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# RADIOCARBON DATING AND ISOTOPIC PALEOECOLOGY ( $\delta^{13}\text{C}$ , $\delta^{18}\text{O}$ ) OF MESOHERBIVORES FROM LATE PLEISTOCENE OF GRUTA DA MAROTA, ANDARAÍ, BAHIA, BRAZIL

DATAÇÃO RADIOCARBON E PALEOECOLOGIA ISOTÓPICA ( $\delta^{13}\text{C}$ ,  $\delta^{18}\text{O}$ ) DE MESOHERBIVOROS DO PLEISTOCENO ATUAL DA GRUTA DA MAROTA, ANDARAÍ, BAHIA, BRASIL

Mário André Trindade DANTAS (1); André Vieira ARAÚJO (2); Estevan Eltink NOGUEIRA (3)

- (1) Laboratório de Ecologia e Geociências, Instituto Multidisciplinar em Saúde, Universidade Federal da Bahia, campus Anísio Teixeira, Vitória da Conquista, BA.  
(2) Sociedade Espeleológica Azimute, Campo Formoso, BA.  
(3) Universidade Federal do Vale do São Francisco, Senhor do Bonfim, BA.

Contatos: [matdantas@yahoo.com.br](mailto:matdantas@yahoo.com.br); [anddrevieira@gmail.com](mailto:anddrevieira@gmail.com); [estevan.eltink@univasf.edu.br](mailto:estevan.eltink@univasf.edu.br).

## Resumo

A Gruta da Marota, inserida dentro do sistema cárstico da Bacia Una-Utinga, Chapada Diamantina, é uma caverna de particular interesse na compreensão de padrões paleoecológicos da Região Intertropical Brasileira. No presente trabalho, utilizamos a datação por radiocarbono ( $^{14}\text{C}$  AMS) juntamente com modelos matemáticos de regressões lineares usando valores isotópicos de carbono ( $\delta^{13}\text{C}$ ), trazendo novas informações sobre a paleoecologia e cronologia das espécies como *Catonyx cuvieri*, *Nothrotherium maquinense* e *Tapirus terrestris* encontrados na Gruta da Marota. As datações realizadas por AMS mostraram uma idade por volta de 11.000 anos para todos os fósseis estudados, sugerindo que viviam na região durante o final do Pleistoceno. Os resultados das análises isotópicas para o carbono foram de -17.21 ‰ para *T. terrestris*, -11.19 ‰ para *C. cuvieri* e -11.99 ‰ e -12.12 ‰ para os dois adultos de *N. maquinense*. Esses dados nos permitem sugerir *T. terrestris* como especialista em plantas C<sub>3</sub> enquanto que *N. maquinense* e *C. cuvieri* apresentam dieta generalista com predomínio de plantas C<sub>3</sub> ( $p_i\text{C}_3 > 66\%$ ). As informações obtidas das análises de  $\delta^{18}\text{O}$  variando entre -4.06 ‰ a -0.08 ‰ ( $\mu\delta^{18}\text{O} = -1.94\%$ ) em conjunto com os dados de  $\delta^{13}\text{C}$ , sugerem um clima mais quente e florestado na região há cerca de 11.000 anos.

**Palavras-Chave:** mesoherbívoros; paleoecologia isotópica; pleistoceno tardio.

## Abstract

The Marota cave, inserted in the karst system of Una–Utinga basin, Chapada Diamantina, is a cave of particular interest to the paleoecological understanding of the Brazilian Intertropical Region. In the present work, we used radiocarbon dating ( $^{14}\text{C}$  AMS) in association with mathematical models of linear regressions using isotopic values of carbon ( $\delta^{13}\text{C}$ ), presenting new information about the paleoecology and chronology of the species, such as *Catonyx cuvieri*, *Nothrotherium maquinense* and *Tapirus terrestris* found in the Marota cave. The  $^{14}\text{C}$  AMS datings showed an age around 11.000 years for all studied fossils, suggesting they lived in this region during the Late Pleistocene. The carbon isotopic values presented here for *T. terrestris* was -17.21 ‰, for *C. cuvieri* was -11.19 ‰, and *N. maquinense* -11.99 ‰ and -12.12 ‰. These data allow us to suggests *T. terrestris* as a C<sub>3</sub> plants specialist, while *C. cuvieri* and *N. maquinense* were generalists, feeding more of C<sub>3</sub> plants ( $p_i\text{C}_3 > 66\%$ ). The information obtained from ( $\delta^{18}\text{O}$ ) varying from 4.06 ‰ to -0.08 ‰ ( $\mu\delta^{18}\text{O} = -1.94\%$ ), associated with  $\delta^{13}\text{C}$  data, suggests a warmer climate in the region around 11.000 yr ago.

**Keywords:** mesoherbivores; isotopic paleoecology; late pleistocene.

## 1. INTRODUCTION

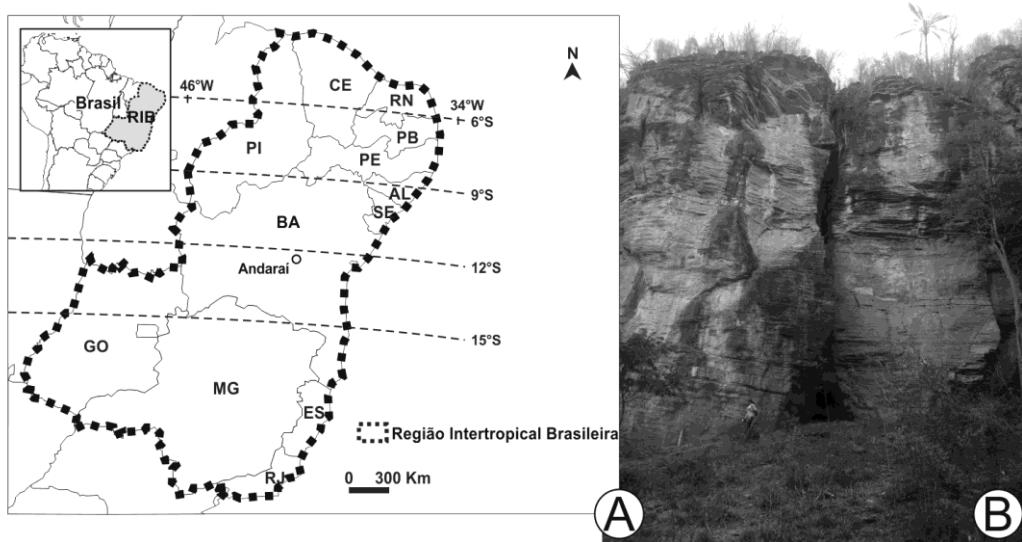
Marota cave (BA-479) is located in Andaraí municipality, Bahia (Figure 1; 12°37'21"S, 41°01'20"W). The cave is inserted within in the Una-Utinga Basin, being formed by sediments of the Upper Proterozoic (Supergroup São Francisco,

Group One). Extending on eastern margin of the Serra do Sincorá (geotectonic domain of the Chapada Diamantina), the carbonate lithophytes of Una-Utinga Basin form an extensive karst system with caves of exalted speleological and paleontological value (e.g. Poço Azul de Milú).

In the last years, the Marota cave has been the focus of scientific researches in Paleontology and Paleoclimatology. They include the record of giant sloths, *Catonyx cuvieri* and *Nothrotherium maquinense* (Dantas *et al.* 2017) and paleoenvironment discussion of the region based on the paleoclimatologic data ( $\delta^{18}\text{O}$ ) acquired from stalagmites (Strikis *et al.* 2018).

In the present communication we present: (i) weight estimation for the fossils of *C. cuvieri*, *N. maquinense* and *Tapirus terrestris* (ii) radiocarbon datings ( $^{14}\text{C}$  AMS); and (iii) carbon and oxygen isotopes data, which provide paleoecological discussion about the past environment in the late Pleistocene.

## 2. MATERIALS AND METHODS



**Figure 1:** (A) Location map of Andaraí municipality, Bahia, Brazil; (B) Entrance of Marota's Cave (photo: Mário Dantas, 2017).

**Table 1:** Circumference (in mm) of humerus and femur obtained from the taxa fossils of the Marota cave.

taxa	circunference	
	humerus	femur
<i>T. terrestris</i>	107.00	128.00
<i>N. maquinense</i>	141.00	186.00
<i>C. cuvieri</i>	150.00	182.00

## 2.2. Radiocarbon datings

The quoted uncalibrated dates are given in radiocarbon years before 1950 (years BP), using the  $^{14}\text{C}$  half-life of 5.568 years. The error is quoted as one standard deviation and reflects both statistical and experimental errors. The date has been corrected for isotope fractionation and used the

## 2.1. Weight estimation

To estimate carbon enrichment (subtopic 2.3) we calculated the estimated weight (1) for mammals species that were found in Marota cave, Andaraí, Bahia (Table 1). For *T. terrestris* was used the regression (1) proposed by Anderson *et al.* (1985). For *N. maquinense* and *C. cuvieri*, an adaptation of Anderson *et al.* (1985) regression (2) was employed (Dantas *et al.*, in preparation):

$$(1) W = 0.078 C_{(h+f)}^{2.73} \quad (2) W = 0.078 C_{(h+0.4f)}^{2.73}$$

Where  $W$  is the weight (in grams - g),  $C$  is the minimum circumference of humerus and femur diaphysis (in mm). In the Table 1 is shown the measures used for the estimation.

hydroxyapatite of fossil teeth. The reliability of the applied technique for purification of hydroxyapatite was previously demonstrated by a comparison of  $^{14}\text{C}$  dating of both collagen and hydroxyapatite fractions, which yielded comparable results, and, therefore, suggests none, or insignificant, isotopic exchange between the biogenic hydroxyapatite and the depositional environment (Sánchez *et al.*, 2004; Cherkinsky, 2009).

The radiocarbon ages were calibrated using CALIB 7.1 program (Reimer *et al.*, 2013) and shown in the Table 2.

## 2.3. Isotopic Paleoecology ( $\delta^{13}\text{C}$ , $\delta^{18}\text{O}$ )

Four samples (Table 2) were analyzed to obtain carbon and oxygen isotopic composition from structural carbonate of their dentin and enamel: two adult individuals of *Nothrotherium maquinense*, one adult individual of *Tapirus terrestris* and one juvenile of *Catonyx cuvieri*.

Isotopic composition of hydroxyapatite can be preserved with minimal or no significant diagenetic alteration (e.g. Bocherens *et al.*, 1996). The stable isotope analyzes were performed at Center for Applied Isotope Studies from University of Georgia. All results are reported using delta notation,  $\delta = [(\text{R}_{\text{sample}}/\text{R}_{\text{standard}} - 1) * 1000]$  (Coplen, 1994). The reference for carbon isotope values ( $R = ^{13}\text{C}/^{12}\text{C}$ ) and oxygen isotope values ( $R = ^{18}\text{O}/^{16}\text{O}$ ) is V-PDB.

To estimate ecological measurements we calculate isotope niche breadth ( $B$ ) using Levins' (1968) measure (3), where  $p_i$  is relative proportion of individuals in isotope bin  $i$ . This measure was then standardized ( $B_A$ ) from 0 to 1 following equation (4), where  $n$  is total number of isotope bins available. Values lower or equal to 0.5 suggests a specialist, and above 0.5 a generalist.

$$(3) B = \frac{1}{\sum p_i^2} \quad (4) B_A = \frac{B - 1}{N - 1}$$

Interpretation of carbon isotopic values for medium-to large-bodied herbivorous mammals is made based on known average for C<sub>3</sub> plants ( $\mu\delta^{13}\text{C} = -27 \pm 3 \text{ ‰}$ ), C<sub>4</sub> plants ( $\mu\delta^{13}\text{C} = -13 \pm 2 \text{ ‰}$ ) and CAM plants (intermediate values between  $\delta^{13}\text{C}$  C<sub>3</sub> and C<sub>4</sub> plants). Carbon isotopic data here presented were obtained from mammals with body mass varying from 182Kg to 230 Kg (Table 2). Based on Tejada-Lara *et al.*(2018) we estimated  $\epsilon^*$  diet-bioapatite of these mammals and found values varying between 13.16‰ to 13.26‰, thus being approximated to 13 ‰. Considering the enrichment of 13‰,  $\delta^{13}\text{C}$  values lower than -14 ‰ are typical of animals with a diet consisting exclusively of C<sub>3</sub> plants, while  $\delta^{13}\text{C}$  values higher than 0 ‰ are consistent with a diet based on C<sub>4</sub> plants.

Thus, proportion of mesoherbivores mammals could be suggested summing +2 ‰ in the carbon data,due to Suess effect (Keeling, 1979), using a single isotope mathematical mixing model (5; Phillips, 2012) for food types: C<sub>3</sub> and C<sub>4</sub> plants.

$$(5) \quad \begin{aligned} \delta^{13}\text{C}_{\text{mix}} &= \delta^{13}\text{C}_1 f_1 + \delta^{13}\text{C}_2 f_2 \\ 1 &= f_1 + f_2 \end{aligned}$$

## 3. RESULTS AND DISCUSSION

### 3.1. Weight estimation

For the fossils of Marota cave, weight between 182 kg to 230 kg were estimated (Table 3).The weight of *T. terrestris* was estimated in 230 kg, which is higher than the average for extant individuals( $w = 170 \text{ kg}$ ), but in the Late Pleistocene the record of larger specimens is not so uncommon(Perini *et al.*, 2011).

In a first attempt Dantas *et al.*(2017) used an adaptation of Anderson *et al.* (1985) regression of that considers 60% of the circumference of the femur of xenarthrans, however, using measurements of extant xenarthrans species, a circumference of 40% of the femur seems to be more realistic (Dantas, in preparation). Thus, for *N. maquinense* and *C. cuvieri* were estimated the weight of 182 kg and 200 kg, respectively.

There is no previous weight estimation for *N. maquinense*, thus, we are not able to discuss its weight, however, *C. cuvieri* presents a weight comparable with another Scelidotherriinae, such as *Scelidotherium leptocephalum*. Farina *et al.* (1998) estimated a weight of 551 kg for an adult of *S. leptocephalum*, as the specimen here presented belonged to a juvenile, we estimate as congruent the weight with that expected for the taxon.

### 3.2. Radiocarbon dating and Isotopic Paleoecology ( $\delta^{13}\text{C}$ , $\delta^{18}\text{O}$ )

The  $^{14}\text{C}$  AMS dating allows us to suggest a higher contemporaneity among studied fossils of Marota cave. *T. terrestris*, *N. maquinense* and *C. cuvieri* were date daround 11,000 yr (Table 2), showing an interesting diversity of mammals living at the same time, in the Late Pleistocene of the region.

**Table 2:** Radiocarbon datings ( $^{14}\text{C}$  AMS) for late Pleistocene mesoherbivores from Marota cave. **Labels.** *Cc* - *C. cuvieri* (UGAMS 34121), *Tt* - *T. terrestris* (UGAMS 34122), *Nm1* - *N. maquinense* (UGAMS 34123), *Nm2* - *N. maquinense* (UGAMS 34124).

Taxa	Age (yr BP)	Age (Cal yr BP)
<i>Cc</i>	11,150±30	11,152-11,019
<i>Tt</i>	11,030±30	11,061-10,835
<i>Nm1</i>	11,130±30	11,142-10,960
<i>Nm2</i>	11,520±35	11,494-11,334

The  $\delta^{13}\text{C}$  of the mesoherbivores from Marota cave varied between -17.21 ‰ to -11.19 ‰, showing a predominance of consumption of C<sub>3</sub>

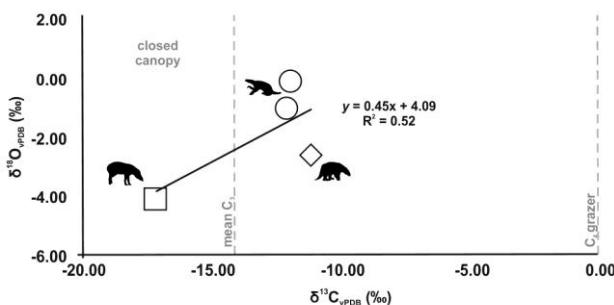
plants, and probably a more forested environment ( $p_i C_3 > 66\%$ ; Table 3).

$\delta^{13}\text{C}$  for *T. terrestris* was  $-17.21\text{\textperthousand}$ , which is in the range found by De Santis (2011) to extant *T. terrestris* from South America ( $\delta^{13}\text{C}$  ranged from  $-18.1\text{\textperthousand}$  to  $-12.8\text{\textperthousand}$ ). Its diet in Marota cave was composed exclusively of  $C_3$  plants ( $p_i = 100\%$ ; Table 3; Figure 2), showing that it was a specialist ( $B_A = 0.00$ ) in this kind of resources.

**Table 3:** Carbon ( $\delta^{13}\text{C}_{\text{VPDB}}$ ) and Oxygen ( $\delta^{18}\text{O}_{\text{VPDB}}$ ) isotopic values (‰), proportion ( $p_i$ ) of  $C_3$  and  $C_4$  plants consumed, standardized niche breadth ( $B_A$ ) and weight ( $W$ ) of Late Pleistocene mesoherbivores from Marota cave. **Labels.** *Cc* - *C. cuvieri* (UGAMS 34121), *Tt* - *T. terrestris* (UGAMS 34122), *Nm1* - *N. maquinense* (UGAMS 34123), *Nm2* - *N. maquinense* (UGAMS 34124).

Taxa	$\delta^{13}\text{C}$	$\delta^{18}\text{O}$	$p_i C_3$	$p_i C_4$	$B_A$	$W$
<i>Cc</i>	-11.19	-2.59	0.66	0.34	0.82	200.00
<i>Tt</i>	-17.21	-4.06	1.00	-	0.00	230.00
<i>Nm1</i>	-11.99	-0.08	0.71	0.29	0.69	182.00
<i>Nm2</i>	-12.12	-1.02	0.72	0.28	0.68	182.00

The carbon isotopic values presented here for *C. cuvieri* and *N. maquinense* (Table 3; Figure 2) are the first report for these taxa, and thus, without other isotopic data for comparison. *C. cuvieri* presented a  $\delta^{13}\text{C}$  of  $-11.19\text{\textperthousand}$ , which allow us to suggests a diet based on 66 % of  $C_3$  plants and 34 % of  $C_4$  plants. For *N. maquinense* we have data from two adult individuals, which presents similar  $\delta^{13}\text{C}$  values ( $-11.99\text{\textperthousand}$  and  $-12.12\text{\textperthousand}$ ), and had a diet composed mainly in  $C_3$  plants ( $p_i = 71$  and 72 %). *N. maquinense* fed more in  $C_3$  plants than *C. cuvieri*, this difference could be strategies to avoid competition, although, both taxa were generalists ( $B_A = 0.68$  to 0.82).



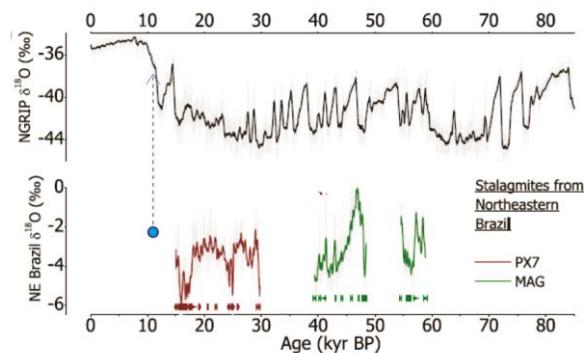
**Figure 2:** Correlation between carbon ( $\delta^{13}\text{C}$ ) and oxygen ( $\delta^{18}\text{O}$ ) values for *T. terrestris* (square), *N. maquinense* (circle) and *C. cuvieri* (losange).

Oxygen isotopic values of mesoherbivores from Marota cave were analysed in carbonate of hydroxyapatite of their dentin and enamel, the  $\delta^{18}\text{O}$

ranged from  $-4.06\text{\textperthousand}$  to  $-0.08\text{\textperthousand}$  ( $\mu\delta^{18}\text{O} = -1.94\text{\textperthousand}$ ). Grazers acquire their oxygen isotopic content mainly in lakes and rivers, while browsers (as all taxa from Marota cave) acquire in  $C_3$  plants tissues (e.g. leaves and fruits).

Water in plant tissues carries the same  $^{18}\text{O}$  isotopic signal than the environment (Marshall *et al.*, 2007), thus, we believe that we can compare the  $\delta^{18}\text{O}$  from mammals (as there is no physiological enrichment in the animal) with the  $\delta^{18}\text{O}$  from stalagmites.

There is a good correlation ( $R^2 = 0.52$ ; Figure 3) between carbon and oxygen isotopic values of mesoherbivores from Marota cave, which allow us to interpret that their oxygen isotopic content came from both drunked water and  $C_3$  plants tissues, representing the oxygen from the environment.



**Figure 3:** Last 80 kyr of the running mean North Greenland Ice Core Project (NGRIP) ice core  $\delta^{18}\text{O}$  record on the annual-layer-counted GICC05 (top);  $\delta^{18}\text{O}$  speleothem record from Paixão (PX7) and Marota (MAG) caves, Bahia, Brazil (below);  $\mu\delta^{18}\text{O}_{\text{VPDB}}$  from Late Pleistocene (11 kyr) mesoherbivores from Marota cave (blue dot)(Figure modified from Strikis *et al.*, 2018).

Strikis *et al.* (2018) presents  $\delta^{18}\text{O}$  from stalagmites from Marota and Paixão (a nearby cave to Marota) caves showing paleoclimatic oscillation through an interval of 60 kyr to 15 kyr and in comparison with  $\delta^{18}\text{O}$  from ice core record (NGRIP; Figure 3). The records from Marota and Paixão caves are in good correlation with the pattern found in ice core record. The  $\mu\delta^{18}\text{O}$  data from mesoherbivores are encompassed in the  $\delta^{18}\text{O}$  variation found for the region, indicating a warmer climate at 11 kyr, in congruency with the ice core record (NGRIP; Figure 3).

#### 4. FINAL REMARKS

In this communication we present weight estimation, radiocarbon datings and isotopic data for

*C. cuvieri*, *N. maquinense* and *T. terrestris* from Marota cave, Andaraí, Bahia.

All taxa lived at 11 kyr, in a more forested environment and warmer climate, where *C. cuvieri*

and *N. maquinense* were generalists ( $B_A > 0.68$ ), feeding more of C<sub>3</sub> plants ( $p_iC_3 > 66\%$ ), and *T. terrestris* was a specialist C<sub>3</sub> feeder ( $B_A = 0.00$ ;  $p_iC_3 = 100\%$ ).

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