



## Electron spin resonance dating of teeth from Western Brazilian megafauna – preliminary results

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### ARTICLE INFO

#### Article history:

Received 22 November 2010

Received in revised form

8 April 2011

Accepted 2 May 2011

#### Keywords:

Dating

Electron Spin Resonance,

ESR

Tooth

### ABSTRACT

Electron Spin Resonance (ESR) was applied to determine ages of *Haplomastodon* teeth from Western Brazilian Megafauna. The Equivalent Doses ( $D_e$ ) of  $(1.3 \pm 0.2)$ kGy,  $(800 \pm 100)$ Gy and  $(140 \pm 20)$ Gy were found and the software ROSY ESR dating was employed to convert  $D_e$  in age, using isotope concentrations determined by neutron activation analysis (NAA) and other information, resulting in  $(500 \pm 100)$ ka,  $(320 \pm 50)$  and  $(90 \pm 10)$ ka considering the Combination Uptake (CU) model for Uranium uptake, set as an Early Uptake (EU) for dentine and Linear Uptake (LU) for enamel. There are scarce reports about Pleistocene Megafauna in this area. This paper presents the first dating of megafauna tooth and this study could contribute to improve the knowledge about the paleoclimate and paleoenvironment of this region and prompt more investigations in this area.

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## 1. Introduction

Pleistocene fossils are common in many Brazilian states and usually found in caves, at the bottom and margin of rivers that form natural depressions in crystalline rocks. As time goes by these depressions are filled with megafauna skeletons. These deposits are also known as wells or “cacimbas” and are a good clue to look for fossils. These deposits are frequent in Mato Grosso (MT), but only a few studies have been published about the Pleistocene Megafauna of this region. Only Hirooka (1991) and Cartelle and Hirooka (2005) reported the existence of a giant sloth, armadillo and giant otter, among other animals in the Curupira Cave, MT, Brazil. Among vertebrate fossils the most preserved parts of the skeleton are the teeth due to the presence of a high resistant enamel layer that precludes its destruction by natural action. The samples used in this work were in good state of preservation with the molars with different wearing degrees.

This work reports on the occurrence of mastodon teeth found at 35 km north of Alta Floresta at a gold mining area. These materials were donated to the Universidade Federal de Mato Grosso museum

by the mining owner. This paper reports the first attempt to date samples of megafauna from this region and the results can contribute to the understanding of the climate changes occurred in this region.

ESR has been successfully used in several cases with tooth enamel for dating (Baffa et al., 2000; Rink et al., 2004; Kinoshita et al., 2005, 2008a, 2008b; Chase et al., 2007; Skinner et al., 2007; Lopes et al., 2010; Kerber et al., 2010) and also for radiation dosimetry (Kinoshita et al., 2001, 2003; Swartz et al., 2007). Thus having precise information about the radioactive content of the soil surrounding the sample and in the sample it is possible to date enamel with good precision. A noteworthy characteristic of ESR dating is that it is possible to cover a period from thousands to one million years old, thus extending the possibility of precise dating beyond that offered by radiocarbon and other radioisotopes.

## 2. Materials and methods

### 2.1. Sample

Fossils coded as 354, MUS99 and MAR belonging to the Museum Collection of Federal University of Mato Grosso, Brazil were used in this work after permission granted by the museum authorities obtained by co-authors (D.S. and J.S.P) from this institution. The samples belong to the Mamilia Class, Proboscidea Illinger 1811

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order, *Haplomastodont waringi* from the Pleistocene era and they were found 35 km north of the city of Alta Floresta. All samples were collected at same site and sample MAR had sediment attached to it. This sediment was used as a first approximation to determine the contribution of the sediment to the total dose rate received by the teeth. Although not ideal this was the only possibility in the given circumstances.

## 2.2. Geological context

Leite et al. (2001) performed a detailed geological study of the Alta Floresta region producing an elaborated geological map with the geological context. According to these authors the Teles Pires province, the west portion of the Amazonian Craton presents characteristics compatible with the intracontinental rifting, meaning that, this area belongs to the Cachimbo Graben with its peculiar rifting pattern. In this area eruptions of the Beneficente Group composed of carbonates, sandstone and silicates with age on the order of 1.6Ga are present.

## 2.3. Methods

Initially, the teeth were washed and enamel was separated from dentine through thermal treatment with liquid Nitrogen, the same procedure adopted in our previous work that is practical and efficient (Kinoshita et al., 2008b). The enamel detaches from dentine after freezing and thawing the teeth at room temperature, due to difference in thermal expansion coefficient of these tissues and possible freezing and expansion of water present in the interstitial layer. The remaining dentine was cleaned using dental drill and a NaOH solution of 30% weight concentration in an ultrasound bath for about 60 min. An additional layer of  $\sim 500 \mu\text{m}$  was eliminated etching the enamel with an acidic solution (HCl 1:10). The enamel was powdered in fine particles ( $\phi < 0.5 \text{ mm}$ ) using an agate mortar and pestle. Additive doses were given in aliquots of about  $\sim 100 \text{ mg}$ . The irradiation was performed with gamma rays, in air, at room temperature, with a dose rate of 2.49 kGy/h using a 0.4 g/mm<sup>2</sup> thick Lucite build-up cap over the samples. The Gammacell Cobalt-60 irradiator of Instituto de Pesquisas Energeticas e Nucleares (IPEN) was employed.

ESR spectra of samples were recorded at two computer controlled X-Band ( $\nu \sim 9 \text{ GHz}$ ) spectrometers, a Varian E-4 and a Jeol JES-FA 200. Typical measuring conditions were: modulation amplitude 0.2 mT, scan range 10 mT, scan time 1 min, incident microwave power below signal saturation.

The NAA of samples was used to determine the concentration of Uranium and Thorium of enamel, dentine and sediment. Samples and standards were irradiated at IEA-R1 nuclear reactor with thermal neutron flux of  $10^{12} \text{ n.cm}^{-2} \text{ s}^{-1}$  for 8 h. Counting of induced gamma-ray activity was performed 10 days after irradiation using a GX20190 hyperpure Ge detector (Canberra), at 10 cm distance between sample and detector. The resolution (full width at half maximum-FWHM) of the system is 1.90 keV for the 1.332 keV gamma rays of <sup>60</sup>Co. VISPECT software was used to process the gamma-ray spectra.

## 3. Results

The native ESR spectra of these samples show a strong signal with spectroscopic factors  $g_{\perp} = 2.0025$  and  $g_{\parallel} = 1.9973$  related to  $\text{CO}_2^-$  radical in Hydroxyapatite (Callens et al., 1989, 2002). Fig. 1a shows the spectrum of fossil sample 354 recorded in the X-band spectrometer. Tooth enamel was irradiated with cobalt-60 to evaluate the effect of the dose on the signal amplitude. This figure shows how the signal changes for increasing doses. It can be

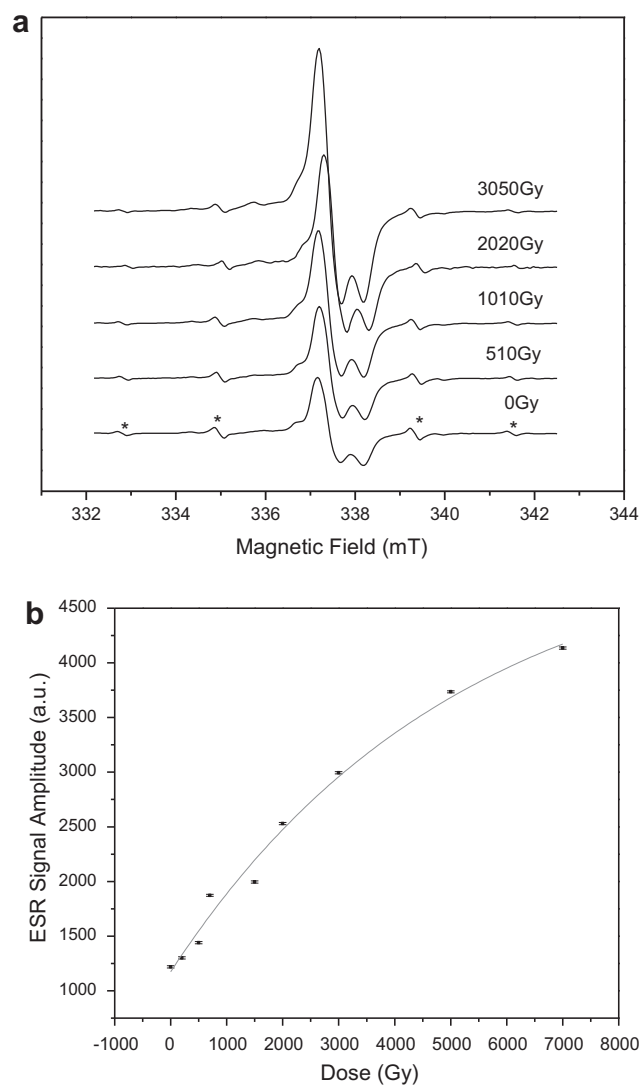


Fig. 1. (a) ESR spectrum of tooth enamel (Fossil 354) for different post irradiation doses. The spectrum shows the signal due to the  $\text{CO}_2^-$  radical as the main component at the centre as well as a septet due to the isopropyl  $(\text{CH}_3)_2\text{C-R}$  radical indicated by \*. Signal amplitude at  $g_{\perp}$  was used to determine the  $D_e$ . (b) Dose-response curve for the enamel fitted by Equation (1). The fitting parameters found are  $D_e = (1.3 \pm 0.2) \text{ kGy}$ ,  $I_0 = (52 \pm 6) \cdot 10^2$ ,  $D_0 = (5 \pm 1) \cdot 10^2 \text{ Gy}$ .

noticed that the central region corresponding to the  $\text{CO}_2^-$  radical increased with the dose while the other regions of the spectrum remained unchanged. The amplitude of the spectral feature at  $g_{\perp} = 2.0025$  was used to construct the dose-response curve (Fig. 1B). The uncertainty associated to each data point was taken as the RMS spectrum noise calculated as the quotient of the peak to peak amplitude of the noise by 2.5. A saturating exponential fitting (1) was used to determine the  $D_e$  and the values are reported at Table 2.

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

$I$  is the ESR signal intensity,  $D$  the added dose,  $I_0$  and  $D_0$  the intensity and the dose, respectively, at saturation. The software Microcal Origin 7.5 (Microcal Software Inc, Northampton, MA, USA) was used with the option instrumental weighting to

**Table 1**  
Radioisotopes concentration in the sample and surrounding soil. The average and standard deviation of values of soil was considered to age calculation.

Sample	Uranium	Thorium	Potassium
Enamel			
354	8.7 ± 0.3	<0.01	<750
MUS99	10.6 ± 0.4	<0.01	<750
MAR	3.2 ± 0.1	<0.01	<750
Dentine			
354	13.9 ± 0.5	<0.01	<750
MUS99	12.3 ± 0.4	<0.01	1311 ± 478
MAR	13.3 ± 0.4	<0.01	<750
Soil			
Sample 1	4.0 ± 0.1	12.6 ± 0.1	29535 ± 3812
Sample 2	4.0 ± 0.1	12.3 ± 0.1	30458 ± 3968
Sample 3	4.0 ± 0.1	12.0 ± 0.1	28580 ± 3705

estimate the uncertainties. In this option, the data points are weighed by the amplitude error bar ( $s_{ij}$ ) through the relation  $w_{ij} = 1/(s_{ij})^2$ , that assures that the lower intensities related to the points at lower doses get higher weights. This procedure has been used by other authors (Berger et al., 1987; Skinner et al., 2000) and is based on the fact that the lower dose points are more important to determine  $D_e$  because they were produced by the natural radiation and are less prone to possible artefacts induced by the artificial irradiation, although there are no reports about this possibility.

We can also note the presence of the signal of isopropyl radical with hyperfine splitting of 2.17 mT. This septet signal was observed by other authors in middle Pleistocene tooth samples (Ikeya, 1993; Kinoshita et al., 2008a).

The age of fossil teeth were calculated using the “ROSY ESR dating software” (Brennan et al., 1999). To convert  $D_e$  into age, the concentration of  $^{238}\text{U}$  and  $^{232}\text{Th}$  present in the samples and soil where the samples were buried were obtained by Neutron Activation Analysis (NAA). Potassium concentration of in the soil was determined by atomic absorption spectroscopy and these concentrations are shown in Table 1. The value of  $0.13 \pm 0.02$  was used for k-value, that is, the ratio of defects creation efficiency for  $\alpha$  particles to internal dose rate calculation, the value given by Grün and Katzenberger-Apel (1994). The energy released by  $\alpha$  particles in the soil was not considered. The typical values of maximum penetration depths of  $\alpha$ -rays for the range of energy of 4–5 MeV in crystals are 40–60  $\mu\text{m}$  (Ikeya, 1993), shorter than the layer extracted in the sample preparation. An initial  $^{234}\text{U}/^{238}\text{U}$  ratio of  $1.2 \pm 0.2$  was assumed for age calculations, based on U-series half lives by Cheng et al. (2000).

Table 2 shows the dose rate from sediment, dentine and enamel, given by ROSY ESR dating program according to the radioisotopes uptake model (EU, LU and CU). The ages calculated taking into account the cosmic rays dose rate of 114  $\mu\text{Gy}/\text{y}$  are reported at Table 3. This value was obtained after performing corrections suggested by Prescott and Hunton (1994) using the latitude,

**Table 2**  
Dose rates ( $\mu\text{Gy}/\text{a}$ ) from Dentine, Enamel and Sediment according to the radioisotopes uptake model and.

Sample	Early uptake			Linear uptake		Combination uptake	
	$D_{\text{ext.sed}}$	$D_{\text{ext.den}}$	$D_{\text{int.ena}}$	$D_{\text{ext.den}}$	$D_{\text{int.ena}}$	$D_{\text{ext.den}}$	$D_{\text{int.ena}}$
354	1127.37	14.51	3594.02	7.22	1772.37	15.14	1771.32
MUS99	1127.37	15.76	3701.67	8.20	1846.28	16.63	1844.86
MAR	1127.37	11.82	798.96	5.80	371.06	12.12	370.58

$D_{\text{ext.sed}}$  is the dose rate from sediment,  $D_{\text{ext.den}}$  is the external dose rate from dentine and  $D_{\text{int.ena}}$  is the internal dose rate in enamel.

**Table 3**  
Equivalent Dose ( $D_e$ ) and the ages calculated using ROSY software according to the radioisotopes uptake model.

Sample	$D_e$ (Gy)	EU (ka)	LU (ka)	CU (ka)
354	1300 ± 200	270 ± 70	430 ± 70	430 ± 70
MUS99	800 ± 100	160 ± 20	260 ± 30	260 ± 30
MAR	140 ± 20	70 ± 10	90 ± 10	90 ± 10

longitude and altitude of Alta Floresta and the depth where the samples were found. This dose rate is very similar to that used by other authors (Watanabe et al., 2003; Kinoshita et al., 2008a) for Brazilian north-eastern region.

#### 4. Comments and discussions

The equivalent dose  $D_e$  was converted to age using ROSY ESR dating program (Brennan et al., 1999). The DATA software (Grün, 2009a) was also employed giving the same results, although, according to the authors, it uses the most updated data on beta radiation interaction obtained from Marsh et al. (2002). Different models for teeth uranium uptake are commonly considered: early uptake (EU) and linear uptake (LU). In the first case, all of the uranium absorption is assumed to have occurred rapidly, whereas in linear uptake, U is assumed to have accumulated gradually at a constant uptake rate from time zero until the time the sample was collected (Grün, 2009b). The ROSY software also presents the option Combination Uptake, where we can choose a different model for uranium uptake in the tissues. In this work, a Combination Uptake using the Early Uptake model for dentine and Linear Uptake for enamel was used, taking into account that the enamel is impervious while dentine is porous. So, the ages resulting with this option seems to be more plausible. However, only with the help of other analytical techniques it is possible to decide which is the best model, ICP-MS (inductively coupled plasma mass spectrometry) has been used in these cases and meaningful results are obtained when the concentration variation from point to point of the sample falls within the resolution of the method (Grün, 2006). The isochron method (Blackwell and Schwarcz, 1993; Schwarcz, 1994; Blackwell et al., 2000) is very interesting and should be used where the external dose is believed to have been spatially homogeneous, but poorly known to determine both the age and the external dose. But unfortunately could not be used in this case due to the limited information about the sediments associated with the samples. In addition, to apply the isochron dating, at least 5 subsamples of enamel with same symmetry are needed (Blackwell and Schwarcz, 1993). There was not enamel in good conditions to ESR experiment available in the samples to try this method.

In relation to the ages found, Paula-Couto (1979), observed that the northern region in South America is the most likely to have mastodon's fossils; and its known distribution is restricted to the Pleistocene of the Argentinean Pampa and the neighbourhood of Paraguay and Uruguay and possibly the southern part of Brazil. The Haplomastodon genus appears to be abundant and widespread all over South America, the Pleistocene one being the more distinguishable than the others. The presence of mastodons are recorded in the Pleistocene epoch (1.8Ma to 10ka) in the United States, these North American migrants invaded South America through the Panama isthmus after its raising 3 million of years ago. The age found for MAR sample is smaller than the other samples but, all of them belong to the Pleistocene period, thus compatible with these events. The Uranium content in MAR enamel is lower than other samples, consistent with the younger age. It is worth to note that these fossils were found in the alluvium and gravel of more recent epochs thus the ages found indicate that these fossils belonged to

**Table 4**  
Concentrations of Uranium, thorium and potassium from different sites in Brazil.

State	U (ppm)	Th (ppm)	K (%)	Reference
Paraíba (PB)	2.27 ± 0.2	5.1 ± 0.1	0.063 ± 0.005	Kinoshita et al., 2005
Pernambuco (PE)	1.95 ± 0.24	13.28 ± 3.5	2.65 ± 0.63	Kinoshita et al., 2008a
Pernambuco (PE)	2.22 ± 1.20	15.06 ± 3.70	2.30 ± 0.60	Kinoshita et al., 2008a
Piauí (PI)	2.4 ± 0.2	9.3 ± 0.1	3.9 ± 0.4	Kinoshita et al., 2008b
Rio Grande do Sul (RS)	1.5 ± 0.05	6.3 ± 0.2	1.0 ± 0.1	Lopes et al., 2010
Rio Grande do Sul (RS)	1.6 ± 0.4	5.8 ± 0.1		Kerber et al., 2010
Rio Grande do Sul (RS)	1.7 ± 0.1	4.9 ± 0.3		Kerber et al., 2010
Rio Grande do Sul (RS)	1.4 ± 0.3	4.78 ± 0.09		Kerber et al., 2010

terraces of Pleistocene period that were reworked, as the age span of the samples suggests.

The possibility of reworking adds to the uncertainty caused by limited information about the sediments associated with the samples. In partial compensation, we note that the dose rate obtained for the sediment is smaller than the internal dose rate for the teeth, showing that the main contribution would come from the internal irradiation. In addition, the values for the sedimentary radioisotopes concentration used in this work to date Fossils 354 and MUS99 are similar to but higher than those found in other states of Brazil, using the same NAA technique. The values are listed in Table 4. Thus using the limited values can be considered to give minimum ages. Calculating the ages without the sediment contribution (sediment dose rate equal to zero), gives maximum ages assuming the Combination Uptake of Fossils 354 and MUS99 of 630 and 370ka. Thus, we can estimate that the maximum and the minimum ages introduced by this approximation into the estimated age for Fossils 354 and MUS99 would be (500 ± 100)ka and (320 ± 50)ka.

## 5. Conclusion

This work demonstrates that ESR dating can be very helpful to date fossils in range beyond the radiocarbon limits, providing useful information. It is also important to note that only a few studies have been published about the Mato Grosso Pleistocene Megafauna and this work reports the first results. At the present the palaeontologists are working in the field to collect information about that could contextualize the dates obtained. We also hope that the visibility of this dating work could stimulate other studies in the area that could find more samples collected with better information about the geological context.

## Acknowledgements

The authors thank the critiques of two anonymous referees that contribute to improve the manuscript. To FAPESP, CAPES and CNPq to partial financial support, Centro de Tecnologia das Radiações-IPEN for the samples irradiation and Carlos A. Brunello and Lourenço Rocha for technical support and Carlos Calia Boscoli for the donation of the sample to Universidade Federal de Mato Grosso.

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