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ESR dating of *Smilodon populator* from Toca de Cima dos Pilão, Piauí, Brazil

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ABSTRACT

Smilodon is a genus of big cats that lived from the early to the late Pleistocene in regions extending from North to South America. The fossil records of the "saber-toothed cats" are uneven, with some taxa being quite abundant in certain regions. In Brazil, *Smilodon populator* is a well-known species whose remains, although scarce in comparison to other large mammals, are found all across the country. In particular, there are multiple records of this species in the region of the Serra da Capivara National Park. This area was home to a rich Pleistocene-Holocene fauna, including many mammals. Here, we report on the Electron Spin Resonance dating of a *Smilodon populator* tooth found in "Toca de Cima dos Pilão", located in the surroundings of the Serra da Capivara National Park. The equivalent dose found after exponential fitting of dose-response curve was $(2.7 \pm 0.3)\mathrm{x}10^2$ Gy. Neutron Activation Analysis was used to determine the concentration of radioisotopes present in the sample and in the sediment to calculate the internal and external dose rates. The result of age found is 93 ± 9 ka, which confirmed the presence of this species in Serra da Capivara National Park in the late Pleistocene.

1. Introduction

Smilodon Lund, 1842 is a genus of big cats that inhabited regions extending from North America to South America. There is evidence that these animals lived from the early Pleistocene Epoch through the beginning of the Holocene Epoch (Barnosky and Lindsey, 2010;Berta, 1985;Faure and Guérin, 2014). This genus belongs to the Machairodontinae subfamily. Its main feature is the presence of hypertrophic blade-shaped upper canines (Christiansen, 2013). Although the fossil records of "saber-toothed cats" are uneven, some taxa have been quite abundant in certain regions (Christiansen, 2013 and references therein). In Brazil, *Smilodon populator* Lund, 1842 is a well-known species that emerged in several regions (see de Castro and Langer, 2008 for a list of localities). There are multiple records of this species in the region of the Serra da Capivara National Park (Faure and Guérin, 2014; Guérin, 1991; Guérin et al., 1993; Parenti et al., 1996).

UNESCO (United Nations Educational, Scientific and Cultural Organization) has recognized the Serra da Capivara National Park, located in the state of Piauí, northeastern region of Brazil, as a world heritage site. The park and its surrounding area have the largest currently known concentration of archaeological sites in the Americas. Rich fauna, including many mammals, have inhabited this region in the late Pleistocene to the Holocene (Guérin and Faure, 2008).

1.1. Toca de Cima dos Pilao

This work reports on the Electron Spin Resonance (ESR) dating of a *Smilodon populator* tooth found in "Toca de Cima dos Pilão", located in the area surrounding the Serra da Capivara National Park (Fig. 1). More specifically, this site is located on the west slope of the main Precambrian limestone outcrop of the region, which dominates the valley. The site consists of two small, interconnected caves (Salão da Teresinha and La Rotonde) whose layers of sandy clay sediments are rich in fossils of the megafauna (da Luz, 1989; Faure and Guérin, 2014; Guérin et al., 1996; Parenti et al., 1996). Toca de Cima dos Pilão has different morphological units and several areas of prehistoric findings. There are also a number of rocky shelters rich in lithic artifacts (da Luz, 1989). The abundance of fossil remains in this site suggest that it was a

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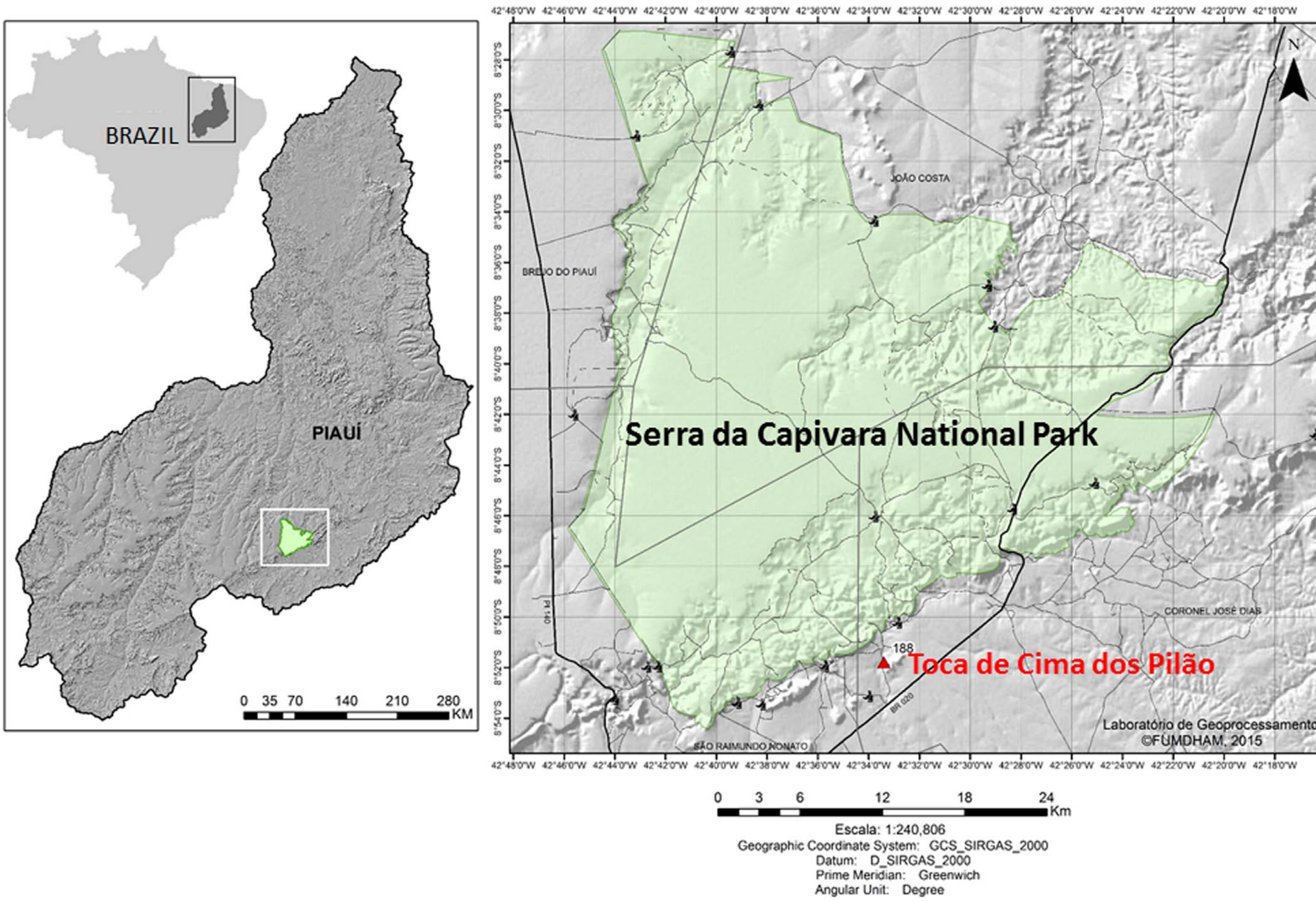

Fig. 1. Location of the Toca de Cima dos Pilão near the Serra da Capivara National Park in the southeastern region of the state of Piauí, Brazil.

hiding place for *Smilodon populator*—fossils of many *Smilodon populator* preys as well as fossils of other mammals exist in the site (Faure and Guérin, 2014; Guérin et al., 1996).

The sample analyzed herein was collected in the exploratory trench located at the back of Salão da Terezinha, at a depth of 385 cm in the fossiliferous assemblage between −341 and −390 cm (Fig. 2A). In 1988, the trench was excavated until a depth of −730 cm, but the bedrock was not reached. The first −200 cm consisted of stalagmite floor; a stone line lay just underneath this floor. Below that, there was sandy clay sediment with several fossiliferous layers (da Luz, 1989).

1.2. ESR Dating

The sample was dated through Electron Spin Resonance (ESR). ESR is a well-established technique to date teeth within the range of ages expected for the fossil tooth of this work (Rink, 1997); it is based on the effects of ionizing radiation on the sample (Ikeya, 1993). Ionizing radiation generated by radioactive isotopes present in the environment and cosmic radiation produces crystal defects in the mineral matrix of the teeth, hydroxyapatite. The radiation dose known as Equivalent Dose (D_e) can be quantified by ESR through the additive dose method. Subsequently, by means of softwares such as ROSY (Brennan et al., 1999 or DATA (Grün, 2009a), the Equivalent dose (D_e) is converted to age by the dose rate calculation. ROSY and DATA are softwares that assume a diffusion model of the radioisotopes into the sample to calculate the internal dose. This conversion of D_e into age can also be performed with other methods where the actual spatial distribution of the uranium series is measured and there is no need to assume a diffusion model, this approach is called US-ESR. This later method is considered the state of the art and it is mandatory for samples older than the one studied here. In the case of older samples the difference between the models are very high and the dates does not have a meaning. However, the use of ICPMS is expensive and not widely available what suggests, until now, that this method should be use for older samples (Shao et al., 2015). One of the important parameters that makes ESR dating possible is the stability of the radical used to determine D_e. In the case of CO_2^- present in tooth enamel, shells, calcites, etc, the estimated lifetime is $\sim 10^7$y at 25 °C, which allows its use for archaeological dating The lifetime of defects is highly dependent on temperature. Natural annealing occurs through the recombination of electron and hole centers (Ikeya, 1993). Recently Negrón-Mendoza et al., 2016 studied the effects of irradiation temperature on calcium carbonate, finding increasing in ESR signal intensity with temperature. In addition, some short-lived radicals were present at low temperatures that disappeared after thermal annealing. Some authors proceed the thermal annealing in the samples before the ESR dating experiments (Blackwell et al., 2010; Skinner, 2000). Furthermore, because CO_2^- is an impurity in the hydroxyapatite structure (Oliveira et al., 2012; Santos et al., 2005) that gives the ESR signal, its intensity for a given dose can vary from sample to sample dependent on the level of this dopant. Thus it is not possible to use an universal calibration curve and each specimen must be calibrated by the additive dose method. ESR dating has allowed for successful dating of a number of samples, which has provided important information about temporal determination of the events that caused the extinction of this species (da Costa Ribeiro et al., 2013; Kerber et al., 2011; Kinoshita et al., 2008b; Lopes et al., 2010; Trindade Dantas et al., 2011). In addition to teeth, fossil shells are also possible to be dated providing important information regarding the formation of deposits and the presence of species in the collection sites or human presence in the place, depending on the context (Aydas et al.,

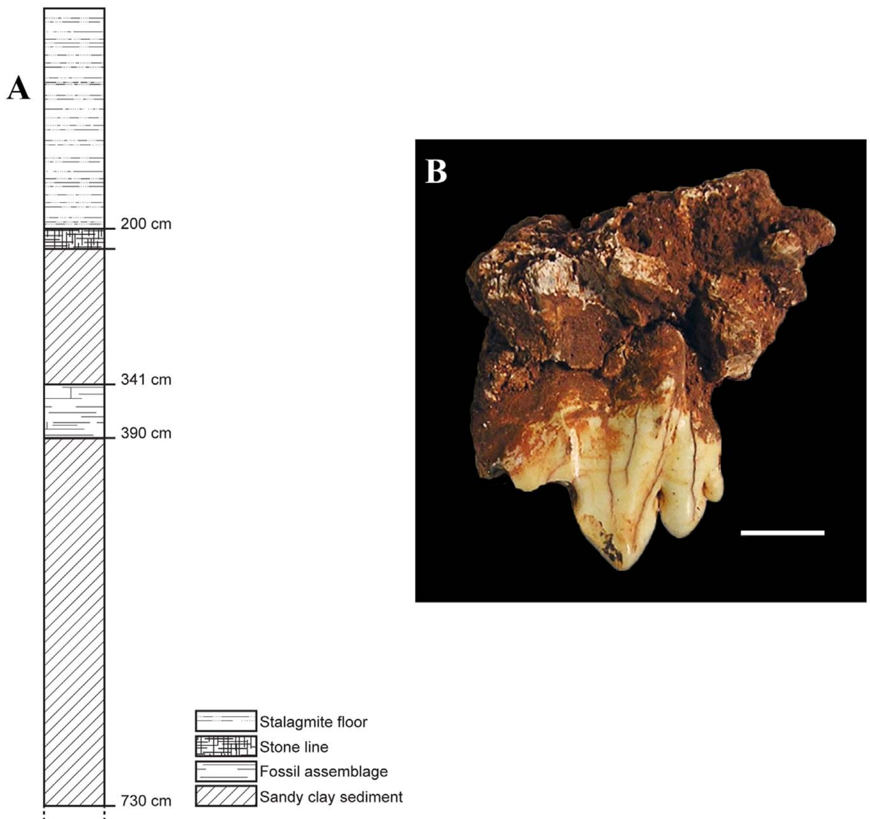

Fig. 2. A, Stratigraphic general scheme of the Toca de Cima dos Pilão indicating the fossiliferous assemblage where the material was collected. B, the sampled *Smilodon populator* fourth upper premolar. Scale bar =1 cm.

2015; Kinoshita et al., 2002; Lopes et al., 2014; Mascarenhas et al., 1982; Shimokawa et al., 1992).

2. Material and methods

The dated specimen is a fourth upper premolar of *Smilodon populator*, deposited at Fundação Museu do Homem Americano (FUMDHAM) under catalog number 19011 (Fig. 2B). It was subjected to heat treatment by freezing in liquid nitrogen and heating at room temperature. After a few repetitions, the enamel came off from the remaining dentin. This procedure has already been adopted previously (Kinoshita et al., 2008a).

The enamel was submitted to acid treatment in HCl 1:5 in an ultrasonic bath for a few seconds, to extract an outer layer from both faces. The tooth thickness before and after treatment was 0.5 and 0.4 mm, respectively. After drying, the enamel was manually ground in an agate mortar to a powder with particle diameter $\varphi < 0.5$ mm. Subsequently, 70 mg of the treated enamel were irradiated with doses from 100 Gy up to 3kGy for application of the additive dose method (Ikeya, 1993).

Dentin and the sediment associated with the sample were also crushed. The sediment was divided into three samples and, together with the dentine and enamel, was subjected to neutron activation analysis (NAA) to determine the concentration of ^{238}U, ^{232}Th, and ^{40}K.

The spectrum was recorded in the Jeol FA-200 X-Band spectrometer by using the following settings: microwave power = 2 mW (below signal saturation), scan width = 10 mT, scan time = 1 min, modulation amplitude = 0.1 mT, modulation frequency = 100 kHz, and time constant = 100 ms. To construct the dose-response curve (DRC), the peak-to-peak intensity at the dosimetric signal $g_\perp$ was associated with the additive dose. The Equivalent Dose (D_e) was determined by fitting the single saturation exponential (SSE) function (1)(Ikeya, 1993):

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

where I is the ESR signal intensity, D the additive dose, I_0 and D_0 is the intensity and dose, respectively, in the saturation and D_e the Equivalent Dose.

There are other functions such as double exponential and SSE plus linear (Duval et al., 2009), however, using SSE high correlation with experimental data found was r = 0.9922.

D_e was converted into age with the ROSY ESR Dating software (Brennan et al., 1999); the concentration of radioisotopes present in the sample and sediment was employed. The value of 136 μGy/year was adopted for the cosmic radiation dose rate, which was found after correction considering the latitude and longitude (33° 42′ 46.26″ "S and 59° 38′ 2.22" W, respectively), the altitude (8 m), and the location and depth (3 m) from which the sample was removed (Prescott and Hutton, 1994). Humidity was measured and considered to be approximately 13.5%. Initial U-234/U-238 ratio and alpha efficiency (α_{eff}) values of 1.2 and 0.13, respectively, were used (Grün, 2009b).

Table 1
Radioisotopes concentration in the enamel, dentin, and sediment measured by Neutron Activation Analysis. There are 3 samples of sediment, the average of concentration was used for age calculation.

	^{238}U (ppm)	^{232}Th (ppm)	^{40}K (%)
Enamel	3.88 ± 0.07	< 0.01	< 0.075
Dentin	34.6 ± 0.2	< 0.01	< 0.075
Sediment	6.2 ± 0.2	12.3 ± 0.3	0.9 ± 0.09

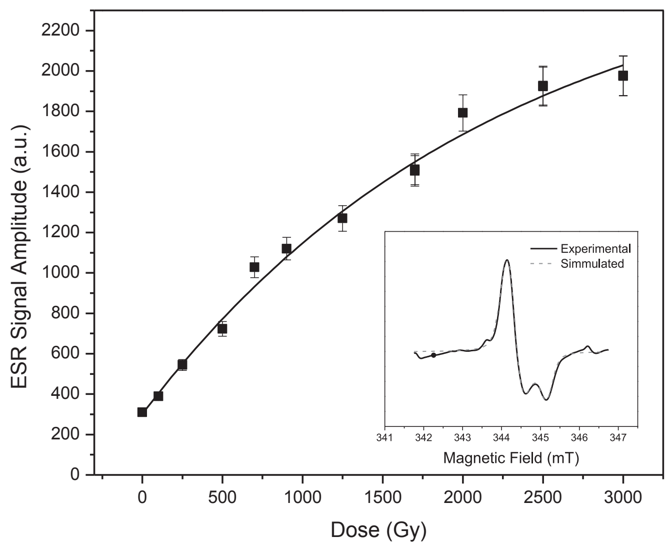

Fig. 3. Experimental and simulated ESR spectrum of aliquot irradiated with a dose of 2kGy. Dose-response curve with exponential fitting. D_e is $(2.7 \pm 0.3)\text{x}10^2$ Gy. The other parameters of function (1) are $D_0 = (2.3 \pm 0.4)\text{x}10^3$ Gy and $I_0 = (2.7 \pm 0.3)\text{x}10^3$.

3. Results and discussion

Table 1 lists the radioisotope concentrations in the enamel, dentin, and sediment around the sample determined by Neutron Activation Analysis.

Fig. 3 shows the ESR spectrum of an aliquot irradiated with 2kGy and the dose response curve constructed (DRC) for the *Smilodon populator* tooth. Spectral simulation performed shows that this spectrum is dominated by the axial CO_2^- radical with g-factors $g_\perp = 2.0025$ and $g_{//} = 1.9973$ and this is the reason for using the intensity peak to peak of the spectrum to construction of the DRC. The software Origin 8.5 was used to fit the experimental data points to Eq.(1); instrumental weighing was employed to calculate D_e uncertainty (Skinner, 2000).

The external and internal dose rates were calculated by using the ROSY ESR Dating software (Brennan et al., 1999). The internal dose rate depended on the way the radioactive elements were incorporated by the sample. Some theoretical models for uranium uptake have been developed and implemented in a computational algorithm. One example is the Early Uptake (EU) model (Bischoff and Rosenbauer, 1981), which provides the minimum age of the sample. Another model is the Linear Uptake (LU) model (Ikeya, 1993), which considers that uranium was absorbed on the analyzed material at a constant rate. As already mentioned, these models were implemented in the ROSY (Brennan et al., 1999) and DATA (Grün, 2009a) software for tooth dating. The Recent Uptake (RU) model gives the maximum age because it assumes that the uranium present in the tissues was incorporated next of sample collection (Blackwell and Schwarcz, 1993). As previously reported, when the U-series is coupled to ESR, the uptake history can be determined by solving the equation proposed by Grün et al., 1988, dismissing the need to assume a uranium uptake model. This method has been applied in several problematic dating situations (Duval et al., 2012, 2011a, 2011b; Falguères et al., 2010). Unfortunately, in the present case it was not possible to apply this method because Neutron Activation Analysis did not allow us to measure the isotopes concentration for each individual radioisotope, a condition required by this technique. Therefore, the Age value obtained by the Combination Uptake model (CU) was defined by using the Early Uptake (EU) model and the Linear Uptake (LU) model in the case of dentin and enamel, respectively. The differences in porosity afforded the most probable age of the fossil Table 2 summarizes the Age results.

The fossil record concerning the megafauna of the Serra da Capivara National Park is extensive. Nevertheless, despite all knowledge about the anatomy and taxonomy of the species found therein, chronological information about the Brazilian Quaternary era is still limited (da Costa Ribeiro et al., 2013). In this sense, ESR dating methods may provide more precise age estimates, which can be very useful in biogeography, taxonomy, extinction, and climate change studies (Kinoshita et al., 2014).

Table 2
Summary of results: External and Internal dose rates and the age according to the uranium uptake model.

Model	Internal (μGy/a)		External (μGy/a)		Age (ka)
	Alpha	Beta	Beta	Gamma	
E.U.	698.5	170.3	1003.6	1438.8	80 ± 7
L.U.	324.6	80.8	710.1	1438.8	100 ± 10
C.U.	307.1	77.7	1075.3	1438.8	93 ± 9

Smilodon populator is a very well documented species that was largely distributed in Brazil and South America from > 48,000 and at least 10,000 years ago; that is, from the late Pleistocene Epoch through the beginning of the Holocene Epoch (Barnosky and Lindsey, 2010; Castro and Langer, 2011; de Castro and Langer, 2008; Faure and Guérin, 2014; Guidon et al., 1994; Neves and Piló, 2003; Scanferla et al., 2013; Suárez and López, 2003). The latest records of this species date from the Lujanian Stage/Age, and there is a doubtful record from the Uquian Stage/Age (Berta, 1985). However, the indirect dating methods used for these records are not precise, and the only Uquian record, from Argentina, may not be from this period—indeed, this record came from the Vorohue Formation at Mar del Plata and Miramar, a rocky unit with intermediate ages between the Uquian and Ensenadan Land-Mammal Ages, being therefore dubious (Berta, 1985).

ESR dating provided an age value of 93 ± 9 ka for the *Smilodon populator* tooth sample found in Toca de Cima dos Pilão, Piauí, Brazil. This age corresponds to the late Pleistocene Epoch and this is the oldest fossil of this taxon from South America for which a direct age was obtained so far; it is much older than all the megafauna identified in the region of the Serra da Capivara National Park (de Castro and Langer, 2008; Faure et al., 1999; Faure and Guérin, 2014; Guérin et al., 1996; Guidon et al., 1994; Neves and Piló, 2003; Suárez and López, 2003). According to Guérin et al.,1993 the megafauna of the Serra da Capivara National Park cannot be older then the Lujanian (from 8.5 ka to 130 ka). Although our results are the oldest recorded in the region and in Brazil, they fully agree with the report by Faure and Guérin, 2014. Nonetheless, they indicate that the dates estimated for Toca de Cima dos Pilão on the basis of the lithic industry (10390 ± 80 BP) should be used with caution for the megafauna of this cavern. Also, the material from Toca de Cima dos Pilão studied in this work was found in an intermediate stratum of the cavern, whereas several other fossils have been detected in lower levels (da Luz, 1989) and need to be dated. Rarely has been *Smilodon populator* directly dated with results between 9 and 15 ka approximately (Auler et al., 2006; de Castro and Langer, 2008; Neves and Piló, 2003) an age when the species was probably in decline (Barnosky and Lindsey, 2010). The result presented here shows that there is much more to be learnt about this species, and that frequent use of direct dating methods to obtain precise age estimates in different areas is welcome. The information obtained here shall contribute to better understanding of the distribution and paleoecological aspects of the South American megafauna.

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