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## Environmental changes recorded in the Holocene sedimentary infill of a tropical estuary

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### ABSTRACT

Early-to late Holocene sedimentary infill of the Potengi-Jundiai Estuary (Brazil) was studied in a 31.5 m long sediment core. The borehole was drilled in a mangrove area within the central part of the flood tide delta. The sedimentary record, encompassing the last 10 kyr, was studied for sediment texture and for the *n*-alkanes composition of organic matter (OM). Sediments rich in OM and bivalve shells accumulated during the period of sea-level rise, mostly in an intertidal environment of mangrove forest, mudflat and subtidal estuarine flood delta. The characterization of *n*-alkanes (C<sub>10</sub>–C<sub>33</sub>) and lignin methoxyphenols by means of direct analytical pyrolysis (Py-GC/MS) technique allowed determining the sources of the extant OM. Downcore fluctuations were observed in *n*-alkanes and in several *n*-alkane based indices, such as long chain carbon preference index (CPI C<sub>24</sub>–C<sub>33</sub>), long to short chain ratio (L/S) and average chain length (ACL), as well as in the relative proportion of lignin derived compounds. The *n*-alkanes distribution points to changing sources of OM input to the estuarine sediments over most of the Holocene period. Major inputs of terrestrial vegetation alternated with periods of mixed input from aquatic macrophytes as the rate of sea-level rise changed over time. The *n*-alkanes distribution and the lignin markers were useful for reconstructing paleoenvironmental conditions, particularly during the early Holocene when the lower Potengi River valley (now an estuarine area) could accommodate terrestrial forest vegetation before switching to halophytic mangroves as sea levels rose.

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### 1. Introduction

Understanding the climatically driven sea-level changes is an important aspect for coastal planning and management because it helps to follow estuarine ecosystem responses to climate forcing over centennial to millennial time scales. Such knowledge is essential for planning appropriate adaptation options and consequently to minimise the impacts that sea-level change may cause at the land–ocean interface (Gilman et al., 2008). There has been growing interest in the response of mangrove depositional systems to sea-level and climate change, leading to several studies from the Brazilian continental margin (e.g. Behling et al., 2001, 2004; Angulo et al., 2006; Cohen et al., 2012; Pessenda et al., 2012; França et al.,

2013; Boski et al., 2015). Owing to fast sedimentation rates and consequent isolation of OM from bacterial remineralisation (Hedges and Keil, 1999; Gonzalez-Vila et al., 2003), estuarine sedimentary deposits accreted during the most recent postglacial transgression (Boski et al., 2002) are valuable archives for the investigation of the origins, pathways and fates of sedimentary organic matter (SOM). Where established chronostratigraphic frameworks are available, SOM in estuarine sediments has a high potential to retain a continuous, high-resolution record of environmental change. Environmental changes that took place in coastal zones since the Last Glacial Maximum have had a profound impact on the spatial distribution of mangrove vegetation and its associated deposits (Tanabe et al., 2003; Pessenda et al., 2012) at both local and regional scales. Mangrove forests have unique physiological and morphological adaptations to life in the intertidal zones of tropical and subtropical regions (Alongi, 2008; Sanders et al., 2012; França et al., 2013) that result in very characteristic

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signatures of their presence in terms of molecular markers. Terrestrial vascular plants are the major source of long chain *n*-alkanes (C<sub>25</sub>–C<sub>33</sub>), with conspicuous odd-over-even predominance in their carbon chain length (Eglinton and Hamilton, 1967). In contrast, mid-chain length (C<sub>20</sub>–C<sub>24</sub>) markers are attributed to submerged aquatic macrophytes (Huang et al., 1999; Ficken et al., 2000). Long chain *n*-alkanes are particularly resistant to microbial degradation due to post-depositional and diagenetic processes. Therefore, are considered a robust biomarker proxy compared to other hydrocarbons (Meyers, 2003; Garcin et al., 2012) and thus suitable for paleoenvironmental studies in sedimentary archives (Yamamoto et al., 2010). Short chain length (C<sub>14</sub>–C<sub>20</sub>) *n*-alkane homologues mainly originate from bacteria, algae and fungi. Their distributions show no odd or even carbon number predominance in chain length (Grimalt and Albaigés, 1987; Meyers and Ishiwatari, 1993), whereas the C<sub>17</sub> alkane is regarded as a biomarker for algae and photosynthetic bacteria (Cranwell et al., 1987; Meyers, 2003).

Other relevant biomarkers for vegetation are the methoxyphenols, released from lignins during pyrolysis and widely used for the direct study this biopolymer (Meier and Faix, 1992; Galetta et al., 2014; Blázquez et al., 2017). Lignin is mainly composed by three precursors: *p*-coumaryl (H), coniferyl (G), and sinapyl (S) alcohols and their proportion differ between groups of plants; whereas the softwood lignin from gymnosperms is made up by G units, that in hardwood from angiosperms contains G and S, and grass lignin G, S and H units.

The carbon stable isotope composition ( $\delta^{13}\text{C}$ ), has been widely used as a tracer for the organic contribution. The majority of terrestrial plant species have a C<sub>3</sub> type photosystem and characteristic low ( $^{13}\text{C}$  depleted)  $\delta^{13}\text{C}$  values ranging between –24 and –34‰, while  $\delta^{13}\text{C}$  of plants from tropical, arid and saline environments with C<sub>4</sub> photosystem have higher ( $^{13}\text{C}$  enriched) values in the range –6 to –19‰ (Deines, 1980). Hence, variations in SOM  $\delta^{13}\text{C}$  can be related to vegetation changes as well as to SOC dynamics. Additionally, factors like temperature, salinity and moisture can also induce  $\delta^{13}\text{C}$  variations in soils and sediments (Farquhar et al., 1982 and references therein). Slight isotopic differences occur between the different parts and organs of single plants (Hobbie and Werner, 2004) as well as among the different OM components: alkanes and lipids have light C isotopic compositions, depleted in  $^{13}\text{C}$  (Collister et al., 1994), while cellulose and lignin present similar values to those from the original vegetation (Hobbie and Werner, 2004). Thus, rapid degradation of certain SOM components may also induce shifts in isotopic composition (Balesdent et al., 1988).

In general, OM becomes  $^{13}\text{C}$  enriched (less negative  $\delta^{13}\text{C}$  values) during decomposition and evolution processes in sediments. The main hypotheses proposed to explain this  $\delta^{13}\text{C}$  enrichment from plant material to sedimentary organic carbon (SOC) with increasing soil depth are; first, a isotopic fractionation during microbial metabolism of SOM that result in respiration of lighter compounds (Wynn et al., 2005) and second, the Suess effect i.e., the increase of atmospheric  $^{12}\text{C}$  due to the combustion of fossil fuels causing a depletion of OM  $\delta^{13}\text{C}$  in the soil surface (Feng and Epstein, 1995; Francey et al., 1999).

Direct analytical pyrolysis (Py-GC/MS) is a powerful ‘fingerprinting’ tool that provides rapid and semi-quantitative information relating to the molecular composition of complex OM (De la Rosa et al., 2012; González-Pérez et al., 2014). This technique involves no solvents, chemical degradation, fractionation, purification, or derivatisation of products and requires only milligram quantities of dry sediment for direct analysis. The Py-GC/MS technique has been widely applied in the study of SOM (De la Rosa et al., 2012; Faria et al., 2015). It has also been applied to the paleoclimatic studies in peat bogs (McClymont et al., 2011), as well as in carbon

sequestration studies (Vancampenhout et al., 2009).

The delivery and deposition of allochthonous and autochthonous OM to NE, Brazilian coastal margin is not well studied, leaving a large gap in our understanding of the fate of OM in coastal environments. In this work, we used a combination of complementary analyses, including conventional SOM characterization, carbon stable isotope composition and analytical pyrolysis, to study Holocene OM inputs into the SOM pool of estuarine mangrove sediments in the Potengi Estuary (NE Brazil) to track the Holocene development of this vegetation type.

## 2. Geological setting and general description of the study area

The Potengi Estuary forms one of the largest embayments of that type on the Brazilian north-eastern coast. It is situated in the state of Rio Grande do Norte, northern Brazil and is formed at the confluence of the Potengi, Jundiá and Doce rivers. The three rivers jointly drain an area of about 5000 km<sup>2</sup>. Over its final 8 km, the main estuarine channel attains the width of 900 m (Boski et al., 2015). Fig. 1 shows the geological settings of the study area. Owing to limited freshwater discharge and the low gradient of the river bed, tidal influence extends up to 30 km upstream of the Jundiá mouth (Barbosa, 1983). The estuary exhibits well-mixed conditions with an overall salinity oscillating between 29.5 and 34.5‰ during rainy and dry seasons respectively (De Miranda et al., 2005). Tides are semidiurnal and tidal amplitude in the estuary is about 2.8 m (mesotidal regime) with current velocity approaching 1.0 m/s. The climate in this region is equatorial hot and humid (Köppen AS' climate category), with precipitation concentrated in the months from February to July. The annual average temperature is 26.8 °C and precipitation varies from 1300 to about 2000 mm per annum.

The main estuarine intertidal zone encompasses patches of mangrove forest, shrimp ponds, mudflats, tidal creeks and channels. Mangrove forest has been reduced to 40% of its original extent by shrimp aquaculture and currently occupies ca. 1488 hectares (Souza and Ramos e Silva, 2011). It is composed of three arboreal species: i) *Rhizophora mangle* (red mangrove), the dominant species (approximately 62% of the total forest cover) found in proximity to channel margins; ii) *Avicennia germinans* (black mangrove, 17% of the total forest cover) present throughout the forest as scattered individuals more towards the higher lying part of halophytic forest; and iii) *Laguncularia racemosa* (white mangrove, 21% of the forested area) occurring at the highest positions above the maximum tide. Heavy metals, urban waste and sewage effluents from adjacent three cities contribute to low conservation status of the mangrove area.

## 3. Methodology

### 3.1. Borehole drilling, sampling and granulometric analysis

Borehole IG-8 was drilled by means of a rotary drilling rig (TECOINSA -TP-50-D with core diameter 2 ½ inch). The 2 ½ inch-diameter core was extracted from 1m-long sections of core barrel, sealed in plasticized cardboard for transport and sectioned in two halves in the laboratory. After sampling, the samples were dried at 40 °C and kept in the refrigerator until further analyses. Granulometric analyses were performed after carbonate removal with hydrochloric acid (1.2N HCl). The samples were sieved through a 250 µm sieve and the fractions <250 µm were analysed in a Malvern Mastersizer Microplus instrument using demineralised water with 1 g of Calgon L<sup>-1</sup>, added to avoid flocculation of clay minerals. The results of granulometric analyses are presented together with core profiles in the form of mean grain size plots.

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