

## MARINE RESERVOIR CORRECTIONS ON THE SOUTHEASTERN COAST OF BRAZIL: PAIRED SAMPLES FROM THE SAQUAREMA SHELLMOUND

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**ABSTRACT.** The Saquarema archaeological site, on the Atlantic coast of the Rio de Janeiro State, is one of many shell-mounds built on the Brazilian coast by hunter-gatherer populations during the Holocene. We used archaeological material from this site with the aim of evaluating the marine reservoir effect (MRE) in the region. Radiocarbon ages of 45 marine and 6 terrestrial samples from this shellmound provided data for assessing the MRE and the influences of freshwater and seasonal coastal marine upwelling in this specific locality. Samples of charcoal, fish otoliths, and mollusk shells were analyzed and the <sup>14</sup>C dates were modeled in the OxCal platform to determine the marine reservoir correction. The result obtained is  $R = 265 \pm 70$  <sup>14</sup>C yr and the offset  $\Delta R$  was found to be  $-140 \pm 66$  <sup>14</sup>C yr. To support the accuracy of this value for correcting conventional <sup>14</sup>C marine ages, taxonomic analyses of the samples were performed.

### INTRODUCTION

For many decades, the marine radiocarbon reservoir effect and its variability have been challenging researchers working on the Brazilian coast. Attempts to search for accurate contributions in this field (Nadal de Masi 2001; Reimer and Reimer 2001; Eastoe et al. 2002; Angulo et al. 2005, 2007) have been conducted using different approaches to calculate the regional  $\Delta R$  values. However, more studies are needed as the complexity of regional ocean dynamics and the lack of available data make it difficult to assess the uncertainties and reliability of the <sup>14</sup>C dates obtained on marine material from the western South Atlantic coast.

The MRE is the offset in <sup>14</sup>C age between marine and atmospheric (terrestrial) samples (Stuiver et al. 1986). This offset must be quantified in order to correctly translate a <sup>14</sup>C age of marine-influenced material into a calendrical timescale. Currently, the global average marine reservoir age for surface water is  $405 \pm 22$  <sup>14</sup>C yr (Hughen et al. 2004). However,  $\Delta R$  deviations from this mean occur as a function of climate and ocean circulation (Gordon and Greengrove 1986; Ascough et al. 2004). The idea that <sup>14</sup>C dates obtained from marine and terrestrial samples are not directly comparable has been recognized for a long time (Stuiver and Ostlund 1980). Nevertheless, the variability of MRE in some regions of the Brazilian coast is still poorly or even totally unexplored. Therefore, this work aims to address the need for coastal regional MRE  $\Delta R$  values from Brazil.

A  $\Delta R$  value is calculated using samples of marine-derived carbon for which the terrestrial/atmospheric <sup>14</sup>C age is known, or can be determined with a high degree of assurance (Russell 2011). There are different methodological approaches of assessing MRE through the quantification of  $\Delta R$  values for geographical locations worldwide (Ascough et al. 2005). They comprise measurements of known-age (pre-1950s) marine samples from museum collections or paired terrestrial/marine samples from secure archaeological contexts.

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Taking advantage of the profuse amount of shellmounds on the Brazilian coastline built up by ancient cultures during the Holocene, the use of the paired-samples approach is very convenient. In these archaeological sites, marine shell and charcoal remains are ubiquitous, allowing comparison between the marine and the atmospheric reservoirs, and thus the study of MRE in the locality.

This article reports data obtained from material discovered at an archaeological shellmound at Saquarema (22°55'S, 42°30'W), on the Atlantic coast of Rio de Janeiro State (Figure 1).  $^{14}\text{C}$  dating of fish otoliths, marine shells, and charcoal fragments was performed and a regional correction for MRE was calculated. Anthracological analyses were also employed to discard uncertainties caused by the inbuilt age of the wood (McFadgen 1982).

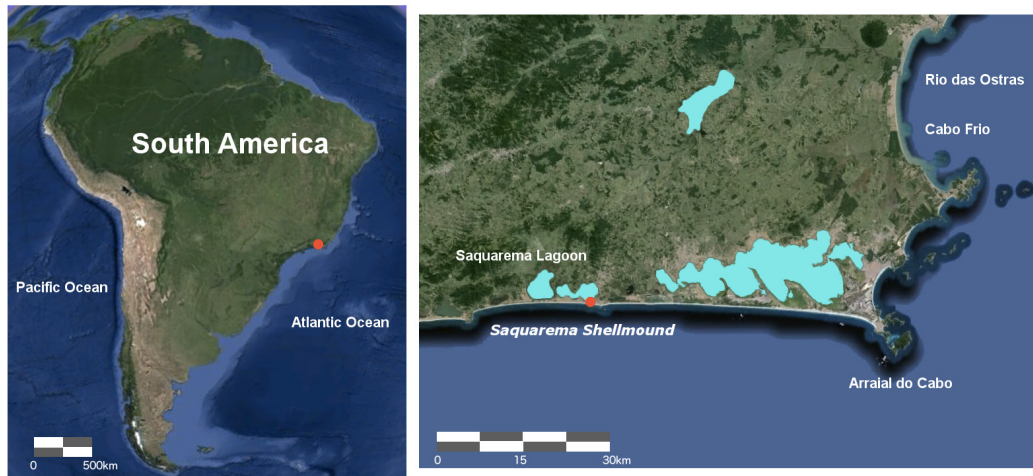


Figure 1 Map showing the studied region on the southeastern coast of Brazil

## MATERIALS AND METHODS

The Saquarema shellmound was first registered by Simões da Silva (1932). Since then, it has been partially destroyed. Mollusk shells from the sandy matrix were removed to serve as pavement for urban streets and roads. During an excavation performed in 1993, three layers of archaeological settlement, labeled I, II, and III (Figure 2), were identified.

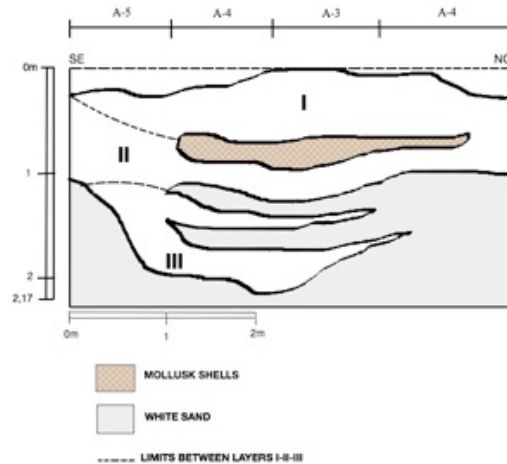


Figure 2 Schematic section of sector A at the Saquarema shellmound (modified from Kneip 1995).

Another excavation, on a larger scale, was conducted in 1994 (Kneip 1995). At that time, two  $^{14}\text{C}$  ages were obtained for this shellmound,  $2550 \pm 60$  BP (GX-20512) for layer II and  $3280 \pm 60$  BP (GX-20513) for layer III. Regarding the archaeostratigraphy of this site, Kneip (1995) argues that the large set of archaeological remains makes the separation of individual layers a difficult task. Furthermore, the author describes the vertical profile of the site as containing smashed and grouped food remains, layers of carbonized material and combustion structures, artifacts both grouped and isolated, soil with diverse coloration—disturbed by human action, animals, or even plants—or with signs of weathering processes. The limits of human occupation were difficult to clarify and combustion structures were used as markers for the beginning and the ending of such occupation levels.

The set of samples used in this study was obtained from the National Museum of Brazil, from the Universidade Federal do Rio de Janeiro (MN/UFRJ) repository, and came from an excavation performed in sector B of the Saquarema Shellmound in the 1960s. The archaeological methods adopted in this excavation were not well documented, but sample labels were available. The samples comprise charcoal fragments, shells, and fish otoliths, collected from several stratigraphic layers and labeled according to depth, material, and collection date.

Samples underwent taxonomic analyses in order to expand the comprehension and interpretation of the dating results. For MRE studies, it is extremely important to know the habitat and dietary preferences of the shellfishes and fishes dated, since the use of sedentary species has been widely recommended by the  $^{14}\text{C}$  community. This is especially important to make sure that the  $\Delta R$  values obtained are representative for only one locality (Ascough et al. 2005). Moreover, marine shells are not necessarily in equilibrium with the ocean water (Barrett et al. 2000). Tanaka et al. (1986) showed that up to 50% of the carbonate present in the shell could be derived from metabolic sources, which requires particular care when dealing with organisms that feed in calcareous strata (e.g. certain species of gastropods). The shells used in this study were subjected to malacological analyses of their structures with the support of specialized literature (e.g. de Souza et al. 2011), and were compared to a reference collection. Shells of the gastropod *Neritina virginea* (Linnaeus, 1758) and the suspension-feeder bivalve *Anomalocardia brasiliensis* (Gmelin, 1791) were identified. They are both geographically widespread (Abbott 1974; Rios 1994) and usually inhabit environments protected from wave action, such as mangroves, estuaries, muddy beaches, and intertidal zones (Boehs and Magalhães 2004; de Souza et al. 2011). A similar protocol was employed for the otoliths; samples from *Micropogonias furnieri* (Desmarest, 1823) species, which represents the primary fish target of the prehistoric communities in the region (Kneip 2001), were selected. This is a plentiful species in the Brazilian coast, living near the coastline up to 60 m depth (Fischer et al. 2011; Froese and Pauly 2014). Juveniles often use estuarine environments as growth and feeding zones (Vieira et al. 1998).

Regarding charcoal samples, the taxonomic classification is essential to avoid their primary source of error, the “inbuilt” age (McFadgen 1982). This may arise due to dating of the dead wood in the center of a living tree or due to the time elapsed between the collection of the wood and its use by people, known as storage age (McFadgen 1982). The so-called old-wood effect is increased when one deals with long-lived species (e.g. oak), leading to errors of up to hundreds of years. Charcoal pieces were manually broken, exposing transverse, tangential longitudinal and tangential radial sections, which allowed wood anatomical investigation. Sections were examined with a reflected light brightfield/darkfield microscope and identification was achieved through the comparison to a reference collection (Antracoteca/Charcoal collection from the National Museum of Brazil, UFRJ) and the use of specialized literature (e.g. Metcalfe and Chalke 1950). Species from the families Anacardiaceae, Rutaceae, and Sapotaceae were identified in the set of samples. They represent sandbank vegetation, which is not usually composed of long-lived trees. Anacardiaceae presents a

tropical distribution, including roughly 70 species in Brazil. They are often fructiferous trees (e.g. *Anacardium occidentale*) and yield quality wood (e.g. *Schinopsis brasiliensis*). Rutaceae, in turn, exhibits a predominantly pantropical distribution, with about 150 species occurring in Brazil. They are bushes or trees, often thorny. In this study, a sample of Rutaceae *Metrodorea* sp. was identified. *Metrodorea* is a native Brazilian genus commonly found in seasonal forests. Sapotaceae also presents a pantropical distribution, with nearly 200 species occurring in Brazil. They are lactescent bushes or trees, including many fructiferous trees (e.g. *Pouteria* spp.) and quality wood species (e.g. *Manilkara* spp.). A wood knot and a tuber fragment were also recognized.

Samples were prepared and analyzed at the Radiocarbon Laboratory of the Universidade Federal Fluminense (LAC/UFF). For charcoal samples, an acid-base-acid (ABA) treatment was employed with 1.0M hydrochloric acid (HCl) (2 hr at 90°C) and 1.0M sodium hydroxide (NaOH) (1 hr at 90°C). Pretreated organic samples were combusted in prebaked quartz tubes containing silver powder and cupric oxide at 900°C for 3 hr in a muffle oven. Shell and otolith samples were chemically treated with 0.5M HCl to remove the outer layer, which could be contaminated. Phosphoric acid ( $\text{H}_3\text{PO}_4$ ) was injected with a gas-tight syringe into evacuated vials to obtain  $\text{CO}_2$ . Calcite blanks and IAEA C2 carbonate were prepared as control samples. Chemistry blanks used were optical calcite and reactor graphite and combustion blank was reactor graphite. The gas was purified by means of dry ice/ethanol traps in the graphitization line (Macario et al. 2013). Graphitization was performed using the zinc/titanium hydrate method with iron catalyst (Xu et al. 2007). Individual torch-sealed tubes were heated at 520°C for 7 hr in a muffle oven. Graphitized samples were pressed in each of the 40 cathodes in the wheel of the SNICS ion source and measured in a NEC 250kV Single Stage Accelerator System (SSAMS). The isotopic fractionation was corrected by measuring the  $\delta^{13}\text{C}$  on-line in the accelerator. Background was measured using processed calcite blanks for carbonate samples and processed graphite for organic samples. Graphite and calcite processed blanks yielded average  $^{14}\text{C}/^{13}\text{C}$  ratios of  $6 \times 10^{-13}$  and  $7 \times 10^{-13}$ , respectively. Average machine background was  $10^{-13}$  for unprocessed graphite. Accuracy was checked by measuring reference materials within the  $2\sigma$  range of consensus values. Calibration was performed with the software OxCal v 4.2.4 (Bronk Ramsey and Lee 2013) using the Marine13 curve (Reimer et al. 2013) with an undetermined offset (Delta\_R(“Saquarema”,U(-600,600))) to account for local corrections for carbonate samples and the atmospheric curve SHCal13 (Hogg et al. 2013) for charcoal samples.

## RESULTS AND DISCUSSION

From the results presented in Table 1, we notice that the  $^{14}\text{C}$  ages of marine samples vary from around 3600 to 4100 BP. Such fluctuations could be explained by the temporal range of the archaeological occupation or by the simple statistical dispersion of the results, taking into account that the experimental uncertainty reflects only the precision in the isotopic concentration determination. Figure 3 shows the distribution of  $^{14}\text{C}$  ages with burial depth for each sample analyzed. A weak correlation between ages and depth was observed [correlation coefficient (Pearson's  $r$ ) = 0.22,  $p > 0.05$ ]; thus, even if the samples were deposited in a chronological order, the statistical dispersion would be larger than any temporal difference between them. There still exists the possibility of mixing of the material from different layers due to funerary burials and other activities within the archaeological site.

There is no distinguishable pattern for the distribution according to sample material or species. The entire set of results from carbonate samples including the mollusk shells and fish otoliths is displayed in Figure 4 and follows a normal distribution (Shapiro-Wilk test,  $p = 0.944$ ) with a mean value of 3791 BP and a standard deviation of 128  $^{14}\text{C}$  yr. As expected, charcoal results were always younger than the respective marine ages from similar depths. The charcoal determinations show

Table 1 Dates obtained from the set of samples.

Lab ID (LACUFF-)	Material	Family/Genus/Species	Depth (cm)	<sup>14</sup> C age (BP)
140466	Shell	<i>Anomalocardia brasiliiana</i>	10–20	3682 ± 42
140467	Shell	<i>Anomalocardia brasiliiana</i>	10–20	3779 ± 81
140468	Shell	<i>Anomalocardia brasiliiana</i>	20–30	3654 ± 44
140469	Shell	<i>Anomalocardia brasiliiana</i>	20–30	3842 ± 46
140470	Shell	<i>Anomalocardia brasiliiana</i>	20–30	3658 ± 79
140471	Shell	<i>Anomalocardia brasiliiana</i>	30–40	3972 ± 65
140472	Shell	<i>Anomalocardia brasiliiana</i>	30–40	3633 ± 43
140473	Otolith	<i>Micropogonias furnieri</i>	30–40	3885 ± 65
140474	Otolith	<i>Micropogonias furnieri</i>	30–40	3581 ± 44
140475	Otolith	<i>Micropogonias furnieri</i>	30–40	3823 ± 49
140476	Shell	<i>Neritina virginea</i>	60–70	3797 ± 43
140477	Shell	<i>Anomalocardia brasiliiana</i>	60–70	3662 ± 64
140478	Shell	<i>Anomalocardia brasiliiana</i>	60–70	3906 ± 79
140479	Shell	<i>Neritina virginea</i>	60–70	3740 ± 80
140480	Shell	<i>Anomalocardia brasiliiana</i>	60–70	3887 ± 77
140481	Otolith	<i>Micropogonias furnieri</i>	60–70	3970 ± 35
140482	Otolith	<i>Micropogonias furnieri</i>	60–70	3845 ± 74
140483	Shell	<i>Neritina virginea</i>	60–70	3806 ± 64
140484	Shell	<i>Anomalocardia brasiliiana</i>	70–80	4069 ± 80
140485	Otolith	<i>Micropogonias furnieri</i>	70–80	3719 ± 64
140486	Shell	<i>Anomalocardia brasiliiana</i>	70–80	3790 ± 78
140487	Shell	<i>Neritina virginea</i>	70–80	3505 ± 69
140488	Shell	<i>Neritina virginea</i>	70–80	3870 ± 48
140489	Shell	<i>Anomalocardia brasiliiana</i>	70–80	3643 ± 41
140490	Shell	<i>Anomalocardia brasiliiana</i>	80–90	3765 ± 65
140491	Shell	<i>Neritina virginea</i>	80–90	3710 ± 65
140492	Otolith	<i>Micropogonias furnieri</i>	80–90	3850 ± 66
140493	Shell	<i>Anomalocardia brasiliiana</i>	80–90	3776 ± 80
140494	Shell	<i>Neritina virginea</i>	80–90	3897 ± 30
140495	Shell	<i>Anomalocardia brasiliiana</i>	80–90	3733 ± 46
140496	Shell	<i>Anomalocardia brasiliiana</i>	90–100	3690 ± 42
140497	Shell	<i>Anomalocardia brasiliiana</i>	100–110	3809 ± 65
140498	Shell	<i>Anomalocardia brasiliiana</i>	100–110	3697 ± 87
140499	Shell	<i>Anomalocardia brasiliiana</i>	110–120	3783 ± 49
140500	Shell	<i>Anomalocardia brasiliiana</i>	110–120	3758 ± 83
140501	Shell	<i>Neritina virginea</i>	110–120	3811 ± 81
140502	Otolith	<i>Micropogonias furnieri</i>	110–120	3842 ± 43
140503	Shell	<i>Neritina virginea</i>	120–130	3596 ± 90
140504	Shell	<i>Anomalocardia brasiliiana</i>	120–130	3745 ± 65
140505	Shell	<i>Anomalocardia brasiliiana</i>	120–130	3807 ± 71
140506	Shell	<i>Neritina virginea</i>	130–140	3969 ± 78
140507	Shell	<i>Anomalocardia brasiliiana</i>	130–140	3954 ± 55
140508	Shell	<i>Anomalocardia brasiliiana</i>	130–140	3636 ± 43
140509	Shell	<i>Neritina virginea</i>	130–140	4015 ± 83
140510	Shell	<i>Anomalocardia brasiliiana</i>	130–140	4052 ± 79
140511	Charcoal	Anacardiaceae	30–40	3628 ± 39
140512	Charcoal	Insufficient sample for analysis	50–60	3703 ± 35
140513	Charcoal	Rutaceae <i>Metrodorea</i> sp.	60–70	3608 ± 36
140514	Charcoal	Wood knot	80–90	3633 ± 38
140515	Charcoal	Sapotaceae	90–100	3670 ± 37
140516	Charcoal	Tuber	100–110	3662 ± 39



less scattering, but the statistical group is not large enough to draw further conclusions. On the other hand, the fact that all the charcoal samples are contemporaneous reinforces the assumption that no “old wood” was incorporated in our analyses, whereas the large variability of marine samples results could be due to real variability of the MRE. This variability could represent seasonal variations like those observed in Pacific corals where subannual measurements show much higher variance than the annual values (Zaunbrecher et al. 2010).

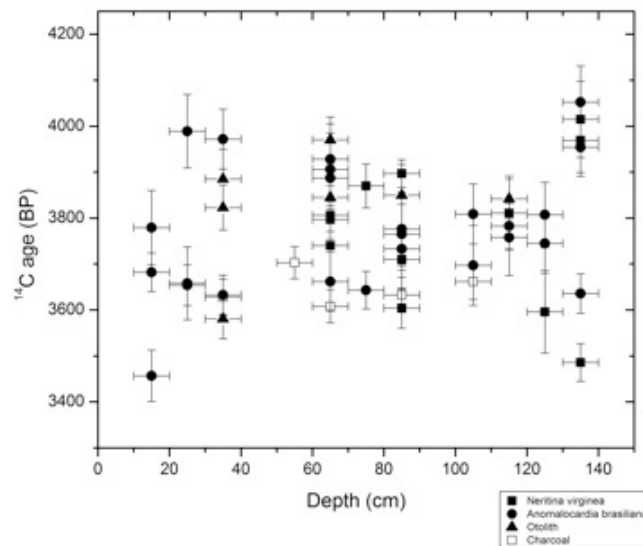


Figure 3 Graph of age versus depth for all samples (correlation coefficient  $r = 0.22$ ).

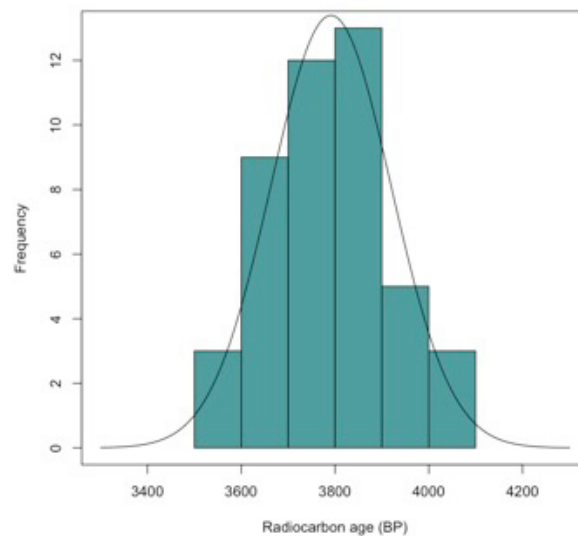


Figure 4 Histogram of the ages of carbonate samples

Averaging the results for each group of samples (marine and terrestrial) and considering specific calibration curves are important issues when dealing with paired samples; therefore, for the purpose of this work, we follow Macario et al. (2015) and consider a Bayesian phase model in the OxCal software containing all the data from the same archaeological context. Such a model assumes that all

dates belong to a common period of time. Each date is calibrated with the appropriate curve and the  $\Delta R$  value is left undetermined; in this case, its potential range spans from  $-600$  to  $600$   $^{14}\text{C}$  yr. This approach considers all samples from each group, not only specific pairs, and it was shown to provide reliable results (Macario et al. 2015) like the multipair approach described by Russell et al. (2011).

The output of the OxCal software is presented in Figure 5, which shows the probability distribution for the  $\Delta R$  correction and the mean value of  $-140 \pm 66$   $^{14}\text{C}$  yr. The large uncertainty of this value is due to the variability of the ages measured, but also to the region of the calibration curve corresponding to the occupation period, where rapid fluctuations due to solar activity are present. On the other hand, the scarce data available in the literature show that  $\Delta R$  values along the Brazilian coast can vary greatly even within the same region. The negative  $\Delta R$  value leads to a reservoir age correction of  $R = 265 \pm 70$   $^{14}\text{C}$  yr. It is worth noting that  $R$  changes both in time and space and thus the obtained value is only valid for samples in the same time range and from the same region.

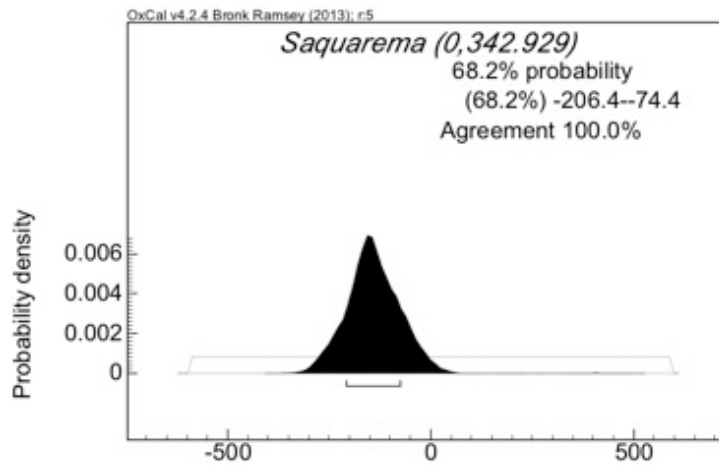


Figure 5 Probability distribution of the  $\Delta R$  correction

Ongoing work in the same region has shown similar values for other shellmounds on the coast of Rio de Janeiro. For example,  $\Delta R$  values obtained for the Manitoba I shellmound, in Ssquarema, and the Tarioba shellmound, in Rio das Ostras, were  $-82 \pm 71$   $^{14}\text{C}$  yr (Carvalho et al. 2015) and  $-127 \pm 70$   $^{14}\text{C}$  yr (Macario et al. 2015), respectively. The negative values indicate a small influence from deep waters, despite the seasonal upwelling effect present in this region. Ikeda et al. (1974) have concluded, through measurements of temperature and water salinity, that the influence of upwelling from the South Atlantic Central Water (SACW) is evenly distributed from Cabo Frio to Ssquarema, where the Ssquarema shellmound is located. On the other hand, freshwater from the Ssquarema Lagoon could have influenced the results. Organisms may have obtained part of their carbon from plant residues introduced in their habitat by means of fluvial discharge (Keith et al. 1964; Schell 1983; Fry and Sherr 1984; Tanaka et al. 1986; Krantz et al. 1987). Terrestrial carbon may increase or lower MRE corrections (Little 1993). Therefore, marine samples near estuaries may absorb carbon of terrestrial origin, leading to negative and variable values of the offset  $\Delta R$  (see e.g. Hogg et al. 1998). Kneip (2004) has demonstrated that fisher-gatherers have intensely exploited the lagoons, this region being a central point for the location of pre-colonial groups in different periods, representing an important role in the economy of the native population.

## CONCLUSIONS

This work aims to expand the knowledge of the poorly studied marine reservoir effect along the western South Atlantic coast. We have analyzed 51 samples comprising of charcoal, mollusk shells, and fish otoliths from the Saquarema shellmound, on the coast of Rio de Janeiro State, southeastern Brazil. Most of this region is influenced by lagoonal systems, which may lead to negative values of the offset  $\Delta R$ , as seen in the obtained value of  $-140 \pm 66$   $^{14}\text{C}$  yr. Although such a value is expected to have limited validity, it is in agreement with other sites within the same time range and from the same region. Considering the large presence of shellmounds on the coast of Brazil, there is a great potential to better understand the marine  $^{14}\text{C}$  reservoir effect, in particular the oceanographic and freshwater influences on it, in this region.

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