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Synthesis of Some Phytolith Studies in South America (Brazil and Argentina)

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Chapter 7

PALEOENVIRONMENTAL CONDITIONS OF CAMPOS GERAIS, PARANÁ, SINCE THE LATE PLEISTOCENE, BASED ON PHYTOLITHS AND C AND N ISOTOPES

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ABSTRACT

There are few studies on paleoenvironmental reconstruction using phytoliths found in sedimentary deposits. Most of the research involving these siliceous structures has been performed based mainly on soil profiles. However, the preservation of phytoliths in sedimentary deposits,

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especially in peat bogs, is preferred because the presence of natural acidity facilitates the removal of ferrous compounds. Therefore, a palaeoenvironmental reconstruction of two peat deposits located in the State of Paraná was carried out, the first on an alluvial plain in Palmeira (P1), approximately 16 km from the second, located at an altitude of 1,186 meters in the Sierra of São Luiz do Purunã in the municipality of Balsa Nova (P2). The sedimentary core samples were obtained using a Russian auger, to a depth of 127 cm at P1 and 81 cm at P2. The 14C dating yielded the following results in cal years BP: P1= 30,833 (127cm), 20,271 (72cm) and 7,357 (42cm); at P2 = 24,142 (81cm) and 17,323 (36cm). In addition to the dating, elemental and isotopic analyses of Carbon and Nitrogen were performed. The phytolith extraction was achieved through heating the sample in a KOH solution (10%), washing to reduce pH (~7), separating the organic and inorganic substances using ZnCl2 (2,3g/cm3), and finally preparing permanent microscope slides. For quantification, a maximum of 200 phytoliths were counted for each measured interval and the water stress (Bi%), aridity adaptation (Iph%) and climate (Ic) indices were calculated. Two paleoenvironmental phases were established for each study area: i) formation of the sedimentary deposit in the Late Pleistocene, under a predominantly dry climate with reduced soil moisture conditions; ii) climate change in the Mid - Holocene, with reduced water stress and increased soil moisture. The paleoenvironmental conditions found in this study are compatible with those already published for the Last Glacial Maximum in Campos Gerais, Paraná, Brazil.

INTRODUCTION

Studies on the Quaternary period in Brazil were originally developed by a German scientist settled in Paraná State, namely Professor Reinhard Maack (1892-1969), who in 1947 was already researching vestiges of more rigorous climate changes in the Late Quaternary. Maack defended the hypothesis that the enclaves of grasslands in the Brazilian forests were remnants of ancient climates. In Paraná, Maack (1981) identified a dry semi-arid climate during the last glaciation, and this condition enabled the development of Cerrado, or woody savannah, vegetation with a predominance of grasses in places where rainforests are now located. During the Last Glacial Maximum the forests retreated, occupying the more humid valleys and slopes. In the post-glacial period, the climate became warmer and wetter, creating conditions for the forests to leave the valleys and occupy the grassland. According to Maack (1981), evidence that an earlier semiarid steppe climate predominated was found in the Paraná grasslands and Cerrado ecotones, within the pluvial and subtropical forests. He suggested that xeromorphic and xerophytic plants, found in parts of the Paraná rainforest, were the result of the existence of the semi-arid climate that had prevailed in the Early Quaternary in Paraná. Along the same lines, Professor Aziz Nacib Ab'Sáber (1924-2012) suggested that the semi-arid climate would have previously occupied more than 50% of the territory. Based on the Refuge Theory (Haffer, 1969), Ab'Saber (1992) emphasized that as the semi-arid climate advanced; the forest took refuge in more humid areas. Sharing this idea, Bigarella et al. (1975) argued that during the Quaternary these refuges were particularly important during climate crises, because, according to them, it was on these sites - isolated in the open vegetation - that genetic differentiation of animal and plant taxons occurred. The authors state that during the cold periods of the Quaternary there was a decline of forests and an expansion of open vegetation landscapes. On the contrary, in warmer periods, the opposite occurred; the forests expanded due to an increasingly humid climate with better rainfall distribution.

There are over a dozen papers on the Quaternary period in the State of Paraná, and up to now the areas of study are largely concentrated in the following regions: coastal; channel of the Paraná River; Londrina; low and medium Ivaí River; Guarapuava; Campo Mourão; Ponta Grossa and Campos Gerais (Stevaux and Parolin, 2010).

In Campos Gerais, a name consecrated by Maack (1948), is a natural phytogeographical zone characterized by the occurrence of grasslands interspersed with clumps of Mixed Ombrophilous Forest (dominated by *Araucaria angustifolia* (Bertol.), Kuntze) and gallery forests there are very few completed studies with absolute dating and a paleoenvironmental/ paleoclimatic focus on the Quaternary (Palynology - Behling and Lichte (1997), Behling and Negrelle (2001) and Behling et al. (2009); Quaternary sedimentation - Melo et al. (2003); Diatoms - Moro et al. (2004)).

The archaeological work developed by Arnt (2003) and Parellada (2006, 2009) in this area, also stands out, indicating that the Campos Gerais region has been occupied since the Late Pleistocene, initially by groups of hunter-gatherers and later by farmer-potters.

In view of the above, this chapter brings new insights into the paleoenvironments, vegetation and climate change of Campos Gerais, in the sense of identifying the paleoenvironmental significance of Histosols located in areas of high altitude (> 1,000 meters).

The focus for the paleonvironmental interpretation was based on phytolith analysis, which in recent decades has shown interesting results from the point of view of reconstruction of the vegetation.

In the process of extraction/recovery of phytoliths from the sediment, freshwater sponge spicules were also detected and, although identification at the species level was not possible, they were used as support for the interpretations. It is noteworthy that in addition to the spicules and phytoliths, absolute dating (^{14}C) and values of $\delta^{13}C$ and $\delta^{15}N$ were analyzed, permitting the delineation of the paleoenvironmental conditions from the Late Pleistocene to the Middle Holocene at Campos Gerais.

STUDY AREA

The study was concentrated in two areas occupying the central-eastern portion of the Second Paraná Plateau, which is located in the Campos Gerais region immediately behind the Devonian Escarpment, with an area of 11,761.41 km^2 (Melo et al. 2003). The first area, denominated as "P1", was located in the municipality of Palmeira, bordering highway BR 476, at kilometer 546, and the second was located about 16 kilometers southeast of the first, at the top of the Sierra of São Luiz do Purunã, in the municipality of Balsa Nova, here denominated as "P2" (Figure 1).

The regional climate can be defined as temperate humid mesothermal, or Cfb, as described by the Köppen classification.

The average annual temperature is between 18 and 19ºC and average annual rainfall between 1,400 and 1,800mm (Cruz, 2007).

It is important to note that the precipitation and other meteorological characteristics, such as insolation and relative air humidity, are influenced by the presence of the Devonian Escarpment, which gives rise to orographic rainfall in the transition from the First to the Second Plateau (Melo et al., 2010).

According to Moro and Carmo (2008), the Grasslands present high physiognomic homogeneity, being established on relatively poor soils and rock outcrops, where they occur in association with clumps of “araucária” forest, riparian forest and cerrado fragments. The physiognomies of the Grasslands are composed of dry fields (steppe - *strictu sensu*), often with rocky outcrops (rocky vegetation refuges), humid grasslands (hygrophilous steppe) and cerrados (woody savannah) (Ritter et al. 2012).

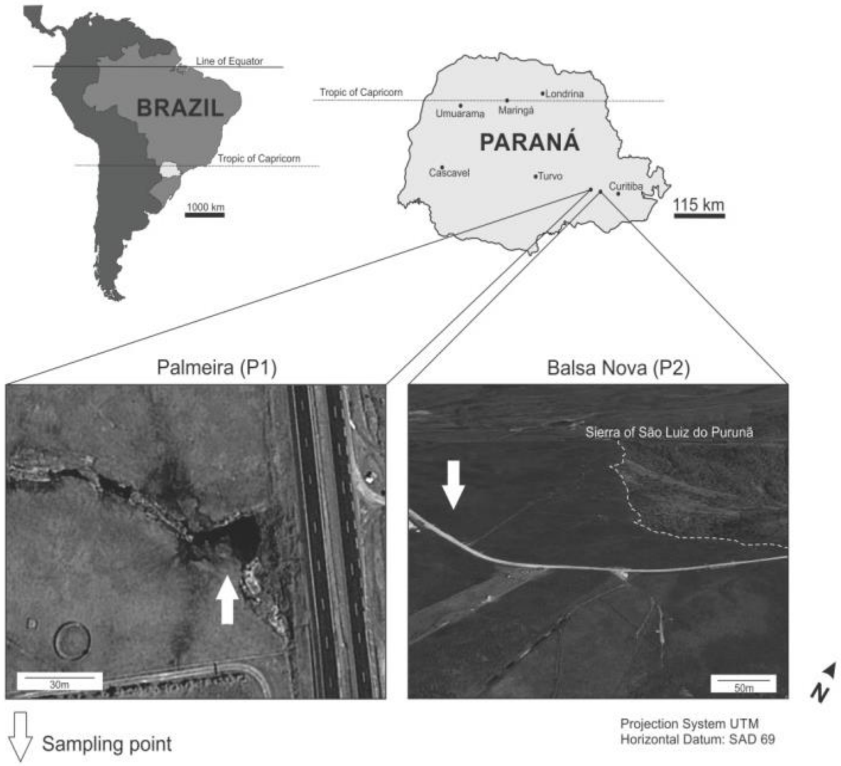


Image: Google Earth®.

Figure 1. Top: Map of South America and Brazil and map of Paraná indicating the points where samples were collected. Bottom: satellite images of areas.

Palmeira (P1)

This area is located next to the Witmarsum Colony between the municipalities of Palmeira and Balsa Nova, São Luiz do Purunã plateau, at an altitude of 1,090m. The sampling point is at the geographical coordinates 25°24'18"S and 49°47'04"W on the banks of a creek (unnamed).

The vegetation is characterized as woody-grass steppe, or Gramineae, although it has been altered by extensive cattle production.

In this region it is possible to observe small streams (Figure 2), whose riverbeds are situated on the sandstones of the Furnas Formation (Siluro-Devonian sequence, ~ 400 million years) (Bigarella and Parolin, 2010). On the banks of these streams, an accumulation of organic matter, predominantly gleysols and/or histosols, can be observed.

Balsa Nova (P2)

This area is situated at the top of the Sierra of São Luiz do Purunã, at an altitude of 1,186 meters, at the division between the First and Second Paraná Plateaus (Figure 1).

Figure 2. Typical landscape of Campos Gerais of Paraná (Palmeira). In the photo it is possible to see the creek riverbed on the Furnas Formation sandstone and peaty sediments on both sides. The sampling was done on the bank of the left margin.

The location has very flat topography, which hinders drainage, and in the aerial images it is possible to observe wetlands formed by small river basins (Figure 3), as in "P1", where the soil (histosol) is found in abrupt contact with the Furnas Formation sandstones. The vegetation is composed of very well preserved natural grasslands (Figure 4).

Figure 3. Satellite image of the area of Balsa Nova (P2), highlighting the river basins.

Figure 4. Partial view from the top of São Luiz do Purunã Sierra, municipality of Balsa Nova/Paraná, with a predominance of grassland vegetation, where the Russian auger was used. In the background one of the escarpments that divide the Second Plateau (top) from the First Paraná Plateau is visible.

METHODS

Sampling

Field activities were performed in 2011 with the collection of samples in "P1" being done on an exposed embankment on the left bank of the creek (Figure 2). Small samples were collected every 3 cm from where the material made contact with the Furnas Formation to the top.

It should be noted that the bank was cleaned with a shovel to avoid any possible contamination from the current vegetation, with 42 sequences being collected at this point. Ten samples, distributed along the outcrop, were chosen for the analyses.

Figure 5. Russian auger used in the collection of samples (bottom) and detail of sampling every 3 cm of depth (top).

The collection of samples at "P2" was accomplished via Russian auger (Figure 5), and as in "P1" the material was removed every 3 cm (27 samples).

^{14}C Dating and Isotopic Analyses of C and N

The samples were dried at 70° C for 24h. From the material collected, 10 cm^3 of distinct sequences were sent for radiocarbon dating (^{14}C) and for isotopic analyses of C and N. The isotopic composition is expressed as delta (δ) per thousand (‰), with a standard deviation of ± 0.2‰, measured against the VPDB standard, and the contents of total organic C (Ctot) and total nitrogen (Ntot) as a percentage of the dry weight of the sample (Pessenda et al. 2009). The analyses were carried out at the Stable Isotope Laboratory of CENA-USP, in an ANCA GSL mass spectrometer, with precisions of 0.1% and 0.02%, respectively.

The dating of the samples was performed through benzene synthesis and liquid scintillation counting at the Carbon 14 Laboratory of CENA/USP (Pessenda and Camargo, 1991), located in Piracicaba, São Paulo, Brazil and through Accelerator Mass Spectrometry (AMS) at the Center for Applied Isotope Studies (CAIS), University of Georgia, Georgia, The United States of America (Table 1).

Table 1. Elemental and isotopic analyses performed on sediment samples from the Palmeira and Balsa Nova study sites and C-14 dating analyses by Laboratory

Palmeira – P1							
Depth (cm)	^{14}C Dating	δ^{13}C (‰)	CTot (%)	δ^{15}N (‰)	NTot (%)	CAIS	LC14
12-15		x	x	x	x		x
39-42	x	x				x	
54-57		x	x	x	x		x
69-72	x	x				x	
78-81		x	x	x	x		x
84-87		x	x	x	x		x
96-99		x	x	x	x		x
111-117		x	x	x	x		x
123-126	x	x				x	
Balsa Nova - P2							
0-3		x	x	x	x		x
12-15		x	x	x	x		x
27-30		x	x	x	x		x
33-36	x	x				x	
39-42		x	x	x	x		X
57-60		x	x	x	x		X
78-81	x	x				x	

CAIS - Center for Applied Isotope Studies University of Georgia; LC14 - C-14 Laboratory of CENA/USP.

The results in years BP (Before Present, where Present refers to the year 1950) were calibrated in calendar years BP (cal BP) based on CALIB 6.0 software (Reimer et al., 2004) and the arithmetic means were calculated.

Laboratory Protocols

For the phytolith extraction, the chemical treatment proposed by Faegri and Iversen (1989) was used, this being a widely used procedure in the area of palynology. Small portions of sediment ($5cm^3$) were placed in beakers, to which hydrochloric acid (10% HCl) was added for carbonate purification. The samples were then boiled with 10% potassium hydroxide for 1 minute for the reduction of pH; and the samples were washed with distilled water every hour until neutralization occurred. The concentration of the material was achieved via centrifugation (1,000 rpm/4min). After concentration, the samples were dried naturally for 24 hours before the addition of zinc chloride with a density of $2.3g/cm^3$, thereby separating the phytoliths. The suspended material was washed several times to eliminate the zinc chloride excess.

After chemical treatment, 50µl of the samples were placed, using a pipette, onto slides and mounted with Entellan® and coverslips. The slides are deposited at the Paleoenvironmental Studies Laboratory of the State Faculty of Science and Letters at Campo Mourão/UEPR - Lepafe (Code L. 213; 214; 215; 216; 217; 218.C.19 for P1 and Code L. 209; 210; 211; 212.C.19 for P2).

Two slides from each interval were analyzed under light microscope at a magnification of x640; a maximum of 100 identifiable phytoliths per slide were counted. The observed phytoliths were classified according to their morphology, based on the International Code for Phytolith Nomenclature (Madella et al. 2005).

Along with the counting of phytoliths, photomicrographs were made. The concentration of phytoliths was determined by counting the occurrence of phytoliths in 5 transects per slide for a total of three slides (magnification X640). Graphs were assembled based on Tilia® software and then drawn with Corel Draw6®.

Based on the phytolith counts, the phytolith rate was determined as follows:

a) Climatic Index (Ic%), first proposed by Twiss (1987) and also used by Bremond (2003), establishes the relationship between C3 and C4 grasses; higher values indicate a predominance of Pooideae, whereas low values have as evidence the presence of Panicoideae and Chloridoideae.

Coe (2009) asserts that the results may be influenced by the abundance of Arundinoidae and Bambusoideae.

Ic (%) = [(Rondel + Trapeziform polylobate + Trapeziform short cell)/(Rondel + Trapeziform polylobate short cell + Trapeziform short cell + Saddle + Cross + bilobate short cell)] x 100

b) Adaptation to aridity index (Iph%) described by Diester-Haas et al. (1973) establishes that high values indicate drier conditions and the predominance of Chloridoideae, while lower values are associated with more humid environmental conditions or moisture in the soil (Twiss, 1992).

Iph (%) = [Saddle/(saddle + cross + bilobate short cell)] x 100

c) Water stress index (Bi%), represents the proportion of cuneiform bulliform phytoliths relative to the sum of the other phytoliths produced by Poaceae (Bremond et al., 2005). It is known that grasses are mainly responsible for the production of Cuneiform in their bulliform cells. However, when water stress acts on the plant, it causes a higher precipitation of these phytoliths in the epidermis of the plant (Bremond, 2003).

This index enables estimation of the dryness of the environment in which the phytolith assemblage was formed.

Bi % (Fs) = Bulliform/[(short cells + acicular + bulliform)] x 100

Environmental considerations were based on the work of Twiss (1992), Tieszen et al. (1979), Fredlund and Tienszen (1997), Alexander et al. (1997) Barboni et al. (1999), Calegari (2008) and Coe (2009).

During the observation and counting of phytoliths, freshwater sponge spicules were detected. Although only fragments of megascleres were seen, a fact that prevented species identification, their presence was taken into consideration for purposes of paleoenvironmental reconstruction.

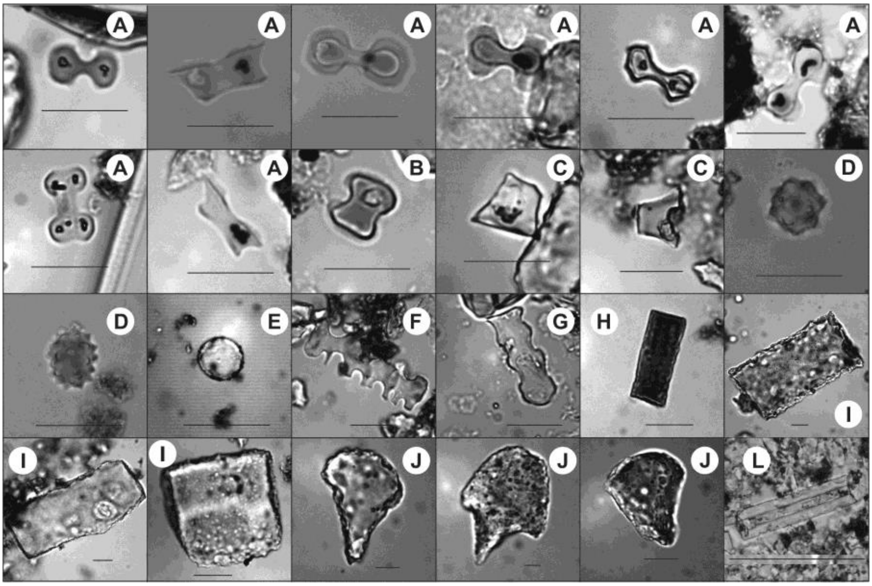
RESULTS AND DISCUSSION

Palmeira (P1)

The base and the middle portion of the outcrop of "P1" presented Pleistocene ages of 30,833 cal BP and 18,322 cal BP, respectively, while the dating at its upper portion corresponds to Middle Holocene, 5,408 cal BP (Table 2). The $\delta^{13}C$ values show a predominance of C4 plants since the Pleistocene (~17‰), with an isotopic depletion to ~-19‰ in the lower and medium portions (99-69 cm), probably indicating a mixture of C3 and C4 plants. The C/N ratio of ~74 to ~28 along the profile indicates that the organic material is predominantly associated with land plants, except in the most superficial part (14) where there is a mixture, probably of terrestrial and aquatic organic materials. Values of $\delta^{15}N$ of 4.94‰ to 6.80‰ indicate the same mixture of organic materials. In the observation of phytoliths it was possible to identify 12 morphologies, with the most representative being illustrated in Figure 6. The concentration of phytoliths increased from the bottom to the top of the core (Figure 7), although from the base up to 72 cm the number of phytoliths did not exceed 100.

Table 2. Results of ^{14}C dating, $\delta^{13}C$, Total Organic Carbon, $\delta^{15}N$, Total Nitrogen and C/N ratio by depth and location sampled

Palmeira – P1							
Depth (cm)	Laboratory	^{14}C (cal years BP)	$\delta^{13}C$ (‰)	CTot (%)	$\delta^{15}N$ (‰)	NTot (%)	C/N
12-15			-16.20	9.95	5.84	0.68	14.63
39-42	CEN (Liquid Scintillation)	7,357	-15.40				
54-57			-16.19	11.07	6.80	0.40	28.32
69-72	UGAMS	20,271	-19.60				
78-81			-19.81	19.81	5.61	0.45	44.02
84-87			-18.88	18.57	5.11	0.30	61.90
96-99			-19.36	53.82	4.94	1.07	50.29
111-117			-17.22	14.92	5.96	0.20	74.60
123-126	UGAMS	30,833	-17.40				
Balsa Nova – P2							
0-3			-15.81	22.78	7.97	1.75	13.01
12-15			-16.53	20.82	7.99	0.87	23.93
27-30			-16.81	13.55	6.35	0.51	26.56
33-36	UGAMS	17,323	-19.60				
39-42			-20.89	12.07	6.04	0.36	33.52
57-60			-20.28	6.81	1.97	0.20	34.05
78-81	UGAMS	24,142	-19.00				



Fragment bars = 17μm.

Figure 6. Phytolith morphologies observed in both sediment cores. A. Bilobate; B. Saddle; C. Rondel; D. Globular echinate; E. Globular psilate; F. Trapeziform polylobate; G. Cilyndric polylobate; H. Elongate psilate; I. Parallepipedal bulliform; J. Cuneiform bulliform. Fragment of freshwater sponge spicule (L) (Megasclere).

This condition may be related to the natural process of dissolution of phytoliths due to the age of the sediment, in which only the most resistant forms are found in the soil matrix, Alexander et al. (1997, 1999), Borba Roschel et al. (2006) and Coe (2009). The predominant morphotypes up to 69 cm were Elongate psilate and Bulliform, and from this point to the top, the Short cell morphotypes, especially Bilobate, became more significant.

Although the occurrence of phytoliths at the base of the outcrop was small, it was sufficient for the establishment of the proposed indices (Figure 7). At the base, the Bi rate reaches 80%, indicating water stress, which undergoes reduction along the sedimentary core from 69 cm to the top, reaching values of below 20%. The rate of Ic has higher values at the base and near the top, but did not exceed 35%, so it is possible to argue that at no time in the analyzed period was there a prevalence of Pooideae. The Iph rate of between 20 and 35% for the entire core suggests a mixture of high and low C4 grasses, predominantly Panicoideae, C4 mesophitic (Barboni et al. 1999).

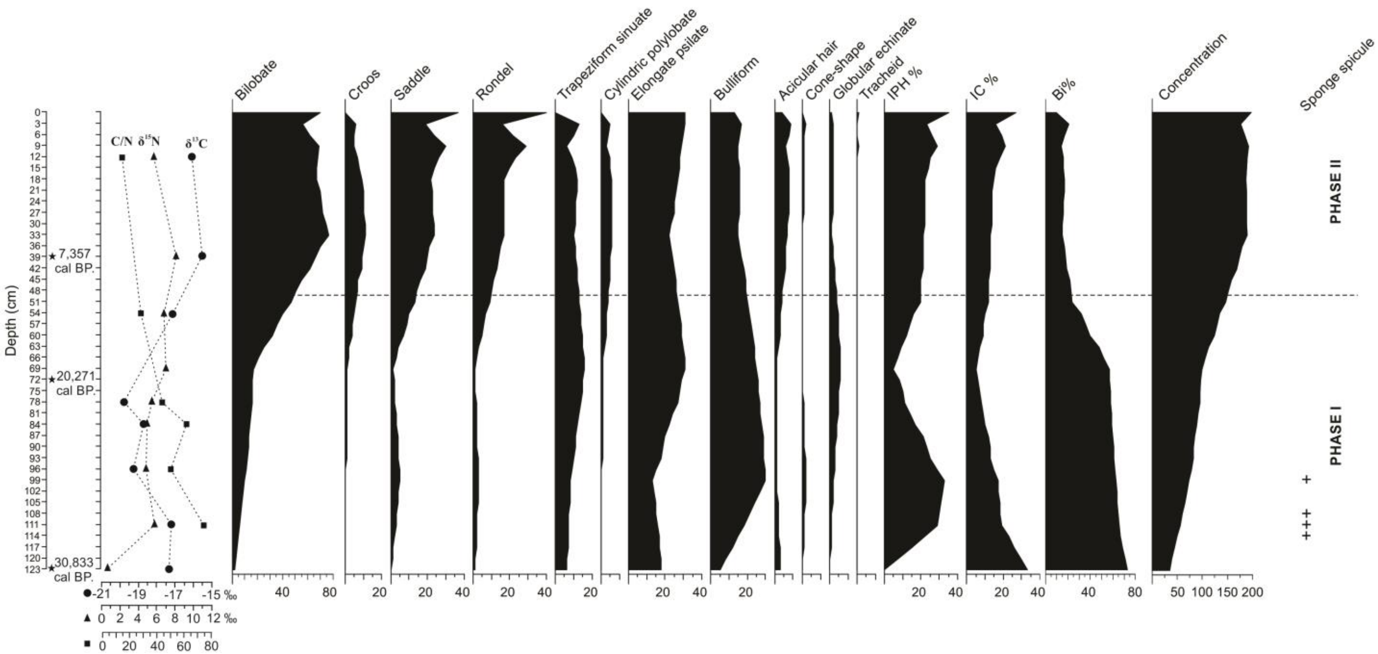


Figure 7. Palmeira "P1" results: ^{14}C dating, $\delta^{13}C$, quantity/concentration of phytoliths, indices and occurrence of freshwater sponge spicules along the core.

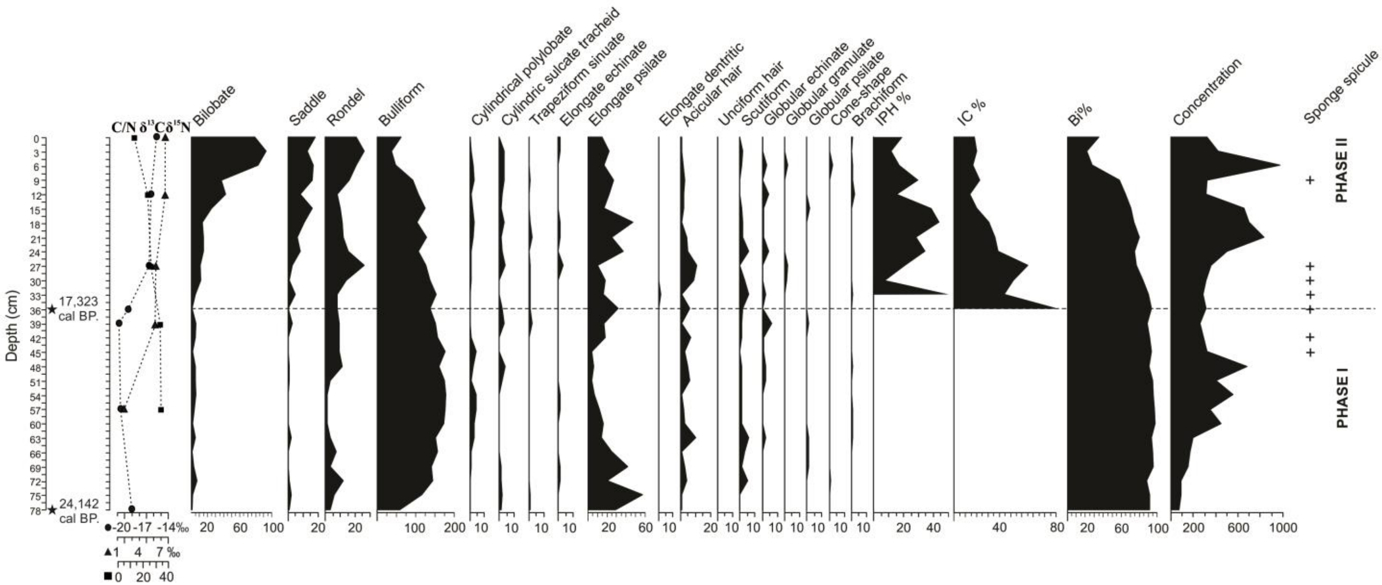


Figure 8. Balsa Nova "P2" results: ^{14}C dating, δ^{13}C, quantity/concentration of phytoliths, indices and occurrence of freshwater sponge spicules along the core.

From the base of the outcrop up to 99 centimeters, small fragments of freshwater sponge spicules (megascleres) were observed (Figure 6L and 7).

In this sequence, minute observations were made with the objective of detecting gemmules for species identification, albeit without success.

The sporadic occurrence of fragmented spicules is evidence of remobilization, and of allochthonous origin. Thus, it is possible that small flood pulses may have occurred during this phase, although such floods may be representative of more concentrated rainfall in specific periods rather than climatic improvements, indicating climatic seasonality.

Balsa Nova (P2)

As in P1, the ^{14}C dating of middle and lower portions of the core deposit corresponded to the Pleistocene and the values of $\delta^{13}C$ for this period (81-33 cm) show a mixture of C3 and C4 plants (~-20 to -19‰), changing to a predominance of C4 plants (~-16‰) from 30 cm (Table 2).

The values of C/N decreased compared to P1 (34-13), indicating a predominance of organic material originating from terrestrial plants with a higher influence of aquatic material than in P1. For $\delta^{15}N$ values, with the exception of the base (~1.9‰), the more enriched values (~6 to 7.9‰) indicate significant influence of organic matter of aquatic origin in the profile.

In this study area there was a greater variety of phytolith morphologies than in "P1," with 18 morphotypes.

The phytolith concentration was not linear, as observed in "P1", despite the lowest concentrations being verified at the base of the core sample; a fact that may be connected to the natural dissolution of phytoliths, as discussed in "P1". The Bulliform morphotype predominated, with a significant reduction at the top, from a depth of 9 cm (Figure 8).

Short cell morphotypes have significant occurrence from 36 cm to the top, permitting an effective determination of the indices for this period.

The Bi index (> 60%) indicates that the gramineae were under water stress between the base of the core sample up to 9 cm from the top.

The Ic rate (>40%) suggests a mixture of Panicoideae and Chloridoideae/Pooideae grasses in the middle portion of the core up to 24 cm, where it then has a predominance of Panicoideae and Chloridoideae. The Iph showed oscillation throughout the core, with the lowest values of < 20% recorded in the upper portion, indicating a condition of higher humidity in the environment or soil (Coe et al. 2014).

Table 3. Palmeira and Balsa Nova paleoenvironmental conditions

Phase	Dating (years cal BP)	Local	Occurrence	Paleoenvironmental Condition
2	7,357	Palmeira	Increase in the concentration of total phytoliths, decrease in bulliform morphotypes, increase in the concentration of Short cell phytoliths , Low rates of Iph and Bi; reduction in C/N ratio, predominance of C4 plants.	Climatic improvement relative to the previous phase, with reduction in water stress and increase in soil humidity.
	-	Balsa Nova		
1	30,833 and 20,271	Palmeira	Low concentration of phytoliths, especially Short cells; presence of fragments of sponge spicules (allochthonous); high C/N ratio, predominance of C4 plants in Palmeira and mixture of C3 and C4 plants in Balsa Nova. Elevated Bi and Iph rates for Palmeira. At Balsa Nova it was not possible to determine Iph.	Beginning of the formation of the sedimentary deposit under dry climatic conditions and reduced soil humidity.
	24,142 and 17,323	Balsa Nova		

As observed in "P1", small fragments of freshwater sponge spicules were also recorded, possibly related to the interconnection of river basin drainage (Figure 3).

Paleoenvironmental Considerations

The combination of the data enabled the establishment of two paleoenvironmental phases for the studied areas (Table 3).

The paleoenvironmental conditions observed are compatible with the paleoenvironmental reconstruction studies performed near the study area. Based on pollen records, Behling and Lichte (1997) and Behling (2002) showed a condition of savannah, under a colder drier climate during the period 48,000 to 18,000 years BP in southeastern Brazil.

Melo et al. (2003), whose Quaternary sedimentation study in the urban area of Ponta Grossa, Paraná, also employed pollen analyses, recognized the dominance of grasslands under paleoclimatic conditions with a dry season of long duration during the Late Pleistocene (~16,000 years BP).

Studies performed by Behling (2007) in the Araçatuba Sierra (1,500 m elevation), located in the Paraná Sea Range (*Serra do Mar Paranaense*), suggest that the climate was drier and colder at the end of the Pleistocene, 14,880 years ago.

Based on the presence of diatom frustules in a core sample obtained in Lagoa Dourada, Ponta Grossa, Moro et al. (2004) showed drier conditions than present at 8,710 years BP but with a significantly more humid climate in the Middle Holocene.

CONCLUSION

The quantification of phytoliths associated with C and N isotopic data, as well as ^{14}C dating showed significant environmental changes from the Late Pleistocene until the Middle Holocene in the regions of Palmeira and Balsa Nova, in the state of Paraná. The changes were gradual, from drier conditions to a state of elevated humidity.

The paleoenvironmental conditions recorded are consistent with those already published for the Last Glacial Maximum in Campos Gerais, Paraná, Southern Brazil.

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