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Contributions to the QR Forum

Interrelation of radiocarbon ages from bone fractions in the Brazilian Intertropical Region

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Abstract

There is a consensus in the literature that radiocarbon dating performed on bioapatite often produces ages younger than dating performed on collagen. We propose a general regression that could be used to convert the bioapatite radiocarbon ages to the simulated ages on collagen in fossil samples worldwide. This general regression presents several good indices of quality, high correlation ($R^2 = 0.98$), lower values of percent predicted error (%PE = 0.01), and standard error of the estimate (%SEE = 21.83), showing that it is a good tool, as the predicted values are similar to those observed. Using this regression, we converted the radiocarbon ages of bioapatite to the expected age from the collagen fraction made for several taxa from the Brazilian Intertropical Region (BIR) and suggest that these dates could be 1–7 cal ka BP older than previously thought.

Keywords: Reduced major axis regression, Late Pleistocene, ^{14}C AMS, Bioapatite, Collagen, Mammals, Brazilian Intertropical Region

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INTRODUCTION

For late Pleistocene researchers, knowing the ages of their samples is very important to help interpret the paleoecology and extinction of the studied taxa (e.g., Barnosky and Lindsey, 2010; Dantas et al., 2020).

The accelerator mass spectrometry radiocarbon technique allows the collection of well-preserved bone samples in the collagen fraction ($^{14}C_{collagen}$), up to approximately 50 ka (Cook and Van der Plicht, 2007). However, in tropical regions, there is one main problem: the lack of collagen as a result of diagenetic processes (Hedges, 2002).

Cherkinsky (2009) presented an option to perform radiocarbon dating on bioapatite ($^{14}C_{bioapatite}$) in the absence of collagen, arguing that the mineral fraction survives much better than organic fractions, suffering only small changes through diagenesis.

Since then, several papers dealing with the chronology and paleoecology of the meso-megamammals from the Brazilian Intertropical Region (BIR) have been published using this technique and presenting the occurrence of this fauna in the late Pleistocene, between 32 and 9 cal ka BP (e.g., Dantas et al., 2017, 2020, 2022).

However, some authors (Zazzo and Saliège, 2011; Zazzo, 2014) suggest that during diagenesis, bioapatite exchanges carbon with a ^{14}C-enriched (i.e., younger) carbon source, which results in younger $^{14}C_{bioapatite}$ dates compared with $^{14}C_{collagen}$. The difference between them increases with the age of the samples. Thus, Zazzo (2014) recommended that $^{14}C_{bioapatite}$ should be considered a minimum age estimate.

Based on this observation, we propose and test regressions that could convert radiocarbon dating in bioapatite to collagen in samples collected from different climatic zones (boreal, temperate, subtropical, and tropical).

MATERIALS AND METHODS

In this study, we used published results for pairs of collagen and bioapatite dates obtained from the same bone samples with relatively good preservation. The degree of preservation estimated on the yield of collagen is more than 5%, and the C/N ratio is below 3.5 (Cherkinsky, 2009; Zazzo, 2014 and references therein; Cherkinsky et al., 2015).

Reduced major axis (RMA; model II) regressions were produced using the entire sample set to create a general regression and specific regressions for each climatic zone (boreal, temperate, subtropical, and tropical; Table 1), because RMA (1) deals better with extrapolation than ordinary least squares (OLS; model I); (2) incorporates an assumption that there is an error in *X*; and (3) is symmetric, meaning that the slope of the line does not differ depending on which variable is identified as *X* or *Y* (Smith, 2009; Halenar, 2011 and references therein). This method uses the slope (b_{OLS}) found in OLS, the mean values of *x* and *y*, and the absolute value of the correlation of Pearson (*r*) to estimate a new slope (b_{RMA}; Eq. 1) and intercept (a_{RMA}; Eq. 2) (Harper, 2016).

$$b_{RMA} = b_{OLS}/|r| \quad \text{(Eq. 1)}$$

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Table 1. Values of the reduced major axis (RMA) regressions, coefficient of determination (R^2), average percent prediction error (%PE), and standard error of the estimate (%SEE) obtained for each climatic zone (CZ).

CZ	Samples	Slope	Intercept	R^2	%PE	%SEE
Boreal	8	1.10	−0.41	0.97	0.02	10.90
Temperate	7	1.15	−0.50	0.98	0.05	89.32
Subtropical	8	0.99	0.05	0.99	0.06	21.26
Tropical	5	0.97	0.13	0.99	0.00	25.16
All	28	1.09	−0.31	0.98	0.01	25.00

Table 2. Radiocarbon dating in bioapatite ($^{14}C_{bioapatite}$) converted to collagen ($^{14}C_{collagen}$), presence of modern carbon (pMC), difference between $^{14}C_{collagen}$ and $^{14}C_{bioapatite}$ ($^{14}\Delta_{bioapatite\text{-}collagen}$), and calibrated ages (SHCal20 curve) for extinct late Pleistocene meso-megamammal taxa from the Brazilian Intertropical Region.

Species	Sample no.	Localities[a]	$^{14}C_{bioapatite}$ (ka)	pMC	$^{14}C_{collagen}$ (ka)	$^{14}\Delta_{bioapatite\text{-}collagen}$ (ka)	Age (cal yr BP)
Catonyx cuvieri	UGAMS 34121[b]	Andaraí, BA	11,150 ± 30	24.97	12,634 ± 30	1484	14,839–15,165
Eremotherium laurillardi	UGAMS 09436[b]	Barcelona, RN	10,050 ± 35	28.62	11,281 ± 35	1231	13,096–13,192
	UGAMS 09435[b]	Currais Novos, RN	15,490 ± 40	—	18,078 ± 40	2588	21,851–22,115
	UGAMS 09431[b]	Poço Redondo, SE	10,140 ± 40	24.98	11,392 ± 40	1252	13,170–13,315
	UGAMS 09432[b]	Poço Redondo, SE	22,440 ± 50	—	27,078 ± 50	4638	31,064–31,206
	UGAMS 09433[b]	Poço Redondo, SE	11,540 ± 40	23.78	13,116 ± 40	1576	15,517–15,841
	UGAMS 13539[b]	Poço Redondo, SE	10,990 ± 30	25.45	12,436 ± 30	1446	14,250–14,644
	UGAMS 13540[b]	Poço Redondo, SE	11,010 ± 30	25.39	12,461 ± 30	1451	14,288–14,878
	UGAMS 13541[b]	Poço Redondo, SE	9720 ± 30	29.82	10,878 ± 30	1158	12,736–12,786
	UGAMS 13542[b]	Poço Redondo, SE	9730 ± 30	29.79	10,890 ± 30	1160	12,738–12,792
	UGAMS 13543[b]	Poço Redondo, SE	11,580 ± 30	23.65	13,166 ± 30	1586	15,610–15,898
	UGAMS 14017[b]	Poço Redondo, SE	10,740 ± 30	26.25	12,128 ± 30	1388	13,810–13,950
	UGAMS 09434[b]	Gararu, SE	11,540 ± 40	23.78	13,116 ± 40	1576	15,517–15,841
	UGAMS 42447[c]	Ourolândia, BA	12,400 ± 30	—	14,185 ± 30	1785	17,072–17,352
	UGAMS 34119[c]	Iuiu, BA	30,080 ± 90	2.37	37,267 ± 90	7187	41,839–42,141
Hemiauchenia mirim	UGAMS 36483[d]	Campo Formoso, BA	20,010 ± 65	8.28	23,898 ± 65	3888	27,764–28,177
Nothrotherium maquinense	UGAMS 34123[b]	Andaraí, BA	11,130 ± 30	25.02	12,609 ± 30	1479	14,819–15,133
	UGAMS 34124[b]	Andaraí, BA	11,520 ± 35	23.84	13,091 ± 35	1571	15,483–15,801
Notiomastodon platensis	UGAMS 09440[b]	Barcelona, RN	16,150 ± 40	—	18,919 ± 40	2769	22,594–22,974
Notiomastodon platensis	UGAMS 09437[b]	Poço Redondo, SE	13,950 ± 40	17.61	16,128 ± 40	2178	19,261–19,544
	UGAMS 13535[b]	Poço Redondo, SE	13,380 ± 35	18.89	15,411 ± 35	2031	18,630–18,813
	UGAMS 13536[b]	Poço Redondo, SE	16,370 ± 40	13.03	19,201 ± 40	2831	22,947–23,164
	UGAMS 13537[b]	Poço Redondo, SE	10,440 ± 30	27.26	11,759 ± 30	1319	13,485–13,613
	UGAMS 13538[b]	Poço Redondo, SE	13,760 ± 35	18.04	15,889 ± 35	2129	18,966–19,252
	UGAMS 09439[b]	Canhoba, SE	17,910 ± 50	—	21,177 ± 50	3267	25,277–25,672
	UGAMS 09438[b]	Coronel João Sá, BA	13,980 ± 40	—	16,166 ± 40	2186	19,345–19,583
	UGAMS 09441[b]	Coronel João Sá, BA	15,210 ± 40	15.06	17,722 ± 40	2512	21,173–21,736
	UGAMS 34140[e]	Coronel João Sá, BA	9640 ± 30	30.13	10,781 ± 30	1141	12,700–12,746
	UGAMS 42448[c]	Ourolândia, BA	25,070 ± 60	—	30,555 ± 60	5485	34,628–35,209
	UGAMS 39057[f]	Caetanos, BA	11,450 ± 30	24.05	13,005 ± 30	1555	15,327–15,662
	UGAMS 34116[c]	Iuiu, BA	23,040 ± 55	5.68	27,868 ± 55	4828	31,562–31,911
	UGAMS 34125[e]	Vit. da Conquista, BA	15,890 ± 40	13.83	18,588 ± 40	2698	22,346–22,535

(*Continued*)

Table 2. (*Continued.*)

Species	Sample no.	Localities[a]	$^{14}C_{bioapatite}$ (ka)	pMC	$^{14}C_{collagen}$ (ka)	$^{14}\Delta_{bioapatite\text{-}collagen}$ (ka)	Age (cal yr BP)
Palaeolama major	LPRBUSP 0755[b]	Iraquara, BA	20,850 ± 50	—	24,993 ± 50	4143	29,077–29,248
Toxodon platensis	UGAMS 09442[b]	Rui Barbosa, RN	10,730 ± 30	—	12,116 ± 30	1386	13,809–13,954
	UGAMS 09446[b]	Poço Redondo, SE	10,050 ± 30	—	11,281 ± 30	1231	13,098–13,188
	UGAMS 09444[b]	Cel. João Sá, BA	12,580 ± 40	20.88	14,410 ± 40	1830	17,340–17,789
	UGAMS 42449[c]	Ourolândia, BA	10,740 ± 30	—	12,128 ± 30	1388	13,810–13,950
	UGAMS 09445[b]	Vit. da Conquista, BA	10,970 ± 30	—	12,412 ± 30	1442	14,189–14,595
Valgipes bucklandi	UGAMS 11763[b]	Felipe Guerra, RN	10,440 ± 35	27.25	11,759 ± 35	1319	13,482–13,617

[a]BA, Bahia; RN, Rio Grande do Norte; SE, Sergipe.
[b]Dantas et al. (2017 and references therein).
[c]Dantas et al. (2020 and references therein).
[d]Greco et al. (2022).
[e]Dantas et al. (2022).
[f]Lessa et al. (2021).

$$a_{RMA} = \bar{Y} - b_{RMA} * \bar{X} \quad \text{(Eq. 2)}$$

As the radiocarbon dates do not present a normal distribution (Shapiro-Wilk test, $p < 0.05$), these data were transformed to logarithmic values (at base 10) to approximate a log-normal distribution, because they assign equal weights to all data points in a regression (e.g., Smith, 1993 and references therein).

As a high correlation does not mean that the regression is a good predictor (e.g., Smith, 1984), we calculated the percent predicted error (%PE) and the standard error of the estimate (%SEE).

The %PE of each sample was calculated using Eq. 3 (Valkenburgh, 1990 and references therein; Halenar, 2011), and then an average of the absolute %PE mean of the variables was calculated. This index provides a comparative value for determining the predictive accuracy of regressions.

$$\%PE = (\text{observed} - \text{predicted}/\text{predicted}) * 100 \quad \text{(Eq. 3)}$$

To estimate %SEE, we uses Eq. 4, which reflects the ability of the independent variable to predict the dependent variable (Valkenburgh, 1990 and references therein). SE is standard error (standard deviation, $\sqrt{n}$).

$$\%SEE = (10^{(2 + SE)}) - 100 \quad \text{(Eq. 4)}$$

To test whether statistical differences between the proposed regressions exist, we performed an analysis of covariance (ANCOVA; 1 factor, $\alpha = 0.05$) in the PAST v. 3.11 software (Hammer et al., 2001).

The best estimated regression (see "Results and Discussion") was used to convert the bioapatite radiocarbon dates to suggested collagen dates of eight extinct meso-megamammals from the BIR (sensu Cartelle, 1999; Table 2).

RESULTS AND DISCUSSION

Converting $^{14}C_{bioapatite}$ into $^{14}C_{collagen}$

The radiocarbon-dated samples (dated for both bioapatite and collagen) came from different locations in boreal, temperate, subtropical, and tropical climatic zones (Supplementary Table S1), which provided, in general, younger bioapatite radiocarbon ages than collagen ages from the same samples (Cherkinsky, 2009; Zazzo, 2014 and references therein; Cherkinsky et al., 2015).

Using these data, we estimated regressions for each climatic zone, plus a general one, and noted that they are similar (ANCOVA, $F_{obs} = 1.98$, $p = 0.10$; Table 1), with strong correlations and similar slope (m) values; however, they showed different %PE and %SEE values.

The slopes of these RMA regressions created with the available data allowed us to infer that the bioapatite radiocarbon dates tended to be slightly younger than those of collagen in boreal ($m = 1.09$), temperate ($m = 1.15$), and subtropical climate zones ($m = 1.04$) and worldwide ($m = 1.09$). In tropical climate zones, bioapatite radiocarbon dates tended to be slightly older than collagen dates ($m = 0.97$).

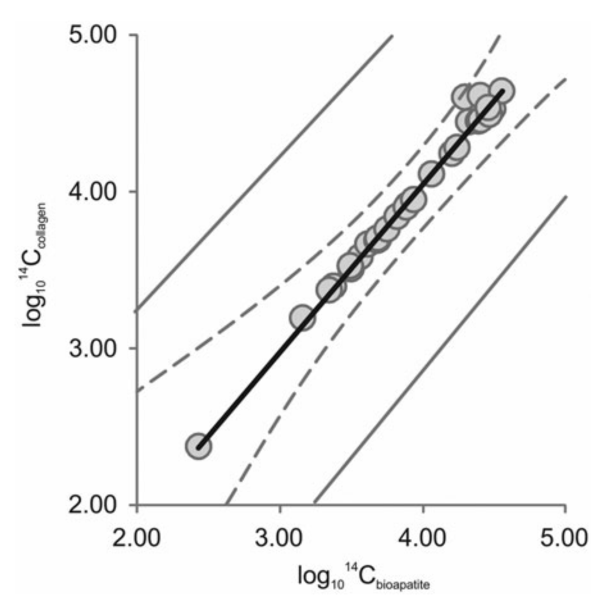

Figure 1. Reduced major axis regression of log radiocarbon dating (bioapatite) and log radiocarbon dating (collagen) using 28 samples (Supplementary Table S1). Regression line (black solid line), confidence intervals (gray dotted lines), and prediction intervals (gray solid lines).

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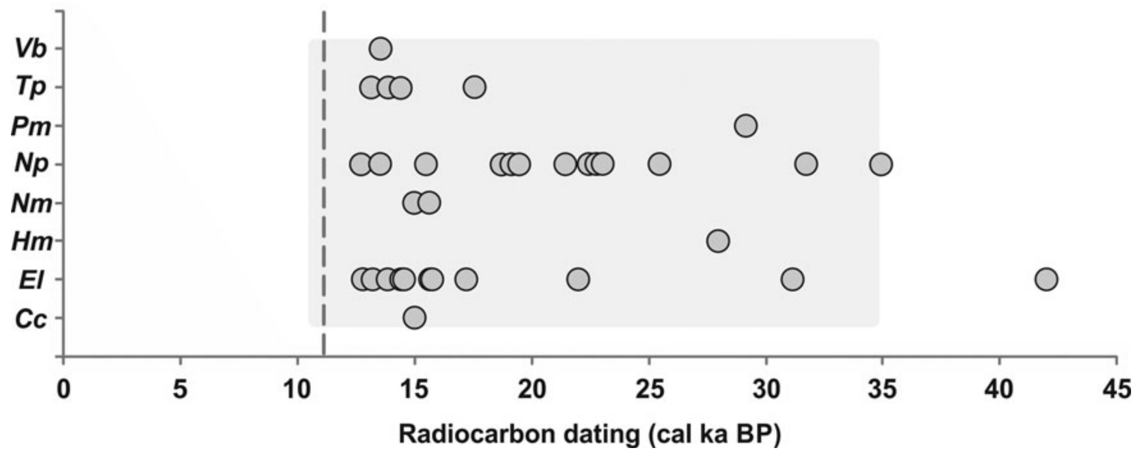

Figure 2. Radiocarbon chronology in collagen (white circles) of extinct meso-megamammals from the Brazilian Intertropical Region. The gray shadow represents the interval found in radiocarbon dating performed on bioapatite. The dotted gray line represents the limit between the Pleistocene and Holocene. Abbreviations: *Cc*, *Catonyx cuvieri*; *El*, *Eremotherium laurillardi*; *Hm*, *Hemiauchenia mirim*; *Nm*, *Nothrotherium maquinense*; *Np*, *Notiomastodon platensis*; *Pm*, *Palaeolama major*; *Tp*, *Toxodon platensis*; *Vb*, *Valgipes bucklandi.*

If we choose to use the regressions for each climatic zone, corrected collagen dates tend to cluster by zone (e.g., those from temperate climatic zones tend to be higher than those in the other zones). To avoid this, as all regressions are similar, we suggest the use of a general regression (Fig. 1), as it shows a strong correlation ($R^2 = 0.98$, $p < 0.05$), lower mean %PE (= 0.01; Table 1), and average %SEE (= 21.83; Table 1).

$$\log_{10}{}^{14}C_{\text{collagen}} = 1.09*\log_{10}{}^{14}C_{\text{bioapatite}} - 0.31$$

The best regressions must have higher values of correlation and lower values of %PE (<15%) and %SEE (Delson, et al., 2000; Ruff, 2003), showing that the predicted values are similar to those observed, which this general equation reached.

Limit of conversion

The radiocarbon calibration curve could allow the estimation of the age of terrestrial samples to approximately 50 ka, which is the limit of the method (Cook and Van der Plicht, 2007; Wood, 2015). As stated before, the bioapatite radiocarbon dates are considered to be minimum ages, and our regression can convert the $^{14}C_{\text{bioapatite}}$ to $^{14}C_{\text{collagen}}$; however, observing the limit of the method (50 ka), our regression should be used to convert only $^{14}C_{\text{bioapatite}}$ to ~39,400 yr. Older converted collagen dating could not be calibrated in the CALIB v. 8.1 program (Reimer et al., 2020) because of the extrapolation of the limit of 50 ka.

Study Case: Converting the $^{14}C_{bioapatite}$ of the meso-megamammals from the BIR

Using the developed regression, we converted the bioapatite radiocarbon dates to the suggested collagen ones for eight extinct meso-megamammal taxa that lived in the BIR and later calibrated them into calendar ages before present, applying the same standard error found in the $^{14}C_{\text{bioapatite}}$ and using the CALIB v. 8.1 program (Reimer et al., 2020), SHCal20 curve (Hogg et al., 2020), and 2σ measured ages reported in Table 2. The use of Northern (Reimer et al., 2020) and Southern Hemisphere (Hogg et al., 2020) curves gives small differences (<7%) between the calibrated data.

The difference between the radiocarbon dating of bioapatite and the dates converted to collagen showed a variation between 1141 and 7187 yr (Table 1), while the difference between the calibrated dates was in the range of 1166 to 7523 cal yr older than previously thought (Cherkinsky et al., 2013; Dantas et al., 2017; Fig. 2).

The diagenesis could promote small alterations in $^{14}C/^{12}C$ in bioapatite carbonate, leading to younger dates; however, this alteration is not significant in the ratio of stable isotopes of carbon ($^{13}C/^{12}C$) for at least the last 40,000 yr (Zazzo, 2014).

When diagenesis affects the bioapatite, the substitutions are mainly in the hydroxyl position in the phosphate, and even with a carbonate substitution, the isotope signature in stable and radioactive carbon maintains the original signature (Cherkinsky, 2009).

The available $\delta^{13}C$ associated with the converted $^{14}C_{\text{collagen}}$ for the megafauna of the BIR provides paleoecological information for a time span ranging ~12,700 to 42,100 yr (Fig. 2) and allows us to suggest that these meso-megamammals lived in the BIR at least until 12 ka. Considering other dating techniques, such as electron spin resonance, this time span can be expanded to 9 ± 2 ka (Ribeiro et al., 2013).

CONCLUSIONS

In this paper, we propose a regression to convert radiocarbon dating performed in bioapatite to collagen, allowing for the comparison of radiocarbon dates worldwide.

Using this new tool, we converted the radiocarbon dating performed on bioapatite in fossils of meso-megamammals from Brazil and suggest that these dates are 1–7 cal ka BP older than previously thought.

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