Quaternary International 305 (2013) 91–96

Contents lists available at SciVerse ScienceDirect

Quaternary International

journal homepage: www.elsevier.com/locate/quaint

Electron Spin Resonance dating of the Late Quaternary megafauna fossils from Baixa Grande, Bahia, Brazil

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ARTICLE INFO

Article history:
Available online 20 July 2012

ABSTRACT

Electron Spin Resonance (ESR) spectroscopy was applied to date megafauna fossil teeth of *Stegomastodon waringi* and Toxodontinae (two teeth) found in Baixa Grande, Bahia, Brazil. The CO_2^- signal with spectroscopic features $g_\perp = 2.0018$ and $g_{//} = 1.9973$ used for ESR dating was detected in all fossil enamel. The additive method was employed to construct the dose response curve and to calculate the Equivalent Dose (D_e). Neutron Activation Analysis of enamel, dentine and soil where the samples were buried was used to determine the main radioisotopes concentration. These data were used in the conversion of D_e into age, using the ROSY ESR dating software. The results of age obtained were 50 ± 10 ka for *S. waringi*, and 43 ± 8 ka and 9 ± 2 ka for Toxodontinae teeth. Although Late Quaternary fossils from the extinct South American megafauna are relatively common in Brazilian's Northeast region, few geochronological studies were conducted. Thus dating samples found in this region will allow a better time and space understanding of that fauna.

1. Introduction

Distributed in all the Brazilian Northeastern region, the mammal megafauna fossil record demonstrates that this was the continental fauna characteristic of this region during Late Quaternary. The main depositional environment where these fossils were found is called *tanques* or *cacimbas*. Those are depressions in the crystalline rocks of Borborema Province and the São Francisco Craton (Paula-Couto, 1979; Bergqvist, 1993; Santos, 2001; Bergqvist and Almeida, 2004; Viana et al., 2005; Silva et al., 2006 and; Silva, 2008). Fossils of these animals can also be found in other deposits, as the karst regions of Bahia (Cartelle and Bohórquez, 1985; Cartelle, 1992, 1998), Piauí (Guérin, 1991; Peyre et al., 1998; Faure et al., 1999), Ceará (Trajano and Ferrarenzzi, 1994), and Rio Grande do Norte (Porpino et al., 2004), paleolacustrine deposits, such as those found in the Serra da Capivara National Park, Piauí (Guérin et al., 1996), and the travertine formations from Jacobina, Bahia (Rolim, 1974).

Despite being well represented and widely known, geochronological research concerning the Quaternary megafauna deposits from Northeast Brazil is rare. In this region, Electron Spin Resonance (ESR) ages were obtained in fossils from Paraíba (Kinoshita et al., 2005), Pernambuco (Kinoshita et al., 2008) and Alagoas states (Oliveira et al., 2010). The other dates on fossils or sediments associated are available in materials from karst deposits: a coprolite from the ground sloth *Nothrotherium* from Gruta dos Brejões, Chapada Diamantina, Bahia (Czaplewski and Cartelle, 1998), and in sediments associated with fossils from 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, Piauí.

The aims of this study are: 1) to perform the first numerical dating of fossils from the fossiliferous deposit of Lagoa do Rumo, Baixa Grande, Bahia, by the ESR method; and 2) to discuss the geochronology concerning the Late Quaternary megafauna from the fossiliferous deposits of Brazilian Northeast.

1.1. Geological setting

The fossiliferous *tanque* from Lagoa do Rumo (11°32′07″S; 40°07′11″W) is the first record of the Quaternary megafauna in Baixa Grande County, Bahia State (Fig. 1A). At this fossiliferous

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http://dx.doi.org/10.1016/j.quaint.2012.07.017

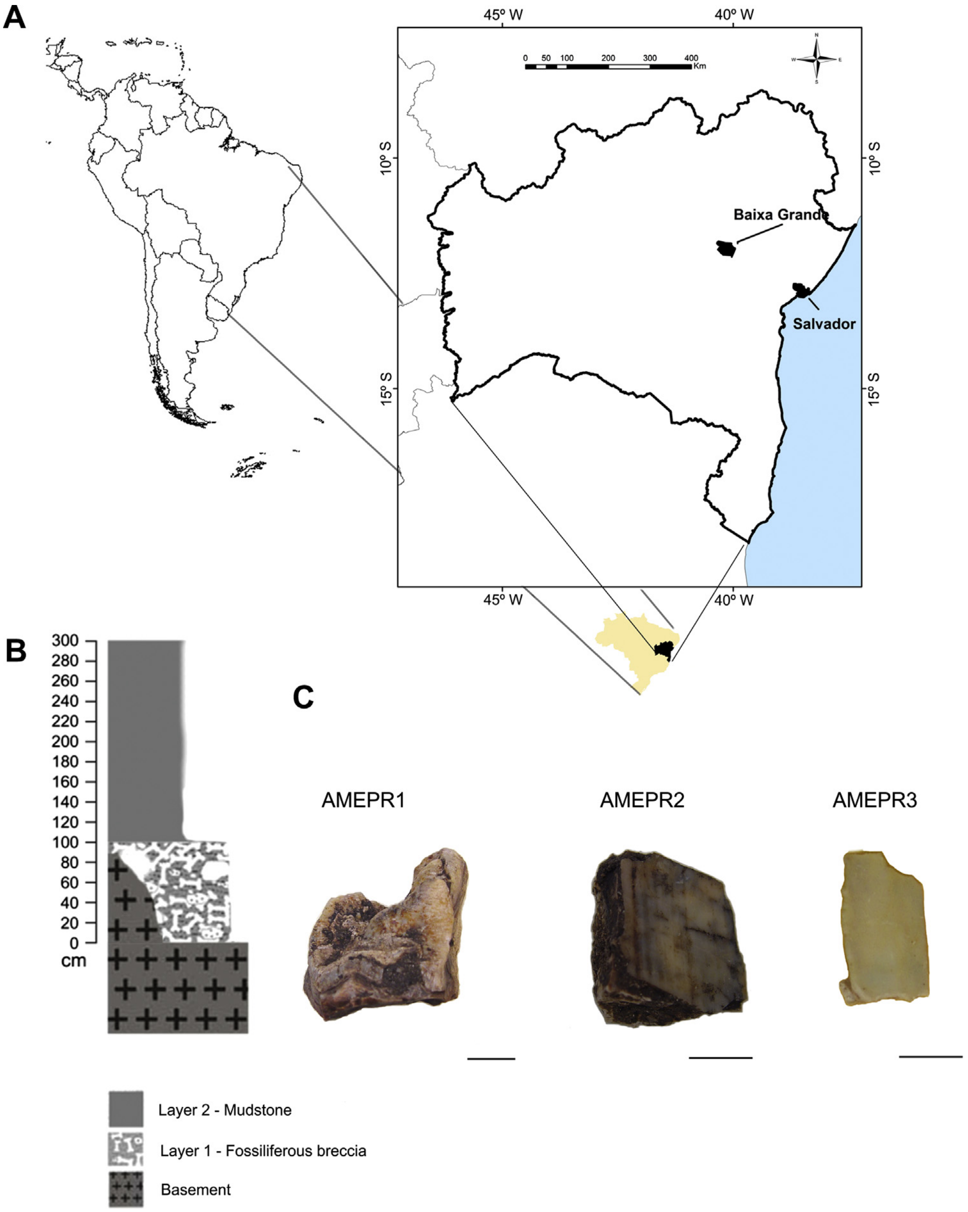

Fig. 1. (A) Geographic location of Baixa Grande County, Bahia State, Brazil; (B) Stratigraphy of the fossiliferous deposits of Lagoa do Rumo, Baixa Grande County, Bahia State; (C) Photography of teeth *S. waringi* (AMEPR1), Toxodontinae (AMEPR2), Toxodontinae (AMEPR3). In AMEPR1 is possible to note the soil associated to the sample (arrow) (scale bar = 1 cm).

deposit, four taxa were identified: *Eremotherium laurillardi* (Pilosa – Megatheriidae), *Panochthus greslebini* (Cingulata – Glyptodontidae), Toxodontinae (Notoungulata – Toxodontidae) and *Stegomastodon waringi* (Proboscidea – Gomphoteriidae) (Ribeiro and Carvalho, 2009).

The sedimentary succession is composed by two layers (Fig. 1B). The fossils are found in the basal one (Layer 1), a thickly bedded fossiliferous breccia, supported by clast and bioclast, approximately 1 m thick. Upon this layer, pelitic sediments were deposited, characterized as an organic-rich mudstone, without any occurrence of macrofossils (Layer 2). Both layers have tabular geometry. The internal structure of Layer 1 shows that the bioclasts are homogeneously distributed in relation to their biostratinomic attributes (disarticulation, transportation and final burial), and to the fossiliferous assemblage in their sedimentological (depositional events of debris flow, producing deposits of coarse sediments, densely packed and poorly selected) and paleoecological (monotypic and polytypic) attributes. The assemblage is densely packed, poorly sorted, with a wide grain-size of the clasts and bioclasts, in a polymodal distribution, without a preferential orientation. This was probably related to the hydrological dynamics of a debris flow during transportation from the source area to the deposit (Ribeiro, 2010).

2. Material and methods

An attempt to date bones by the ^{14}C (AMS) method (analysis in the Center of Applied Isotope Studies, Georgia University, USA), failed to obtain good results due the natural degradation of collagen in the samples. The same dating method was successfully applied to a sample of sediment from the top of the fossiliferous layer (Layer 1) (UGAMS 5030), where the age is 8600 ± 30 BP (Ribeiro, 2010).

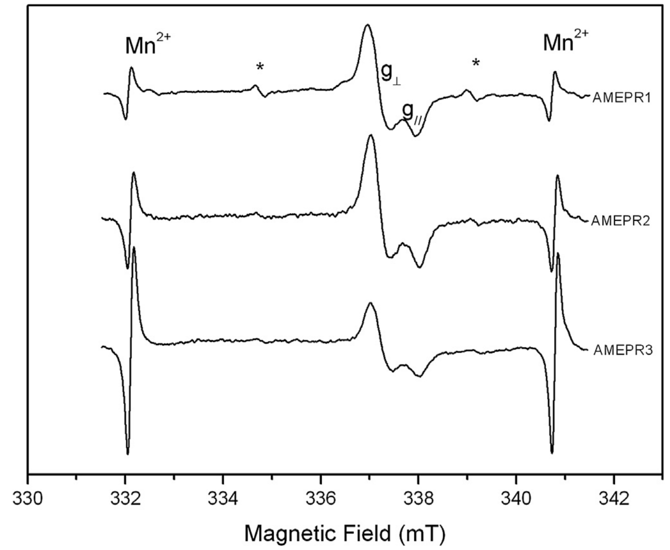

Fig. 2. ESR spectrum of enamel of teeth. The signal of radical CO_2^- is present in all spectra between the 3rd and 4th Mn lines. The radical isopropyl (*) is also present.

The dating of the remains of these animals by the ESR method is a valuable tool for geochronological studies, due to its applicability to biomineral materials such as the enamel and dentin from teeth, enabling dating when other Quaternary methods of geochronological research, such as ^{14}C, cannot be performed. A tooth of *Stegomastodon* (sample AMEPR1), and two teeth of Toxodontinae (samples AMEPR2 e AMEPR3), from the fossiliferous layer (Layer 1) of the Lagoa do Rumo were selected for ESR dating (Fig. 1C).

A fraction of enamel and dentin of the tooth AMEPR1 was removed using a Dremel diamond disk operating with low rotation and constant water irrigation. This fraction and the other teeth (AMEPR2 and AMEPR3) were submitted to thermal treatment by freezing in liquid nitrogen and defrosting at room temperature. After a few repetitions, the enamel detached from dentine. The enamel was subjected to acid treatment (HCl) 1:5 in an ultrasonic bath for extraction of outer layer of both sides of approximately 250 μm. After drying, the enamel was ground manually in an agate mortar until the particles reached a diameter $\varphi < 0.5$ mm. Ten aliquots of about 70 mg were irradiated with different doses, ranging from 0 to 1.2 kGy in a Cobalt-60 irradiator with dose rate of 2.49 Gy/minute at IPEN. The spectra were recorded in a Jeol FA200 X-Band spectrometer.

The intensity of peak-to-peak signal dosimetric $g_\perp$ was used to construct the dose–response curve. The equivalent dose (D_e) was determined by fitting with exponential function (1) (Ikeya, 1993).

$$I = I_0\left\{1 - e^{-\left[\frac{(D+D_e)}{D_0}\right]}\right\} \qquad (1)$$

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 obtained by Neutron Activation Analysis (NAA).

3. Results

Fig. 2 shows the ESR spectrum of original enamel samples, without any additional irradiation. A manganese marker was employed to precisely determinate the g-factors of the signal, and the radical CO_2^- is observed, with $g_\perp = 2.0018$ and $g_{//} = 1.9973$. The isopropyl radical is also present in two samples. The signal intensity at $g_\perp$ was employed to construct the dose response curves (Fig. 3). Table 1 relates the concentration of U, Th and K in enamel, dentine and soil, obtained by NAA. Table 2 shows the results of Equivalent Doses (D_e), the internal and external dose rates (D_{int} and D_{ext}) and

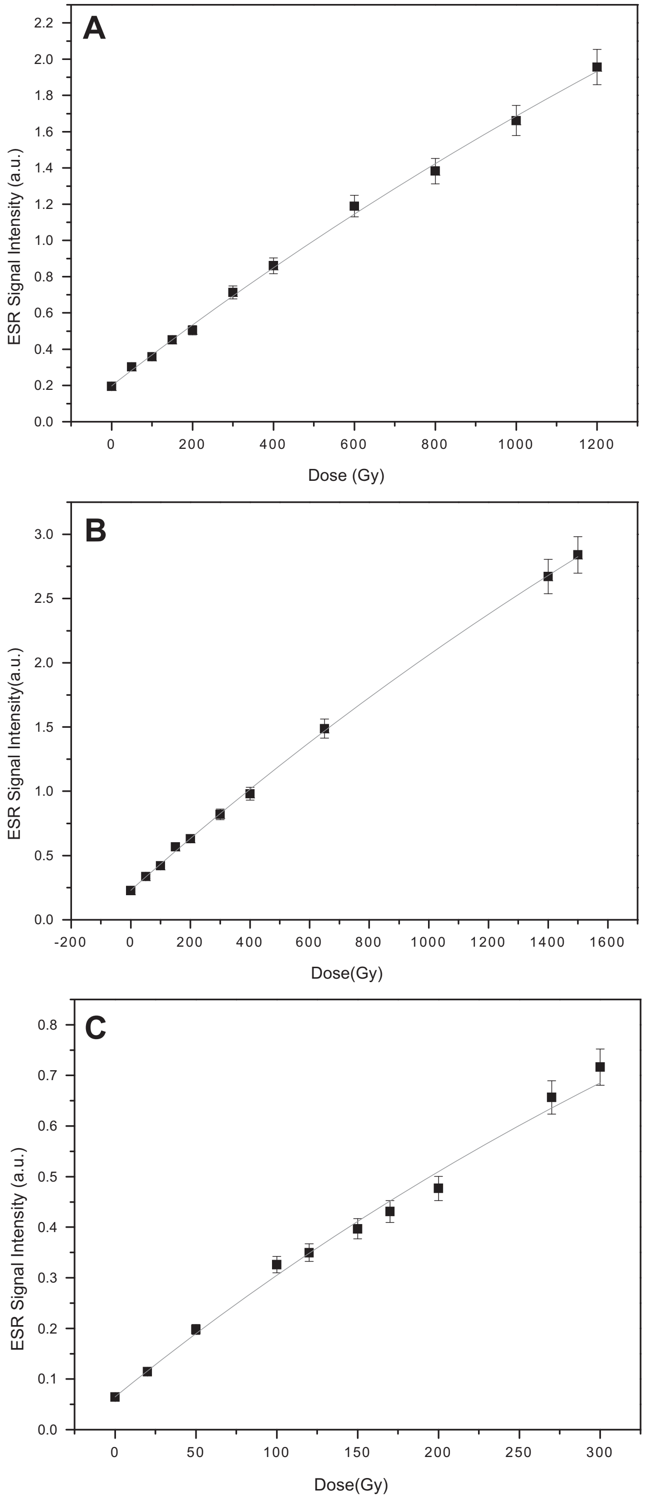

Fig. 3. Dose response curve. The experimental data points were fitted by Equation (1). (A) *S. waringi* (AMEPR1) (B) Toxodontinae (AMEPR2) (C) Toxodontinae (AMEPR3).

Table 1
Concentration of Uranium, Thorium and Potassium of samples obtained by NAA.

Sample	U (ppm)	Th (ppm)	K (ppm)
Enamel			
AMEPR1	0.49 ± 0.01	<0.01	<750
AMEPR2	<0.05	<0.01	<750
AMEPR3	0.59 ± 0.01	<0.01	<750
Dentine			
AMEPR1	36.0 ± 0.9	1.65 ± 0.06	<750
AMEPR2	15.1 ± 0.4	1.14 ± 0.04	<750
AMEPR3	19.7 ± 0.5	<0.01	<750
Soil			
Sample 1	3.25 ± 0.08	20.7 ± 0.8	11,517 ± 2239
Sample 2	3.44 ± 0.09	39 ± 1	<750
Sample 3	3.63 ± 0.09	26 ± 1	15,204 ± 2280
Average	3.4 ± 0.2	29 ± 9	13,000 ± 3000

the ages according to the uranium uptake model: Early Uptake (EU), Linear Uptake (LU) and Combination Uptake (CU). The water content in soil was 11%. The value of 190 µGy/year was adopted for cosmic ray dose rate, taking into account the latitude, longitude, altitude (386 m) and the depth where the samples were collected (2 m) (Prescott and Hutton, 1994).

4. Discussion

The model Combination uptake (CU) was defined as Early Uptake (EU) to dentin and Linear Uptake (LU) to enamel, due to differences in porosity. Usually the age given by this model is adopted as the most probable. However, considering the experimental uncertainties of the results, the ages given by the three models are the same for the three samples studied. In addition, the ages calculated using the DATA software (Grün, 2009) produced the same ages, although, according to the authors, it uses the most updated data on beta radiation interaction obtained from Marsh et al. (2002).

The results obtained for the samples from Lagoa do Rumo (Table 2) show, besides a wide time-average in the fossil assemblage from 30,000 to 50,000 years, the youngest age obtained directly from a Late Quaternary megafaunal fossil in Northeastern Brazil, AMEPR3, a tooth of Toxodontinae, showing that part of this fauna might have survived until the early Holocene in the Baixa Grande area.

The time-averaging in the Lagoa do Rumo deposit is more significant than that observed in the other northeastern deposits (Table 3). Time-averaging is a taphonomic term that deals with the temporal mixing in fossil assemblages. Mixing may be caused by sedimentation rates, bioturbations or physical reworking (Martin, 1999). At Puxinanã, Paraíba State, this time-averaging was approximately 5000 years (Kinoshita et al., 2005), while at Brejo da Madre de Deus, Pernambuco State, it was approximately 3000 years (Kinoshita et al., 2008). The distinct degrees of time-averaging, except for a deposit from Alagoas State, with a time-averaging of approximately 30,000 years (Oliveira et al., 2010), with the Holocene result obtained from the analysis of the sample AMEPR3 from Lagoa do Rumo, suggest a different depositional dynamics between the tanque deposits located at Borborema Province and ones in the São Francisco Craton. The results obtained by ESR in these northeastern states show the presence of these extinct mammals in the Borborema Province in the Late Pleistocene, between 63,000 ± 8000 and 10,000 ± 500 years.

Table 2
Equivalent Doses D_e(Gy), Internal and External dose rates (D_{int}(µGy/a) and D_{ext}(µGy/a)) and Ages of samples according to the Uranium uptake model.

	AMEPR1	AMEPR2	AMEPR3
Equivalent Dose (Gy)	113 ± 8	110 ± 5	25 ± 2
Early Uptake (EU)			
D_{int} (µGy/a)	134.2	0	90.1
D_{ext} (µGy/a)	1906.5	2538.2	2483.9
Age(ka)	50 ± 10	43 ± 8	9 ± 2
Linear Uptake (LU)			
D_{int} (µGy/a)	58.6	0	43
D_{ext} (µGy/a)	1890.0	2450.5	2401.1
Age(ka)	50 ± 10	45 ± 9	9 ± 2
Combination Uptake (CU)			
D_{int} (µGy/a)	58.4	0	42.9
D_{ext} (µGy/a)	1906.6	2538.2	2484.1
Age(ka)	50 ± 10	43 ± 8	9 ± 2

Holocene ages were obtained indirectly in Northeastern Brazil in sediments associated to fossils from that extinct fauna. In the National Park Serra da Capivara, Southeastern Piauí, sediments associated to *Palaeolama*, *Equus* and *Glytptodon*, from a karst deposit (Toca do Serrote do Artur), were ^{14}C dated at 8490 ± 120 BP (Faure et al., 1999). In another cave from the Serra da Capivara karst at Toca do Garrincho, sediments associated with *Hippidium*, *Palaeolama*, *Pampatherium*, *Toxodon* and *Catonyx* fossils were ^{14}C dated at 10,020 ± 290 BP (Peyre et al., 1998). In addition to this, an early Holocene age was obtained from sediments from the top of the fossiliferous layer of the Lagoa do Rumo deposit, ^{14}C (AMS) dated 8600 ± 30 BP (UGAMS 5030).

Holocene ages for Toxodontinae fossils from Abismo Ponta da Flecha, a karst deposit from Ribeira Valley, São Paulo, Southeastern Brazil, were also obtained by ESR. The results of 6700 ± 1300 and 5000 ± 1600 a for *Toxodon platensis* teeth are the latest for a fossil from the extinct Quaternary megafauna (Baffa et al., 2000). However, a previous ^{14}C AMS date was 13ka BP, suggesting a minimum Late Pleistocene age for these specimens (Neves et al., 2007). A karst deposit from Lagoa Santa, Minas Gerais, Southeastern Brazil, was dated twice with results younger than 10,000 BP: bones from a *Scelidodon* (*Catonyx*) *cuvieri* and *Smilodon populator* were dated at 9990 ± 40 BP and 9260 ± 150 BP (^{14}C – AMS), respectively (Piló and Neves, 2003).

Other megafauna fossils from South America have produced dates in the boundary of Pleistocene-Holocene. In Ayacucho complex, Peru, a Megatheriidae indet. dated at 12,200 ± 180 BP (MacNeish et al., 1970). At Taima Taima, Venezuela, a *Glyptotherium cf. cylindricum* dated 12,580 ± 150 BP (Carlini and Zurita, 2006). A *Glossotherium aff. G. lettsomi* from Santa Elina Rockshelter, Cuiabá, Brazil, dated 10,120 ± 60 BP (Vialou, 2003). In Monte Verde, Chile, a *Cuvieronius humboldti* was dated 11,900 ± 200 BP (Borrero, 1997). In Southern Argentina, Cueva Lago Sofia, a *Smilodon* dated 11,210 ± 50 BP (Borrero, 1997), and another *Smilodon*, from Mylodon Cave, Chile, was dated 11,420 ± 50 BP (Barnett et al., 2005). In Rio Negro, Uruguay, an age of 11,600 ± 130 BP was obtained for an *Arctotherium tarijense* (Ubilla and Perea, 1999; Soibelzon et al., 2005). For a comprehensive list of additional records of taxa and ages in the uppermost Pleistocene beds (dated between 20,000 and 10,000 BP) of several South American sites, see Cione et al. (2008).

The results obtained from the fossils from Lagoa do Rumo show a marked degree of time-averaging, and the latest age for a fossil from this extinct fauna from Northeastern Brazil (AMEPR3). The results obtained from samples AMEPR1 and AMEPR2 do not provide the first known appearance date for the Lagoa do Rumo deposit. The provenance of the older teeth is not clear. Probably, these teeth have been reworked from neighboring deposits. AMEPR3 is probably from Lagoa do Rumo, due to the proximity to the age obtained in the ^{14}C analyses performed on sediments from Layer 1.

Although well documented, the Late Quaternary megafauna from that region are rarely directly dated. A small part of the more

Table 3
Geochronological studies performed in fossiliferous deposits from the Late Quaternary in Northeastern Brazil.

Site/ State	Deposit	Method	Age	Taxon
Puxinanã, Paraíba[a]	*Tanque*	ESR – Tooth	30,000 ± 5000[a] – sample 1, recorded in X-band; 36,000 ± 7000[a] – sample 1, recorded in K-band; 39,000 ± 7000[a] – sample 2; 39,000 ± 9000[a] – sample 3	*Stegomastodon waringi* (sample 1 and 2), *Xenorhinotherium bahiense* (sample 3)
Brejo de Madre de Deus, Pernambuco[b]	*Tanque*	ESR – Tooth	63,000 ± 8000[a] – sample 1; 60,000 ± 9000[a] – sample 2	*Stegomastodon waringi*
Gruta dos Brejões, Bahia[c]	Cave	^{14}C – Coprolite	12,200 ± 120 BP	*Nothrotherium*
Toca do Garrincho[d], Piauí	Cave	^{14}C – Charcoal	10,020 ± 290 BP	*Hippidion*, *Palaeolama*, *Pampatherium*, *Toxodon* and *Catonyx*
Toca do Serrote do Artur[e], Piauí	Cave	^{14}C – Charcoal	8490 ± 120 BP	*Hoplophorus*, *Glyptodon*, *Conepatus*[h], *Panthera*[h], *Dicotyles*[h], *Tayassu*[h], *Palaeolama* and *Mazama*[h]
Fazenda Ovo da Ema[f]	*Tanque*	ESR – Tooth	10,000 ± 500[a] – AL 1 (EU age); 10,000 ± 500[a] – AL 1 (LU age); 39,800 ± 1000[a] – AL 2 (EU age); 39,800 ± 1000[a] – AL 2 (LU age)	*Stegomastodon waringi*
Lagoa do Rumo, Baixa Grande[g]	*Tanque*	^{14}C – Sediments	8600 ± 30 BP	*Eremotherium laurillardi*, *Panochthus greslebini*, *Stegomastodon waringi*, Toxodontinae

[a] Kinoshita et al., 2005.
[b] Kinoshita et al., 2008.
[c] Czaplewski and Cartelle, 1998.
[d] Peyre et al., 1998.
[e] Faure et al., 1999.
[f] Oliveira et al., 2010.
[g] Ribeiro, 2010.
[h] Present fauna.

than 150 records of megafauna fossils spread across the northeastern region was 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 comprehension 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, that usually are taken as the main reason responsible for the disappearance of that fauna.

Acknowledgments

To Mrs. João França, who discovered the fossils, and Rômulo Fontoura, for all assistance in the field trips. Partial financial support from the Brazilian agencies Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Fundação de Amparo à Pesquisa do Estado do Rio de Janeiro (FAPERJ) and Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP) are appreciated.

Appendix A. Supplementary material

Supplementary material associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.quaint.2012.07.017.

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