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# The Influence of Urban Effluents on the Elemental C/N Ratio in a Tropical Coastal Area of Northeastern Brazil

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**ABSTRACT**

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The present study was performed in a tropical and highly eutrophic estuarine system in the urban coastal area of Greater Recife, Pernambuco, northeastern Brazil. Organic carbon is introduced into these urban estuaries through domestic and industrial sewage waste. One indicator of anthropogenic influence is the concentration of organic matter, including the suspended elemental forms of carbon and total nitrogen and the C/N ratio. The distribution of the concentrations of both particulate organic carbon and total nitrogen (POC and TN, respectively) were studied in two parallel transects in the estuarine plumes of Pernambuco's major rivers, the Capibaribe (CP) and the Jaboatão (JP), from 2010 to 2011. The archipelago of Fernando de Noronha, with relatively little human influence, was selected as the control area. The particulate organic matter exported via estuarine flow was defined by elemental carbon median values of 2.04% and 0.97%. The C/N ratio of the Capibaribe and Jaboatão plumes featured median values of 6.60 and 9.30, respectively. The principal components analysis (PCA) results suggest that the percentage of POC was influenced by rainfall and that the percentage of TN was influenced by the transport of suspended particulate matter. These differences may result from different loads of human activities and estuary transport dynamics, and the coastal region highlightsthe complexity of the distribution of organic matter in these environments.

**ADDITIONAL INDEX WORDS:** *Elemental Carbon, total Nitrogen, estuarine plume, archipelago*

## INTRODUCTION

Estuaries and coastal areas with rapid population growth are the ultimate repository for a vast array of organic substances discharged without treatment. Consequently, changes have occurred in the land-sea interface, damaging marine biodiversity (Thomas *et al.*, 2009; Guenther *et al.*, 2015). The continental supply of particulate organic matter (POM) to coastal areas includes anthropogenic organic debris and waste transported by rivers. Due to the existence of numerous sources, we conducted a qualitative and quantitative evaluation of POM in a coastal area to determine the predominant source. Because there are many processes that control the transport and remineralization of POM, the source of POM can be determined by quantifying the elemental content (% C, % N, and C/N ratio).

POM represents 50% of the organic export of estuaries to coastal areas, and 35% of this matter is mineralized in the estuaries (Degens and Ittekkot, 1985). Based on elemental content, POM can be characterized as allochthonous (natural or anthropogenic), *i.e.* delivered via estuaries, or autochthonous, *i.e.* associated with primary productivity, in a coastal area (Middelburg Herman., 2007).

The characterization of the source of POM using elemental analysis is based on the structural differences in the organic matter produced by terrestrial and marine primary producers (Meyers, 1994). Given the scarcity of information on the origin of POM in the coastal areas of northeastern Brazil, our aim was to evaluate the spatial and temporal variations in the elemental composition of C and N and the C/N ratio in areas under the direct influence of two urban rivers in Greater Recife. This region has been the main trade center of NE Brazil since the 16th century and considered the 6th most populated area in Brazil (IBGE, 2010).

### Background

The continental shelfin the vicinity of Greater Recife is narrow, with an average width of 35 km, and features a sedimentary cover composed of terrigenous and biogenic carbonate sediments (Manso *et al.*, 2003). The inner continental shelf in front of Greater Recife is fertilized with the riverine inputs of the Jaboatão and Capibaribe rivers, which contain high concentrations of nutrients and chl-a (Montes *et al.*, 2011). According to the Köppen classification, the climate is humid

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tropical or type As, with a rainy season between April and August and a dry season between September and March. The average annual rainfall is 2,272 mm, the air temperature is 25.6°C and the relative air humidity is 90% (Torres and Machado, 2011). The coastal waters exhibit small variations in the hydrological conditions, with temperatures of approximately 28°C and salinity values near 35 (Resureição *et al.*, 1996). The studied rivers (Figure 1) receive discharges of domestic and industrial effluents from the hydrographical basin (Ribeiro Neto, 2014).

The Capibaribe River basin has a drainage area of 7,557 km$^2$ and flows through the metropolitan area of Recife (07°41'20" S and 08°19'30" W). The Capibaribe River is 240 km long, has a flow rate of 19 m$^3$s$^{-1}$ and supplies water to a population of approximately 1,328,361 inhabitants (Montes *et al.*, 2011).

The Jaboatão River drains an area of approximately 1,022 km$^2$ (8°12'30"S - 8°15'0"S and 34°57'30"W - 34°55'0" W) and is used by a population of 1,347,053 inhabitants (CPRH, 2011).

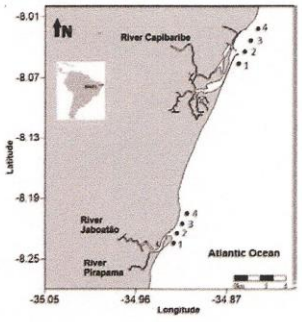


Figure 1. Study site location. The inset shows the state of Pernambuco in Brazil.

The Fernando de Noronha Archipelago (FNA) is located onthe mid-Atlantic ridge (3°50' and 3°52' S and 32°24' and 32°28' W), on the top of a 4,000-m-tall underwater mountain (Batistella, 1993) (Figure 2).

FNA has a tropical climate with two well-defined seasons: a dry season from September to February and a rainy season from March to August. The average annual air temperature is 28°C, with an air temperature amplitude of 4°C. The average rainfall in the region is 1,300 mm. Prevailing winds (trade winds) are southeast, especially between June and August (CPRM, 2015), and this area is directly influenced by the South Equatorial Current (SEC).

## METHODS

Sampling occurred between May 2010 and September 2011 along a profile parallel to the coastline, always in the slack water of low tide following the direction of the estuarine plume of the rivers Capibaribe (CP) and Jaboatão (JP). The samples were collected at four points spaced 1 km apart, and samples were taken from the sub-surface layer (0.5 m) and 1 m above the bottom (Figure 1).

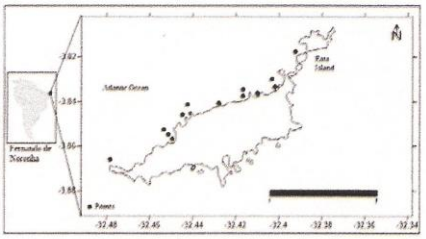


Figure 2. Location of sampling stations in the Fernando de Noronha Archipelago.

In the Fernando de Noronha Archipelago (FNA), sampling occurred in the northwest side (Figure 2).

The water samples were collected with Niskin bottles. Salinity and temperature were measured *in situ* using a SeaBird 19 profiler, and the dissolved oxygen samples were analyzed using the modified Winkler method (Strickland and Parsons, 1972). Suspended particulate matter (SPM) was determined according to Baumgarten *et al.* (1996), and chlorophyll *a*was measured according to the method described in (UNESCO, 1966). Particulate organic carbon (POC) and total nitrogen (TN) were calculated using the method of the U.S. Environmental Protection Agency, described by Zimmerman *et al.* (1997).

To determine POC and NT, the samples were filtered using a vacuum pump in a filtration kit decontaminated with HCl. The filters were Whatman grade GF/F. Before use, they were calcined in a muffle kiln at 450°C to remove any organic residue.

The final filtered volumes were 200-300 mL of water from the plume and 2 L of water from the FNA. The filters were subsequently preserved at a temperature of -20°C until the testing period, when they were thawed and fumigated in a desiccator with HCl for 24 hours. After fumigation, the filters were dried in an oven at 60°C for 24 hours. Two aliquots (measuring 1 cm$^2$) were removed from the filters, weighed in tin capsules and analyzed in an EuroVector elemental analyzer coupled to a Delta V Advantage isotope ratio mass spectrometer.

## RESULTS

The maximum observed rainfall, 755.7 mm, occurred in May 2011. Compared to the average of the last 20 years (1992-2011), during which the average total annual rainfall was 2,232.7 mm, the total rainfall in 2011 (3,245.7 mm) was 45.37% above average. The average DO concentration was similar for both the CP (4.10 mg L$^{-1}$) and JP (4.36 mg L$^{-1}$) (Table 1). The SPM in the Capibaribe and Jaboatão plumes was 24.53 mg L$^{-1}$and17.83 mg L$^{-1}$, respectively (Table 1). The salinity differed slightly

between the Capibaribe plume (32.33) and the Jaboatão plume (33.22), both categorized as euhaline water. The average concentration of chlorophyll *a* was higher in the CP (3.72 mg $m^{-3}$) than the JP (2.39 mg $m^{-3}$). The highest POC percentages were recorded in the CP, where the minimum and maximum percentages of elemental carbon were0.15% and 39.47%, respectively. The minimum and maximum COP percentages in the JP were 0.34% and 6.21%, respectively. The minimum and maximum percentages of TN were 0.08% and 5.20%, respectively, in the CP and 0.07% and 0.64%, respectively, in the JP (Table 1).

The samples collected from the control area in the Fernando de Noronha Archipelago featured minimum and maximum POC percentages of 0.46%and3.69%. The highest value was recorded in December 2010 in the Baía dos Golfinhos, and the second highest POC value,3.37%, was recorded in April 2011 in Biboca beach. These values correlated with the chlorophyll *a* concentrations, which were also higher in the dry season on Biboca beach, with a peak value of 2.61 mg $m^{-3}$.

Table 1. *Minimum, maximum and mean values of the parameters analyzed in the studied areas.*

| | Capibaribe plume | | Jaboatão plume | |
|---|---|---|---|---|
| Parameters | mean | range | mean | range |
| Photic zone depth (m) | 1.85 | 0.50-6.00 | 2.52 | 0.65-6.7 |
| Salinity | 32.33 | 14.27-37.16 | 33.22 | 10.77- 38.50 |
| DO mL $L^{-1}$ | 4.10 | 1.73-7.78 | 4.36 | 2.26-5.63 |
| SPM mg $L^{-1}$ | 24.53 | 2.80-64.00 | 17.83 | 0.80-39.69 |
| %N | 0.92 | 0.08 -5.20 | 0.28 | 0.07 -0.64 |
| %C | 3.2 | 0.15-39.47 | 1.56 | 0.34 -6.21 |
| C/N | 6.37 | 0.62-27.93 | 9.17 | 2.63-12.79 |
| Chl *a* mg $m^{-3}$ | 3.72 | 0.24-19.29 | 2.39 | 0.21-9.67 |

More than 50% of the collected TN samples were below the detection limit, and the minimum and maximum recorded percentages were 0.11% and 0.31% on Ilha Rata and Boldró, respectively. The higher values were observed during the rainy season.

During the sampled period, the average C/N ratio was 6.37, ranging from 0.62 to 27.93 in the CP. The maximum value was measured in front of the inlet that connects the estuarine system to the coastal area. In the JP, the C/N ratio ranged from 2.63 to 12.79, with an average of 9.17 (Table 1). Here, the maximum value was measured north of the plume.

## DISCUSSION

The input of organic carbon has been observed in urban estuaries that receive domestic and industrial sewage waste and has been linked to decreased dissolved oxygen levels. In addition to these sources,mangroves and benthic macroscopic autotrophs generate natural debris and other biodetritus (*e.g.*, feces). The studied coastal region is affected by heavily impacted river plumes, as shown by others studies (Montes *et al.*, 2011, Paulo *et al.*, 2011, Oliveira *et al.*, 2014, Guenther *et al.*, 2015).

According to Noriega *et al.* (2013), previous research on the port of Recife and adjacent estuaries has shown that rainfall and the consequent contribution of the tributaries can modify the quality of circulating water and produce temporal and spatial changes in the dynamics of coastal waters and in phytoplankton biomass, as also observed in the present study.

The dissolved oxygen levels were high throughout the area, ranging from unsaturated to oversaturated water, which shows a strong dilution of estuarine waters by marine waters. The river flow increased in the rainy season, consequently increasing the amount of SPM in both the CP and the JP.

The salinity in the CP and JP varied seasonally from mesohaline, influenced by flooding during the rainy season, to euhaline, influenced bya greater evaporation rate in the dry season. According to Noriega *et al.* (2005), in the rainy season, factors such as river discharge, low evaporation and rainfall create a significant vertical gradient, whereas in summer the waters are well mixed and uniformly saline with a higher rate of insolation. Guenther *et al.* (2015) suggested this estuarine system is vertically uniform with flood and ebb currents entering and leaving the system equally through the whole water column (*i.e.*, no thermohaline circulation was established).

The chlorophyll *a* concentration also varied seasonally in the plumes and did not exhibit trends that indicate strong changes in the trophic structure via eutrophication processes.

Seasonally, the percentages of COP were highest during the rainy season in both plumes; however, the maximum values in both plumes were recorded in September 2011. Comparing the two study sites, the highest percentages of elemental carbon were recorded in the CP.

In relation to spatial variation, during the rainy season, the POC percentages in the JP varied significantly ($p<0.05$), which suggests a predominance of autochthonous organic matter.

These higher percentages recorded in the rainy season highlight the importance of the estuary as an exporter of organic matter and nutrients to the coastal area, promoting primary production and, consequently, the food web on the continental shelf of Pernambuco. During this period, an extreme event was recorded: The observed rainfall (547.70 mm)was greater than the historical average (410.60 mm), which increased the river flow, delivering more SPM to the coastal areas. The opposite pattern was observed in the samples collected during the dry season: Low values of elemental carbon were observed atthe sampling sites most distant from the plume outlet, suggesting the presence of allochthonous organic matter. The maximum values obtained in both plumes (Table 1) at the beginning of the dry season indicate the diversity of factors, including climate change and human activities, which influence this distribution. Because the average rainfall during this month was 29.6 mm, which is considered low, the previously transported matter remained in the area and experienced the processes of suspension, deposition and remineralization.

The allochthonous fraction can be diluted, deposited and/or degraded by bacteria until the remineralization process is complete. Under a heavy organic matter load, the dissolved oxygen levels can become depleted. Guenther *et al.* (2015)

reported that the dissolved organic carbon concentrations for the Capibaribe estuary ranged from 475 to 975 µM. Human activities in urban areas also influence the cycle of elements due to the improper use of soil, organic waste dumps and climate change, all of which directly or indirectly influence the cycle and flow of carbon between the mainland and the coastal areas. Unlike the elemental composition of carbon, which can indicate autochthonous production or the export of plant waste material and organic debris to the platform, the elemental signature of organic matter is subject to modification, especially in areas under the influence of anthropogenic sources (Fogel *et al*., 1993)

The suspended material collected on the continental shelf was characterized by low percentages of elemental nitrogen. In approximately 50% of the samples collected, the nitrogen levels were below the detection limits. This indicates that most of the nitrogen could be in the dissolved fraction. This finding is consistent with the nature of the predominant sediments in the port channel, which are moderately siliciclastic with very coarse grains and carbonate values greater than 30% (Oliveira *et al*., 2014). These authors also claim that the nature of the analyzed sediment reflects a high-energy system, resulting in a low rate of organic matter deposition in the lower estuary.

The highest percentages of elemental nitrogen in both plumes were recorded in the rainy season. Total nitrogen in the plume of the Jaboatão River varied little during the studied period, and the percentages found in the JP were lower than the percentages found in the plume of the Capibaribe River.

The samples collected in the FNA showed an interesting contrast between natural sources and anthropogenic activities. This archipelago is a protected area, but tourism is intense year-round. The largest concentrations of organic material were observed at the Baía dos Golfinhos (Dolphin bay) station. This result indicates the importance of natural sources for the cycling of elements because a large group (>500 individuals) of spinner dolphins (*Stenellalongirostris*-Delphinidae) gather at this site every day for resting, mating, feeding, defecation and regurgitation, thus contributing to the influx of allochthonous debris (Silva-Jr *et al*., 2007). In contrast, the high percentages at the Biboca beach station are related to autochthonous sources (high chlorophyll *a* concentrations) and allochthonous sources because Biboca beach receives the wastewater from the FNA treatment plant.

During the rainy season, the surface runoff from the watersheds via estuarine flux did not transport high loads of organic matter to the estuarine plumes based on the low C/N ratios. Therefore, urban effluents (domestic and industrial) represent the major influence on the organic matter cycle. The highest values were found in the dry season, thus corroborating the hypothesized presence of strongly autochthonous particulate matter. The C/N ratios measured in the coastal region of Pernambuco were directly related to the chlorophyll *a* concentrations, especially for the Capibaribe estuary plume, where the maximum value was found in front of the mouth of the estuarine system. In the JP, the maximum C/N ratio value was recorded in the farther station, indicating that remineralization processes are producing inorganic nutrients and $CO_2$, enhancing the density of primary producers.

The C/N was also observed to increase towards the bottom of the water column, likely due to N assimilation processes, nitrification, decomposition of particulate organic matter in the surface layer, depositional processes, and vertical distribution. This pattern was also observed for POC and TN in other sampling sites.

The lowest C/N ratio values exhibited a negative relationship with the SPM values and a positive relationship with the TN values. These relationships could be the result of increased biological activity and advective processes that caused the resuspension of sediments and a>50% increase in the SPM levels.

In the FNA area, the higher variations in the C/N ratio coincided with the period of lower concentrations of chlorophyll *a*. This correlation does not reveal the sources or origins of the low levels of organic matter at the site because >50% of the samples featured elemental nitrogen levels too low to detect, preventing the calculation of the C/N ratio.

The principal component analysis (PCA) showed more variance for the CP. The first two factors explained relatively little (53.25%) and showed significant variation between the dry and rainy seasons ($p<0.05$).

In the JP, the first two factors explained 72.51%, and the spatial variations (vertical and horizontal) showed significant differences due to both dispersion and remineralization rates.

## CONCLUSIONS

The sources of the organic fractions of SPM in the coastal region of Pernambuco are the terrigenous matrix and autochthonous debris, indicating that anthropogenic sources directly and indirectly influence the concentrations of elemental carbon and total nitrogen in these environments.

The results reveal that the distributions of these elements (%C, %N, and the C/N ratio) are highly influenced by the weather conditions, especially in the Capibaribe plume, and the high load of organic and inorganic matter during the rainy season. These results also indicate that human activity has a great influence on the carbon and nitrogen cycles.

The plume of the Jaboatão River had less influence than the plume of the Capibaribe River due to a lower river flow and less SPM.

The variation in POC and TN sources and the indication of the influence of chlorophyll in this ratio show that the organic material is allochthonous and of continental origin and that the high primary production within the estuary is the result of eutrophication processes. Phytoplankton blooms occur in environments with high nutrient loads, forming a source of particulate and dissolved organic material that is subsequently decomposed.

In the FNA, the levels of organic matter are low, and the anthropogenic organic matter differs from natural organic matter, highlighting the importance of studies on these elements. Although low, these values can be used as a reference in the study of organic contamination processes.

The obtained C and N values indicate that there are cause-and-effect relationships present in the coastal area of Pernambuco.

The transport of allochthonous organic matter can induce autochthonous production and cause variations in the OM levels and C/N ratios.

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