 1980s (Guidon 1989; Guidon and Delibrias 1986; Parenti 2001; Parenti, Fontugne, and Guérin 1996), other sites have yielded late Pleistocene ages from charcoal fragments associated with the stone artifacts, and from optically stimulated luminescence (OSL) ages of the sedimentary layers with archaeological material. A new wave of multidisciplinary archaeological research in the area is being conducted by the French-Brazilian Mission, led by Éric Boëda. This research has re-excavated classic sites and opened new

ones. Since 2008 the research team has recovered lithic tools made of quartz and quartzite pebbles and cobbles. These tools date from before the Late Glacial Maximum (pre-LGM) to the Holocene. Evidence for these industries is widespread across the region, including at the Serra da Capivara Cuesta, with its upper conglomerates containing quartz and quartzite cobbles (e.g., Sítio do Meio and Vale da Pedra Furada), and the nearby meta-limestone rock shelters (Toca da Tira Peia and Toca da Janela da Barra do Antonião North - TJBA-North) (Boëda et al. 2014a, 2014b, 2016; Lahaye et al. 2013, 2019). Use-wear analyses of the pre-LGM quartz stone tools from Vale da Pedra Furada and Sítio do Meio have shown that most were used for butchery, skin processing, and wood processing (Boëda et al. 2014a, 2016; Clemente-Conte, Boëda, and Farias-Gluchy 2017).

Scholars have noted that analysis of the geoarchaeological site formation processes is required at the controversial sites of Serra da Capivara (Borrero 2015; Dias and Bueno 2014), and micromorphological and sedimentological research has even been suggested by the research team at BPF (Parenti 2001; Parenti et al. 2018). In our work, we combine high-resolution techniques to study the formation processes of the TJBA-North site, located about 12 km northeast of BPF, in the surface karst of Barra do Antonião. The site has been dated as more than 20,000 yr BP based on OSL ages (Lahaye et al. 2019). We combine micromorphological analyses with Fourier transform infrared spectrometry (FTIR), micro-FTIR (an FTIR coupled with a microscope) on the micromorphology thin sections, and magnetic analyses of loose sediments. This is the first time that a geoarchaeological and micro-contextual approach (sensu Goldberg and Berna 2010) has been applied to one of the late Pleistocene sites around Serra da Capivara.

Micromorphology enables microstratigraphic analysis of archaeological sediments (Courty, Goldberg, and Macphail 1989), to reveal components and formation/ transformation processes at the micro-scale. FTIR and micro-FTIR are currently popular techniques in geoarchaeology and microarchaeology given their potential to identify authigenic minerals, heated material, and organic components in archaeological sediments (Berna 2017; Monnier 2018; Toffolo and Berna 2018). Magnetic analyses are common in paleomagnetic and environmental studies and have long been applied in archaeology as non-invasive survey methods for identification of combustion features, site taphonomy, and the study of the paleoenvironment (Dalan and Banerjee 1998; Evans and Heller 2003). In this work, we will focus on understanding the formation processes of TJBA-North using a micro-contextual approach, and infer paleoclimatic information from magnetic analyses of one of its stratigraphic profiles.

# 2. The Toca da Janela da Barra do Antonião sites (TJBA)

The Serra da Capivara is a large cuesta consisting of sandstones, conglomerates and shales of the Serra Grande Group, and Itaim, Pimenteiras and Cabeças Formations from the Parnaíba Basin (Paleozoic age) (Figure 1(A)). The climate around Serra da Capivara is warm semi-arid with a mean annual temperature of 28°C and mean annual rainfall of 650 mm (concentrated in the rainy season, October to April). Local vegetation is *Caatinga*, a dry forest with grasslands,

xerophytic thorn shrubs, savanna, and shrub woodland (IBGE 1993; Nimer 1989).

The São Francisco depression extends southeast of the cuesta front, on the pediplain, and contains several meta-limestone relict hills of pre-Cambrian origin from the Riacho do Pontal fold belt. The Barra do Antonião is a light gray surface karst of about 500-600 m long N-S and 100-450 m E-W, made of stratified meta-limestones (Figure 1(B)) (Guérin et al. 1993, 1996, 2015; Santos 2007). The whole meta-limestone massif has a west-east tilt and a conduit network filled with local red clayey sediments (Rodet 1997; Santos 2007). The Barra do Antonião karst comprises a sheltered area 180 m long and 28 m wide where the French-Brazilian Mission has excavated several archaeological and paleontological sites since 1986, removing about 750 m<sup>2</sup> of sediments. The first radiocarbon date for the Barra do Antonião sites was 11,275 - 10,564 calendar years (cal yr) BP  $(9670 \pm 140^{-14}\text{C} \text{ yr BP (Gif-8712) calibrated})$ here with OxCal 4.4, SHCal20 curve to a 95.3% confidence interval). This date was obtained in the 1990s from a piece of charcoal recovered next to a human burial in Sector C (Guérin et al. 1993, 1996, 1999; Guidon 1989; Parenti et al. 2002; Peyre 1993, 1994). Studies of the stone tools of the Barra do Antonião sites (Sectors A, B, C, and D) reveal an industry that used quartz, quartzite, and chert, including hammers, unretouched flakes, and core tools (see Guérin et al. 1999, 2015; Parenti et al. 2002; Santos 2012; Santos and Cura 2014).

The Barra do Antonião sites are rich in fossil remains with different species of mammals, birds, reptiles, and fish, including Pleistocene megafauna (Mylodontidae, Megatheriidae, Megalonychidae, Glyptodontidae, Toxodontidae, Gomphoterridae, etc.) (Bélo and Oliveira 2013; Guérin and Faure 2008, 2014; Guérin et al. 1993, 1996, 1999, 2015; Parenti et al. 2002). Scholars have interpreted the sheltered area as covering an ancient lake or swamp, where animals gathering to drink water became trapped in the muddy deposit, and died in situ (Guérin and Faure 2014; Guérin et al. 1996, 2015). Sedimentation in the sheltered area would be mostly lacustrine, with episodes of highenergy transport of sediments and fauna remains through the karst conduits, thus bringing exogenous material to the rock shelter. The lower topography of the rock shelter favored the formation of ponds and small lakes, as described by the excavators. The diversity of animal species in the deposit was interpreted as corroborating the presence of a paleo-lake, while the pebble-like morphology of some bone remains resulted from the transport of sediments and animal parts through the shafts and conduits (Guérin and Faure 2014; Guérin et al. 1996, 1999, 2015).

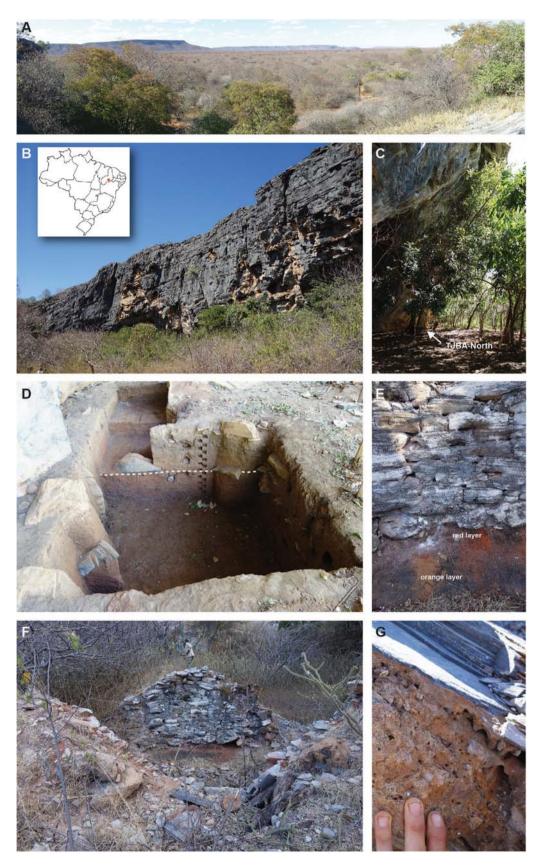


Figure 1 (A) View of the Serra da Capivara cuesta from the hill at Antonião. (B) View of the meta-limestone outcrop of Antonião. (C) The rock shelter at TJBA-North. (D) Excavation at TJBA-North in 2015 with the meta-limestone wall to the left of the photograph (notice the stratigraphic discontinuity indicated by a dashed line). (E) Profile of the lime oven sampled for FTIR analyses. (F) View of the lime oven near the site. (G) Red clayey rock interbedded in the meta-limestone, as seen at the top of the outcrop.

#### 2.1. TJBA-North

TJBA-North is located about 200 m north of the surveys carried out in the 1990s (Figure 1(C)). The French-Brazilian Mission under the direction of Éric Böeda and María F. Gluchy, first excavated the site in 2013. The ongoing excavation covers an area of 8 m<sup>2</sup> and a depth of more than 1 m (Figure 1(D)). The sedimentary succession at TJBA-North contains six lithostratigraphic units and seven archaeological levels containing lithic and bone artifacts (Figure 2), together with fauna and megafauna remains. The chronology of TJBA-North was established by combining radiocarbon dating of charcoal fragments and OSL dating of the sediments collected in the 2013 and 2014 field seasons (Lahaye et al. 2019). In this work, all radiocarbon ages are reported in calendar years (cal yr BP) to allow comparison with OSL ages.

A change in the sedimentary regime can be observed in the contact between the lower units (C6 to C5) and the upper units (C4 to C1), despite grain-size analyses showing little variation through the profile (mostly sandy loam with poor sorting) (Figure 1(D), Figure 2). Roof spall is frequent in units C1 and C2, where human presence is registered between 9600 and 9900 cal yr BP. Unit C3 contains archaeological remains of the Itaparica cultural tradition (archaeological levels C3a and C3b, dated 11,200–12,500 cal yr BP), a common techno-complex during the Pleistocene-Holocene transition and the early Holocene in central and northern Brazil (Calderon 1972; Lourdeau 2016). Macro- and

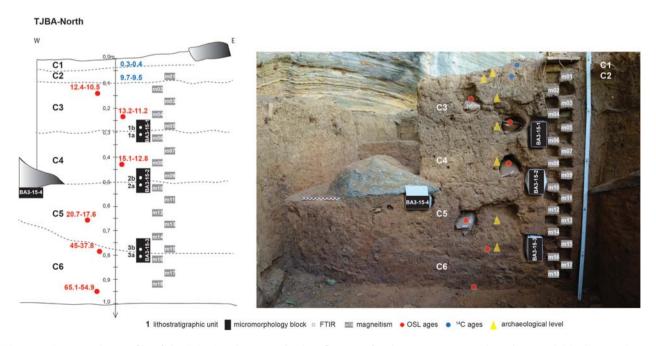
micro-artifacts in quartz were identified in units C4 and C5 (archaeological level C4a dated 13,500–14,500 cal yr BP; level C5a dated 19,000–20,400 cal yr BP). Simple bevels, *rostrums*, and other tools appear in the upper part of unit C6 together with bone fragments (archaeological level C6a, older than ~20,000 cal yr BP, but younger than 41,000 cal yr BP) (Figure 2) (Lahaye et al. 2019).

A total of 452 bone remains were collected from the excavations at TJBA-North. Most of the bones were highly fragmented and corroded, which prevented identification beyond the family level. Rodents were abundant in units C2 and C3, while the basal units C4 and C5 contained mostly Megatherridae bones. A significant proportion of the Megatherridae bones were diaphyses, highly corroded and with typical fractures of dry bone, with very few cancellous bones.

#### 3. Materials and methods

## 3.1. Micromorphology

Four undisturbed blocks of sediment were collected from the profile at TJBA-North, with a focus on the contacts between sedimentary units. Sample BA3-15-1 was collected at the sharp contact between units C4 and C3, sample BA3-15-2 at the contact between units C4 and C5, and sample BA3-15-3 between units C5 and C6. Sample BA3-15-4 was collected from underneath a large meta-limestone fragment at the surface of unit C5 (Figure 2). Samples were dried and impregnated with a mixture of



**Figure 2** Stratigraphic profile of the TJBA-North site, with identification of sedimentary units, radiocarbon, and OSL (BayLum) ages (see Lahaye et al. 2019), archaeological levels, and locations of samples for micromorphology, FTIR, and magnetic analyses.

resin, diluent, and catalyst, and sliced into sections 30 µm thick. Micromorphological analyses were carried out using a Leica s9i stereomicroscope and Leica 2700P petrographic microscope, at magnifications ranging from 10 to 200 times, under plane polarized light (PPL) and crosspolarized light (XPL). Descriptions followed the guidelines of Stoops (2003). The quantification of microscopic components was carried out using visual estimation diagrams available in Bullock et al. (1985), therefore percentages must be considered as estimates.

#### 3.2. FTIR and micro-FTIR

Six bulk sediment samples were collected from the same locations as the micromorphology blocks in the profile, covering sedimentary units C3, C4, C5, and C6 (Table 1). Off-site samples were collected from a lime oven nearby (Figure 1(D, E)), to identify the effects of heating in natural sediments. Small-scale lime production was common among local communities, who

Table 1 List of samples for micromorphology, FTIR, and magnetic analyses collected from TJBA-North. See Figure 2 for locations of the samples in the profile.

		<u> </u>	
Sample code	Analyses	Stratigraphic unit	Observations
BA3-15-1	Micromorph.	C3-C4	Transition between C2 and C3
BA3-15-2	Micromorph.	C4–C5	Transition between C4 and C5
BA3-15-3	Micromorph.	C5–C6	Transition between C5 and C6
BA3-15-4	Micromorph.	C5	Top of C5, underneath limestone boulder
BA3-15- 3a	FTIR	C4	Behind micromorphology block
BA3-15- 3b	FTIR	C3	Behind micromorphology block
BA3-15- 2a	FTIR	C5	Behind micromorphology block
BA3-15- 2b	FTIR	C4	Behind micromorphology block
BA3-15- 1a	FTIR	C6	Behind micromorphology block
BA3-15- 1b	FTIR	C5	Behind micromorphology block
m1	Magnetism	C2	_
m2	Magnetism	C3	_
m3	Magnetism	C3	_
m4	Magnetism	C3	_
m5	Magnetism	C3	_
m6	Magnetism	C4	_
m7	Magnetism	C4	_
m8	Magnetism	C4	_
m9	Magnetism	C4	_
m10	Magnetism	C5	_
m11	Magnetism	C5	_
m12	Magnetism	C5	_
m13	Magnetism	C5	_
m14	Magnetism	C5	_
m15	Magnetism	C5	_
m16	Magnetism	C6	_
m17	Magnetism	C6	_
m18	Magnetism	C6	<u> </u>

spent about a month building the ovens using blocks from the meta-limestone. We sampled the sediments inside the oven at the bottom of the structure, over which lime blocks were heated for about 48 hours to obtain lime (Figure 1(F)). Two spectra were collected for both archaeological and off-site samples. Analyses were carried out with a Cary 670 series spectrometer (Agilent Technologies) by attenuated total reflectance (ATR) with 64 scans, and 4 cm<sup>-1</sup> resolution, between 4000 and 400 cm<sup>-1</sup> using ResolutionsPro software. Spectra processing and comparisons with open-source and personal databases were carried out using Essential FTIR software.

Micro-FTIR measurements were carried out on the thin sections to further investigate the composition and diagenesis of bone fragments in the thin sections for micromorphology. Spectra were collected with a Cary 620 FTIR microscope equipped with an MCT detector in ATR mode through a germanium crystal. All spectra were collected using ResolutionsPro software at 4 cm<sup>-1</sup> resolution with 32 scans between 4000 and 400 cm<sup>-1</sup>. The contact area of the germanium crystal with the thin sections measures  $100 \times 100 \,\mu\text{m}^2$ . We did not perform ATR corrections on the spectral data. The crystallinity index or infrared splitting factor (SF) was calculated in 40 bone fragments following Weiner and Bar-Yosef (1990). This index provides information on the crystallinity of the bone mineral, with increasing values associated with highly ordered crystals as a result of weathering (archaeological/fossil bone) or heating (Stiner et al. 1995).

## 3.3. Magnetic analysis

Magnetic measurements were carried out on 18 specimens from TJBA-North collected from the same profile as the micromorphology samples in 10 cm intervals (from 7.5 to 92.5 cm deep; Table 1). Magnetic analyses of low-field magnetic susceptibility, natural remnant magnetization (NRM), anhysteretic remanent magnetization (ARM), and isothermal remanent magnetization (IRM) were used to determine the different magnetic contributions throughout the studied profile at TJBA-North. Magnetic parameters offer information about concentration, domain, grain-size, and mineralogy of magnetic grains, which are related to the deposition and diagenetic alteration of sediments (e.g., soil development, environmental changes, fires, etc.) (Dalan and Banerjee 1996; Evans and Heller 2003; Liu et al. 2012; Maher 1998; Maxbauer, Feinberg, and Fox 2016; Thompson and Oldfield 1986).

Magnetic susceptibility is one of the most common parameters in environmental magnetism (e.g., Evans

and Heller 2003; Liu et al. 2012) and is defined as the ratio between induced magnetism (M) in a sample and the applied magnetic field (H). This depends on the type, quantity, size, and shape of ferromagnetic minerals in a sample (Dunlop and Özdemir 1997). The grain-size boundary from which particles go from single-domain magnetic grains (SD) to superparamagnetic behavior (SP) is controlled by the time (frequency) in which a magnetic field is applied, and therefore is dependent on frequency and temperature. The lowfield magnetic susceptibility (y) measurements of each specimen were taken using a 200 A/m field oscillating at both 976 Hz (F1) and 15,616 Hz (F3) excitation frequencies for calculating the  $\chi fd\%$  ( $\chi fd\% = [(\chi F1 \chi$ F3)/( $\chi$ F1 × 100)]). Increases in  $\chi$ fd% and magnetic susceptibility indicate an increase in the percentage of superparamagnetic (SP) grains within the total assemblage of magnetic grains. SP contribution is often observed in burned or well-developed soils (e.g., Dalan and Banerjee 1998; Maxbauer, Feinberg, and Fox 2016).

NRM, ARM, and IRM measurements were performed using a vertical 755-1.65 UC SQUID magnetometer (2G Enterprises) housed in a magnetically shielded room with ambient field < 500 nT. The ARM acquisition curves were obtained with a bias field of 0.05 mT in 22 steps up to 100 mT. The IRM data were acquired using a pulse magnetizer with applied DC fields of +1000 mT (corresponding to the saturation of IRM, SIRM) and a backfield of -300 mT. The S-ratio of each sample was determined using a ratio of IRM magnetizations, i.e., S-ratio = [|IRM - 300 mT|/|SIRM|]. The S-ratio indicates the relative contribution of high-coercivity minerals (e.g., hematite, goethite) to low-coercivity minerals (e.g., magnetite) (Evans and Heller 2003; Liu et al. 2012; Maxbauer, Feinberg, and Fox 2016). Susceptibility and remnant magnetizations were mass normalized for each sample.

#### 4. Results

## 4.1. Magnetic analysis

Figure 3 shows all magnetic results from the TJBA-North profile. The mass-normalized magnetic susceptibility (F1 - Susc) shows an increase with depth, varying from  $1.37 \times 10^{-7}$  m<sup>3</sup>/kg (at 7.5 cm) to  $1.16 \times 10^{-6}$  m<sup>3</sup>/kg (at 92.5 cm). The most significant increase in magnetic susceptibility is observed between ~50 and 92.5 cm, covering units C5 and C6. The xfd% varies between  $\sim$ 13% and 16% from 12.5 to 92.5 cm deep; this suggests a constant contribution of SP grains throughout the profile. A similar behavior is observed for NRM values.

ARM at 100 mT (ARM@100) and SIRM determinations display the same behavior observed for magnetic susceptibility, suggesting that the magnetic mineralogy is dominated by low-coercivity grains (e.g., magnetite, titanomagnetite) throughout the profile. The S-ratio values also increase with depth, corroborating the contribution of low-coercivity minerals. However, at shallow depths (0 to ~12.5 cm) the low S-ratio values indicate a significant contribution (up to ~40%) for high-coercivity minerals (e.g., hematite, goethite). In general, the TJBA-North samples are relatively well magnetized. The values for ARM@100, SIRM, and Sratio increase with depth. This suggests an augmented contribution of single domain (SD) over pseudo single domain (PSD) magnetic grains.

Comparing the magnetic results with the age model for the studied profile, we observe that all parameters show low variations from ~10,800 to ~14,000 cal yr BP, with a peak in magnetic susceptibility, ARM@100, SIRM, and S-ratio at ~16,500 cal yr BP. From ~18,000 to ~57,500 cal yr BP, magnetic parameters show a continuous increase. This indicates that variations in magnetic parameters are controlled by climate and not associated with specific human activities (e.g., combustion features). Thus, magnetic data from TJBA-North are a suitable environmental parameter.

## 4.2. Micromorphology

Micromorphology enabled the characterization of early Holocene to terminal Pleistocene units C3 and C4 as well as late Pleistocene units C5 and C6. Sample BA3-15-1 captured the contact between sedimentary units C4 and C3, the Pleistocene to Holocene transition at the site (Figure 4(A)). Sample BA3-15-2 contains two microfacies belonging to the same unit (C5) (Figure 5 (A)), and both samples BA3-15-3 and BA3-15-4 display one microfacies (C6 and C5, respectively) (Figure 6(A), Figure 7(A)).

Unit C3 and C4 show similar porosity (~15–20%) with complex packing voids (few chamber and channel voids), poorly sorted, subangular and subrounded quartz grains (~25-35%) and quartzite fragments  $(\sim 3\%)$  in the coarse fraction (from very coarse to fine sand). Both contain a crumb microstructure with enaulic c/f related distribution (Figure 4(B, D)), but the main difference is in the c/f ratio and color of the predominant micromass. Unit C3 has a 30/70 c/f ratio and a speckled brown micromass (PPL). Unit C4 has a 40/ 60 c/f ratio and the micromass is light brown (PPL) with more bioturbations (e.g., passage features). Both contain very few fine to medium sand-sized bone fragments (~2%) with various colors and signs of

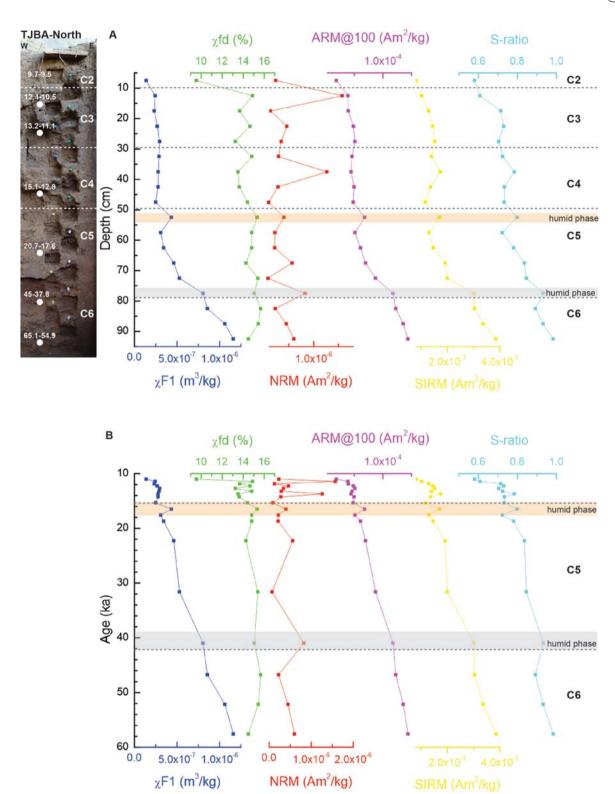
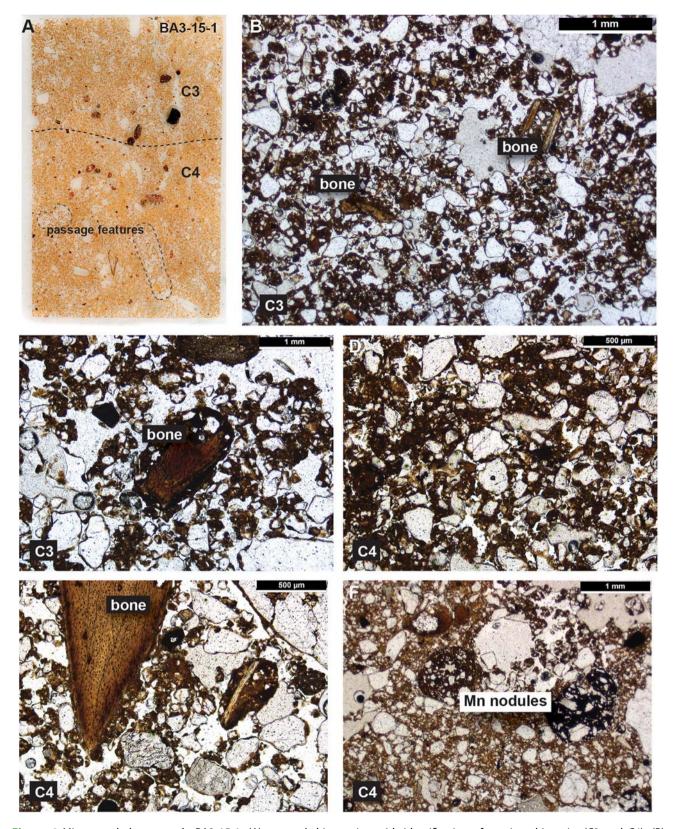


Figure 3 Studied profile and magnetic results. (A) Photograph of the excavated profile at TJBA-North indicating the stratigraphic units and sampling locations (left), and graphs of magnetic parameters in function of depth (right): F1 – Susc is the magnetic susceptibility determined in frequency F1 (976 Hz);  $\chi$ fd% is magnetic susceptibility frequency dependence; NRM is natural remanent magnetization; ARM@100 is anhysteretic remanent magnetization obtained at 100 mT; SIRM is the saturation of isothermal remanent magnetization; S-ratio is the soft ratio. (B) Age model built by combining the magnetic results with the chronology of the studied profile. Dotted lines indicate the basal depths for stratigraphic units. Shaded colored areas show the different climate intervals. Identification of humid phases after Behling et al. (2000) and Wang et al. (2004).



**Figure 4** Micromorphology sample BA3-15-1: (A) scanned thin section with identification of stratigraphic units (C3 and C4); (B) groundmass of unit C3 with crumb microstructure and bone fragments (PPL); (C) reddish bone fragment in unit C3, investigated with micro-FTIR (PPL); (D) groundmass of unit C4 with crumb microstructure and subangular quartz grains (PPL); (E) large bone fragment in unit C4 and, to the right, aggregate with enclosed bone fragment (PPL); (F) orthic manganese nodules in unit C4 (PPL).

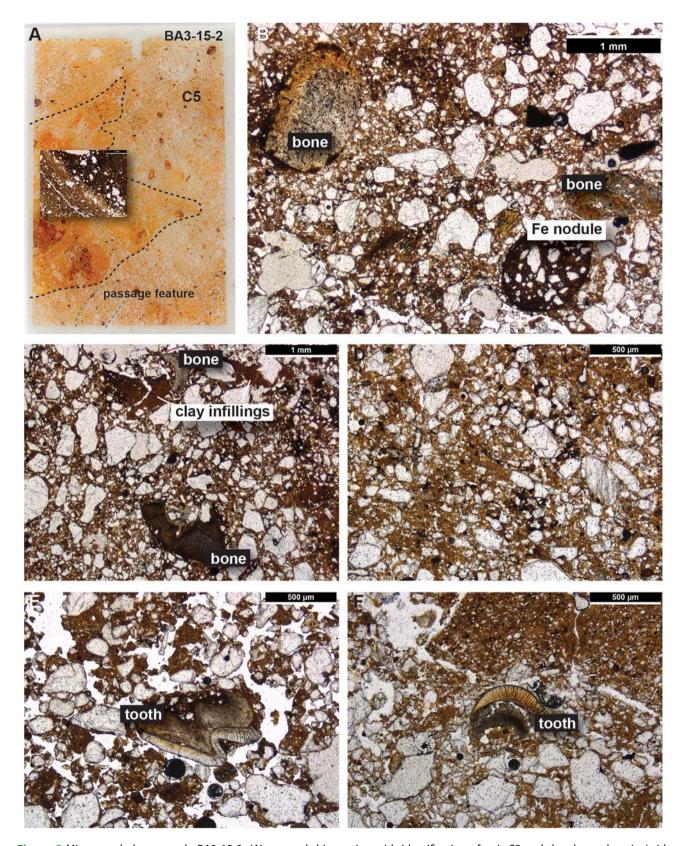


Figure 5 Micromorphology sample BA3-15-2: (A) scanned thin section with identification of unit C5 and the clayey domain (with photomicrograph in PPL); (B) groundmass of unit C5 with rounded bone fragments and iron nodule in heterogeneous reddish and yellowish brown massive micromass (PPL); (C) bone fragments and clay infillings (PPL); (D) iron and manganese impregnations (PPL); (E, F) teeth fragments, possibly from microfauna (PPL).

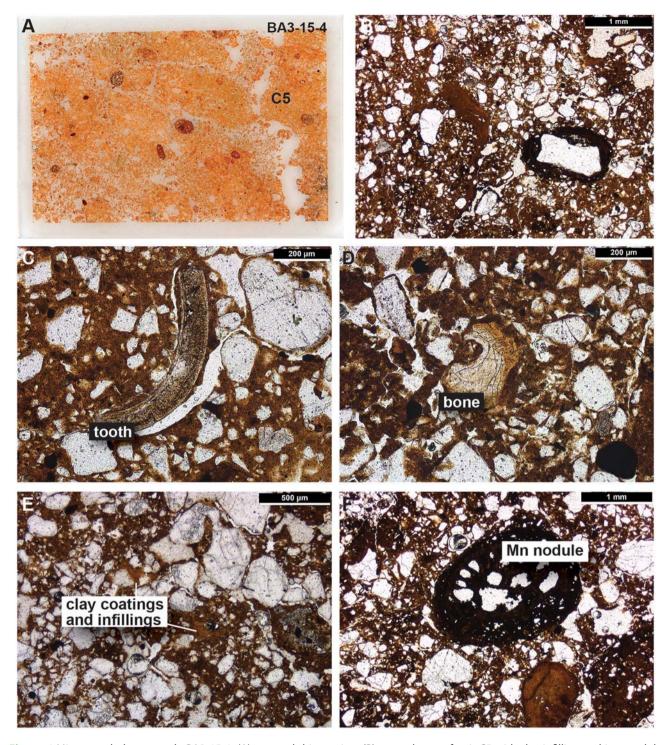


Figure 6 Micromorphology sample BA3-15-4: (A) scanned thin section; (B) groundmass of unit C5 with clay infilling and iron nodule (PPL); (C) tooth fragment within the reddish micromass (PPL); (D) bone fragment with darker margins (PPL); (E) clay infillings and clay coatings around voids (PPL); (F) large manganese nodule (PPL).

dissolution (Figure 4(C, E)). In unit C4 the frequency of bones is slightly higher than in unit C3. Iron and manganese (hydr)oxide nodules and impregnation are frequent in both units (Figure 4(F)).

Unit C5 is very compact, with only ~5% porosity made of fissures, chamber, and channel voids, in a massive microstructure with porphyric c/f related distribution (Figure 5(B), Figure 6(B)). The coarse fraction contains poorly sorted, subangular, and subrounded quartz grains (~35-50%), fragments of quartzite, limestone, schist, clay aggregates, and heavy minerals (~1-3%), as well as very few bone fragments ( $\sim$ 2%) (Figure

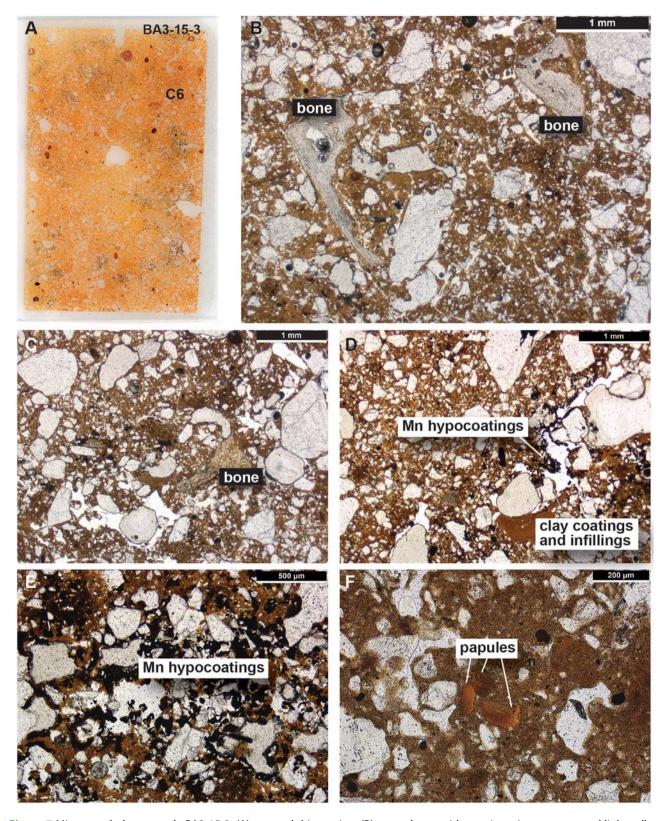


Figure 7 Micromorphology sample BA3-15-3: (A) scanned thin section; (B) groundmass with massive microstructure and light yellow bone fragments (PPL); (C) yellowish brown micromass with bone fragments and subangular quartz grains (PPL); (D) manganese hypocoatings around voids and clay coatings and micro-laminated infillings (PPL); (E) detail of manganese hypocoatings (PPL); (F) reworked clay coatings, also known as papules (PPL).

5(B), Figure 6(D)). The micromass is speckled reddish and yellowish brown (PPL). The frequency of iron and manganese (hydr)oxide nodules and impregnations is higher than the overlying units (and includes manganese dendrites) (Figure 6(F)). Passage features are frequent. Unit C5 contains micro-laminated dusty coatings around the coarse fraction and infillings in voids (Figure 5(C), Figure 6(E)), in addition to undifferentiated iron (hydr)oxide hypocoatings around fissures (Figure 5(D)). A compact, clayey micromass with sharp boundaries was described in the central-left portion of thin section BA3-15-2 (Figure 5(A)), containing very few voids (~2%), common quartz grains (~25%), and very few quartzite ( $\sim$ 3%) and bone ( $\sim$ 2%) in the coarse fraction. The pedofeatures in the clayey domain include clay coatings and iron and manganese nodules. Unit C5 contains a few tooth fragments, possibly from microfauna, not described in other units (Figure 5(E, F), Figure 6(C).

Finally, unit C6 also has low porosity ( $\sim$ 5%) with few fissures and chamber voids in a massive microstructure with a 45/65 c/f ratio and porphyric c/f related distribution (Figure 7(B)). The coarse fraction is the same as other units, with poorly sorted, subangular and subrounded quartz grains (~40%) and very few quartzite ( $\sim$ 3%), as well as bone fragments ( $\sim$ 2%) (Figure 7 (C)). The micromass is yellowish brown (PPL) with several iron and manganese (hydr)oxide nodules and impregnations (Figure 7(D, E)). The frequency of limpid and dusty micro-laminated clay coatings is slightly higher than in unit C5. It contains fragments of clay coatings, also known as papules, indicative of colluviation (Kuhn, Aguilar, and Miedema 2010) (Figure 7(F)).

## 4.3. FTIR and micro-FTIR

The FTIR analyses of bulk sediments showed a similar composition for units C3, C4, C5, and C6, with kaolinite (peaks at 3694, 3645, 3620, 1028, 1000, 911, 749, and 530 cm<sup>-1</sup>) and quartz (797, 777, and 648 cm<sup>-1</sup>) (Beauvais and Bertaux 2002) (FTIR data for the archaeological samples is available as supplemental online material). The intensity of quartz peaks diminishes in the spectra from units C6 and C5 (Figure 8). There is a noticeable difference between archaeological spectra and the lime oven spectra, with the disappearance of the OH region, broadening of the main Si-O-Si at ~1030 cm<sup>-1</sup> and the disappearance of the Al-O-H peak at  $\sim$ 910 cm<sup>-1</sup> (Figure 8) (FTIR data for the lime oven is available as supplemental online material). All these transformations in the lime oven are characteristic of heated kaolinite (Berna et al. 2007; Karkanas et al. 2004; Shoval and Beck 2005; Villagran et al. 2017), indicating that there is no heating of clays at TJBA-North, and therefore, no rubified sediments from combustion features.

Micro-bone fragments were described for all the units at TJBA-North at a similar frequency, with a slight increase in units C4 and C5. Micro-bones have moderate selection (from very fine to medium sand-size) and are mostly angular and subangular, with few rounded fragments. They display different colors and degrees of chemical alteration. A total of 40 measurements were taken from the thin sections for micromorphology to investigate whether bones were exposed to fire, especially the ones with reddish colors under PPL (data for micro-FTIR measurements on bone fragments is available as supplementary material online). All the analyzed bones displayed similar spectra, with the typical peaks of carbonate hydroxyapatite. Only one bone fragment from unit C3 (Figure 4(C)) exhibited a clear shoulder at ~1089 cm<sup>-1</sup> and a weak peak at ~630 cm<sup>-1</sup>, while one fragment from unit C4 and one from unit C5 exhibited weak shoulders at ~1090 cm<sup>-1</sup> and ~1089 cm<sup>-1</sup>, respectively. The splitting factor (SF) of the 40 bones varies between 2.612 and 3.369 (see supplementary material online). The SF values showed no statistically significant difference between bones from different stratigraphic units (ANOVA F = 0.35, p =0.79), indicating that the age of burial did not influence the diagenetic alteration. The dispersion diagram (Figure 9) shows a tendency towards lower SF values in units C3 (c. 11,000-12,500 cal yr BP) and C5 (c. 18,000-40,000 cal yr BP), higher SF values in units C4 and C5, and values falling within the range of other units for unit C6 (older than 41,000 cal yr BP).

### 5. Discussion

### 5.1. Paleoenvironment

A progressive decrease in magnetic susceptibility, ARM@100, SIRM, and S-ratio parameters are detected from the bottom to the top of the sequence. After a peak around 16,500 cal yr BP (top of unit C5), magnetic parameters stabilize and reach the lowest values in unit C2. This tendency indicates a decrease in low-coercivity minerals (magnetite) in units C4 to C2 in relation to basal units C5 and C6. In environmental terms, this can be interpreted as a shift from more humid and warm conditions (units C6 and C5) in the late Pleistocene to progressively drier conditions (units C4 to C2). This is consistent with paleoenvironmental studies in northeastern Brazil that indicate wetter conditions after the LGM (Auler and Smart 2001; Behling et al. 2000; Pessenda et al. 2005). Studies indicate a general dry climate in northeastern Brazil during the late

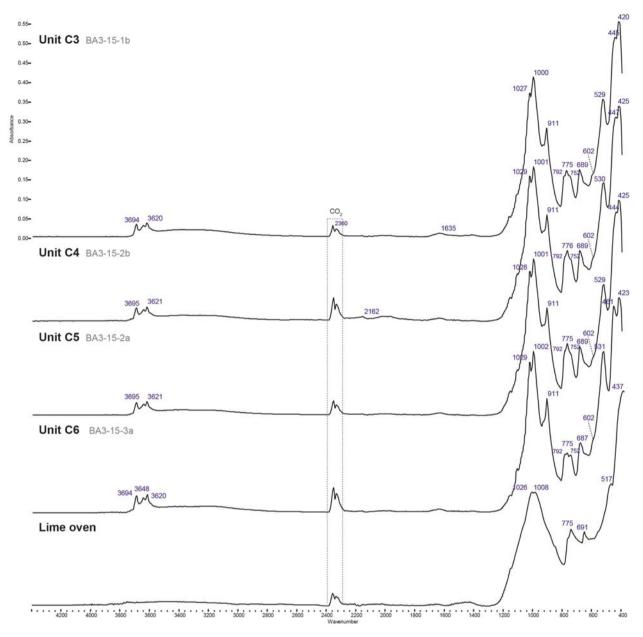


Figure 8 Representative FTIR spectra of sediment samples from units C3, C4, C5, C6, and the heated sediments of a lime oven near the site.

Pleistocene (though more humid than in the Holocene) with episodes of higher pluviosity, lasting for several hundred up to a few thousand years. Data from speleothem and travertine deposits (dated by U/Th method) indicate high pluviosity at c. 48,000 and 40,000–39,000 cal yr BP (Wang et al. 2004), and the pollen data point to wet periods at about 44,000, 37,000, and 28,000 cal yr BP (40,000, 33,000, and 24,000 <sup>14</sup> cal yr BP) (Behling et al. 2000).

The peak in magnetic parameters observed at  $\sim$ 16,500 cal yr BP in TJBA-North is consistent with the highest precipitation rates detected by Behling et al. (2000) between c. 17,000 and 13,000 cal yr BP

(15,500 and 11,800  $^{14}$ cal yr BP) possibly associated with a Heinrich event at  $\sim$ 16–15,000 cal yr BP (Wang et al. 2004). According to Behling et al. (2000), this would be the longest wet period of the terminal Pleistocene and is associated with the expansion of the humid forest. Ledru et al. (2006) report an increase in moisture rates and an expansion of the rainforest after 15,500 cal yr BP. Jacob et al. (2007) detect this rapid shift to wetter conditions occurring at 17,300 cal yr BP, coinciding with the transformation of the semi-arid vegetation to humid rain forest. Higher pluviosity is also recorded in higher stalagmite growth between c. 17,000 -16,000 cal yr BP ( $^{130}$ Th dating) (Wendt et al. 2019).

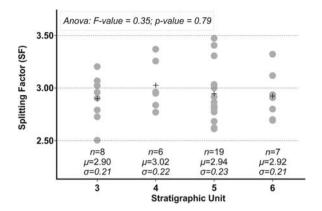


Figure 9 Splitting Factor (gray circles) and mean values (black crosses) for bones analyzed in stratigraphic units C3, C4, C5, and C6. n,  $\boldsymbol{\mu}$  and  $\boldsymbol{\sigma}$  represent sample size, mean value, and standard deviation, respectively. The ANOVA test shows that the difference between stratigraphic units is not statistically significant.

Paleoenvironmental studies of northeastern and northern Brazil point to a relatively wet late Pleistocene climate and more humid tropical climate at the Pleistocene/Holocene transition (Auler and Smart 2001; De Oliveira, Barreto, and Suguio 1999; Jacob et al. 2004; Sifeddine et al. 2003). Paleontological studies of the megafauna assemblage from Serra da Capivara and Barra do Antonião sites also indicate humid conditions in the terminal Pleistocene (Guérin and Faure 2008, 2009, 2014; Guérin et al. 1999). Semi-arid conditions, with an increase in natural fires, only prevailed from the start of the Holocene onwards (Auler et al. 2009; Behling et al. 2000; Ledru et al. 2006; Pessenda et al. 2005; Sifeddine et al. 2003). There was a short period of moist climate in the mid-Holocene, and the onset of drier conditions with expansion of modern-day Caatinga occurred after c. 4200 cal yr BP (De Oliveira, Barreto, and Suguio 1999; Jacob et al. 2004; Utida et al. 2020).

The contrast between sedimentary units C6 and C5, more compact with red color, compared with yellowish units C4 to C2, relates to the progressive shift from more humid conditions at the end of the Pleistocene to the semi-arid Holocene climate. This is also revealed in the micromorphology of units C6 and C5 (see next topic), containing clay coatings and infillings and a higher frequency of iron and manganese (hydr)oxide nodules and impregnations, indicating more humid conditions before 16,500 cal yr BP.

#### 5.2. Formation processes

No evidence of lacustrine deposition, the main depositional agent at the remaining Antonião sites (sectors

A, B, C, and D) (Guérin et al. 1996, 2015), was identified at TJBA-North. This is consistent with the higher topographical position of the site. The micromorphological and compositional characteristics of sediments from TJBA-North indicate a colluvial origin with dual composition: oxidized clay; poorly sorted, subangular, and subrounded quartz grains. The reddish clay may have two origins: (1) dissolution of the pre-Cambrian metalimestone bedrock and residue accumulation, the classical model for Terra Rossa formation in arid and semiarid landscapes (Douchafour 1968; Moresi and Mongelli 1988), which has long been a subject of debate (Merino and Banerjee 2008); (2) from an eroded geological layer overlying the meta-limestone, such as the residual layers of reddish, pelitic rocks observed interbedded on top of the outcrop (Figure 1(G)), and described by Rodet (1997) as filling the conduit network. In both cases, the reddish color is due to water promoting oxidation and formation of hematite. The quartzose sand-sized grains may have an allochthonous origin. Following Santos (2007) and De Oliveira et al. (2014), the sand-sized fraction of TJBA-North is probably colluvium from the pediplain, originating from weathering of Paleozoic sandstones. Sediments from the pediplain are mostly loamy sand with poor sorting, transported during episodes of intense precipitation after long periods of semi-aridity (De Oliveira et al. 2014; Santos 2007).

The stratigraphic difference between units C6 and C5, and units C4 and C3, was also expressed in the micromorphology of the sediments. Units C6 and C5 contain dusty and micro-laminated clay coatings, most abundant in unit C6; these are absent in units C4 and C3. Iron and manganese (hydr)oxide nodules and impregnations are present in the whole profile, but their frequency increases in units C5 and C6, and especially in unit C6, which also contains manganese hypocoatings. These micromorphological features indicate wet/dry cycles in the profile, and more water pasthrough the sediments underneath stratigraphic boundary between units C4 and C5 (marked by the large meta-limestone fragment on the paleosurface). Microlaminated clay coatings are indicative of seasonal clay illuviation, while the dusty coatings are common in the surface and in the upper part of subsurface horizons (Kuhn, Aguilar, and Miedema 2010). This means that units C5 and C6 were formed under more humid conditions than the upper layers C4 and C3; the dusty coatings corroborate the paleosurface at the top of unit C5. The higher moisture level in units C5 and C6 is consistent with paleoclimatic reconstructions for northeastern Brazil (Behling et al. 2000; De Oliveira, Barreto, and Suguio 1999; Jacob et al. 2007;

Ledru et al. 2006; Sifeddine et al. 2003), and with data obtained here by magnetic analyses that point to a wetter climate at the end of the Pleistocene, with a peak at around 16,500 cal yr BP.

Bioturbations are more frequent in the upper layers C3 and C4, mostly in the form of passage features and few channel and chamber voids. Units C5 and C6 are compact, and, despite the high frequency of clay coatings, no evidence of artifact mixing or vertical translocation was identified.

The splitting factor of the 40 bones analyzed by micro-FTIR falls within the range of archaeological weathered bones or low temperature heating (< 300°C) (Dal Sasso et al. 2016; Stiner et al. 1995; Surovell and Stiner 2001; Thompson, Gauthier, and Islam 2009). Both processes cause similar effects on the crystallinity of carbonate hydroxyapatite (Lebon et al. 2010; Stiner et al. 1995; Thompson, Gauthier, and Islam 2009). Only one fragment from Holocene unit C3 exhibited a shoulder at ~1089 cm<sup>-1</sup> and a weak peak at ~630 cm<sup>-1</sup>. One fragment from unit C4 and another from unit C5 only exhibited a weak shoulder at ~1090 cm<sup>-1</sup> and  $\sim$ 1089 cm<sup>-1</sup>, respectively. The appearance of these peaks in the FTIR spectra of bone has been attributed to heating at temperatures above 300°C (Stiner et al. 1995; Thompson, Gauthier, and Islam 2009, 2013). Both peaks have been reported as evidence of heating in bones as old as 1.0 million years ago (Ma) from Wonderwerk cave (Berna et al. 2012) and 1.5 Ma from Koobi Fora (Hlubik et al. 2017). However, the peaks at  $\sim$ 1089 cm<sup>-1</sup>, and especially at  $\sim$ 630 cm<sup>-1</sup>, in the micro-bone fragments from TJBA-North are too weak to fully confirm that bones were heated above 300°C. In addition, the spectra were collected on the thin section and do not have optimal resolution, meaning that FTIR analyses on macroscopic bones from TJBA-North are needed to further investigate the presence of heated bones at the site.

The micro-bone fragments in the TJBA-North sediments may be associated with the megafauna remains recovered at the site during excavations. In situ fragmentation and weathering of the megafauna bones could result in the minute bone splinters observed in the thin section. In fact, taphonomic analyses of the bones recovered during excavation point to high fragmentation and corrosion. In addition, the microscopic tooth fragments suggest that micro-bones also result from weathering of microfauna remains. Another possibility, evidenced by the rounding of some bone fragments, is that micro-bones were transported from outside the sheltered area by the colluviation processes responsible for sand deposition at the site.

## 6. Conclusion

Geoarchaeological and site formation processes analyses provide the contextual framework to understand and interpret the history and content of archaeological deposits. At sites with controversial chronologies and/ or artifactual remains, site formation is a key factor for further discussions concerning the input of anthropogenic vs. natural sediments, age of the deposit, its content and preservation.

Stone tools are found throughout the archaeological sequence at TJBA-North, from layers dating ~20,000 cal yr BP, to the upper early Holocene layers. Sediment deposition at the rock shelter is colluvial, combining autochthonous reddish clays from eroded pelitic rocks interbedded in the pre-Cambrian meta-limestone, and allochthonous sand-sized quartz from the pediplain, originating in the Paleozoic cuesta (c. 2.5 km northeast). A shift in environmental conditions is registered at ~16,500 cal yr BP. Below this boundary, noticeable between units C5 and C4, environmental conditions were more humid with an increase in magnetic parameters (e.g., magnetic susceptibility, ARM, SIRM, and S-ratio), greater oxidation of the deposit (e.g., iron and manganese nodules) and illuviation of dusty clay. Above this boundary, interpreted as a paleosurface by the presence of a large meta-limestone boulder at the top of unit C5, conditions became progressively drier, leading up to the present-day climate.

The environmental interpretations derived by magnetic and micromorphological data are consistent with regional paleoclimatic reconstructions that describe a wetter climate during the terminal Pleistocene, relative to Holocene semi-arid conditions (Auler and Smart 2001; Behling et al. 2000; De Oliveira, Barreto, and Suguio 1999; Pessenda et al. 2005; Sifeddine et al. 2003). This is also corroborated by a peak in magnetic parameters at ~16,500 cal yr BP corresponding to the highest precipitation rates reported by Behling et al. (2000) between c. 17,000 -13,000 cal yr BP, and to an increase in moisture and expansion of the rainforest after 15,500 cal yr BP, reported by Ledru et al. (2006). These environmental changes were produced by the rapid shift to wetter conditions detected by Jacob et al. (2007) at 17,300 cal yr BP, and described as higher stalagmite growth between c. 17,000-16,000 cal yr BP by Wendt et al. (2019).

Anthropogenic micro-remains were not detected in the micromorphological analyses of TJBA-North. Charcoal and micro-charcoal were absent, and there was no evidence of heated sediments in the FTIR spectra. Micro-charcoal at the site could have been produced by natural fires, which were prevalent in the semi-arid

landscape during dry periods (Auler et al. 2009; Behling et al. 2000; Ledru et al. 2006; Pessenda et al. 2005; Sifeddine et al. 2003). However, their absence at TJBA-North suggests that the byproducts of natural fires did not reach the rock shelter, or that fires around the site were not widespread enough to create visible deposits. Sampling off-site areas outside the sheltered area would be necessary to confirm this.

Micro-bone fragments were found in all six stratigraphic units; however, their attribution to human activities remains uncertain for two main reasons: (1) megafauna remains are common at the site and the surroundings and intense weathering (high fragmentation and corrosion) of these remains may have contributed to the micro-bone assemblage; (2) the micro-teeth described in the thin sections point to microfauna bones in the deposit. Two bones from Holocene units C4 and C3 showed evidence of heating at temperatures above 300°C by micro-FTIR. This suggests the need for further spectroscopic analyses of the macro-bone assemblages to test the ubiquity of fire in the faunal

Geoarchaeological data confirm the colluvial origin of the sediments from TJBA-North. Given the complete absence of anthropogenic micro-remains in the layers containing stone tools, the data indicate that the site may only have been used for short-term activities.

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## **Disclosure statement**

No potential conflict of interest was reported by the author(s).

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