



ESR dating of late Quaternary megafauna fossils from João Dourado, Bahia, Brazil

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ABSTRACT

We applied the ESR (Electron Spin Resonance) dating method to fossils of *Notiomastodon platensis* (two teeth) and *Toxodontinae* (two teeth) found in the fossiliferous deposit of Lajedão do Patrício, João Dourado, State of Bahia, Brazil, to identify the period of formation of this fossil accumulation. Neutron Activation Analysis was applied to determine the concentrations of the main radioisotopes in enamel, dentine and sediment. The ages found for *N. platensis* are 16.8 ± 2.6 ka and 12.5 ± 2.3 ka, while the ages for *Toxodontinae* are 9.6 ± 1 ka and 9.1 ± 1 ka. The results for *Notiomastodon. platensis* and *Toxodontinae* are similar to other fossiliferous of Brazil. The estimated maximum time-averaging for Lajedão do Patrício is 11.3 ka, indicating a long period of accumulation of skeletal remains, attributed to resedimentation and reworking. The crossing between the period of formation of fossil assemblage Lajedão do Patrício and ages of climatic variations diponible in paleoclimatic curves produced for the Quaternary of northeastern Brazil indicates different climatic and environmental conditions during the formation of the deposit.

1. Introduction

The fossil record of the Quaternary megafauna in northeastern Brazil is impressive and widely distributed in geographic terms. One of the main fossil-containing deposits of the Quaternary megafauna of South America is named tank deposit, whose geographical distribution is restricted to northeastern Brazil. Tank deposits are small stratified sedimentary bodies that infilling natural depressions formed in basement rock outcrops (Araújo-Júnior et al., 2013, 2015, 2017).

Geochronological studies concerning the Quaternary megafauna preserved in deposits of northeastern Brazil are rare, despite the wide geographical distribution and large richness and diversity of fossils. In this region, Electron Spin Resonance (ESR) ages were successfully obtained in fossils of megafaunal of remains of some of tank deposits (Kinoshita et al., 2008, 2005; Oliveira et al., 2010; Ribeiro et al., 2013). This method is useful for materials from the Middle Pleistocene to Early

Holocene, cover an important gap separating the ¹⁴C dating method to the beginning of other radioisotope methods as U/Th (Ikeya, 1993; Rink, 1997). ESR is based on the effects of ionizing radiation in solids, produced by radioactive elements such as uranium (U), thorium (Th) and potassium (K) present in the environment, in the archaeological material itself, and by cosmic radiation (Duval, 2018).

In northeastern of Brazil, twenty one absolute dating in fossils of the Quaternary megafauna are available in literature, being nine using the ¹⁴C method (Dantas et al., 2013; Scherer et al., 2017), and 12 using the ESR method (Kinoshita et al., 2008, 2014; Oliveira et al., 2010; Ribeiro et al., 2013). Other ages were obtained from sediments associated with fossils from karst deposits: a coprolite of the ground sloth *Nothroterium* from Gruta dos Brejões, Chapada Diamantina, Bahia (Czaplewski and Cartelle, 1998), and in sediments associated with the fossils of Toca do Garrincho (Peyre et al., 1998), and Toca do Serrote do Artur (Faure et al., 1999), Serra da Capivara National Park, São Raimundo Nonato,

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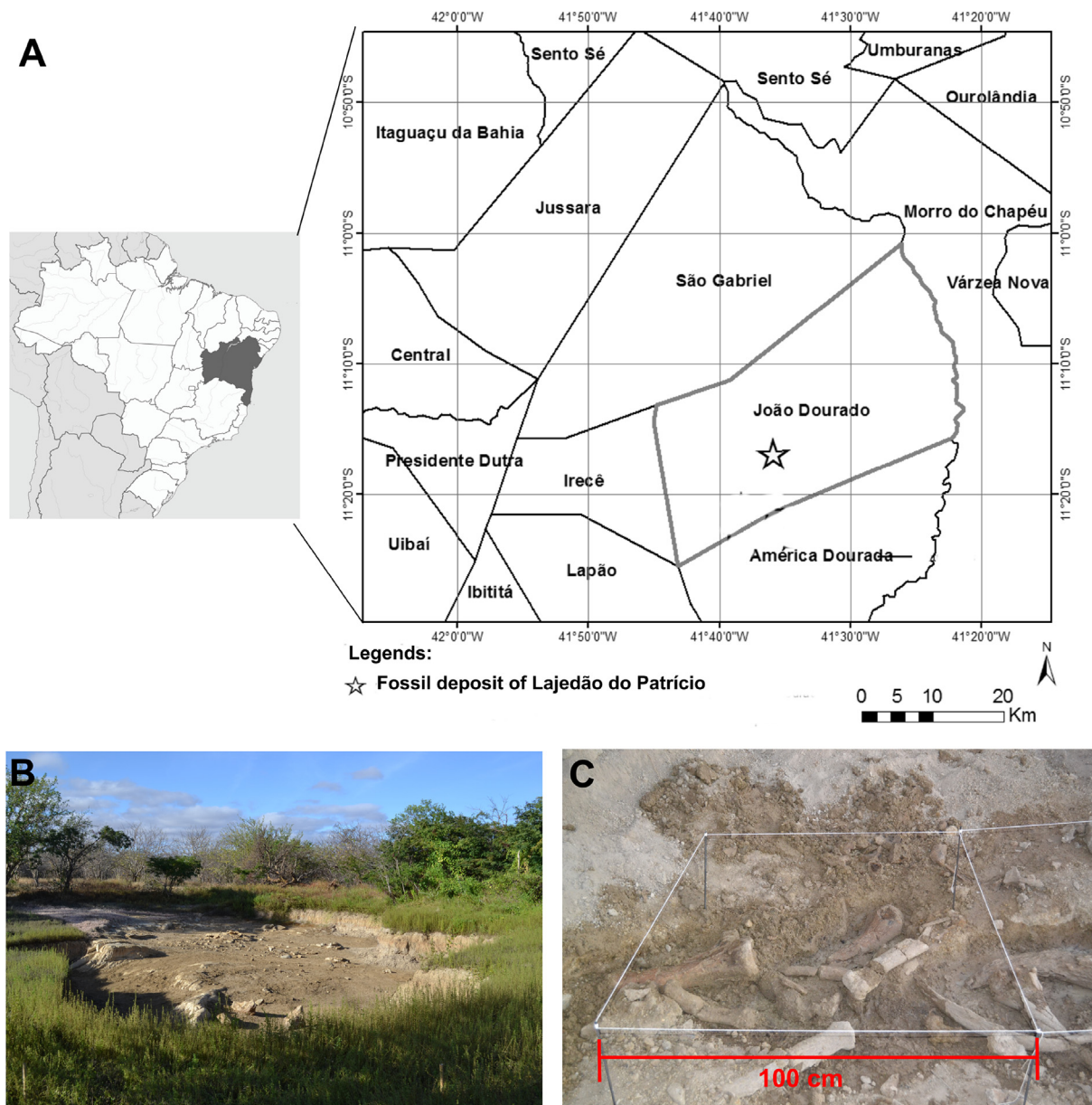


Fig. 1. Geographic location and general view of the Lajedão do Patrício paleontological site; (A) Geographic location of the municipality of João Dourado, Bahia, Brazil; (B) and (C) visualization of the fossiliferous deposit and fossiliferous layer, respectively.

Piauí.

This study aims to: (i) perform the first absolute dating in the fossils of the fossiliferous deposit of Lajedão do Patrício, João Dourado, Bahia State, Brazil, by the ESR method; (ii) infer the periods of the deposition and formation of the analyzed fossiliferous deposit; and (iii) discuss the geochronology concerning the Late Quaternary megafauna from the fossil deposits of northeastern Brazil.

2. Site description

The fossiliferous deposit of Lajedão do Patrício is located at Fazenda Faveleira, in the village of Lajedão do Patrício, municipality of João Dourado, Bahia State, Brazil ($11^{\circ} 16' 52.4''$ S, $41^{\circ} 35' 79''$ W datum WGS 84, elevation of 820 m; Fig. 1). The deposit is placed in Neo-Proterozoic carbonate metasedimentary rocks of the Salitre Formation, Nova América unit. This unit is characterized by calcisiltites, cream-colored calcilutites and fine grain laminated gray calcarenites (CPRM, 1985).

In this fossiliferous deposit, four taxa were identified: *Eremotherium*

laurillardii (Pilosa, Megatheriidae), Toxodontinae indet. (Notoungulata, Toxodontidae), *Notiomastodon platensis* (Proboscidea, Gomphoteriidae) and *Glyptodon* sp. (Cingulata, Glyptodontidae) (Faria and Carvalho, 2019). The Lajedão do Patrício fossil concentration is paucitaxic and multi dominant, with predominance of the species *Eremotherium laurillardii* and *Notiomastodon platensis* (Faria and Carvalho, 2019).

Five layers were identified in the sedimentary succession (Fig. 2). From the base to the top, they are: Layer 1, corresponds to a conglomerate with clasts and fossils displaying flow direction from NW/SE to WSW/ENE, with fossils supported by a 40 cm thick muddy matrix. The taphonomic attributes identified in layer 1 are disarticulated bones with a high degree of fragmentation, different patterns of staining, different stages of weathering and moderate abrasion. The skeletal remains were accumulated and transported by flash flood under low sediment rate conditions, which generated densely packed fossil concentrations, suggesting events of transport and reworking. The orientation of the fossils and the predominance of hard-to-transport

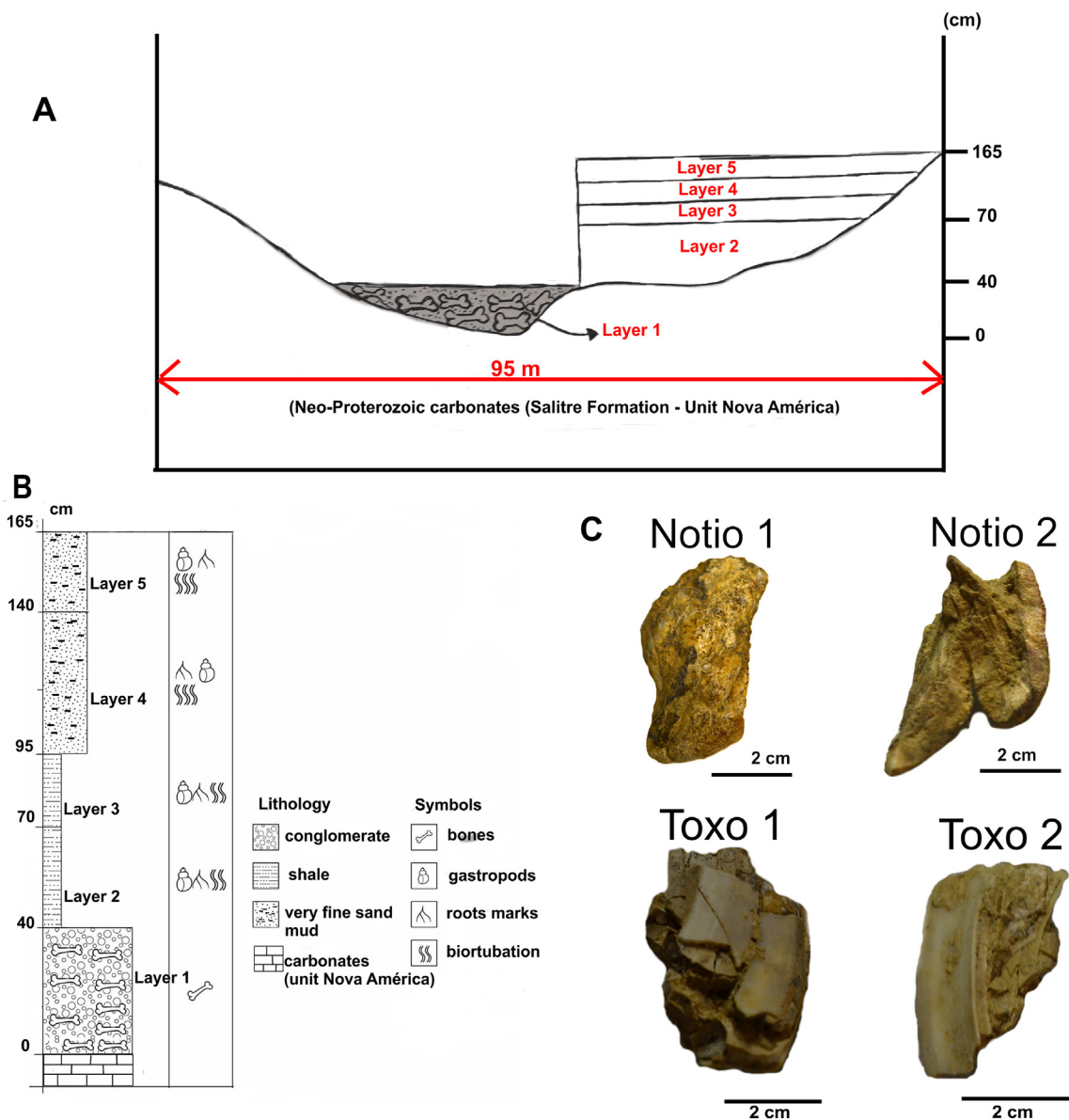


Fig. 2. Stratigraphic context of Lajedão do Patrício paleontological site; (A) Schematic cross-section profile of the fossil deposit of Lajedão do Patrício; (B) stratigraphy of the fossiliferous deposit of the Lajedão do Patrício; (C) analyzed samples of teeth.

bones indicate that the layer where the fossils were found was formed in a context of channel (Faria and Carvalho, 2019); Layer 2 (30 cm thick) is characterized by the deposition of pelitic sediments presenting evidence of bioturbation and invertebrate remains (*Biomphalaria*); Layer 3 (25 cm thick) is characterized by deposition of pelitic sediments with the presence of bioturbations and remains of invertebrates of the same species of the previous layer, but of smaller size; finally, Layers 4 and 5 consist of a mixture of very fine sand and extremely bioturbated mud; Layer 4 (45 cm thick) was differentiated from 5 (25 cm thick) due to the greater presence of plant remains in the organic horizon of the sediment. Layers 1 to 5 had Munsell colors 7.5Y8/2, 7.5Y5/1, 7.5Y4/1, 7.5Y7/3, and 7.5Y8/3, respectively. These colors – together with *Biomphalaria* shells – indicate that the sediment was deposited in waterlogged conditions, such as in a bog or marsh.

3. Material and methods

ESR dating was chosen due to the absence of the collagen in the samples, making impossible to date with other Quaternary methods of analysis, such as ^{14}C . Two teeth fragments attributed to *Notiomastodon*

platensis (Notio 1 and Notio 2) and two teeth fragments attributed to Toxodontinae indet. (Toxo 1 and Toxo 2), collected at different points of the Layer 1 (depth of 165 cm) were used for dating by ESR method. They were removed from the layer with the associated sediments, and sent to the Physics Department, University of São Paulo, Ribeirão Preto city, São Paulo State, Brazil.

The teeth were initially immersed in ultrapure water (Milli-Q, Millipore) to remove the sediment associated and, then, they were submitted to heat treatment by freezing in liquid nitrogen and thawing at room temperature to separate enamel from dentine (Kinoshita et al., 2008). A motorized drill was used to remove dentin under constant irrigation with water to prevent heating. When the enamel was reached, we removed a safety margin ($\sim 50\ \mu\text{m}$) from the area to ensure that only enamel is present.

Enamel samples were subjected to ultrasonic treatment with a diluted acid solution (HCl 1:10) for a few minutes to surface cleaning and to eliminate the influence of the alpha dose (Kinoshita et al., 2005, 2008). The enamel thickness was controlled during the process until the reduction is approximately $\sim 200\ \mu\text{m}$. With a digital micrometer, the thickness of the enamel at three points of the sample was measured.

Table 1
Enamel thickness before and after preparation.

Sample	Before (μm)	After (μm)
Notio 1	7239 \pm 332	6945 \pm 225
Notio 2	6846 \pm 442	6659 \pm 244
Toxo 1	1158 \pm 322	989 \pm 215
Toxo 2	723 \pm 256	627 \pm 264

Table 1 shows the thickness of the samples before and after the procedures.

After drying, the enamel was ground manually in an agate mortar until to reach the size of 38–76 μm , and divided into aliquots of 60 mg. Each aliquot was irradiated with an additive dose of γ rays from cobalt-60 source to determine the equivalent dose. The dose rate is 634 Gy/h and corrected monthly by the decay factor. This source belongs to a metrological laboratory that is frequently audited to assure compliance with standard values and the practices of the International Atomic Energy Commission. U, Th and K content in the sediment, enamel, and dentine of each tooth was determined by neutron activation analysis (NAA) at the Instituto de Pesquisas Energéticas e Nucleares – IPEN, São Paulo, Brazil.

The ESR spectra were recorded in a Jeol FA200X-Band spectrometer. The signal is due to the CO_2 radical that has axial symmetry. The intensity of the peak-to-peak signal at g perpendicular was used to construct the dose-response curve. The equivalent dose (D_e) was determined by fitting with the exponential function (1) (Ikeya, 1993).

$$I = I_0 \cdot (1 - e^{-\frac{D+D_e}{D_0}}) \quad (1)$$

I is the ESR signal intensity, D , the dose, I_0 and D_0 the intensity and dose at saturation. The conversion of D_e into age was made using the ROSY ESR Dating software (Brennan et al., 1999), using the concentrations of U, Th and K found in enamel, dentine and soil.

4. Results

Table 2 shows the concentration of uranium, thorium, and potassium in enamel, dentine, and soil, obtained by Neutron Activation Analysis (NAA). The ages obtained are shown in Table 3, according to the radioisotopic concentrations of enamel, dentine and sediment samples. The local cosmic radiation found was $168 \pm 50 \mu\text{Gy/a}$, calculated taking into account the geographic position (latitude, longitude, altitude) of the site (Prescott and Hutton, 1994). The values for U-234/U-238 were 1.20 ± 0.20 ; Alpha efficiency 0.13 ± 0.02 ; Density: enamel $2.95 \pm 0.02 \text{ g/cm}^3$, sediment $2.66 \pm 0.02 \text{ g/cm}^3$, and dentine $2.82 \pm 0.02 \text{ g/cm}^3$.

Table 2
Uranium, thorium and potassium concentrations in samples obtained by NAA.

Sample	U (ppm)	Th (ppm)	K (ppm)
Sediment Notio 1	1.8 \pm 0.2	10.1 \pm 0.6	5522 \pm 395
Sediment Notio 2	2.1 \pm 0.1	9.1 \pm 0.2	5909 \pm 614
Sediment Toxo 1	14.5 \pm 0.3	6.9 \pm 0.2	2713 \pm 282
Sediment Toxo 2	9.2 \pm 0.1	9.5 \pm 0.2	7322 \pm 760
Enamel Notio 1	0.083 \pm 0.010	< 0.01	< 750
Enamel Notio 2	0.476 \pm 0.010	< 0.01	< 750
Enamel Toxo 1	0.476 \pm 0.002	0.145 \pm 0.009	< 750
Enamel Toxo 2	0.92 \pm 0.05	< 0.01	< 750
Dentine Notio 1	40.7 \pm 0.8	0.20 \pm 0.01	< 750
Dentine Notio 2	72 \pm 1	0.43 \pm 0.03	< 750
Dentine Toxo 1	52 \pm 1	< 0.01	< 750
Dentine Toxo 2	46 \pm 1	0.187 \pm 0.04	< 750

Table 3
Equivalent Doses (Gy), internal and external dose rates ($\mu\text{Gy/yr}$) and Age (ka) according to the U-Uptake models.

	Notio 1	Notio 2	Toxo 1	Toxo 2
D_e Early Uptake				
D_e (Gy)	14.0 \pm 2.0	10.9 \pm 1.9	21.0 \pm 1.0	18.7 \pm 2.1
Internal	13 \pm 2	76 \pm 11	68 \pm 8	122 \pm 17
β Dentine	31 \pm 1	52 \pm 1	356 \pm 34	474 \pm 43
γ Sed	639 \pm 58	634 \pm 61	1583 \pm 59	1300 \pm 53
β Sed	6 \pm 3	6 \pm 3	216 \pm 60	285 \pm 29
Age (ka)	16.3 \pm 2.5	11.6 \pm 2.1	8.7 \pm 0.9	7.9 \pm 0.9
D_e Linear Uptake				
Internal	6 \pm 1	38 \pm 6	32 \pm 5	59 \pm 8
β Dentine	14 \pm 1	25 \pm 1	174 \pm 16	233 \pm 21
γ Sed	639 \pm 58	634 \pm 61	1583 \pm 59	1300 \pm 53
β Sed	6 \pm 3	6 \pm 3	216 \pm 60	285 \pm 29
Age (ka)	16.8 \pm 2.6	12.5 \pm 2.3	9.6 \pm 1	9.1 \pm 1

5. Discussion

We identified relatively low concentrations of Th and U in sediment samples associated with the analyzed teeth fragments. Blackwell et al. (2007) explain that relatively low concentrations of these elements indicate environmental conditions with saline or brackish groundwater. We attribute the high concentrations of U in the dentin samples (Table 2) to diagenetic factors such as the presence of fractures generated by the weight of the overlying sediments, because these mechanical changes facilitate the percolation of water and sediment inside the bones and teeth and, then, allowing alterations in the chemistry of the groundwater (e.g. high concentrations of salts). Also, the less compact nature of the dentine in relation to enamel, with dentine being more susceptible to diagenetic changes – allows more susceptibility to diagenetic alterations. We identified large amounts of authigenic calcite in the sediments of the fossiliferous deposit of Lajedão do Patrício, as well as the presence of gypsum and anhydrite in layers 3 and 4, respectively, through X-ray diffraction analysis. High concentrations of U have also been identified by Blackwell et al. (2007) due to the saline characteristics of groundwater of depositional environment. Therefore, the high concentrations of U in the analyzed dentin samples are linked to diagenetic factors of the depositional environment.

The different concentrations of U (Table 2) between dentine and enamel samples indicate that they were reworked from different sedimentary deposits. So, probably the teeth fragments initially belonged to four distinct deposits before being deposited in the current fossil layer (Blackwell, 1994). Araújo-Júnior et al. (2017) identified that the fossil concentration of tank deposits presents a complex taphonomic history, due to the presence of accumulated and reworked bones. Therefore, the different concentrations of U in the enamel and dentin samples of the teeth analyzed corroborates the hypothesis of reworking phases during the formation of Lajedão do Patrício fossil deposit.

The different U concentrations between dentin and enamel (Table 2) indicate that the analyzed teeth fragments underwent different U uptake conditions due to reworking. (Blackwell et al., 2000). Thus, during the secondary U uptake event, dentin absorbed more U than enamel, causing different ages between the models in Table 3. According to Blackwell et al. (2007), the high concentrations of U in the analyzed dentine samples, the presence of carbonate sediments and groundwater salinity, support the idea that the teeth fragments followed the LU uptake model. Therefore, the most appropriate ages for the analyzed tooth are those calculated by the LU model (Table 3).

The different ages obtained as well as the different concentrations of U (Table 3) indicate time-averaging in the fossiliferous deposit of Lajedão do Patrício. According to Kowalewski (1996), time-averaging is defined as “the processes by which events that occurred at different times appear to be synchronous in the geological record”. Martin (1999) explains that time-averaging may be caused by low

sedimentation rate, bioturbation or reworking. Thus, the process of accumulation of the skeletal remains of Lajedão do Patrício did not occur at the same time due to taphonomic and sedimentary processes. Araújo-Júnior et al. (2015, 2017) explain that fossiliferous tank deposits are formed in a low supply of clastic sediments, where the transport agent is responsible for accumulation, re-sedimentation and reworking of the skeletal remains. The different ages obtained for Lajedão do Patrício can be associated to the same agents related by Araújo-Júnior et al. (2015, 2017).

The maximum time span of time-averaging is 11.3 ka, indicating that the degree of time-averaging of Lajedão do Patrício is in the order of 10^4 years. Behrensmeier (1982) identified that fossil deposits in alluvial and fluvial environments have a time-averaging in the order of 10^3 – 10^4 years. So, it is likely that the time-averaging inferred for Lajedão do Patrício, from reworking of preexisting taphocoenoses in alluvial and fluvial contexts. The skeletal remains of Patrício do Lajedão are found in a conglomerate with sub-rounded clasts and preferentially oriented fossils, due to the action of tractive flows in the accumulation process. According to Gerra and Guerra (2008), alluvial and fluvial deposits include many reworked particles from other deposits. Probably, it was the origin of reworking inferred for Lajedão do Patrício and for the different concentrations of U in the analyzed tooth fragments (Table 2).

Some considerations can be made by comparison with other geochronological data for fossil assemblages of northeastern Brazil. In Fig. 3 these fossil deposits represent a combined time interval of 70 ka to 9 ka, comprising the Late Pleistocene - Early Holocene interval. In Fig. 3, we can observe that time-averaging is a common process in tank deposits, with fossil concentrations presenting different degrees of time-averaging, specially Lagoa do Rumo paleontological site (also in Bahia State).

Among the taxa dated in northeastern Brazil, *Notiomastodon platensis* has the highest number of available ages. The time range covered by this taxon (Fig. 3) indicates its presence during the Late Pleistocene, from 63 ± 8 ka at Fazenda Logradouro (Pernambuco State) to about 10 ± 0.5 ka at Fazenda Ovo da Ema (Alagoas). *Toxodon* has the second largest number of ages available for the northeast region, with a time range extending from 39.8 ± 1 ka and 9 ± 2 ka at Lagoa do Rumo (Bahia), indicating the presence of this taxon during the Late Pleistocene-Early Holocene time interval (Fig. 3). *Eremotherium laurillardii* is the taxon most identified in fossil deposits in northeastern Brazil, but it has few available ages, being restricted to the Late Pleistocene (Fig. 3). This can be attributed to the difficulty of dating its skeletal remains, due to the absence of enamel in its teeth, being possible to date them only when collagen is preserved in the bones.

In the literature, we identified Holocene ages for megafauna in northeastern Brazil, obtained indirectly from fossil-associated sediments. At the Toca do Serrote do Artur, southwest of Piauí State, sediment-associated with *Palaeolama major* and *Glyptodon* were dated at 8.49 ± 0.12 ka BP, using method ^{14}C (Faure et al., 1999). In Toca do Garrincho - another cave from the Serra da Capivara Karst - sediments associated with *Hippidion*, *Palaeolama*, *Pampatherium*, *Toxodon*, and *Catonyx* fossils were ^{14}C dated at $10,020 \pm 290$ BP (Peyre et al., 1998). In the fossiliferous deposit of Lagoa do Rumo, Bahia State, the sediment at the top of the fossil layer dated at 8600 ± 30 ka BP, using method ^{14}C (Ribeiro et al., 2013). Holocene ages were identified by the ESR method for Toxodontinae fossils from Abismo Ponta da Flecha, a Karst deposit in the Ribeira Valley, Sao Paulo State, Brazil. The results obtained at 6700 ± 1300 and 5000 ± 1600 for *Toxodon platensis* teeth are the youngest ages of the extinct Quaternary megafauna (Baffa et al., 2000). However, they were dated by the ^{14}C AMS method with ages of 13 ka BP and minimum Late Pleistocene age for these specimens (Neves et al., 2007).

In South America, we observe other fossil records of Quaternary megafauna at the Pleistocene-Holocene boundary dated by the C^{14} method. In the Ayacucho complex, Peru, a Megatheriidae indet. was

dated at $12,200 \pm 180$ BP (MacNeish et al., 1970). In Taima Taima, Venezuela, a *Glyptotherium* cf. *G. cylindricum* was dated at $12,580 \pm 60$ BP (Carlini and Zurita, 2006). In Monte Verde, Chile, a *Curvieronius humboldti* was dated at $11,900 \pm 200$ BP (Borrero, 1997). In southern Argentina, Cueva Lago Sofia, remains of a *Smilodon* were dated at $11,210 \pm 50$ BP (Borrero, 1997), and another *Smilodon* in the Milodon Cave, Chile, was dated at $11,240 \pm 50$ BP (Barnett et al., 2005). Therefore, the ages obtained for Lajedão do Patrício are very similar to other records from Brazil and from neighboring countries in South America, indicating that these deposits are chronocorrelated.

When comparing the formation period of the Lajedão do Patrício fossiliferous deposit (16.8 ± 2.6 to 9.1 ± 1 ka), we observe that it is inserted in a period marked by intense climate changes in northeastern Brazil. Cruz et al. (2009) reported, based on $\delta^{18}\text{O}$ values of speleothems collected in Rio Grande do Norte State, a predominantly dry climate between 26 and 15.1 ka, interrupted by short wet periods of 25.9 to 25 ka and 17.3 to 15.1 ka. Between 15.1 ka and 13.2 ka no speleothem deposition was identified, being attributed to an extremely dry climatic period. Cruz et al. (2009) identified that between 13.2 ka and 10.5 ka there was a small increase in humidity, being a period marked by abrupt variation between dry and wet periods, where, between 10.5 ka and 5 ka, dry weather conditions are again established. Therefore, it is likely that different climatic and environmental conditions are condensed during the process of accumulation and formation of the fossiliferous deposit of Lajedão do Patrício.

Only a small part of the 150 fossil records of megafauna fossils spread across the northeastern region has been the object of geochronological studies. More studies focusing on mammal fossils and their deposits are required. Direct dating with different methods (when possible) will enable a better understanding of the space and time distribution of these animals, and will also contribute to investigations related to environmental changes that occurred in the Pleistocene-Holocene transition, which usually are taken as the main reason responsible for the disappearance of that fauna.

6. Conclusion

The different concentrations of U in the dentin and enamel samples in the analyzed teeth indicated that the teeth were initially from different deposits before being reworked and deposited in the fossiliferous concentration of Lajedão do Patrício. We interpret that time-averaging is caused by reworking, explaining the different U concentrations and ages obtained. The obtained ages (Notio-1, 16.8 ± 2.6 ka; Notio-2, 12.5 ± 2.3 ka; Toxo-1, 9.6 ± 1 ka; Toxo-2, 9.1 ± 1 ka) are similar to those obtained for Quaternary megafauna from northeastern Brazil, and also from neighboring countries. The fossil assemblage of Lajedão do Patrício is a deposit of the Late Pleistocene-Early Holocene time interval, representing a large time span of formation. The identified maximum time-averaging (11.3ka) overlaps with others identified for northeastern Brazil, as well as a period of climate change and megafauna extinction.

Declaration of competing interest

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

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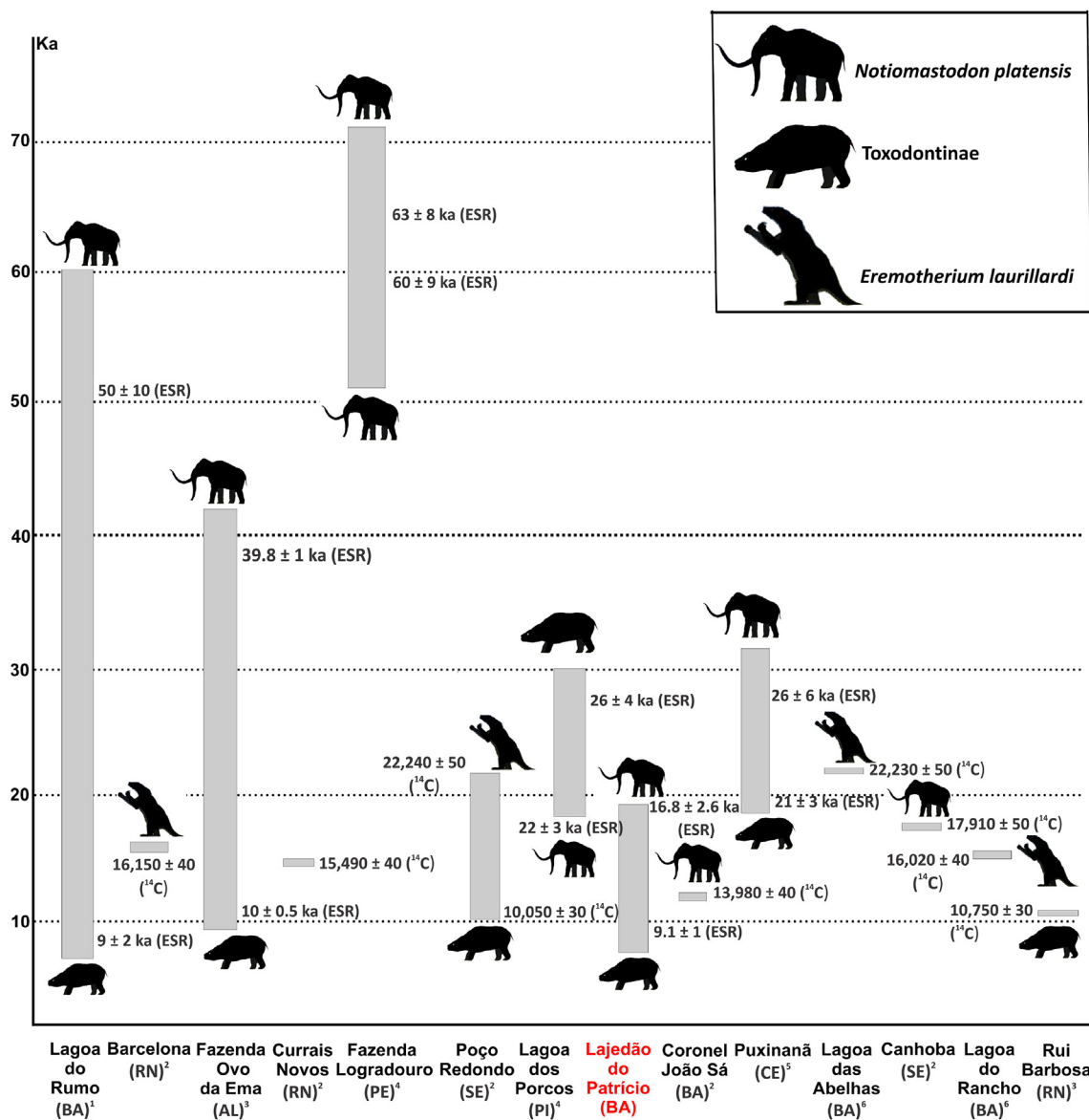


Fig. 3. Time-averaging and chronological dispersion of northeast Brazil megafauna dated by ESR and ¹⁴C. The symbols represent the taxa dated in their relative position. ⁽¹⁾Ribeiro et al. (2013); ⁽²⁾Dantas et al. (2013); ⁽³⁾Oliveira et al. (2010); ⁽⁴⁾Kinoshita et al. (2014); ⁽⁵⁾Kinoshita et al. (2008); ⁽⁶⁾Scherer et al. (2017).

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