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Palynofacies and stable C and N isotopes of Holocene sediments from Lake Macuco (Linhares, Espírito Santo, southeastern Brazil): Depositional settings and palaeoenvironmental evolution

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ABSTRACT

Particulate organic matter in sediments constitutes a valuable proxy for paleoenvironmental reconstructions. A 192 cm lacustrine sediment core (~7521 cal yr. B.P.) from the northern coast of the State of Espírito Santo, southeastern Brazil, was radiocarbon dated and then analyzed for palynofacies, stable isotopes (C and N), and grain size. The values for total organic carbon – TOC (1.91% to 20.89%), total nitrogen – TN (0.08% to 1.1%), $\delta^{13}C$ (−27.10‰ to −29.01‰), $\delta^{15}N$ (1.92‰ to 5.09‰) and C/N (12.87 to 50.11) suggest predominance of C_3 plants and changing mixtures of terrestrial and aquatic organic matter in the sediments. Palynofacies and cluster analyses were used to identify four palynofacies associations that reflect different phases in the Lake Macuco evolution. Palynofacies 1 (192 to 106 cm, ~7521 cal yr. B.P. to ~7054 cal yr. B.P.—interpolated age) is characterized mainly by amorphous organic matter (AOM), non-opaque phytoclasts (NOPs), mangrove pollen grains (*Rhizophora* sp. L. 1753 and *Avicennia* sp. L. 1753), and foraminiferal test linings. Palynofacies 2 (106 to 65 cm, ~7054 cal yr. B.P.—interpolated age to ~4847 cal yr. B.P.) is dominated by NOPs and AOM, with an increase in the values of mangrove pollen grains relative to Palynofacies 1. Both palynofacies reflect marine and fluvial influences in a depositional environment related to the development of an estuarine system during the mid-Holocene transgressive stage. Palynofacies 3 (65 to 27 cm, ~4847 cal yr. B.P. to ~3800 cal yr. B.P.—interpolated age) comprises high abundances of fluvial elements (NOP and opaque phytoclasts—OP) and low values of AOM. The depositional environment is interpreted as being an estuary during the marine regression, with the displacement of the estuarine basin and mangroves towards the sea. Palynofacies 4 (27 to 0 cm, <~3800 cal yr. B.P.) is characterized by the predominance of AOM and NOP, increase in the values of freshwater algae, and no evidence of marine influence. This last phase reflects the beginning of the establishment of Lake Macuco and floodplain of the Barra Seca River under conditions similar to the present-day.

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1. Introduction

Studies aiming to reconstruct paleoenvironmental conditions represent an important source of data for understanding the environmental changes that have occurred throughout the geological time, especially during the Quaternary. Large-scale events, such as changes in climate and vegetation, as well as coastal development delimited by sea-level shifts, can be inferred by characterizing assemblages of microfossils recorded in sediments.

The particulate organic matter in sediments may be used to deduce information about past ecosystems where the sedimentary organic matter was created and deposited (Meyers, 1997, 2003). The occurrence of organic components in sediments records a set of factors that acted during their production, accumulation and preservation. Several

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studies in Quaternary Brazilian lakes have utilized isotopes (e.g. Pessenda et al., 2005, 2010; Amaral et al., 2012; Smith et al., 2012) and palynofacies (e.g. Jacob et al., 2004; Meyer et al., 2005; Boussafir et al., 2012) in order to trace the origin of the organic matter and the conditions under which it was deposited. However, little is known about the characterization of palynofacies parameters in lacustrine deposits of Brazilian coastal areas that have evolved under the influence of relative sea-level changes. In this context, palynofacies analyses of lake sediments sampled from the northern coast of the State of Espírito Santo (ES) are important when integrated with isotopic (C and N) and paleoecological data obtained from characterization of microfossils recorded in the sediments.

The Quaternary deposits in the northern coast of ES are still too poorly studied to allow reconstruction of paleoenvironmental changes. The aim of this study is to use an interdisciplinary approach for reconstructing the Holocene paleoenvironmental conditions in the surroundings of Lake Macuco, Sooretama Natural Reserve, northeastern ES, using palynofacies, stable isotopes (C and N), and the chronology of sedimentary layers dated by AMS ^{14}C.

2. Regional setting

2.1. Geological setting and sedimentary context

The northern coast of ES is characterized by different geomorphological compartments (Fig. 1A) that provide a well-defined landscape for the region. A highland area with Precambrian rocks occurs in the western portion. It is characterized by an uneven relief covered by Atlantic rainforest and a system of dendritic rivers. Numerous valleys in this compartment appear as "dead ravines", indicating interruption of erosion and weak drift of coarse sediment to the river courses (Suguio et al., 1982). The Neogene plateau to the east of the mountain region is formed by continental deposits of the Barreiras Formation and is characterized by a flat terrain with a corrugated slope towards the sea. A subparallel hydrographic net with tectonic control dissects this compartment, and wide valleys are presently silted up by Quaternary sediments (Martin et al., 1996). The coastal plain comprises marine, fluvial–marine, lagoon and eolian sediments accumulated along the coastline during the Quaternary. This compartment has a semilunar shape reaching about 150 km long in the N–S direction and up to 55 km wide at the mouth of the Doce River (Suguio and Martin, 1981; Suguio et al., 1982). The coastal plain evolution was strongly controlled by relative sea-level changes, fluvial sedimentary supply, long shore drift, and changes in atmospheric circulation (Martin et al., 1993).

Lake Macuco (19°02′S/39°56′W) lies within the Barra Seca River valley at Sooretama Natural Reserve, about 23 km west from the Atlantic Ocean, and 40 km southwest of the city of Linhares on the northern coast of ES (Figs. 1B–D). This lake is an E–W elongated feature with average 3-m depth and has a water surface level about 1 m above the current sea level. The studied sedimentary deposit occurs in a residual lake formed under the influence of sea-level changes during the Holocene, and its development over time is fundamental to understanding the evolution of lacustrine complexes that occur in the region.

2.2. Climate and vegetation

According to the Köppen system (Köppen, 1948), the climate in the study area is classified as Aw (i.e. tropical humid), which is characterized by dry winter and rainy summer. The regional climate is seasonal with the rainy season occurring during austral summer, and the dry season between the months of June and September. Annual average temperature for the region is 23.3 °C, and the mean annual precipitation is 1215 mm (Buso Junior et al., 2013a). Northeastern trade winds are predominant most of the year, while southeastern winds are related to cold fronts that affect the region (Nimer, 1989).

In general, the vegetation in the northern coast of ES is composed of lowland Atlantic forest (*Tabuleiros* forest) occupying Plio–Pleistocene lands. Pioneer vegetation can be observed in areas close to the sea. Noteworthy is the occurrence of two natural reserves in the region, i.e., the Vale Natural Reserve—VNR and the Sooretama Natural Reserve, which protect one of the last remnants of the *Tabuleiros* forest in Brazil, thereby avoiding vegetation destruction by modern agricultural systems.

The *Tabuleiros* forest is associated with sediments of the Barreiras Formation (Neogene), occurring on top of Yellow Argisol (EMBRAPA, 2006), which is an acid and nutrients poor soil, with a flat relief and altitudes between 28 and 65 m (Peixoto et al., 1995). It is a high lowland forest with some very large trees (canopy of 40 m), in addition to high plant diversity and many endemic species. Areas of pioneer vegetation comprise a strip of varying width, being practically continuous over Holocene sediments along the coast. These vegetational formations have several phytophysiognomies that are dependent on environmental conditions, which may have marine, fluvial–marine or fluvial influences (IBGE, 1987). According to IBGE (1987), the pioneer vegetation is represented mainly by mangrove plants (e.g. *Rhizophora mangle* L. 1753, *Laguncularia racemosa* C.F. Gaertn 1807, *Avicennia germinans* (L.) Stearn 1958), salt marshes (e.g. *Typha* sp. L. 1753, *Cyperus* sp. L. 1753), *restingas* (e.g. *Chrysobalanus icaco* L. 1753, *Nectandra* sp. Rottb. 1776), and plants living on floodplain habitats (Poaceae Barnhart. 1895 and Cyperaceae Juss. 1789).

3. Materials and methods

3.1. Sediment coring and sampling

A 192 cm long core (MAC-A) was collected approximately 200 m from the north margin of Lake Macuco (19°02′35.04″S/39°56′41.70″W) using a modified Livingstone piston sampler on a floating platform (Livingstone, 1955; Colinvaux et al., 1999). The sediment core was transported to the ^{14}C Laboratory at the Center for Nuclear Energy in Agriculture at the University of São Paulo (CENA-USP), where it was sampled at intervals of up to 10 cm. Twenty-four samples from the sedimentary section were processed for palynofacies analysis, grain size, C (δ^{13}C, total organic carbon) and N (δ^{15}N, total organic nitrogen) isotopes, and five samples were processed for radiocarbon dating.

3.2. Palynofacies analysis

About 5 g of sediment from each sample were processed following the standard non-oxidative palynological procedures described by Tyson (1995), Mendonça Filho (1999) and Meyer (2004). The samples were treated with HF and HCl to eliminate silicate and carbonate contents, and the organic matter was separated from the mineral fraction using $ZnCl_2$ ($d = 1.9$–2.0 g/cm^3). After this procedure, the isolated organic matter was sieved at 5 μm to concentrate it, and permanent slides were mounted using glycerol-jelly and *Entellan* as a mounting medium. Centrifuging was not used during the chemical treatment to avoid breakage of particles. According to Tyson (1995), the opaque phytoclasts (i.e. carbonized particles) are crumbly, and for this reason, use the centrifuge could have increased their numbers in the studied samples.

Qualitative analysis was made under transmitted white light microscopy (Fig. 2). Identification of different groups of particulate organic matter was based on the model proposed by Tyson (1995) and Mendonça Filho (1999) with adaptations. Palynomorphs were divided into pollen grains (colporates, porates, colpates, mangrove pollen grains, polyads and bissacates), pteridophyte spores, fungal spores, freshwater phytoplankton (*Botryococcus braunii* Kützing, 1849 and other freshwater algae), marine phytoplankton (dinoflagellate cysts), foraminiferal test linings and zooclasts (e.g. Platyhelminthes eggs and invertebrate mouth parts). The phytoclast group, the organic fraction derived from plant tissues, was divided into opaques and non-

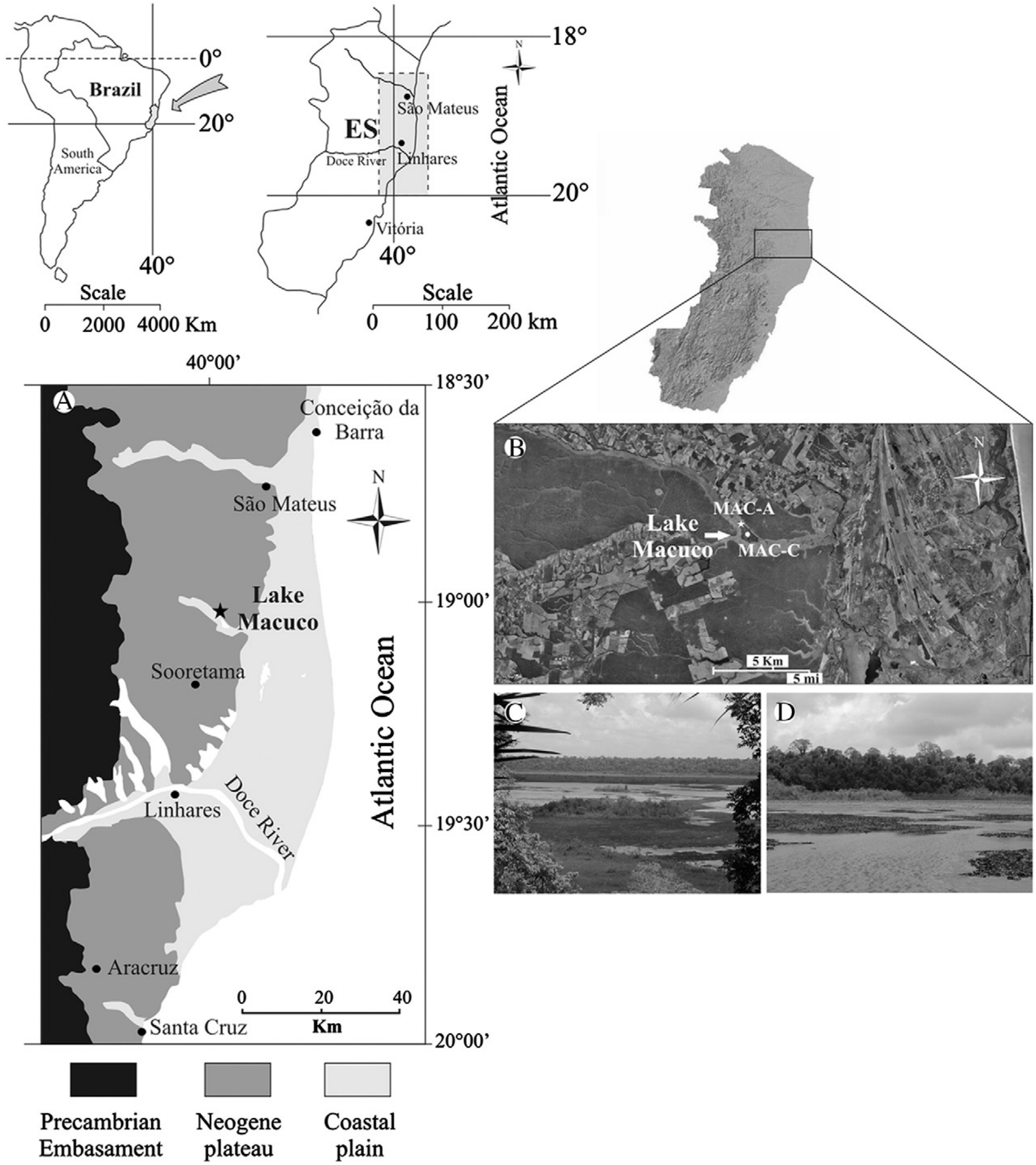

Fig. 1. Study area location. A. Geomorphological map of the northern of State of Espírito Santo (modified from Suguio et al. (1982)). B. Image of the study area. The star indicates the approximate location of MAC-A core and the dot indicates the approximate location of MAC-C core (Buso et al., 2013a,b). C–D. Modern photographs of Lake Macuco.

opaques. Opaque phytoclasts are carbonized particles (charcoal), whereas the non-opaques have brown and yellow colors. In addition, they were also classified according to their morphology based on the presence or absence of structural elements (biostructured and unbiostructured), shapes (elongates and equidimensionals) and their preservation state. The amorphous organic matter group represents all structureless organic material derived from microbiological attack.

A total of 500 particulate organic matter particles per sample were counted under transmitted white light microscopy and the absolute values were converted to percentages and normalized to 100%. The results of qualitative and quantitative analysis from particulate organic matter were synthesized in the form of percentage diagrams prepared by the programs Tilia, Tilia GView 2.0.2 and CONISS (Grimm, 1987, 1992). Furthermore, cluster analysis by similarity index was undertaken, and the palynofacies associations generated were used for paleoenvironmental interpretation.

3.3. Radiocarbon dating

Five sedimentary samples were subjected to physical and chemical treatment for the radiocarbon analyses (Pessenda et al., 2009). In the first procedure, roots, rootlets, leaves and other contaminants were physically removed. Thereafter, the samples were treated with 4% HCl at 60 °C for 4 h to remove fulvic acids, carbonates and resins, and

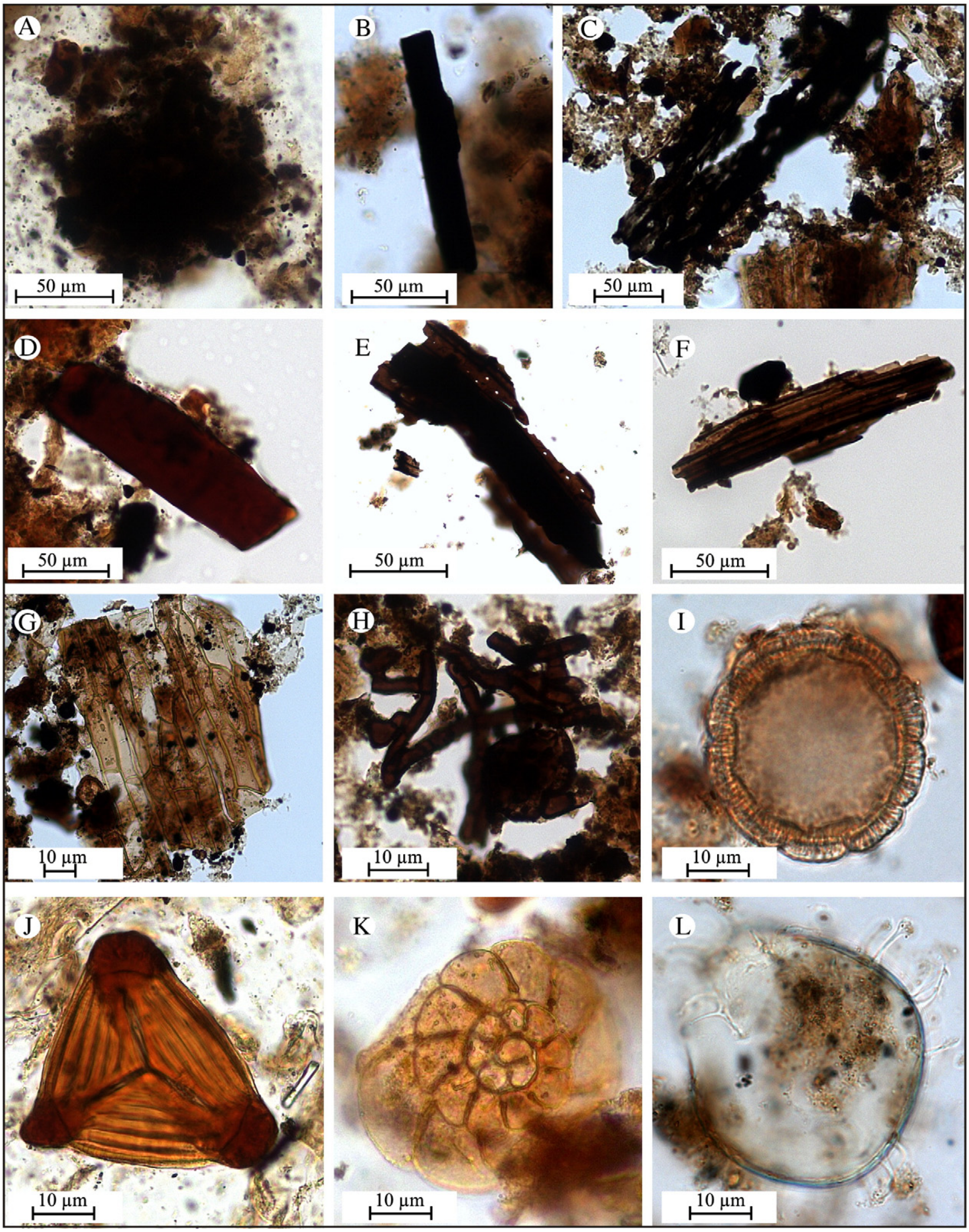

Fig. 2. Components of particulate organic matter viewed under transmitted white light microscopy. A. Amorphous organic matter. B. Elongated opaque phytoclast. C. Corroded opaque phytoclast. D. Un-biostructured non-opaque phytoclast. E. Biostructured perforated non-opaque phytoclast. F. Biostructured striped non-opaque phytoclast. G. Cuticle. H. Fungal hyphae. I. Colporate pollen grain. J. Trilete ornamented spore. K. Foraminiferal test lining. L. Dinoflagellate cyst. The photos A to H were taken at 40× magnification and the photos I to L were taken at 100× magnification.

successive washes were done until the pH was neutral. The samples were dried at 50 °C and combusted to obtain purified CO_2. The capsules containing the CO_2 were sent to the UGAMS Laboratory at the University of Georgia (USA), and to LAC-UFF Laboratory, Fluminense Federal University (Brazil) for Accelerator Mass Spectrometry (AMS). The two radiocarbon dating laboratories were inter-calibrated (Macario et al., 2013). Radiocarbon ages are expressed as yr. B.P. (years Before Present) and in cal yr. B.P. (calibrated years Before Present) with precision of

$\pm 2\sigma$ (Reimer et al., 2009). The mean calibrated ages are used in the text.

3.4. Elemental C and N and isotopes

Plant fragments were removed from all sediment samples submitted for elemental and isotopic carbon and nitrogen analyses. The samples were treated with 4% HCl to eliminate carbonates, washed with distilled water up to pH 6, dried at 50 °C and homogenized. Then the samples were transferred to tin capsules and weighed in an analytical balance with a precision of 10^{-5} g at the Stable Isotope Laboratory of CENA-USP where the total organic carbon (TOC), total nitrogen (TN), and the stable isotopes of carbon and nitrogen were analyzed in an ANCA SL 2020 mass spectrometer. TOC and TN values were expressed in percentage by dry weight, and $\delta^{13}C$ and $\delta^{15}N$ isotopic values were calculated relative to the international standards of VPDB for ^{13}C and atmosphere N_2 for ^{15}N, with precisions of 0.2‰. Elemental results were used to calculate the C/N ratios (weight/weight) for the sediment samples.

The origin of sedimentary organic matter can be distinguished from the isotopic composition recorded in the sediments. Algae and vascular plants are made of various quantities of different organic compounds with specific isotopic values; therefore, it is possible to determine their contributions as organic matter sources in the depositional environment. The following major parameters were used in this study:

(1) TOC: These values represent the fraction of organic matter that escaped remineralization during sedimentation (Meyers and Teranes, 2001) and are used as a fundamental proxy for describing the abundance of organic matter in sediments (Meyers, 2003);
(2) C/N ratio: The presence or absence of cellulose in the plant sources of organic matter influences the C/N ratios of sediments (Meyers and Ishiwatari, 1993). Autochthonous organic matter (e.g. phytoplankton and bacteria) is characterized by low C/N ratios (<10), whereas allochthonous organic matter (e.g. vascular plants) in which lignin and cellulose are the dominant components is generally characterized by C/N ratios higher than 20 (Talbot and Johannessen, 1992);
(3) $\delta^{13}C$: Organic matter produced from atmospheric CO_2 by land plants using the C_3 Calvin–Benson pathway has an average $\delta^{13}C$ value of −27‰ (−32‰ to −22‰), whereas those using the C_4 Hatch–Slack pathway yield approximately −14‰ (−17‰ to −9‰) (Meyers, 1997); and
(4) $\delta^{15}N$: Algal and land plant sources of organic matter can be distinguished by $\delta^{15}N$ values (algae: +8.5‰; land plants: +0.5‰). This organic matter source distinction is possible because the isotopic composition of the sources of inorganic nitrogen to aquatic plants and land plants differ (Peterson and Howarth, 1987; Meyers, 1997).

3.5. Sediment grain size analysis and color

For grain-size analysis, twenty-four sedimentary samples were treated with H_2O_2 and HCl to eliminate organic matter and carbonate, respectively, and the particles were dispersed using ultrasound. A laser diffraction particle analyzer SALD-2201 SHIMADZU, which is based on the scattering of monochromatic light, was used for determining the grain-size distribution. The results were converted into statistical values obtained by the SysGran Program (de Camargo, 1999), and the sedimentary particles were separated into sand (2–0.0625 mm), silt (62.5–3.9 μm) and clay fractions (3.9–0.12 μm) following Wentworth (1922). Each sediment sample was plotted as a point within a Shepard's diagram (Shepard, 1954). Sediment color was characterized by the use of Rock-Color Chart (Goddard et al., 1984).

4. Results

4.1. AMS ^{14}C dating

Radiocarbon dates of five samples from the Lake Macuco core (MAC-A) are given in Table 1. The base of the sediment core (191 cm) has an age of ~7521 cal yr. B.P., and the mean age calibrated for the other samples were ~7356 cal yr. B.P. (113 cm), ~4847 cal yr. B.P. (63 cm), ~4980 cal yr. B.P. (55 cm) and ~3603 cal yr. B.P. (17 cm). Age inversion, eventually associated with the sediment transportation during the inundation of the Barra Seca valley by the rising sea level, was observed between the samples at 63 cm and 55 cm depths. For this reason, the sample dated at 55 cm was not used for the age interpolations. Deposition time rates (Table 1) vary from 2.5 $yr \cdot cm^{-1}$ to 50 $yr \cdot cm^{-1}$.

4.2. Stratigraphy and grain size analysis

Mainly clayey silt with organic matter and plant remains constitute the entire Lake Macuco sedimentary record (Fig. 3). The core was divided into five lithofacies taking into account grain size and color. (1) The basal lithofacies between 192 and 111 cm consists of very dark gray muddy silt (sand: ±0.01%; silt: ±73%; clay: ±26.9%; 10YR3/1), few roots and unidentified benthic fossil tubes. (2) The 111–106 cm interval is characterized by clayey silt (sand: 0%; silt: ±60%; clay: ±40%) and a color ranging from very dark gray to dark grayish brown toward the top of the interval. (3) From a 106 to 21 cm depth, the sediments are comprised of dark grayish brown clayey silt (sand: ±0.004%; silt: ±67.9%; clay: ±32.09%; 2.5Y3/2) with plant debris. (4) The 21–0.6 cm interval is marked by a gradual transition, with clayey silt (sand: ±1.2%; silt: ±74%; clay: ±24.8%) varying in color between dark grayish brown and black toward the core top. (5) The interval above 0.6 cm is composed of organic black peat (sand: ±0.18%; silt: ±82%; clay: ±17.8%; 5Y2.5/1).

4.3. C and N analyses

Total organic carbon (TOC) concentration in the Lake Macuco core varied from a minimum value of 1.91% (12–10 cm) to a maximum value of 20.89% in the topmost layer of the core. Total nitrogen concentration (TN) varied from 0.08% (162–160 cm, 122–120 cm, 24–22 cm, 12–10 cm) to 1.1% (2–0 cm). $\delta^{13}C$ values ranged from −27.10‰ (162–160 cm) to −29.01‰ (24–22 cm), and $\delta^{15}N$ values varied from 1.95‰ (50–48 cm) to 5.09‰ (152–150 cm). A C/N ratio (weight/weight)

Table 1
AMS ^{14}C ages and sedimentation rates of the core MAC-A.

Laboratory number	Sample	Depth (cm)	Age (^{14}C yr. B.P.)	Age (cal yr. B.P., 2σ)	Mean calibrated age (cal yr. B.P.)	Deposition time ($yr \cdot cm^{-1}$)
LACUFF13024	Sediment	17	3367 ± 23	3558–3648	~3603	33.33
LACUFF13025	Sediment	55	4438 ± 54	4871–5089	~4980	–
LACUFF13017	Sediment	63	4273 ± 31	4818–4877	~4847	50
UGAMS3409	Sediment	113	6430 ± 30	7289–7424	~7356	2.5
UGAMS3410	Sediment	191	6640 ± 30	7467–7576	~7521	

UGAMS, University of Georgia, USA; and LACUFF, Fluminense Federal University, Brazil.

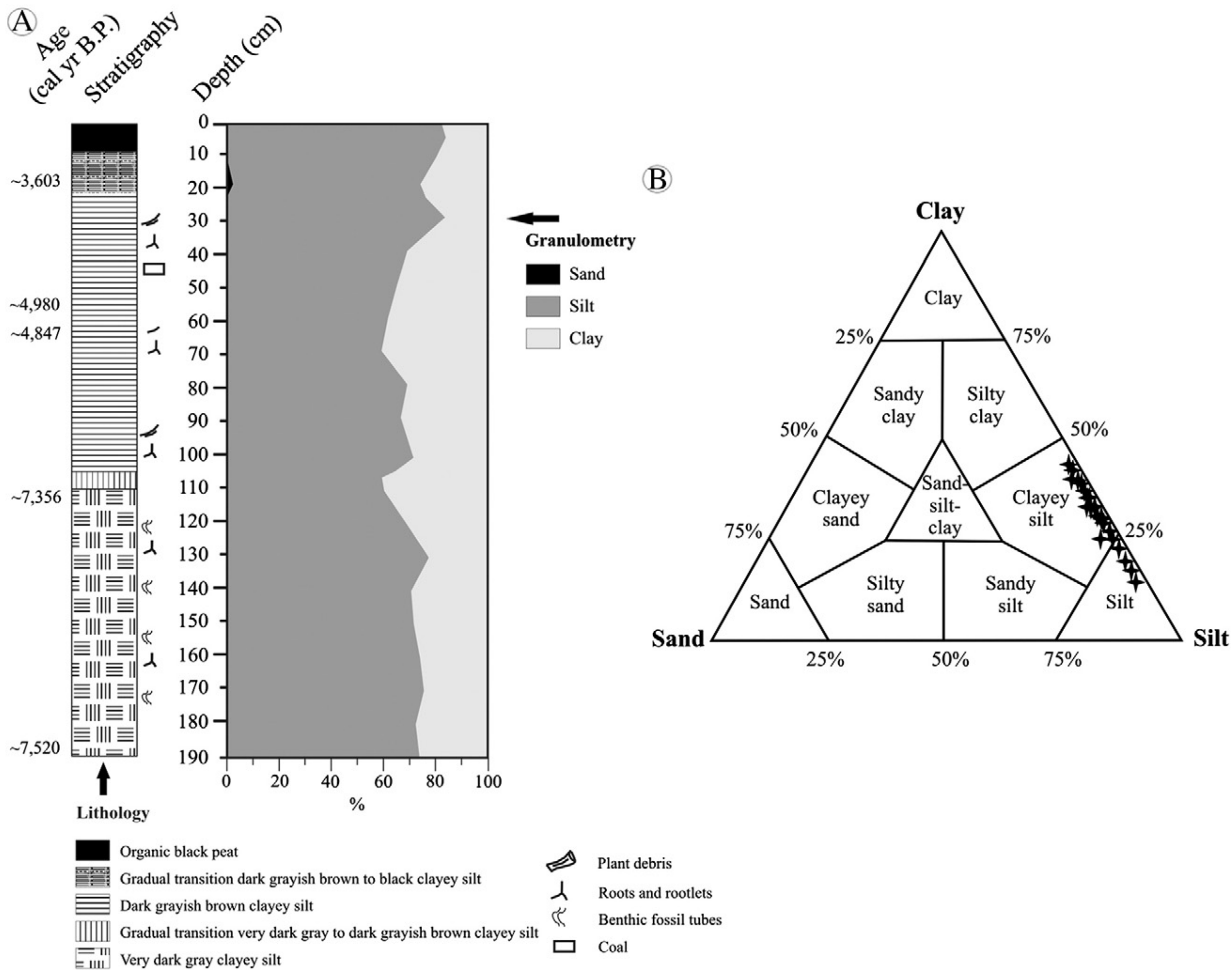

Fig. 3. A. MAC-A core stratigraphy and grain size analysis. B. A Shepard's diagram showing the sedimentary samples distribution (modified from Shepard (1954)).

was obtained for each sample, with values between 12.62 (152–150 cm) and 50.11 (20–18 cm). The isotopic data are expressed in Fig. 4.

4.4. Palynofacies associations

Four palynofacies associations were identified from cluster analysis generated by similarity index of the organic matter content (Fig. 5) and are described in detail in the following sections.

4.4.1. Palynofacies association 1

Palynofacies association 1 (192–106 cm; ~7521 cal yr. B.P. to ~7054 cal yr. B.P.—interpolated age) is characterized by the predominance of amorphous organic matter (AOM; ±55.62%), followed by non-opaque phytoclasts (NOP; ±29.2%), palynomorphs (PAL; ±8.8%) and opaque phytoclasts (OP; ±6.38%). Among the non-opaque phytoclasts (NOP), the frequency of cuticles (±11.98%) and unbiostructured non-opaque phytoclasts (±10.94%) is higher compared to biostructured striped non-opaques (±3.16%) and fungal hyphae (±2.16%). Membranes and biostructured perforated non-opaque phytoclasts occur as subordinate elements with averages that do not exceed 0.70%.

The palynomorphs (PALs) identified belong to the following groups: fungal spores (±2.64%), foraminiferal test linings (±1.12%), pollen grains (±0.54%), pteridophyte spores (±0.4%), freshwater algae (±0.08%), zooclasts (±0.08%), and dinoflagellate cyst (±0.02%). Fungal spores are brown colored and appear as isolated or aggregated components. Foraminiferal test linings occur only in this association, recording the highest percentage (1.6%) in the basal sample. Mangrove pollen grains represented by *Rhizophora* sp. and *Avicennia* sp., as well as other colporate pollen grains, are common. Pteridophyte spores appear more often represented by ornamented and laevigate trilete spores; monolete spores are fewer. However, the percentages of these spores do not exceed 0.05%. It is worth noting that the dinoflagellate cyst *Spiniferites* sp. occurs only in the 140–142 cm sample and is not observed in other intervals of the sedimentary profile.

The opaque phytoclasts (OPs) have lower percentages in this association compared to the other major groups of particulate organic matter. Opaque phytoclasts with irregular shapes and signs of degradation are predominant (±2.3%), followed by elongated and equidimensional clasts with means of 1.98% and 1.48%, respectively. The opaque phytoclasts with cellular structures have low percentage values (±0.7%).

4.4.2. Palynofacies association 2

Palynofacies association 2 (106–65 cm; ~7045 cal yr. B.P.—interpolated age to ~4847 cal yr. B.P.) is dominated by non-opaque phytoclasts (NOP; ±41.6%). The amorphous organic matter percentage values (AOM; ±38.2%) exhibit a decrease in comparison to palynofacies association 1. It also consists of palynomorphs (PAL; ±10.96%), and opaque phytoclasts (OP; ±9.24%). The percentages of the palynomorphs and opaque phytoclasts increased in this association.

Among the non-opaque phytoclasts (NOPs), cuticles are common compared to other components of this subgroup, with an average of 19.16%. Biostructured striped non-opaque phytoclasts increased, recording an average of 10.92% throughout the interval. Fungal hyphae

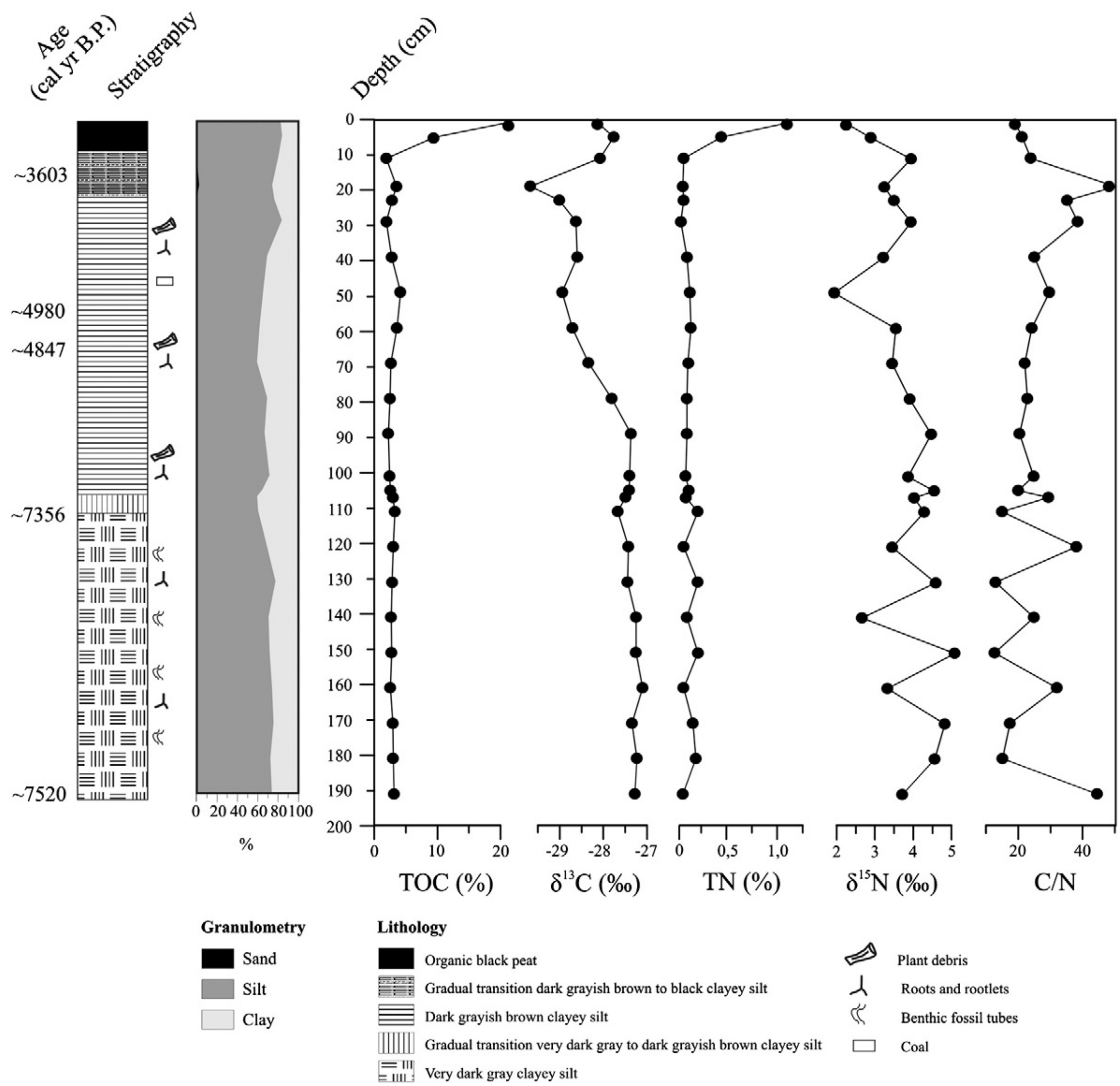

Fig. 4. ^{14}C ages, stratigraphy, total organic carbon (TOC), $\delta^{13}C$, total nitrogen (TN), $\delta^{15}N$ and C/N values (weight/weight).

(±1.08%), membranes (±1.56%) and un-biostructured non-opaque phytoclasts (±0.84%) are subordinate elements.

The palynomorph (PAL) group is mainly characterized by pollen grains. Relative abundances of the mangrove pollen grains reached their highest values (±1.88%). Colporate and porate pollen grains are well represented with an average of 2.2% and 2.12%, respectively. Fungal spores (±1.32%), pteridophyte spores (±0.69%), freshwater algae (±0.24%), and colonies of *Botryococcus braunii* (±0.04%) comprise this association.

The opaque phytoclast (OP) subgroup is dominated by elongated, corroded, and equidimensional clasts with average relative abundances not exceeding 3%. The biostructured opaque phytoclasts occur at an average of 1.4%, which is similar to palynofacies association 1.

4.4.3. Palynofacies association 3

Non-opaque phytoclasts (NOP; ±59.4%) are the dominant organic matter component in palynofacies association 3 (65–27 cm; ~4847 cal yr. B.P. to ~3800 cal yr. B.P.—interpolated age), where this subgroup records the highest values throughout the studied sedimentary record. Opaque phytoclasts (OPs) constitute ±15.45% of the organic matter components. Palynofacies association 3 has the lowest values for amorphous organic matter (AOM; ±13.85%), and palynomorphs (PAL) average 11.3%.

The subgroup of non-opaque phytoclasts (NOPs) is characterized mainly by cuticles (±41.95%), whereas biostructured striped non-opaque (±9.2%), un-biostructured non-opaque (±4.65%), biostructured perforated non-opaque (±1.15%), membranes (±2.1%), and fungal hyphae (±0.35%) are subordinate elements. Among opaque phytoclasts (OPs), there is a greater occurrence of corroded phytoclasts (±5.9%), followed by equidimensional (±3.5%), elongated (±3.15%) and less frequently the biostructured opaque (±2.9%). The two subgroups of phytoclasts (opaque and non-opaque) have an inverse relation with amorphous organic matter.

The palynomorph (PAL) group comprises pteridophyte spores (±1.2%), fungal spores (±1.1%), pollen grains (±1.04%), *Botryococcus braunii* colonies (±0.1%), and freshwater algae and zooclasts each averaging 0.05%. Pollen grains are mainly colporate and porate, mangrove pollen grains decline (±0.3%) compared to palynofacies associations 1 and 2. Pteridophyte spores, represented mainly by trilete spores, have their highest percentage values in this palynofacies association.

4.4.4. Palynofacies association 4

Palynofacies association 4 (27–0 cm; <~3800 cal yr. B.P.) is comprised of the following components: amorphous organic matter (AOM; ±44.16%), non-opaque phytoclasts (NOPs; ±27.56%), opaque phytoclasts (OPs; ±15.16%) and palynomorphs (PALs; ±13.12%).

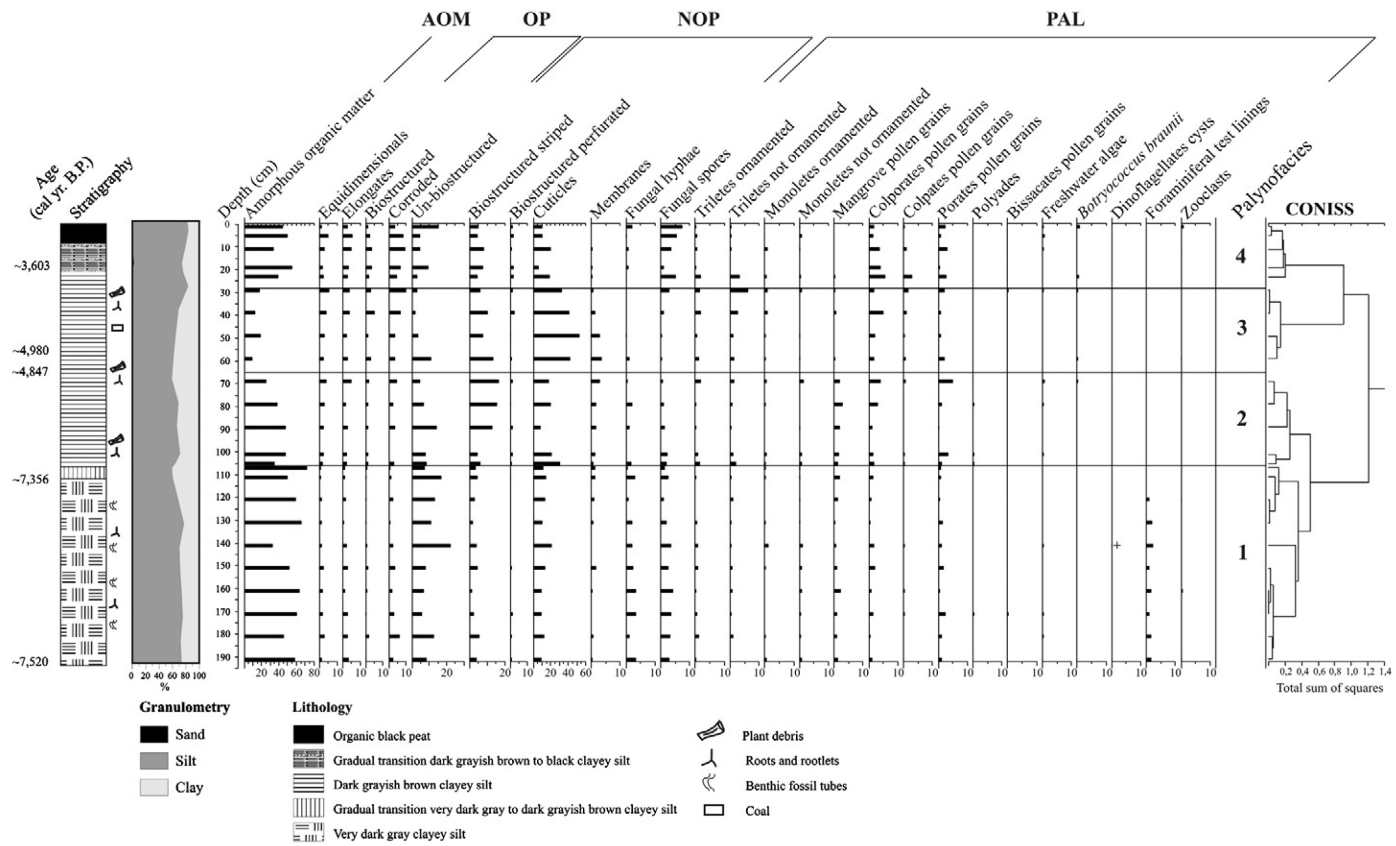

Fig. 5. Relative frequencies of particulate organic matter components. AOM, amorphous organic matter. OP, opaque phytoclasts. NOP, non-opaque phytoclasts. PAL, palynomorphs.

An increase occurs in amorphous organic matter in comparison to palynofacies association 3. Cuticles remain dominant (±12.32%) in the non-opaque phytoclast (NOP) subgroup, although fewer appear here than in associations 2 and 3. This subgroup also contains un-biostructured non-opaque phytoclasts (±7.04%), biostructured striped non-opaque clasts (±5.76%), and biostructured drilled non-opaque clasts (±1.44%). Fungal hyphae and membranes average 0.8% and 0.2%, respectively.

Opaque phytoclasts (OPs) are characterized by corroded elements (±6.28%), followed by elongate (±3.84%), equidimensional (±2.6%), and biostructured (±2.44%) clasts. For the palynomorph group, fungal spores have their highest values (±4.48%) in the core; other palynomorphs include pollen grains (±1.48%), pteridophyte spores (±0.51%), *Botryococcus braunii* colonies (±0.28%), freshwater algae (±0.24%), and zooclasts (±0.16%). Mangrove pollen grains occasionally appear, but only in the basal sample. It should be noted also that Palynofacies association 4 has the highest values of freshwater algae and *Botryococcus braunii* colonies.

5. Discussion

5.1. $\delta^{13}C$ vs. C/N and $\delta^{15}N$ vs. $\delta^{13}C$

The $\delta^{13}C$ values (−27.10‰ to −29.67‰) associated with C/N ratios (12.62 to 50.11) indicate the mixture of aquatic and terrestrial C_3 plant inputs of organic matter to the sediments since ~7521 cal yr. B.P. Some sedimentary samples (192 to 106 cm) have lower C/N values (~12), suggesting greater phytoplankton influence during their deposition. The $\delta^{15}N$ values (1.95‰ to 5.09‰) also reveal that phytoplankton played a role in the organic matter contributions by reflecting a mixture between the two nitrogen sources in the sedimentary deposit (Fig. 6).

5.2. Evolution of the depositional environment

The composition of particulate organic matter components reflects if terrestrial or aquatic environments had more influence during the production and transport of organic matter to the depositional site at different times. Taking into account the palynofacies analysis in combination with isotopic data, it was possible to subdivide the MAC-A core into three intervals that reflect the paleoenvironmental evolution of Lake Macuco during the Holocene (Figs. 7–8).

5.2.1. Phase 1: estuarine system—transgressive phase (192–65 cm; ~7521 cal yr. B.P. to ~4847 cal yr. B.P.)

This phase is defined by palynofacies associations 1 and 2. The main groups of particulate organic matter that occur in this interval are AOM and NOP (Fig. 7). Generally, AOM is higher in sediments far from fluvial sources due to the diminished input of terrestrial organic matter, and it is usually the dominant organic component of sediments that accumulates in anoxic conditions (Tyson, 1995; Batten, 1996). Higher percentages of AOM occur (24–71.6%) in Phase 1, and they are associated with silt and mud grain sizes, which are favorable for organic matter preservation. The amorphous group is held in suspension, and it is deposited in distal facies together with fine-grained sediments. It is highly likely that the high percentage of AOM during this interval is indicative of a low energy environment with reducing conditions.

High percentages of non-opaque phytoclasts (20–47.4%), mainly cuticles and un-biostructured clasts, are markers of fresh organic input to the sediment (Boussafir et al., 2012). Significant percentages of NOP are usually found near fluvial sources, where the action of river currents can carry these particles and disperse the opaque phytoclasts, palynomorphs, and amorphous organic matter in suspension (Tyson, 1995; Meyer et al., 2005). The inversely proportional relation between NOP and AOM is

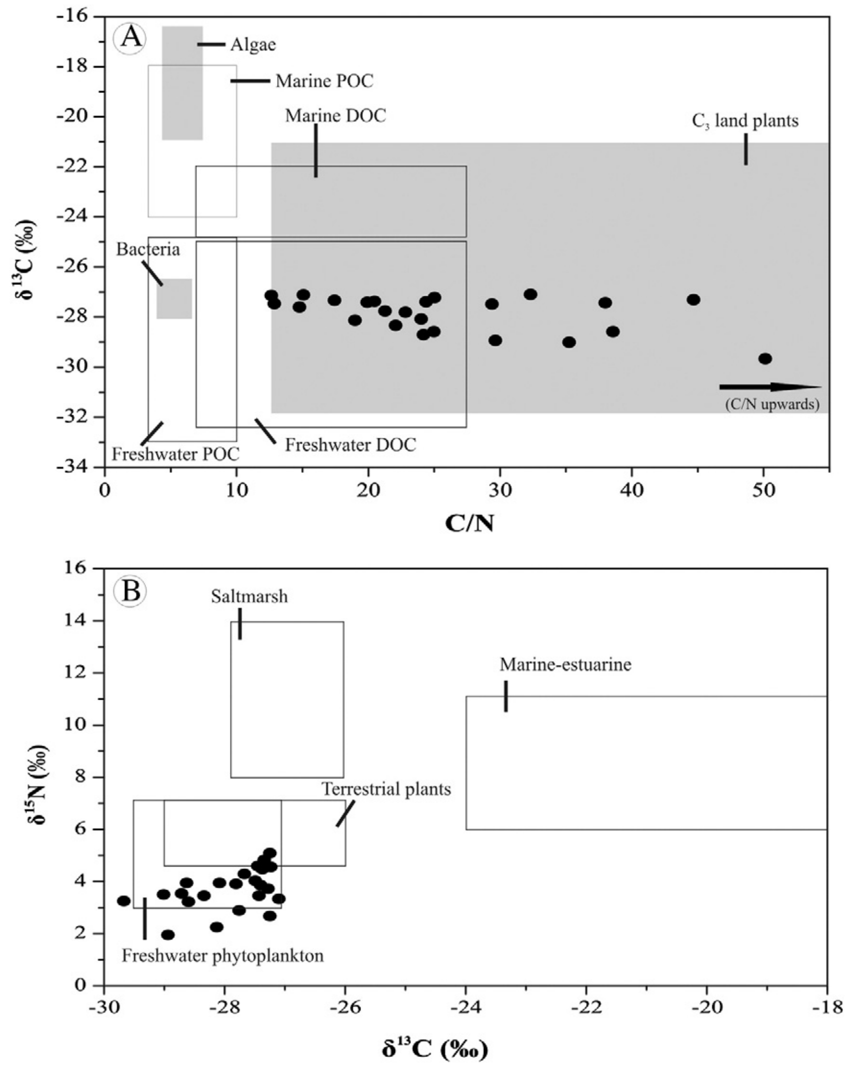

Fig. 6. A. $\delta^{13}C \times$ C/N values (weight/weight) of Lake Macuco organic matter. B. $\delta^{15}N \times \delta^{13}C$ values of Lake Macuco organic matter. The dots suggest mixture of terrestrial and aquatic organic matter source. Interpretation was based according to data presented by Meyers (1994), Cloern et al. (2002) and Ogrinc et al. (2005).

prominent between 192 and 65 cm, confirming AOM amorphous dispersal by fluvial currents.

These results indicate that the low energy environment experienced periods of higher oxygenation during which plant remains were deposited by river currents. They also suggest that the Barra Seca River likely played an important role in the transportation and deposition of particles into the Lake Macuco region since the Early Holocene. Furthermore, the C/N ratios in the interval between 192 and 108 cm (~7500–~7300 cal. yr. B.P.) record low values (±11.11) interspersed with higher values (±31.06). This variability probably reflects a varying mixture of land plants (allochthonous) and phytoplankton (autochthonous) as the main organic matter sources in the sedimentary succession.

A marine influence can be recorded by microfossils like foraminiferal test linings and dinoflagellate cysts. Foraminiferal test linings probably originated from benthic species, and their occurrence indicates a marine influence, although they can be abundant in estuarine marshes (Tyson, 1995; Batten, 1996). Alongside foraminiferal test linings, the dinoflagellate cyst *Spiniferites* sp. was recorded only during this phase and is an indication of salinity variations. These organic components show that the Lake Macuco area was a transitional environment with both marine and freshwater influences during this phase.

Noteworthy are the mangrove pollen grains (*Rhizophora* sp. and *Avicennia* sp.) that dominated the palynomorph group during this phase. According to Behling et al. (2001), *Rhizophora* pollen grains can be transported by wind; however, the capacity of wind to disperse or transport *Avicennia* pollen is very poor. Hence, the occurrence of both these pollen grains is indicative of the presence of mangrove vegetation near the depositional site. Mangroves are a linkage between terrestrial and marine environments, and sea-level changes play an important role in the development and dynamics of this ecosystem (Behling et al., 2001; Vedel et al., 2006; Cohen et al., 2012). The unbiostructured non-opaque phytoclasts identified in some studies (Jacob et al., 2004; Sebag et al., 2006) as gelified cells are typical of root tissues. They were described by Lallier-Verges et al. (1998) in zones with high concentrations of *Rhizophora* and *Laguncularia* as a result of the aerial systems of these plants. In addition to pollen grains, fungal spores were abundant, and their occurrence can be indicative of proximity to a fluvio-deltaic source or redeposition from deltaic, estuarine and lagoonal oxic facies (Tyson, 1995; Batten, 1996). Furthermore, they may reflect the degradation of leaf detritus from mangroves (Muller, 1959).

From the foregoing discussion, the Lake Macuco site is interpreted as being an estuary associated with mangrove vegetation during the Early Holocene and the beginning of Middle Holocene (Fig. 8). In this study, we have followed the definition of estuary by Boyd et al. (1992) and Dalrymple et al. (1992), which represents the seaward portion of a

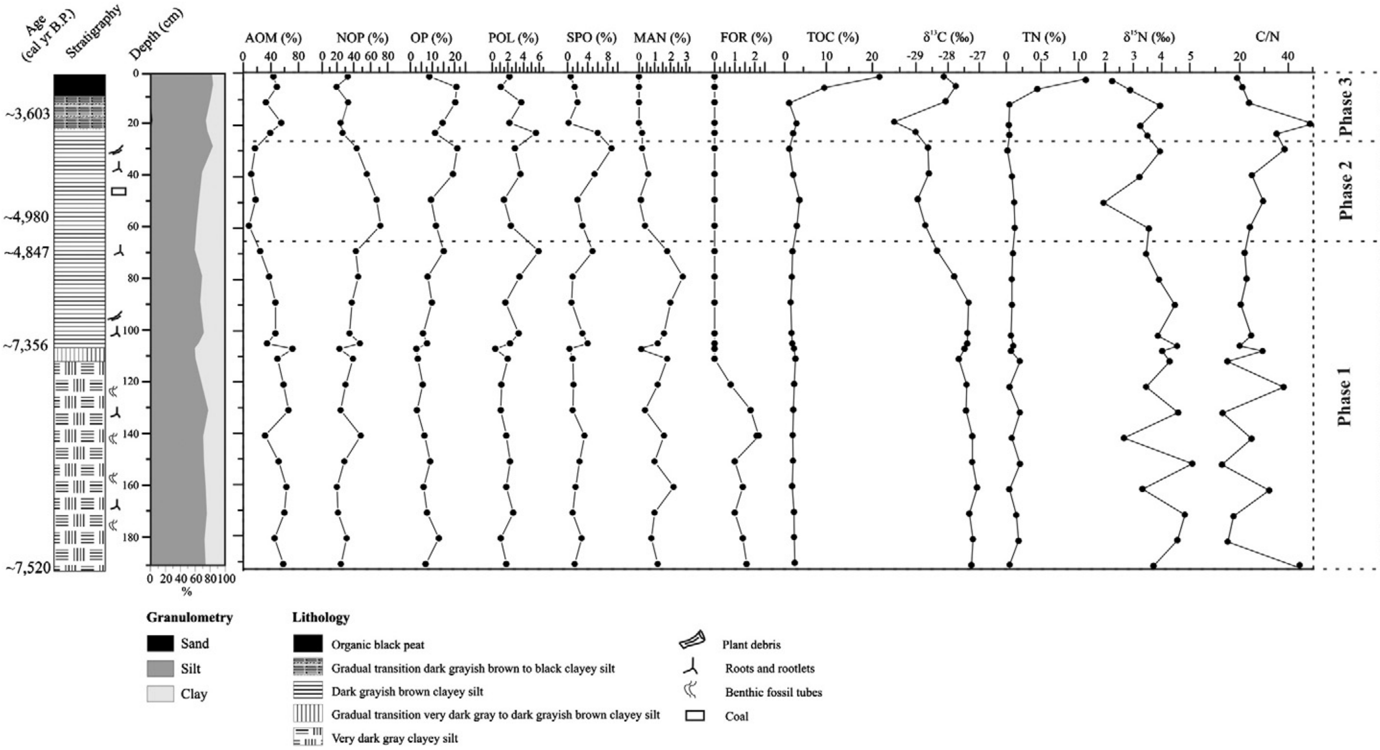

Fig. 7. Relative frequencies of major particulate organic matter components and total organic carbon (TOC), $\delta^{13}C$, total nitrogen (TN), $\delta^{15}N$ and C/N values (weight/weight). AOM, amorphous organic matter. NOP, non-opaque phytoclasts. OP, opaque phytoclasts. POL, pollen grains. MAN, mangrove pollen grains. SPO, cryptogam spores. FOR, foraminiferal test linings.

drowned valley that receives fluvial and marine sediments. According to these authors, an interaction between river currents and marine processes (e.g. tides, waves, salt-water intrusions) characterizes the estuary where the landward limit occurs at its head and the seaward limit occurs at its mouth. The equilibrium between fluvial influx and tidal currents occurs in the central basin of the estuary. Estuarine circulation generated by a salinity gradient keeps the fine-grained sediment in suspension for a long time before being deposited in the central basin or in swamps, mangroves, and mudflats on the margins of estuary (Rossetti, 2008).

The sedimentary succession between 192 and 65 cm appears to have been deposited in the lower energy part of the estuarine system, probably near the margins of the estuary and the mangroves. Higher percentages of AOM corroborate this interpretation because the amorphous group behaves like fine-grained sediments. Non-opaque phytoclasts indicate a terrestrial influence on the sedimentary deposit, which is in agreement with the palynofacies data described by Oboh-Ikuenobe et al. (2005) for an estuarine lithofacies in Nigeria. According to these authors, estuary and/or proximal lagoon lithofacies are characterized by high percentages of unstructured and structured phytoclasts, and the co-existence of terrestrial and marine material. Sediments deposited in estuaries have a strong terrigenous influence, regardless of whether transgressive or regressive conditions are predominant (Degens and Mopper, 1976; Tyson, 1995). TOC values do not exceed 5%, and such relatively low values usually occur in lagoon and estuarine environments above internal shelves (Tyson, 1995).

Estuaries are formed at the beginning of a transgression (Dalrymple et al., 1992) and their development occurs mainly in coastal plains with wide shelves and drowned valleys, allowing sediment accommodation (Rossetti, 2008). Several studies along the Brazilian coast (e.g. Angulo and Lessa, 1997; Angulo et al., 1999, 2006; Bezerra et al., 2003; Martin et al, 2003) show that a sea-level highstand occurred at ~7000 cal yr. B.P., followed by a period of relative sea stabilization between ~7000 and ~5000 cal yr. B.P. Part of the MAC-A core (192 up to 65 cm) corresponds to the time interval between ~7521 cal yr. B.P. and ~4847 cal yr. B.P., which includes this phase of the high sea-level period described for the Brazilian coast during the Holocene. Moreover, the deposition time for this interval shows a high sedimentation rate of 2.5 $yr \cdot cm^{-1}$, which may indicate the creation of accommodation space due to increasing sea-level during the Early Holocene. Additionally, the inundation of the Barra Seca valley by the rising sea level can be recorded by organic muds in the MAC-A core containing mangrove pollen, much like the data by Woodroffe et al. (1989) for northern Australia.

From an interdisciplinary study (i.e. palynology, sponge spicules, C and N isotopes and geochemistry) of another sediment core from Lake Macuco (i.e., MAC-C), Buso Junior et al. (2013b) described the study area as having the most dense mangrove cover, reducing conditions, intertidal sedimentation as well as the presence of a bay-head delta between 7623 and 7015 cal yr. B.P. According to these authors, during the interval between 7015 and 3190 cal yr. B.P., a landward migration of the paleo-estuary occurred, and the depositional environment was described as an estuarine central basin. However, in the present study, the estuarine environment is identified only until ~3800 cal yr. B.P. The difference between these two interpretations is consistent with the distinct positions of the two cores. The MAC-C core was collected in a more central position in the Lake Macuco basin, whereas the MAC-A core presents a more marginal position and is placed closer to the paleo-estuary head. The two cores are distant from each other by about 150 m. The marginal position of MAC-A core may have been subjected to a higher influence of terrestrial plants and mangroves, lower hydrodynamic energy, and a higher relative influence of organic matter transported by the river. Furthermore, higher percentages of herb pollen were recorded by Buso Junior et al. (2013a) for the study area between 7085 and 4396 cal yr. B.P., probably reflecting the occupation of the paleo-estuary margins by C_3 plants, a feature that must have influenced the isotopic values and the particulate organic matter composition in the MAC-A core due to its marginal position. These differences that are related to the position of the sampling sites may have prevented MAC-A core from recording the changes in the estuarine system described from MAC-C. In addition, the higher ^{14}C dating resolution used

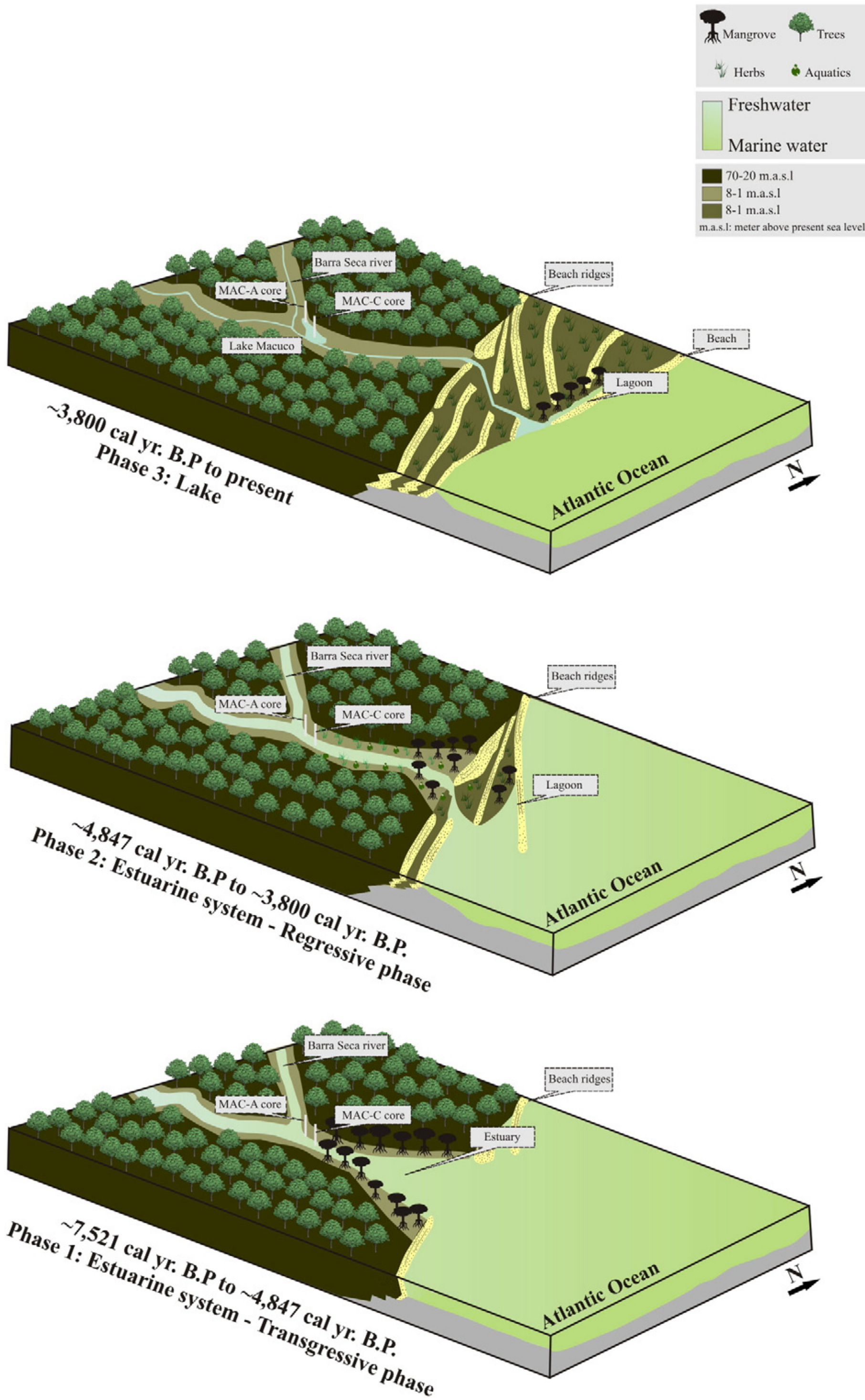

Fig. 8. Schematic representation of paleoenvironmental changes in the study area during the Holocene.

in the study of Buso Junior et al. (2013b) cannot be ignored in the data comparison.

5.2.2. Phase 2: estuarine system—regressive phase (65–27 cm; ~4847 cal yr. B.P. to ~3800 cal yr. B.P.)

Palynofacies association 3 defines this interval, with sediments having high relative abundances of fluvial elements (non-opaque and opaque phytoclasts) and low values of AOM (Fig. 7). The Lake Macuco deposits formed during this time interval record an environment with relatively more fluvial influence. The inversely proportional relation between phytoclasts and amorphous groups may indicate that AOM was dispersed with plant debris by fluvial currents. Alternatively, due to fluctuations in the level of the water column, structureless particles could have been oxidized when the water level was lower. High input of phytoclasts may dilute all the other components of particulate organic matter; therefore, the occurrence of palynomorphs as subordinate elements may be reflective of deposition in the vicinity of the vegetation.

The occurrence of non-opaque phytoclasts may indicate oxic conditions, fluvial source proximity, and sediment input through floods (Tyson, 1995). Mainly cuticles and biostructured striped phytoclasts constitute the NOP group in palynofacies 3. According to Batten (1996), most cuticles in palynological slides are derived from leaves, and they are associated with a low energy fluvio-deltaic environment. Because these cuticles are well-preserved, they indicate proximity to terrestrial source (Mendonça Filho et al., 2010). In their palynofacies analysis of floodplains of southeastern Brazil, Meyer et al. (2010) described palynofacies dominated by biostructured striped non-opaque phytoclasts. These phytoclasts were derived from local vegetation, but the authors did not rule out the role of the fluvial channel in depositing the plant tissues during periods of high flow. The same type of data was observed in the MAC-A core. More negative $\delta^{13}C$ (~−28.6‰) and higher C/N ratios (~22 to ~38) during this phase suggest greater contribution of organic matter from C_3 vascular plants. Therefore, isotopic data and significant relative abundances of non-opaque phytoclasts indicate that terrestrial plants were the major source of organic matter during this interval. In wetlands, constituents derived from vascular plants represent the main organic fraction (Sebag et al., 2006).

Opaque phytoclasts are the most resistant components of particulate organic matter and remain in the depositional basin after the selective destruction of other particles. They reflect environments with oxidizing conditions and are generally associated with sand grains (Tyson, 1995). However, no evidence of coarse sediments was found in the MAC-A core. This observation suggests that opaque phytoclasts probably were deposited distally from their source area, likely being transported for long distances by the river. Based on these data, we also infer that periods of high river flood probably were interspersed with dry seasons. Oxidation of woody material and plant tissues is favored by strong seasonality, which occurs during dry periods and fluctuations of the water table (Tyson, 1995).

This phase is characterized by significant percentages of sporomorphs, mainly pteridophyte spores and colporate pollen grains. High relative abundances of pteridophyte spores indicate the development of pterydophytic vegetation near the deposit, wetter conditions, and water transportation (Meyer et al., 2005). In well-developed coastal areas such as the ES and alluvial plains, local pollen rain is more important than spores and wind dispersed pollen, and the sporomorphs are supplied primarily by local vegetation (Streel and Richelot, 1994). Although rare mangrove pollen grains occur in this phase, they are represented only by *Rhizophora*. This genus has the capacity to grow in low salinity habitats (Smith and Snedaker, 1995), and its pollen grains can be transported by wind (Behling et al., 2001), which can explain their distribution in the region even during times of greater fluvial influence.

Using the palynofacies data, we infer that the sedimentary deposit that accumulated during this period probably still represents an estuarine system but with higher fluvial inflow than during the mid-Holocene transgression (Fig. 8). In general, studies of transgressive and regressive episodes in Brazil during the Holocene show that after ~5000 cal yr. B.P. sea level had fallen to its present position (e.g. Angulo et al., 2006). Periods of lowering sea-level are characterized by a decrease in sediment accommodation space, thus promoting fluvial erosion (Scherer, 2008). A high river influx can provide large amounts of sediment to fill an estuarine basin, and floods can cause the convergence zone to move toward the sea (Rossetti, 2008). In this way, the estuarine basin became gradually abandoned, and it was progressively closed due to sand accumulation in barriers at its mouth as a result of the Doce River delta progradation. According to Castro et al. (2013) the development of beach ridge systems by dispersion of sands brought in by the Doce River Delta caused the replacement of the estuaries by lakes and the establishment of the wave-dominated delta system. As the estuary was being closed, the marine influence was reduced, resulting in more freshwater input in the sedimentary deposits.

From 4396 to 1287 cal yr. B.P., Buso Junior et al. (2013a) describes pollen types associated with an ecological succession in sites that were previously flooded, which could be an indication of an environment with episodic freshwater flooding, like an alluvial forest. Moreover, Buso Junior et al. (2013b) noted more effective fluvial input and an increase of continental spicules sponges as well as just a few mangrove pollen grains in the MAC-C core from 3190 cal yr. B.P. The presence of well-preserved gemmoscleres of continental sponges from ~4200 cal yr. B.P. (interpolated age) could represent the establishment of freshwater environments close to the study area site, which agrees with the regressive trend noted in the MAC-A core. According to these authors, the patterns found by them may record the progradation of the paleo-estuary. Inasmuch as the MAC-A has a more proximal and marginal estuarine position, the decrease of marine influence and the beginning of the estuary displacement seaward were first noted in this core because it had a stronger continental influence.

Estuaries are complex systems and their development is influenced by climatic conditions, tidal ranges, and parameters like morphology and sedimentation rates. The shape of the flooded valley has an important influence on the nature of the sedimentary facies developed in an estuary, mainly during the stage of infilling. As the estuary evolves, its transgressive characteristics decrease, and the facies zones translate landward (Dalrymple et al., 1992). The differences between sedimentary deposition and transgressive–regressive trends noted in the two cores could be the result of variations in the basin morphology as well. This fact can be mainly noted through the difference between the stratigraphy described for two cores. Wavy laminations and muddy sediments were described by Buso Junior et al. (2013b) during the transgressive stage for MAC-C core, and an increase in sand sedimentation occurred during the regressive trend, indicating more effective fluvial input. However, no evidence of grain size changes were noted in the MAC-A core, which is consistent with its deposition in a marginal position. According to Rossetti (2008), fine sediments in the estuarine system can be transported to areas as swamps, mangroves, and mudflats on the estuary banks, where massive muddy sequences with organic matter are deposited.

5.2.3. Phase 3: Lake Macuco (27–0 cm; <~3800 cal yr. B.P.)

Phase 3 is represented by palynofacies association 4, which is dominated by AOM and NOP (cuticles and un-biostructured) (Fig. 7). Based on the data set, we characterize this phase as recording the establishment of the present environment at the study site, which includes Lake Macuco and the floodplain of the Barra Seca River (Fig. 8).

The amorphous group reflects a low energy environment with reducing conditions. In shallow lakes, frequent winds can promote turbulence in the water column, causing sediment resuspension (Gons et al., 1986). However, the predominance of AOM shows that fine-grained sediments and organic matter in Lake Macuco probably were deposited in a stratified water column with anoxic–dysoxic bottom conditions and little disturbance at the water–sediment interface. Similar results were observed in an Eocene perennial lake in northwest Argentina (del Papa et al., 2002) and in a Holocene lake in southwest Brazil (Silva et al., 2010).

The lakes in the Neogene plateau in the northern part of State of Espírito Santo, including Lake Macuco, were formed from sedimentation in ancient tributaries through fluvial and marine influence (Suguio and Kohler, 1992). For the lakes in the plateau, Bozelli et al. (1992) proposed a model to explain the primary productivity and nutrient cycling through the water column. According to these authors, these lakes are thermally stratified, where the isolation of trophogenic zone promotes a decline of phytoplankton primary productivity. Most of the biomass is confined to the metalimnion and decomposed by cyanobacteria (Saijo and Tundisi, 1997). This fact may explain why the values of freshwater algae did not exceed 1% during this phase. Most of the algae probably must have been decomposed in the metalimnion before being deposited.

The palynomorphs consist mainly of fungal spores, have similar hydrodynamic behavior to other palynomorphs and are associated with sediments rich in phytoclasts (Tyson, 1995). Fine particulate organic matter in the MAC-A core may have been produced during the degradation of leaf detritus by fungi. However, we do not exclude the degradation of plankton by bacteria as one of the origins of amorphous organic matter. Higher values of fungal spores are generally found in soils, and for this reason, their occurrence may be also indicative of erosional processes in soils surrounding the lake.

Non-opaque phytoclasts suggest that during this phase the major source of organic matter was terrestrial plants. The highest percentages of TOC (2.82–20.89%) agree with palynofacies data, because organic matter of terrestrial origin is more resistant to degradation (Tyson, 1995). $\delta^{13}C$ values averaging −28.16‰ and high C/N values (~19 up to ~50) also suggest C_3 plants as the major source of the organic matter. The highest concentration of TN (1.1%) occurred in the sample from the top of the sedimentary profile. These carbon isotopic values are similar to those reported by Buso Junior et al. (2013a) for plants that are currently growing in the surrounding areas of the lake. This similarity suggests that the modern vegetation plays an important role in the origin of sedimentary organic matter during Phase 3.

According to Suguio and Kohler (1992), most of the lakes in the study area have been formed as a consequence of damming of the river courses by coastal marine sediments or by fluvial deposits. Furthermore, it is possible that neotectonic mechanisms have played a role during the evolution of the Lake Macuco. Hatushika et al. (2007) studied the high-resolution seismic stratigraphy of one lake in the region and described its younger seismic sequence as a parallel pattern. This younger sequence could be associated with lacustrine deposition after the damming of the alluvial valley. According to these authors, an irregular distribution of the lacustrine deposits in distinct depocenters was observed and suggested a neotectonic control. Therefore, neotectonic movements could have contributed to different subsidence rates along the valley, and these movements could have induced rapid facies changes. As an alternative, the facies differences noted between MAC-A and MAC-C cores may have been tectonically controlled, mainly during sea-level decrease and establishment of the lacustrine basin. The authors do not exclude the possibility that tectonic movements influenced the evolution of the Lake Macuco, once faults occur in the northern part of State of Espírito Santo (Hatushika et al., 2007). However, just few data about neotectonic control are published for the study area.

6. Conclusions

The interdisciplinary approach of this study indicates a mixture of terrestrial and aquatic organic matter sources in the Lake Macuco sedimentary deposits since the Early Holocene. The different proxies were used to identify three intervals that reflect the evolution of this depositional system:

- In the interval between ~7521 and ~4847 cal yr. B.P. (192 cm to 65 cm), the particulate organic matter components were comprised mainly of the amorphous group and non-opaque phytoclasts, reflecting a low energy environment with reducing conditions. Stable isotope and palynofacies data indicate terrestrial and aquatic organic matter sources. A marine influence was confirmed by foraminiferal test linings, dinoflagellate cysts, and mangrove pollen grains. This interval represents an estuarine system, probably in a central estuarine basin that developed during the Holocene transgression.
- An estuarine system with higher fluvial inflow characterizes the interval between ~4847 and ~3800 cal yr. B.P. (65 cm to 27 cm). In this phase, non-opaque and opaque phytoclasts were dominant components, reflecting a higher fluvial influence. The estuary and mangrove vegetation were displaced seaward during the marine regression.
- The sedimentary deposit between 27 cm and 0 cm (<~3800 cal yr. B.P.) is characterized by high relative abundances of amorphous organic matter and non-opaque phytoclasts. There was no evidence of a marine influence, and freshwater algae increased. We define this phase as the beginning of the establishment of Lake Macuco under conditions similar to present-day conditions.

Acknowledgments

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