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ESR dating of Pleistocene mammals and marine shells from the coastal plain of Rio Grande do Sul state, southern Brazil



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ABSTRACT

The Coastal Plain of Rio Grande do Sul state (CPRS), in southern Brazil, encompasses four barrier-lagoon depositional systems formed by marine highstands during the Quaternary, but the ages of the Pleistocene systems older than ~125 ka are not precisely known due to the lack of numerical dates. In order to refine the chronology of these deposits, several fossils of terrestrial mammals collected in the fluvial facies of the Lagoon System III and fossil shells from the marine facies of the Barrier System II, exposed in the southern sector of the CPRS, were subject to dating by electron spin resonance (ESR). The ages of the mammalian remains range from 90 ± 10 to 43 ± 3 ka, thus representing the last glacial cycle (MIS 4 and 3). The mean EU age of the shells is 224 ± 24.6 ka, but this value is considered younger than the 'real' age because of excessive Uranium uptake observed among the younger shells. An estimated mean age of some 235–238 ka would agree with other ESR and TL ages obtained for fossils and sediments collected from beds overlying the shells, and would be consistent with the sea-level highstand at the early MIS 7 (substage 7e). The presence of bivalves and foraminifers that indicate ocean temperatures higher than at present seems to corroborate this estimate, given that MIS 7e was characterized by hypsithermal conditions.

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1. Introduction

In recent years, Electron Spin Resonance (ESR) has been used for dating speleothems, travertines, marine organisms, terrestrial megafauna and archaeological remains (Grün, 1989; Rink, 1997; Kinoshita et al., 2008; Blackwell et al., 2010; Kerber et al., 2011). The physical basis of the ESR method is the measurement of the concentration of unpaired electrons by ESR spectroscopy. Electrons

trapped in defects of the lattice of solid materials are produced by ionizing radiation emitted by radioactive elements (mainly ^{238}U , ^{232}Th and ^{40}K) present in the surrounding sediments and within the sample being dated; and from cosmic rays. The total dose of natural radiation is called equivalent dose (D_e) and is proportional to the age. By measuring the concentration of unpaired electrons trapped in the material and the dose rate from the environment and radioactive elements in the material itself, the age can be calculated. The internal dose rate depends on the way in which the radioactive elements were incorporated by the sample. Thus some theoretical models for uranium uptake have been developed and implemented in a computational algorithm.

In the Early uptake (EU) model it is assumed that uranium incorporation occurs in a short time relative to age and remains constant (Bischoff and Rosenbauer, 1981). This model corresponds to a closed system and provides the minimum age of samples. In the Linear Uptake model, the absorption of Uranium occurs at a

Abbreviations: LGP-E, Laboratório de Geologia e Paleontologia (Notoungulata); LGP-G, Laboratório de Geologia e Paleontologia (Proboscidea).

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constant rate (Ikeya, 1993). These models were implemented in the ROSY (Brennan et al., 1999) and DATA (Grün, 2009a) software for tooth dating. In the ROSY software, there is another option, the Combination Uptake model, in which it is possible to calculate the ages with different combinations of the EU and LU for enamel and dentine. The Recent Uptake (RU) provides a maximum age since it is assumed that the Uranium present in the tissues was recently incorporated (Blackwell and Schwarcz, 1993). When U-series is used coupled to ESR, the uptake history can be determined by solving the equation proposed by Grün et al. (1988). This methodology has been applied successfully in several problematic dating (Grün, 2009b; Falguères et al., 2010; Duval et al., 2011, 2012) and it becomes increasingly important as the ages increase and the difference between the ages obtained by models above mentioned are very large, thus justifying the resources needed to perform this method.

ESR has been employed for dating Quaternary fossils of mammals, human remains, mainly using tooth enamel, and shells of both marine and terrestrial molluscs (Molodkov, 1988, 1993; Engin et al., 2006; Kinoshita et al., 2006). This method is useful for Quaternary fossils because it can determine ages older than the maximum limit of ^{14}C (about 50,000 years) its upper limit is dependant on the saturation of defects and signal stability in older samples and on the thermal stability of the defects (Ikeya and Ohmura, 1981; Radtke et al., 1985; Shimokawa et al., 1992; Schellmann and Radtke, 1997).

The coastal plain of Rio Grande do Sul state (CPRS, Fig. 1), in southern Brazil, is a geomorphological unit formed by successive marine transgressions correlated to glacial cycles of the Quaternary; the exact ages of the depositional systems, however, are mostly unknown. The only ages published so far were obtained for sediments from the northern and central sectors of the CPRS using thermoluminescence (TL) (Poupeau et al., 1988; Buchmann and Tomazelli, 2003), and shells, peat and mammalian remains from the southern sector using ^{14}C and ESR (Forti Esteves, 1974; Buchmann et al., 1998; Caron, 2007; Lopes et al., 2010; Lima et al., 2013).

Two major problems concerning the ages of the Pleistocene units of the CPRS are the predominance of sandy deposits mostly reworked by aeolian activity and/or covered by younger sediments, and the absence of fossil materials with good stratigraphic control. Although Holocene shells and peat are found in or near the present-day shoreline, Pleistocene materials in their original stratigraphic setting are scarce. The only deposit known so far that contains such materials is Chuí Creek, located in the southern sector of the CPRS. Lopes et al. (2010) published seven ESR ages from mammalian remains from the continental shelf (dated between ~650 and 18 ka) and four from the banks of Chuí Creek, with ages between 226 and 34 ka. Although these ages were useful for refining the stratigraphy, paleobiogeography and paleo sea-level curves for the CPRS, additional dating on fossils from Chuí Creek are necessary in order to establish more robust interpretations. Here are presented new results on dating of other mammalian fossils from the creek and from marine shells collected from a stratigraphic interval that outcrops below the mammalian remains.

2. Geological setting

The study area encompasses the Chuí Creek, located in the southernmost part of the CPRS (Fig. 1A); in this area only three of the four barrier-lagoon depositional systems that form the CPRS are preserved (Fig. 1B). The creek flows over a plain that is part of the Lagoon III depositional system, located between the Pleistocene barriers II and III; each barrier was formed in response to a marine transgression correlated to an interglacial epoch. While the age of

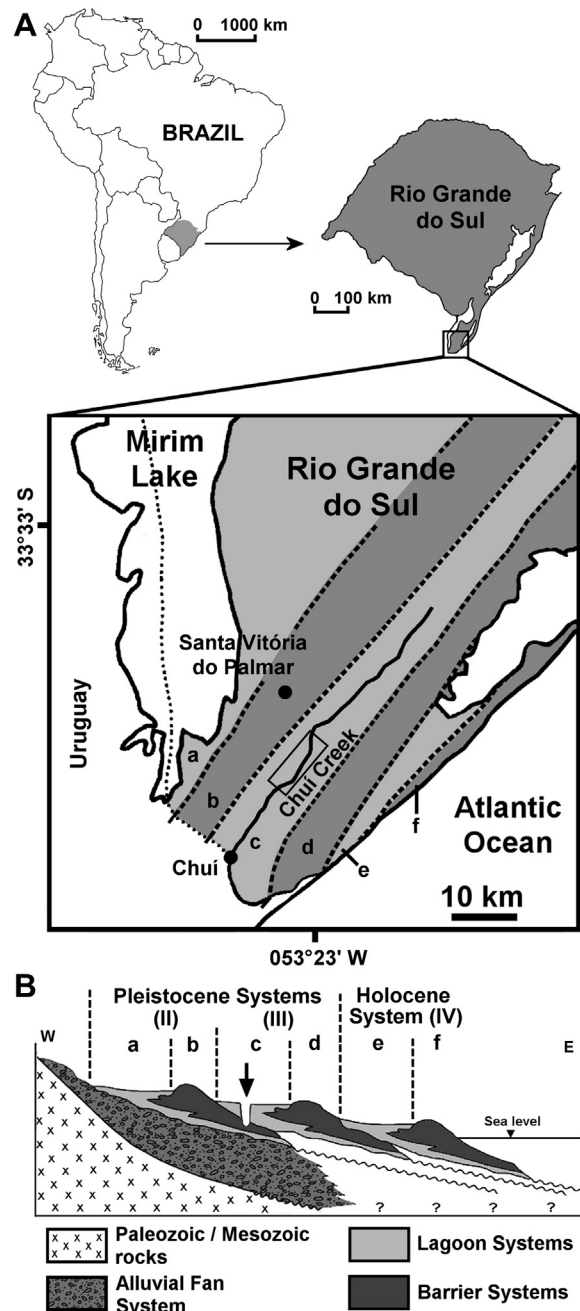


Fig. 1. A) Location of Chuí Creek, in the southernmost Rio Grande do Sul state, a rectangle indicates the area where the fossils were collected. Dashed lines indicate boundaries between the depositional systems: a) Lagoon II; b) Barrier II; c) Lagoon III; d) Barrier III; e) Lagoon IV and f) Barrier IV; B) West-East transect (modified from Tomazelli and Villwock, 2005) of the coastal plain in the area where Chuí Creek is located (indicated by arrow).

the Barrier System III was found to be correlated to the ~125 ka marine transgression (MIS 5), according to TL dates (Poupeau et al., 1988; Buchmann and Tomazelli, 2003), the age of the Barrier System II was considered to be of some 325 thousand years, by correlation with the oxygen isotopic curves of Imbrie et al. (1984) (Villwock and Tomazelli, 1995; Tomazelli et al., 2000). The Barrier-Lagoon System IV comprises the present coastline and was formed by the marine transgression around ~6 ka (Caron, 2007; Lima et al., 2013).

The Chuí Creek is the most studied Pleistocene fossiliferous locality of the CPRS. It flows parallel to the coastline in an NE–SW direction, which changes to NW–SE near the town of Chuí following the Chuí faulting zone. Prior to the 1960s, the creek was a shallow stream surrounded by wetlands, but it was further excavated in 1963 for agricultural purposes. During the excavations, a stratum containing remains of extinct mammals was exposed (Paula Couto and Cunha, 1965). The fossiliferous interval has been extensively studied in the last 10 years and has provided several new records of extinct mammals, besides information on past environments and climate conditions in the area (Lopes et al., 2009, 2011, 2013; Pereira et al., 2012; Lopes, 2013).

Closs and Forti (1971) reported the presence of several species of marine molluscs in the creek, but did not correlate these remains with any specific stratigraphic interval. Thus, this material probably consisted of shell remains scattered on the creek bed due to erosion of the banks, as can be seen today. Recently, four outcrops containing concentrations of marine shells were discovered in their original stratigraphic position, exposed at the base of the banks of the creek (Lopes and Simone, 2012).

The total height of the banks is of some 4.5 m (Fig. 2A, B). The lowermost layer is composed of well-sorted, quartz sand with small amounts of biogenic carbonate, heavy minerals and micas, contains ichnofossils *Ophiomorpha nodosa* and *Rosselia* sp., shell concentrations (Fig. 2C) and exhibits parallel and cross stratifications, thus indicating a shallow marine environment (upper shoreface). Some 2 m of this layer remain above the creek bed, and although the total thickness is yet unknown, it measures more than 4 m, based on soundings with hand auger. In the first survey of the stratigraphy of Chuí Creek, Soliani (1973) considered the marine sediments to be part of the “Chuí Formation” of Delaney (1965), while the overlying mammal-bearing stratum has been assigned to a new unit he called “Santa Vitória Formation”. The reviews on the geology and stratigraphy of the CPRS in the 1980s led to the replacement of the lithostratigraphic units by chronostratigraphic units (barrier-lagoon depositional systems) formed by associations of facies (Tomazelli and Villwock, 2005). Following the new scheme, the marine sediments exposed along the banks of the creek are part of the marine facies of the Barrier System II.

After the marine highstand, sea-level retreated and the uppermost part of the marine facies was occupied by paleosoils, fluvial systems and possibly small lakes that constitute the Lagoon System III. The contact between the marine facies and the overlying terrestrial environment is gradual, except in places where ancient fluvial streams eroded the marine sediments. These fluvial facies contain remains of Pleistocene terrestrial mammals, mostly fragmented and isolated, indicating intense erosion and reworking after the fossilization (Lopes, 2009; Lopes et al., 2009). The fossiliferous bed measures between 1.0 and 1.5 m in thickness, and is composed of silty sand with root traces and iron oxide concentrations; at some points of the banks a ~40 cm-thick caliche horizon consisting of nodules and rhizocretions is found at its upper part. Above the fossiliferous bed the amount of silt increases and vertebrate remains disappear. The silty layer measures some 1 m in thickness, and is covered by a ~50–70 cm-thick layer of organic matter-rich sand and clay of Holocene age, indicated by the presence of paleoindian remains (Schmitz et al., 1997).

3. Material and methods

3.1. Fossil teeth

Six teeth of extinct mammals, three of the notoungulate *Toxodon* and three of the proboscidean *Notiomastodon* (= *Stegomastodon*)

were selected for dating. All remains have been collected from the fossiliferous level in the banks of Chuí Creek, near the town of Santa Vitória do Palmar, and belonged to the paleontological collection of the Universidade Federal do Rio Grande (FURG). Three samples of the surrounding sedimentary matrix containing the fossils were also collected. Considering that the sediments containing mammalian remains are rather homogeneous, composed mostly of siliclastic sand deposited and reworked by fluvial systems, because the area is isolated from other fluvial systems since the Barrier II was formed, it is considered here that the fossil-bearing layer did not receive sediment contributions from external sources that could affect the results. A humidity of 10% was used to calculate the cosmic ray attenuation. This water content was estimated considering that the fossils are most of the time located above the water table (except during winter, when large floods can cover the fossiliferous horizon from periods of hours to a few days), and the beds above the shells are rather impermeable. The fossils are also too deep beneath the surface to be reached by plant roots, thus the only humidity that could usually affect the fossil remains comes from precipitation, but this reaches only the first few centimeters of the banks.

Between 2.2 g and 5 g of enamel and dentine were extracted from the specimens by mechanical drilling; enamel was separated from dentine using the thermal expansion technique. The samples were immersed in liquid nitrogen for a few minutes and then warmed to room temperature; this procedure was repeated a few times until enamel was detached from dentine due to differences in thermal expansion between these two materials. The enamel was then etched in a 1:10 HCl solution for 2 min to remove a thin surface layer (approximately 100 µm in thickness). The etched enamel was manually powdered until obtaining a fine (about 500 µm in diameter) powder which was then divided in 200 mg samples for irradiation with additive doses using a radioactive source with known energy (⁶⁰Co). The final dose of each aliquot was obtained in a single step, with the same dose rate, to increase the ESR signal. ESR spectrum was recorded in a JEOL FA-200 X-band spectrometer with the following acquisition parameters: central field 344 mT, scan width 10 mT, scan time 1min, modulation amplitude 0.1 mT, microwave power 2 mW, time constant 100 ms. The amplitude peak–peak at g_{\perp} was used to construct the dose response curves. The dose–response curve was adjusted using one single saturation exponential function (Ikeya, 1993):

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

where I_0 is the signal intensity of the saturation signal, D the additive dose and D_0 is the dose at saturation, using instrumental weighing ($1/I^2$) (Grün and Brumby, 1994; Skinner, 2000). Neutron activation analysis (NAA) was used to obtain the concentration of ²³⁸U, ²³²Th and ⁴⁰K in enamel, dentin and sediments. Once the D_e was obtained, the ages of the samples were calculated using one of the softwares available. Although DATA (Grün, 2009a) has incorporated the most recent beta attenuation factor, the software ROSY (Brennan et al., 1999) allows combinations of uptake models for dentine and enamel and was used in this work. Due to differences on porosities, the combination uptake (CU) was set as EU for dentine and LU for enamel is the best choice. This option is in accordance with U-concentration maps obtained by ICPMS that show that U-migration through the external enamel surface is very small when compared to the bulk of the uranium that migrated internally through the dentine into the enamel (Grün et al., 2008).

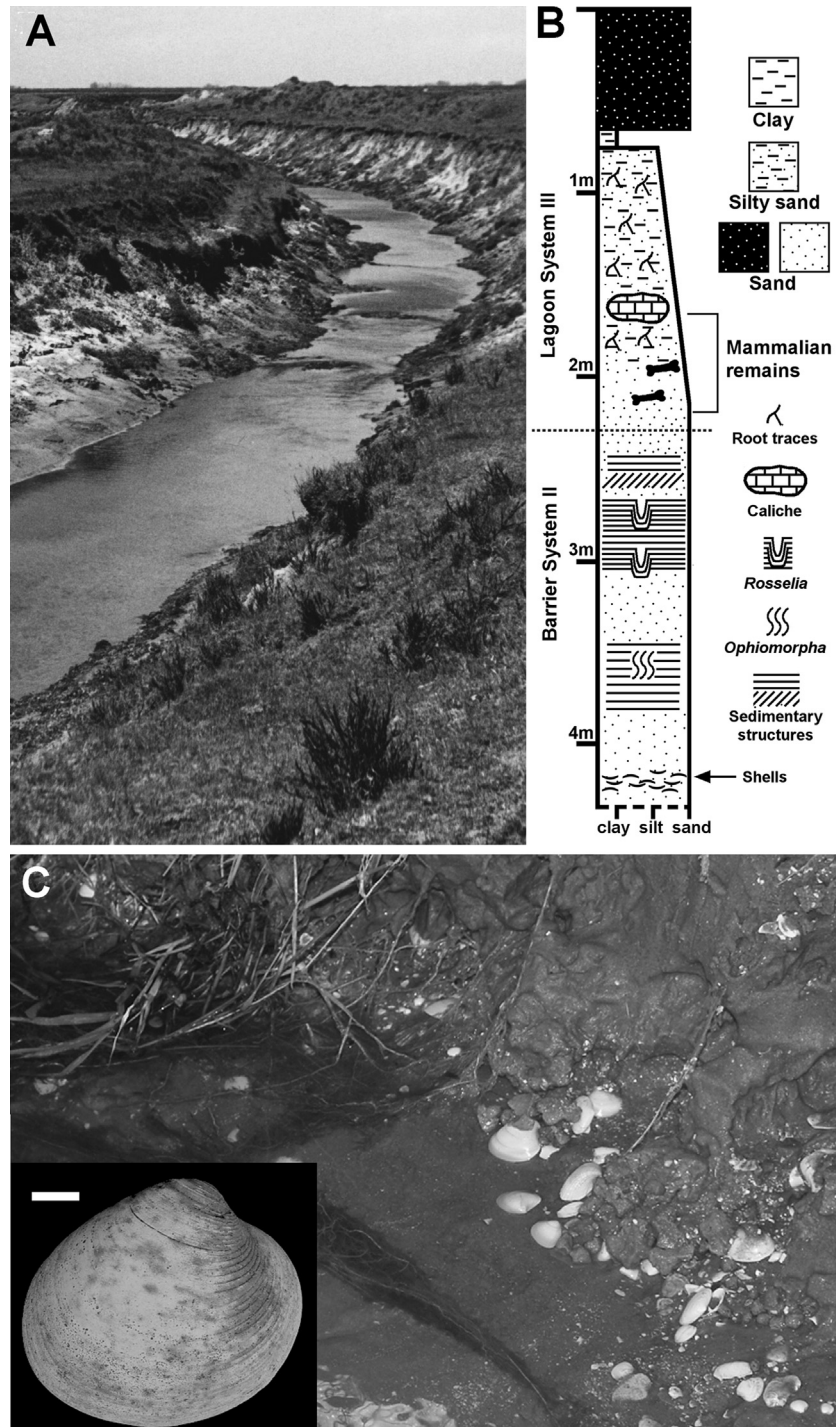


Fig. 2. A) panoramic view of the creek. B) stratigraphic sequence exposed along the banks of the creek, with indication of the position of the fossils. C) marine shells outcropping at the base of the left bank; on the insert, one specimen of *Amiantis purpuratus*.

3.2. Fossil shells

Shells were collected from three outcrops exposed at the banks of Chuí Creek (M001, M002 and M004). The three concentrations are at the same stratigraphic horizon, and seem to have been accumulated by a storm event, thus are considered as a single assemblage. Today, the concentrations are positioned at the level of the creek, thus are permanently saturated in water. Shell remains consisted mostly of bivalves, with few gastropods (Lopes and Simone, 2012). One major concern regarding the suitability of

these shells for dating is the fact that most of the bivalves are disarticulated. Schellmann and Radtke (1997) considered that only well-preserved, articulated (in living position) bivalves are useful for dates, but the only articulated specimens from Chuí Creek are six *Corbula caribaea*, a taxon that is too small to provide reliable results using ESR.

One sample of the sedimentary matrix surrounding the shells was collected from each outcrop; the sediments samples are homogeneous, consisting of well-selected, rounded mature siliciclastic sand. The presence of articulated bivalves and ostracod

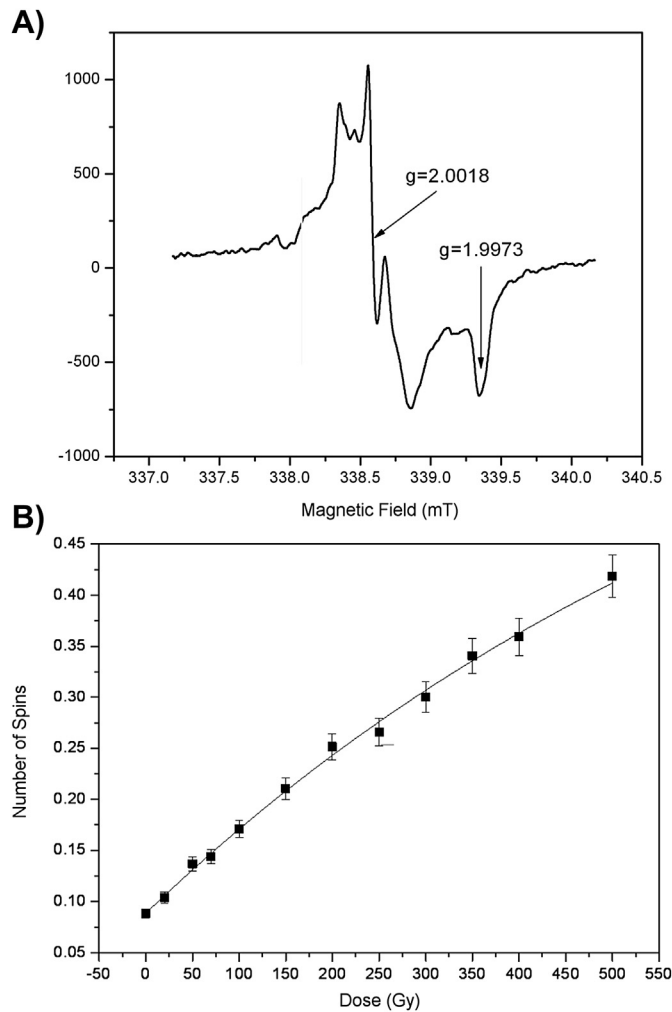


Fig. 3. (A) ESR spectrum of fossil shell irradiated with dose of 400Gy. The spectrum is composed and the signal of CO_2^- radical ($g_{\perp} = 2.0018$ and $g_{\parallel} = 1.9973$) is present. (B) Dose response curve (DRC) built by double integration of signal OF sample no.1 from outcrop M001. The fitting parameters are $D_e = 95 \pm 7$ Gy, $I_0 = 0.77 \pm 0.11$ and $D_0 = 770 \pm 160$ Gy.

carapaces indicates that the shells have not undergone reworking before being collected.

The dating of reworked shells could give a wide range of ages due to time-averaging (e.g. redeposition of older shells together with much younger material). As an attempt to minimize these problems, the taxon selected for dating was the bivalve *Amiantis purpuratus*, because it is abundant in all outcrops and is a shallow-living species, thus the probability of survival of the shells of dead individuals for more than 10^3 years is low, as pointed by studies of the rate of mechanical and chemical destruction of shells in marine habitats (Flessa and Kowalewski, 1994). Because of the higher rate of shell destruction in shallow areas, it is expected that well-preserved shells of taxa that inhabit these areas represent material that was quickly buried shortly after the individuals have died. Thus, the use of *A. purpuratus* would in theory avoid large uncertainties that could result from the larger time-averaging of species that inhabit deeper areas of the continental shelf, where chemical and mechanical destruction rates are lower (Flessa and Kowalewski, 1994). Besides, considering that the assemblage seems to have been formed by a short-termed depositional event and that the shells were covered by marine sediments and were not disturbed until the creek was excavated, they are probably reliable indicators of the time of deposition of the marine facies. The use of

a single taxon would also exclude variations related to interspecific physical differences, as pointed by other studies (Shimokawa et al., 1992; Brumby and Yoshida, 1994).

Five pristine, similar-sized and well-preserved (without signs of abrasion and/or bioerosion), disarticulated valves of *A. purpuratus* from each outcrop were selected for dating. The samples were subject to etching in a 1:10 HCl solution for 1 min for removal of the surface layers of the shells (Table 1). After drying, the shells were manually powdered with agate mortar and pestle; the sediment samples were also manually disaggregated. The resulting powder (<0.5 mm) was separated in subsamples and irradiated with additive doses up to 550Gy using a radioactive source with known energy (^{60}Co). Aliquots from the fifteen shells and sediment samples from each outcrop were subject to Neutron Activation Analysis (NAA) to determine ^{238}U , ^{232}Th and ^{40}K concentrations. The ESR spectra of subsamples were recorded using an X-band (9.5 GHz) JEOL FA-200 spectrometer. The acquisition parameters were: center field 338 mT, sweep width 1.5 mT, scan time 1 min, modulation frequency 100 kHz, modulation width 0.025 mT, time constant 100 ms, microwave power 1 mW. The value of the double integration signal was associated to the additive dose for obtaining the dose–response curve. As performed in the fossil teeth experiment, the dose–response curve was adjusted using the Equation (1) using instrumental weighing ($1/I^2$) (Grün and Brumby, 1994; Skinner, 2000).

Table 1

Thickness of the shell samples before and after the acid etching.

Outcrop	Sample	Before etching	After etching
M001	1	2.0 mm	1.4 mm
	2	1.4 mm	1.2 mm
	3	1.4 mm	1.2 mm
	4	1.2 mm	1.0 mm
	5	1.1 mm	0.7 mm
M002	1	2.2 mm	1.6 mm
	2	1.3 mm	1.0 mm
	3	1.1 mm	0.6 mm
	4	1.0 mm	0.7 mm
	5	1.3 mm	0.7 mm
M004	1	1.4 mm	1.0 mm
	2	1.1 mm	0.9 mm
	3	2.0 mm	1.6 mm
	4	1.2 mm	0.8 mm
	5	1.5 mm	1.2 mm

4. Results and discussion

4.1. Mammalian remains

In South America there are few Pleistocene fossil localities with both good stratigraphic control and numerical ages for the associated fossils. This had hampered the biostratigraphic correlations that have been based mostly on the Stage/Ages defined from the fossil beds of the Pampean region of Argentina (Cione and Tonni, 1999; Lopes, 2013; Lopes and Buchmann, 2010). The reworked nature of most fossiliferous units of fluvial origins indicate that numerical dates on fossil remains rather than on sediments from such deposits is the most reliable way of establishing biostratigraphic correlations.

Table 2 lists concentration of ^{238}U , ^{232}Th and ^{40}K in enamel, dentin and sediments. Table 3 lists the Equivalent Dose (D_e), early uptake (EU), linear uptake (LU) and combined uptake (CU) of the fossil teeth. The value of 190 $\mu\text{Gy}/\text{year}$ was found for cosmic radiation after corrections of latitude and longitude, altitude (Prescott and Hutton, 1994). The water content of the sediment was considered 10%, values of $^{234}\text{U}/^{238}\text{U} = 1.20 \pm 0.20$, $\alpha_{\text{eff}} = 0.13 \pm 0.02$

Table 2
²³⁸U, ²³²Th and ⁴⁰K contents (in ppm) of tooth enamel, dentin and sediments.

Sample	Enamel			Dentine		
	U (ppm)	Th (ppm)	K (%)	U (ppm)	Th (ppm)	K (%)
LGP-G0001	1.00 ± 0.03	n.d.	n.d.	82 ± 4	0.082 ± 0.005	<0.075
LGP-G0036	2.68 ± 0.08	n.d.	n.d.	104 ± 5	<0.01	<0.075
LGP-G0037	1.72 ± 0.05	n.d.	n.d.	150 ± 8	<0.01	<0.075
LGP-E0038	1.68 ± 0.05	n.d.	n.d.	109 ± 6	<0.01	<0.075
LGP-E0040	1.36 ± 0.04	n.d.	n.d.	118 ± 6	<0.01	<0.075
LGP-E0053	3.91 ± 0.11	n.d.	n.d.	155 ± 8	<0.01	<0.075
Sediment 1	2.2 ± 0.1	3.03 ± 0.09	0.63 ± 0.09			
Sediment 2	1.70 ± 0.09	3.23 ± 0.07	0.60 ± 0.07			
Sediment 3	2.2 ± 0.1	2.90 ± 0.09	0.64 ± 0.07			

Table 3
 Equivalent Dose (*D_e*), early uptake (EU), linear uptake (LU) and combined uptake (CU) ages (in thousands of years) of the fossil teeth.

Sample	<i>D_e</i> (Gy)	EU	LU	CU
LGP-G0001	39 ± 3	42 ± 3	50 ± 4	48 ± 4
LGP-G0036	90 ± 10	67 ± 9	90 ± 10	90 ± 10
LGP-G0037	54 ± 8	47 ± 6	60 ± 9	57 ± 8
LGP-E0038	100 ± 20	45 ± 8	70 ± 10	48 ± 9
LGP-E0040	130 ± 20	64 ± 8	90 ± 10	70 ± 10
LGP-E0053	102 ± 7	38 ± 3	59 ± 4	43 ± 3

Although some obtained ages are older than most of the previously published ages, they are in agreement with others presented by Lopes et al. (2010). These CU ages are also consistent with ages obtained for large mammal fossils from the Touro Passo Formation in the western Rio Grande do Sul state (Kerber et al., 2011), thus reinforcing the biostratigraphic correlation between both areas and also with those from the Sopas Formation of Uruguay (Ubilla et al., 2009).

The new ages, together with the results previously published by Lopes et al. (2010), also help to refine the chronology of the formation of the fossil mammalian assemblage from Chuí Creek. The ages indicate that most of the fossils represent the time interval between the end of MIS 5 and MIS 3 (roughly between 90 and 30 ka); the only specimen dated so far older than this interval is the aforementioned 226 ka-old tooth of *Toxodon*, which

represents MIS 7 (Fig. 4). These results in part fill the ~180 thousand years gap between the oldest fossil dated of 226 ± 35 ka and the next older of 42 ± 3 ka from Chuí Creek (Lopes et al., 2010). The relative absence of specimens older than 80 ka may be a result of successive reworking and destruction of the fossils from this location by fluvial action (Lopes, 2009; Lopes et al., 2009). The sedimentary record exposed along the banks of the creek shows evidences of humid and dry phases, linked to climate oscillations; it is likely that older fossils have a lower chance of being preserved because of successive reworking by episodes of fluvial reactivation. The co-occurrence in the same stratigraphic interval of fossils with such age variation corroborates the existence of several episodes of burial-exhumation-redeposition of most of the remains and associated sediments (Lopes, 2009).

The identification of the time interval during which these animals lived in southern Brazil may help to identify the causes that led to their disappearance in this area. Most of the specimens are chronocorrelated to intervals of milder climates, mostly the MIS 3 interstadial (Fig. 4), a relatively warm interval marked by warm (and cold) pulses in both hemispheres caused by Dansgaard-Oeschger (and Heinrich) events (EPICA Community Members, 2006; Rabassa and Ponce, 2013). This pattern suggests that the late Pleistocene climatic oscillations affected the faunal dynamics of the large mammal community that inhabited the area. Several studies have pointed that the interval between ~50 and ~18 ka in south and southeastern Brazil and the Argentinean Pampas was marked by intercalated dry/humid and cold/warm conditions, followed by very dry conditions during the Last Glacial Maximum (LGM), between ~25 and ~11 ka (Ledru, 1993; Behling and Lichte, 1997; Turcq et al., 1997; Carignano, 1999; Iriondo, 1999). Other geological records show that during the pre-LGM interval the environmental changes in southern Brazil were also influenced by insolation-driven millennial-scale oscillations that occurred in the Northern Hemisphere and affected the patterns of precipitation (Cruz jr et al., 2005; Wang et al., 2007).

4.2. Shells

Fig. 3 shows the dose response curve for sample 1 from outcrop M001. The equivalent dose (*D_e*) was determined by fitting with exponential function (Ikeya, 1993) (Equation (1)).

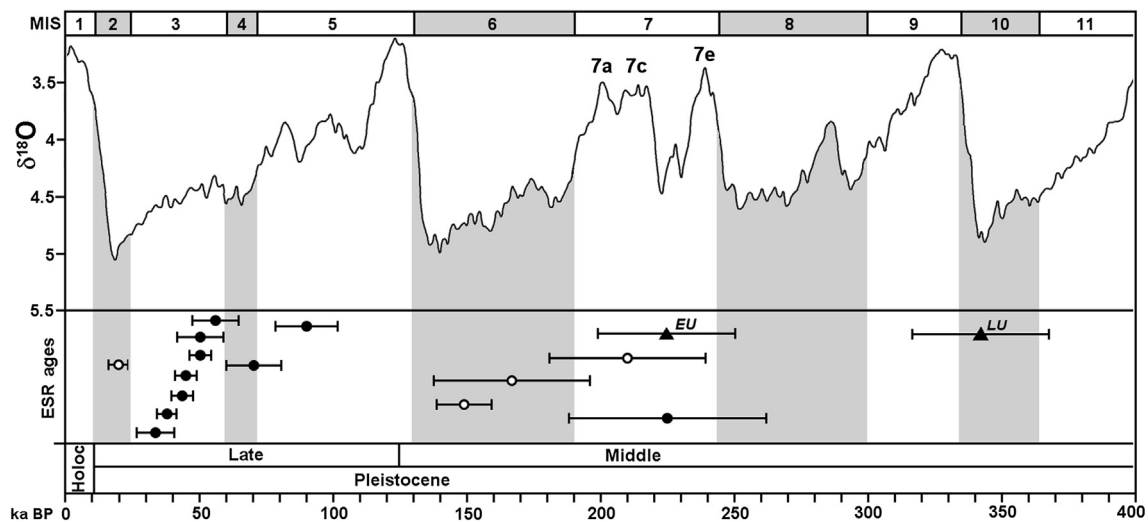


Fig. 4. Correlation of the marine isotope stages (MIS) and oxygen isotope curve (=ice volume) from Atlantic Ocean core samples (Lisiecki and Raymo, 2009) with the ages of the mammalian remains (black circles) and EU/LU ages of the shells (triangles) from the Chuí Creek. Open circles indicate the ages of mammalian fossils from the continental shelf (Lopes et al., 2010).

Table 4 lists the concentration of ^{238}U , ^{232}Th and ^{40}K of shells and sediments that were used to convert D_e in ages using the software DATA (Grün, 2009a). The value of $116 \mu\text{Gy}/\text{year}$ was found for cosmic radiation after corrections of latitude and longitude, altitude ($\sim 7 \text{ m a.s.l.}$) and depth ($\sim 4.5 \text{ m}$). The water content of the sediment was considered 100%, because the water table is at the same level of the shells since the creek was excavated in the early 1960s. Also for the age calculations the values of $^{234}\text{U}/^{238}\text{U} = 1.20 \pm 0.20$, $\alpha_{\text{eff}} = 0.10 \pm 0.02$ and density = $2.71 \pm 0.02 \text{ g}/\text{cm}^{-3}$ were adopted following the literature (Blackwell et al., 2010).

Table 4
 ^{238}U , ^{232}Th and ^{40}K content of shells and sediment samples.

Outcrop	Sample	U (ppm)	Th (ppm)	K (%)
M001	1	0.28 ± 0.02	<0.01	<0.075
	2	0.47 ± 0.02	<0.01	<0.075
	3	0.55 ± 0.03	<0.01	<0.075
	4	1.35 ± 0.03	<0.01	<0.075
	5	1.07 ± 0.03	<0.01	<0.075
	Sediment	0.39 ± 0.02	1.31 ± 0.1	0.91 ± 0.07
M002	1	1.91 ± 0.04	<0.01	<0.075
	2	0.47 ± 0.02	<0.01	<0.075
	3	1.73 ± 0.05	<0.01	<0.075
	4	1.34 ± 0.05	<0.01	<0.075
	5	0.55 ± 0.08	<0.01	<0.075
	Sediment	0.41 ± 0.04	1.60 ± 0.1	0.82 ± 0.2
M004	1	1.45 ± 0.06	<0.01	<0.075
	2	1.47 ± 0.06	<0.01	<0.075
	3	1.23 ± 0.04	<0.01	<0.075
	4	1.21 ± 0.08	<0.01	<0.075
	5	1.37 ± 0.05	<0.01	<0.075
	Sediment	0.5 ± 0.1	1.4 ± 0.2	0.9 ± 0.3

The calculated early uptake (EU) and linear uptake (LU) ages for the shells are listed in Table 5. Because the oldest dated tooth from the fluvial facies above the marine sediments was dated of $226 \pm 35 \text{ ka}$ (Lopes et al., 2010), it was expected that all shells were older than this; the results however, show a wide variation. The ages obtained through the EU model range from $140 \pm 40 \text{ ka}$ to $430 \pm 30 \text{ ka}$; the average ages are $266 \pm 30 \text{ ka}$ for the shells from M001, $222 \pm 26 \text{ ka}$ for M002, and $184 \pm 18 \text{ ka}$ for M004. The mean EU age for the fifteen specimens is of $224 \pm 24.6 \text{ ka}$ and corresponds to the average of the minimum age of shells (Ikeya, 1993; Blackwell, 1995). The LU ages range from $570 \pm 20 \text{ ka}$ to $230 \pm 30 \text{ ka}$, with a mean value of $340 \pm 31 \text{ ka}$.

Table 5
Accumulated dose, early and linear uptake ages (in thousands of years) of the fossil shells.

Outcrop	Sample	D_e	Age (E.U.)	Age (L.U.)
M001	1	95 ± 7	430 ± 30	570 ± 20
	2	75 ± 7	280 ± 30	390 ± 20
	3	90 ± 10	300 ± 30	440 ± 20
	4	67 ± 17	140 ± 40	230 ± 40
	5	73 ± 9	180 ± 20	280 ± 20
	Mean		266 ± 30	296 ± 24
M002	1	97 ± 8	150 ± 10	250 ± 20
	2	84 ± 9	300 ± 30	440 ± 40
	3	79 ± 12	140 ± 20	230 ± 30
	4	90 ± 11	180 ± 20	300 ± 40
	5	104 ± 14	340 ± 50	500 ± 70
	Mean		222 ± 26	344 ± 40
M004	1	92 ± 13	170 ± 20	280 ± 40
	2	87 ± 8	170 ± 20	270 ± 20
	3	89 ± 10	190 ± 20	300 ± 30
	4	91 ± 5	190 ± 10	310 ± 20
	5	104 ± 11	200 ± 20	320 ± 34
	Mean		184 ± 18	382 ± 28.8
Total mean age			224 ± 24.6	340 ± 31.0

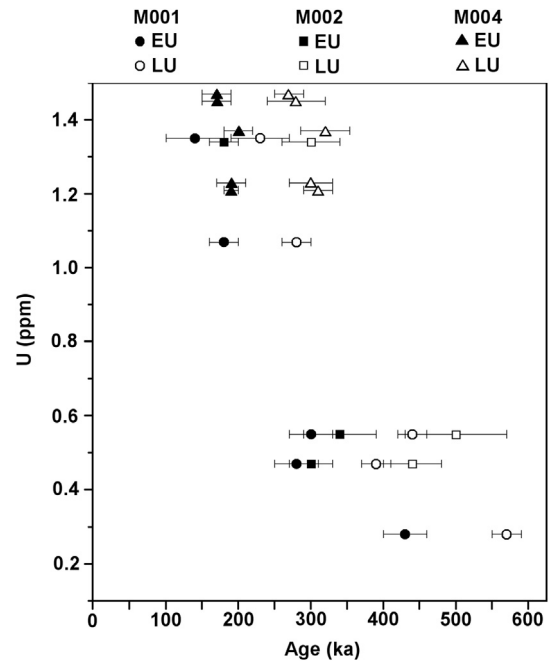


Fig. 5. Relationships between uranium concentration and EU/LU ages of the shell samples.

Differences of approximately 120 ka between EU and LU models due to uranium uptake modeling were also observed by Schellmann and Radtke (1999), indicating how sensitive this information can be to the age obtained, thus the results shall be considered as minimum ages, unless it is assumed that the enrichment has occurred shortly after the burial of the material (Radtke et al., 1985). Because Early Holocene shells already display uranium concentrations equivalent to those found in shells from the Last or Penultimate Interglacial, the most reliable ages are those obtained by the EU model (Schellmann and Radtke, 1997, 1999, 2000). The amount of uranium found in the fifteen shells was relatively low, ranging from 0.28 to 1.91 ppm (1.09 ppm on average), thus suggesting that the U-uptake occurred shortly after the shells were deposited; recent shells exhibit uranium concentrations around 0.2 ppm (Schellmann and Radtke, 1999). Nevertheless, the specimens with higher U-concentrations are also the ones that exhibit the younger ages (Fig. 5); as already mentioned, EU ages are the youngest possible (Blackwell, 1995). The coupled ESR-US method provides results of age independently of uranium uptake, but as in this study the US determination was not available, so the entire discussion here is based on the EU ages, taking into account that these are probably younger than the 'real' ages of the samples.

The U-uptake may be related to the thickness (higher concentrations are usually found in thinner shells), the type of surrounding matrix (higher in sandy sediments) and the mobility of the interstitial water (Shimokawa et al., 1992). The sedimentary matrix in which the shells are embedded is composed of fine sand; the shells themselves were progressively buried beneath a $\sim 4.5 \text{ m}$ -thick sedimentary cover, and although it is not clear how much the water table varied since their burial, it is probable that they remained below the water table until the creek was excavated in the early 1960s. Today, the shells are at the mean level of the creek, constantly saturated in water (Fig. 2D).

On the other hand, some shells exhibit very low uranium concentrations, and conversely are the ones with older ages. This pattern needs further investigation, but may indicate that these shells were reworked from older deposits or that there was some

diagenetic alteration on these specimens, which interfered in the uranium uptake. Considering that the assemblage is composed mostly of shallow-living molluscs accumulated in the upper shoreface, with many specimens well-preserved, including small and fragile juveniles, it seems unlikely that the large age discrepancies represent time-averaging, because the shells would not have withstood the mechanical destruction for such a long time in this high-energy environment (Flessa and Kowalewski, 1994). Large time-averagings are expected to be found in transgressive lag deposits that are formed on the shelf during marine transgressions, as those found in the continental shelf today (Figueiredo, 1975).

The ages of the shells reinforce the interpretation that the marine facies of the Barrier II is older than 226-thousand years, based on a tooth from the overlying fossiliferous bed (Lopes et al., 2010). Despite of the large ages difference of the shells, the results seem consistent with TL ages obtained for marine sediments collected about 1 m above the shells (Lopes, unpublished data). Given the time interval indicated by the EU ($\sim 224 \pm 24.6$ ka) and the LU ($\sim 340 \pm 31$ ka) models, and that the EU ages are the youngest possible, these shall be considered younger than the 'real' ages due to excessive U-uptake. An estimated age closer to ~ 234 – 238 ka, therefore within the error margin of the EU model, seems reasonable and also agrees with the interglacial peak of the marine isotope substage 7e, according to oxygen isotope curves for the Atlantic (Lisiecki and Raymo, 2009, Fig. 4). The interval between ~ 238 and 234 ka, within the substage 7e, is considered a hypsithermal, marked by warmer ocean temperatures than at present in the southern Atlantic (Robinson et al., 2002; Becquey and Gersonde, 2002, 2003). The shell assemblage from Chui Creek also points to oceanographic conditions warmer than at present, indicated by the presence of the bivalves *Anadara brasiliana*, *Anomalocardia brasiliana*, *Chione cancellata* and *Chione paphia*, which today are found living only in areas of the Brazilian coast to the north of Rio Grande do Sul state, and also by foraminifers found together with the shells (Lopes and Bonetti, 2012; Lopes et al., 2012).

Kanfoush et al. (2002) and Becquey and Gersonde (2002, 2003) reconstructed past oceanographic conditions from the Atlantic sector of the Southern Ocean and concluded that the early phases of the last four interglacials were hypsithermals, characterized by higher temperatures in comparison to the present. Thus, the presence of warm-water bivalves in the coastal waters of southern Brazil around ~ 234 – 238 ka seems consistent with the hypsithermal recorded at the beginning of MIS 7 (substage 7e). A hypsithermal was also identified during the late MIS 7 (substage 7a) around 200 ka, which would fall within the lower error margin of the EU ages; nevertheless, one tooth of *Notiomastodon* from the continental shelf dated as of 207 ± 28 ka (Lopes et al., 2010) indicates that sea-level had already retreated around 200 ka.

On the other hand, an LU age around ~ 340 ka would not be consistent with a sea-level highstand, considering that at this time sea-level was more than 120 m below the present due to the glacial maximum of MIS 10 (Rohling et al., 1998). Nevertheless, an age closer to ~ 325 ka, within the lower error margin, would be consistent with the marine transgression of MIS 9.

4.3. Stratigraphic implications

The ages presented here increase the available ages for the deposits of the CPRS (Table 6), and help refine the sea-level curves for the coast of Rio Grande do Sul state presented by Lopes et al. (2010) (Fig. 6). Because of the scarcity of datable indicators of past sea-levels, mainly for the Pleistocene deposits, the exact timing of the sea-level highstands that formed the barrier-lagoon depositional systems have been mostly unknown. Based on the oxygen curves of Imbrie et al. (1984), Villwock and Tomazelli (1995) correlated the

Barrier-Lagoon System II with the marine highstand of MIS 9, while the ages presented here suggest instead that the Barrier System II was formed by the marine transgression correlated to MIS 7. That leaves open the question of whether the oldest system (Barrier-Lagoon I) is correlated to MIS 9 or if it may represent an even older marine highstand.

Table 6

Available ages for the Pleistocene-Holocene depositional systems of the CPRS.

Ages (years BP)	Material	Method	Unit	Author
960	Sediments	TL	Barrier IV	Poupeau et al., 1988
1222	Sediments	TL	Barrier IV	Poupeau et al., 1988
2470 ± 60	Peat	14C	Lagoon IV	Buchmann et al., 1998
2567	Sediments	TL	Barrier IV	Poupeau et al., 1988
2667	Sediments	TL	Barrier IV	Poupeau et al., 1988
3250	Sediments	TL	Barrier IV	Poupeau et al., 1988
3833	Sediments	TL	Barrier IV	Poupeau et al., 1988
4330 ± 60	Shells	¹⁴ C	Lagoon IV	Poupeau et al., 1988
4920 ± 110	Shells	¹⁴ C	Lagoon IV	Buchmann et al., 1998
5045	Shells	¹⁴ C	Lagoon IV	Forti Esteves, 1974
5750 ± 750	Shells	¹⁴ C	Lagoon IV	Caron, 2007
6820	Shells	¹⁴ C	Lagoon IV	Lima et al., 2013
6870	Shells	¹⁴ C	Lagoon IV	Lima et al., 2013
10,160	Peat	¹⁴ C	Barrier III	Lima et al., 2013
16,250 ± 1670	Shells	¹⁴ C	Continental shelf	Figueiredo, 1975
17,000 ± 340	Shells	¹⁴ C	Continental shelf	Figueiredo, 1975
18,000 ± 3000	Mammalian fossils	ESR	Continental shelf	Lopes et al., 2010
>30,000	Shells	¹⁴ C	Continental shelf	Figueiredo, 1975
34,000 ± 7000	Mammalian fossils	ESR	Lagoon III	Lopes et al., 2010
38,000 ± 2000	Mammalian fossils	ESR	Lagoon III	Lopes et al., 2010
>38,000	Peat	¹⁴ C	Lagoon III?	Buchmann et al., 1998
42,000 ± 3000	Mammalian fossils	ESR	Lagoon III	Lopes et al., 2010
42,260 ± 1400	Shells	¹⁴ C	Lagoon IV	Lima et al., 2013
43,000 ± 3000	Mammalian fossils	ESR	Lagoon III	This paper
48,000 ± 4000	Mammalian fossils	ESR	Lagoon III	This paper
48,000 ± 9000	Mammalian fossils	ESR	Lagoon III	This paper
57,000 ± 8000	Mammalian fossils	ESR	Lagoon III	This paper
70,000 ± 10,000	Mammalian fossils	ESR	Lagoon III	This paper
90,000 ± 10,000	Mammalian fossils	ESR	Lagoon III	This paper
74,840	Sediments	TL	Barrier III	Poupeau et al., 1988
85,075	Sediments	TL	Barrier III	Poupeau et al., 1988
86,440	Sediments	TL	Barrier III	Poupeau et al., 1988
109,000 ± 7,5	Sediments	TL	Barrier III	Buchmann and Tomazelli, 2003
146,000 ± 9000	Mammalian fossils	ESR	Continental shelf	Lopes et al., 2010
165,000 ± 28,000	Mammalian fossils	ESR	Continental shelf	Lopes et al., 2010
207,000 ± 28,000	Mammalian fossils	ESR	Continental shelf	Lopes et al., 2010
224 ± 24.6	Shells	ESR	Barrier II	This paper
226,000 ± 35,000	Mammalian fossils	ESR	Lagoon III	Lopes et al., 2010
428,000 ± 30,000	Mammalian fossils	ESR	Continental shelf	Lopes et al., 2010
464,000 ± 65,000	Mammalian fossils	ESR	Continental shelf	Lopes et al., 2010
650,000 ± 100,000	Mammalian fossils	ESR	Continental shelf	Lopes et al., 2010

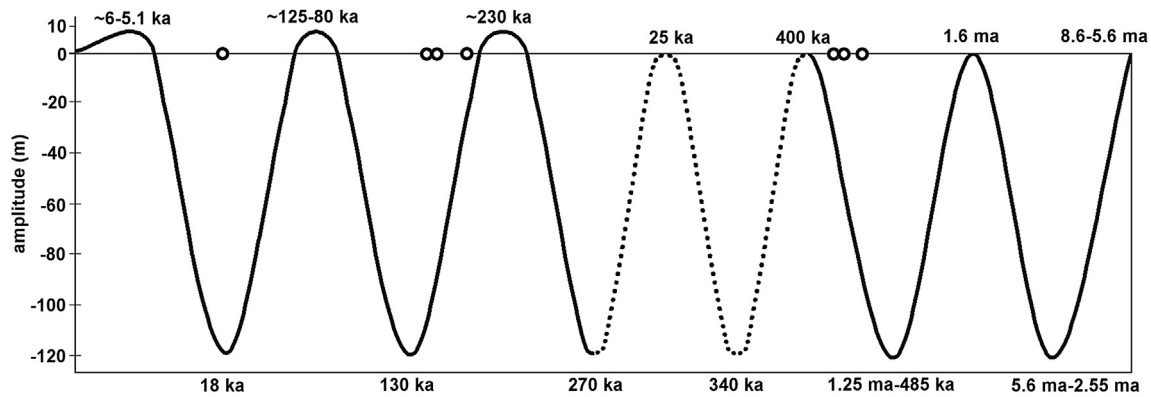


Fig. 6. Simplified scheme of sea level oscillations in southern Brazil between Late Miocene and Holocene. Oscillations older than 400 ka are indicated by ostracode assemblages from drilling cores (Carreño et al., 1999), while the three last highstands are represented by geomorphological features and fossils (Villwock and Tomazelli, 1995; Caron, 2007 and this paper). The dashed lines indicate inferred oscillations (from Rohling et al., 1998); open circles indicate mammalian fossils from the continental shelf dated by ESR (modified from Lopes et al., 2010).

The ~125 ka-old marine transgression that formed the Barrier System III, correlated to MIS 5 and called Cananéia Transgression or Penultimate Transgression, is relatively well documented along the Brazilian coast (Bittencourt et al., 1983; Suguio et al., 1986), but deposits formed by older transgressions are scarce. In the north-eastern Brazilian coast, several raised marine terraces of a unit called Barra de Tabatinga Formation have yielded TL and optical stimulated luminescence (OSL) ages of ~210–220 thousand years (Barreto et al., 2002; Suguio et al., 2011). The similar ages suggest that the Barrier System II and the Barra de Tabatinga Formation have been formed by the same transgressive event. The presence of chronocorrelated transgressive marine deposits in two distant areas (more than 3000 km from each other) indicates that there are probably other similar deposits along the Brazilian coast that have not yet been identified due to lack of numerical dates.

5. Conclusions

The new ESR dates confirm a Late Pleistocene age for most of the mammalian remains found in Chuí Creek and their correlation with similar faunas found in deposits from Northern Uruguay and Western Rio Grande do Sul state. These new results help to put this assemblage in a more precise time interval, which should contribute to biostratigraphic correlations with other localities and for the assessment of the causes that led to the disappearance of the large mammals in the area. One hypothesis is that the progressive increase in aridity after ~34 ky BP, correlated to the LGM, may have played a role in their (pseudo)extinction. These ages will also help to reconstruct and understand the evolution of the terrestrial environments in the southern CPRS during the late Quaternary.

The mean EU age of ~224 ka obtained from the fossil shells seem to be in agreement with other unpublished dates and provide an estimate for the timing of the marine transgression that formed the Barrier System II. These results indicate a Late Middle Pleistocene age; considering that 224 ka is the youngest possible age, and that the younger shells are probably younger than the 'real' age due to excessive Uranium uptake, an estimated age of ~234–238 ka for the shells seems closer to the 'real' ages and would be consistent with the marine highstand during early MIS 7 (substage 7e). This time interval was characterized by higher ocean temperatures, related to the hypsithermal phase at the beginning of the interglacial; such conditions are indicated by the presence in the fossil assemblage of bivalves that today live only in warmer areas to the north of Rio Grande do Sul state. Although the LU values suggest a mean age of

340 ± 31 ka for the shells, the higher reliability of the EU model and the correlation with other yet unpublished ages suggest that the EU values are closer to the real ages than the LU values.

These results help to refine and improve the knowledge regarding the chronostratigraphy and sea-level variations for the Coastal Plain of Rio Grande do Sul state, showing that the maximum amplitude of the marine highstand that formed the Barrier System II was reached around 230 ka. The ages also indicate chronological correlation of this depositional system with other marine deposits from northeastern Brazil. This correlation among two distant areas indicates that other depositional systems formed by this marine transgression are likely to be found in other parts of the Brazilian coast. ESR dates on shells, in conjunction with other methods and datable materials, are a potential tool for refining the chronology of the middle to late Quaternary deposits along the Brazilian coast.

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