

# Petroleum hydrocarbons in Brazilian Northeast continental shelf waters: baseline values

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## ABSTRACT

Water quality on the continental shelf off the eastern Brazilian Northeast region was investigated in two oceanographic campaigns during winter and summer, when vertical profiles of salinity, temperature, turbidity, chlorophyll and dissolved oxygen were recorded. Dissolved / dispersed petroleum hydrocarbons (DDPH) were also analyzed in sub-superficial water samples using fixed wavelength fluorescence spectroscopy methods. Shelf waters are dominated by Tropical Waters, with high salinity ( $> 36.5 \text{ g kg}^{-1}$ ) and temperature ( $> 26 \text{ }^{\circ}\text{C}$ ) and little continental influence. Turbidity was higher during winter, which may be due to the higher continental contribution and/or higher wave action. Chlorophyll was mostly  $< 1 \mu\text{g L}^{-1}$  and dissolved oxygen saturation was predominantly high ( $> 90\%$ ), but innermost costal stations were influenced by the Capibaribe estuary. Median DDPH concentrations were as low as  $0.07$  and  $0.04 \mu\text{g L}^{-1}$  Carmópolis oil equivalents during winter and summer respectively, and  $0.02 \mu\text{g L}^{-1}$  chrysene equivalent for both periods. An exception was observed off Suape Harbor ( $0.35 \mu\text{g L}^{-1}$  Carmópolis oil equiv. and  $0.13 \mu\text{g L}^{-1}$  chrysene equiv.), suggesting navigation activities as the main local source. The overall DDPH median of  $0.06 \mu\text{g L}^{-1}$  Carmópolis oil equivalents and  $0.02 \mu\text{g L}^{-1}$  chrysene equivalents are proposed as the baseline concentration for non-polluted coastal water for the tropical western Atlantic Ocean margin.

**Descriptors:** Chrysene, Carmópolis oil, Chlorophyll, Dissolved oxygen, Turbidity.

Petroleum is the main known energetic source and its exploitation and production is greater than ever to supply the increasing worldwide demand. Polycyclic aromatic hydrocarbons (PAH) are a key group of compounds (~8 to 15%) of oil composition and have become a major environmental concern because they are ubiquitous. PAH in environments can be harmful due to their potential biochemical and behavior alterations (Mrdakovic *et al.*, 2016, Dissanayake *et al.*, 2010, Torreiro-Melo *et al.*, 2015) beside carcinogenic and teratogenic effects

(Magi *et al.*, 2002; Boonyatumanond *et al.*, 2006; Chen *et al.*, 2013; Rahmanpoor *et al.*, 2014). PAH can be introduced in coastal waters directly by oil spill accidents, shipping activity, industrial and domestic effluents, fluvial runoff (Zaghden *et al.*, 2007; Lemos *et al.*, 2014), or indirectly by atmospheric deposition of products of oil or organic matter combustion (Boehm, 2005; Meire *et al.*, 2007; Lemos *et al.*, 2014; Maciel *et al.*, 2015a; Arruda-Santos *et al.*, 2018).

PAHs, individually or expressed as total concentration (dissolved / dispersed petroleum hydrocarbons - DDPH), have been reported in water and sediments from many Brazilian estuaries and bays (e.g., Todos os Santos Bay by Venturini and Tommasi, 2004; Patos Lagoon by Medeiros *et al.*,

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2005; Guanabara Bay by Silva et al., 2007; Cocó and Ceará estuaries by Cavalcante et al., 2009; Santos Estuary by Martins et al., 2011; Arruda-Santos et al., 2018). Assessments were also made near oil industry hotspots as harbors (São Sebastião channel by Zanardi et al., 1999a,b; Silva and Bicego, 2010; Lemos et al., 2014; Maciel et al., 2015a; 2016; Zanardi-Lamardo et al., 2018) and offshore extraction zones (Potiguar basin by Wagener et al., 2011) or open waters (South West Atlantic waters, Bicego et al., 2002). This study presents the very first assessment on dissolved / dispersed petroleum hydrocarbons (DDPH) on the eastern sector of the Brazilian Northeast region, off Pernambuco State in open waters on the continental shelf. This region is relatively far from offshore active oil extraction zones, ~400 km southward from Potiguar Basin (Rio Grande do Norte State) and ~ 350 km northward from Sergipe Basin (Sergipe State). The nearest potential sources are the continental runoff through the estuaries and the port and navigation activities. However, during August and September 2019, many tons of crude oil washed on northeastern Brazil shores (Lourenço et al., 2020). The origin of the oil is still unknown, but given its distribution on landing it appears to have originated from a far offshore source, drifting ashore through regional winds and the South-Equatorial Current (Schettini et al., 2017).

Pernambuco State coast has several estuaries that could act as potential PAH sources from the continental drainage. Instead, a combination of geomorphology, tidal regime and fluvial discharge act as a filter, trapping most of the continental load of pollutants (Maciel et al., 2015a; 2016; Arruda-Santos et al., 2018). The Capibaribe estuary is the main coastal system in terms of social and economic importance because it crosses the densely populated Recife Metropolitan Area. This system has also high trapping capacity (Schettini et al., 2016a), but the higher loads of pollutants discharged in its lower stretch can effectively export material to the adjacent shelf (Maciel et al., 2016; Schettini et al., 2016b). Another potential coastal source is the Suape Estuary, located ~50 km south of Recife, where a major industrial and harbor development started in the mid- 1980s (Lemos et al., 2014; Zanardi-Lamardo et al., 2018).

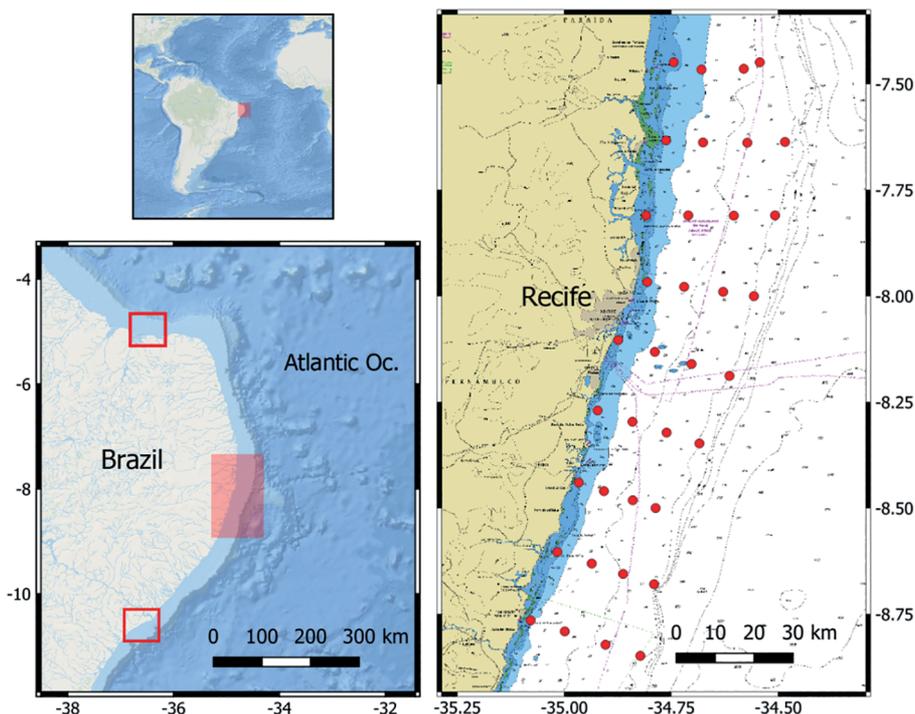
The local continental shelf is ~40 km wide with the shelf break at ~60 m deep (Brazilian Navy Nautical Chart N.o 22.200). A better

geomorphological description of the shelf is provided by Barcellos et al. (2020). It is dominated by the presence of oligotrophic Tropical Water with salinity  $> 36.5 \text{ g kg}^{-1}$  and temperature  $> 26 \text{ }^{\circ}\text{C}$ . The continental inflow of fresh water is very limited by the regional semi-arid climate countryside and small river drainage basins. The regional tides are semi-diurnal, ranging between 1.5 and 3 m. The wind regime is dominated by the South Atlantic High and blows steadily westwards. Wintertime currents are stronger and northwards and summertime currents are sluggish and southwards (Schettini et al., 2017; Domingues et al., 2017).

Two oceanographic campaigns were carried out during austral winter (18-22 August 2013) and summer (10-14 January 2014), when water properties (salinity, temperature, turbidity, chlorophyll and dissolved oxygen) were recorded and water samples collected to analyze dissolved and/or dispersed petroleum hydrocarbons (DDPH). A total of 36 sampling stations were distributed across the shelf, from as near as possible from the shore until the shelf break between  $7.5^{\circ}$  to  $9^{\circ}$  south, comprising mostly the continental shelf off Pernambuco State (Figure 1). During the campaigns, currents were monitored at a central point study area with a moored acoustic Doppler current profiler (Schettini et al., 2017).

The water properties were recorded with a CTD probe, JFE Advantech, Rynko Profiler, with sensors resolutions of: (a) depth:  $0.01 \text{ m} \pm 0.3 \%$  FS; (b) temperature:  $0.001 \pm 0.01 \text{ }^{\circ}\text{C}$ ; (c) conductivity:  $0.001 \pm 0.01 \text{ mS m}^{-1}$ ; (d) turbidity by optical backscattering:  $0.03 \pm 0.3 \text{ FTU}$ ; (e) chlorophyll by fluorimeter:  $0.01 \text{ ppb} \pm 0.1 \%$  FS; and (f) dissolved oxygen by phosphorescence:  $0.004 \text{ mg L}^{-1} \pm 2\%$  FS. Salinity was calculated using temperature and conductivity by PSS-78 (UNESCO, 1981). Surface water samples (1 m depth) were taken with 4 L pre-cleaned amber glass bottles attached to a stainless steel device. Water samples were collected at the bow just prior to stopping the boat to avoid potential contamination from the boat engine oil.

Water samples were liquid-liquid extracted according to previous studies (Lemos et al., 2014; Zanardi-Lamardo et al., 2018). In brief, 20 mL of n-hexane (pesticide grade) was added to the 4 L amber glass bottle immediately after water sampling. An aliquot of 100  $\mu\text{L}$  of a deuterated PAH solution (1,000  $\text{ng mL}^{-1}$  of AccuStandard, Internal Standard Mix containing naphthalene-d8, acenaphthene-d10,



**Figure 1.** Localization of the study area in South America (a) and in northeastern Brazil (b). The pink rectangle is the assessed area, and the red squares indicate the areas of active oil rigs of Sergipe Basin (in the south) and Potiguar Basin (in the north). The sampling stations (red circles) are presented in (c); ports of Recife and Suape are indicated.

phenanthrene-d10 and chrysene-d12) was added as recovery surrogate to all samples, including the field blank. The bottle was vigorously agitated for 2 minutes to extract hexane-soluble organic matter. In the laboratory, the organic extract was separated from water using a separating funnel. Anhydrous  $\text{Na}_2\text{SO}_4$  (previously combusted at 450 °C) was added to the extract for binding traces of water. Subsequently, the extract was concentrated down to 10 mL on a rotary evaporator. DDPH were analyzed by fluorescence spectroscopy (SpectraMax M3 Molecular Devices), using UV-Vis fixed wavelength methodology. The organic extract was excited at 310 nm and fluorescence emission was recorded at 360 nm (Ehrhardt (1983); Knap et al., 1986; Zanardi et al., 1999a, 1999b; Lemos et al., 2014). Spectroscopy is a sensitive, fast and cheap technique for measuring petroleum (aromatic) hydrocarbons in water (Law et al., 1987; Ehrhardt and Petrick, 1989; Bícigo et al., 2002; Li et al., 2010). This methodology was originally developed for monitoring petroleum hydrocarbons in marine waters and expressed as dissolved/dispersed petroleum hydrocarbons. It does not discriminate sources of hydrocarbons, though. In fact, results may include products of oil degradation, combustion products

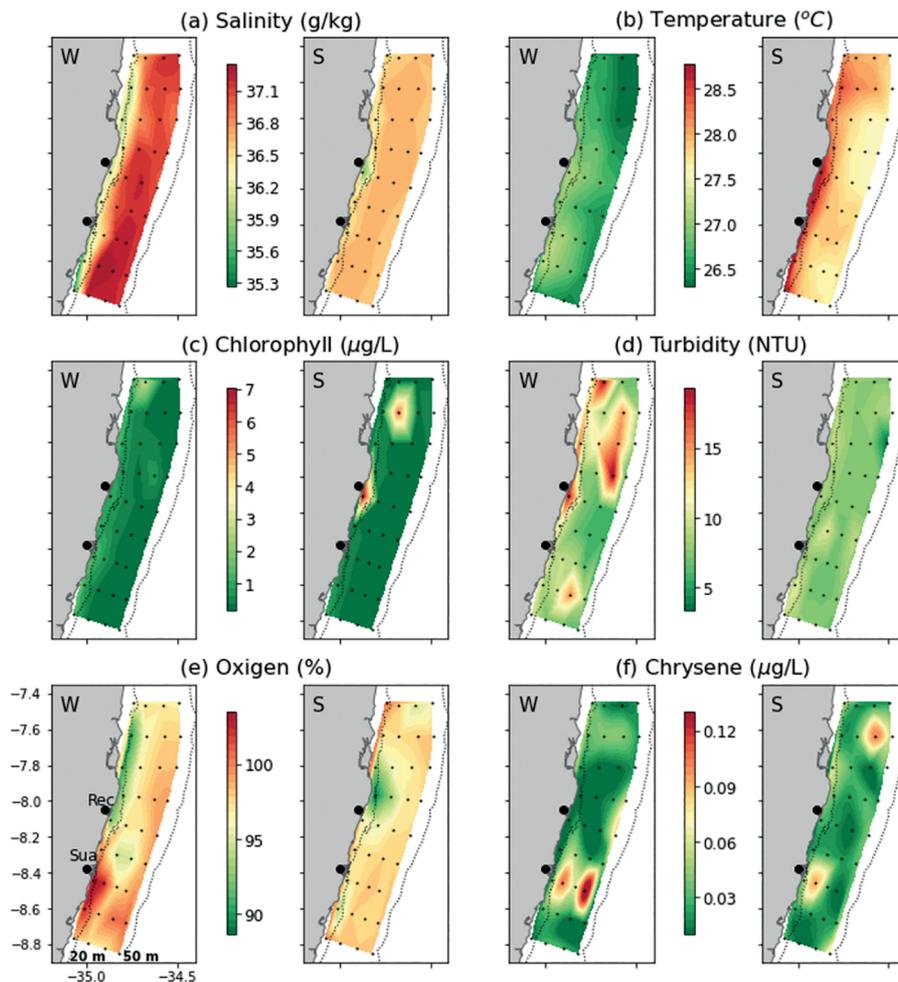
and other natural materials that absorb/emit light at the 310/360 nm wavelength pair. This excitation/emission pair is typical of benzene-containing hydrocarbons, therefore results were expressed as dissolved/dispersed petroleum hydrocarbons (DDPH) in bulk water. Despite limitations, ultraviolet fluorescence is a good tool for understanding the fate of aromatic hydrocarbons after release in the water and has been proposed for investigation of petroleum hydrocarbon accumulation (hot spot) (Knap et al., 1986; Zanardi et al., 1999a;b; Lemos et al., 2014; Zanardi-Lamardo et al., 2018; Arruda-Santos et al., 2018).

DDPH concentrations were calculated based on two analytical curves: one prepared with chrysene (99% purity, Sigma-Aldrich) and the other prepared with Carmópolis weathered crude oil. Concentration in the analytical standards ranged between 0 and 2.5  $\mu\text{g L}^{-1}$ . Results were expressed as chrysene and Carmópolis oil equivalents, respectively. Considering that oil is a complex mixture, the use of chrysene as a standard (IOC, 1984) has raised some disagreement among researchers although it has been accepted because it provides a worldwide basis for comparison of results. On the other hand, Carmópolis oil comes

from one of the oldest active oil fields along the northeastern coast of Brazil, and it has been widely used as a standard oil for the Brazilian coast, southwestern Atlantic and western Antarctic Peninsula (Zanardi et al., 1999a; b; Bicego et al., 2002; 2009; Lemos et al., 2014; Maciel et al., 2015b; Arruda-Santos et al., 2018; Zanardi-Lamardo et al., 2018). Field blank samples and internal standard recovery (surrogate) were used for quality control purposes. The field blank fluorescence was subtracted from sample readings. The method detection limit (DL) was calculated as three times the standard deviation of blank concentrations (Quevauviller et al., 1992). DLs expressed in chrysene and Carmópolis oil equivalents were  $0.01$  and  $0.03 \mu\text{g L}^{-1}$ , respectively.

Mean recovery of the surrogate was  $70.7 \pm 8.4\%$  (95% level of confidence).

Table 1 presents the summary of basic statistics of the near surface waters. A detailed analysis of the salinity and temperature distributions and currents regime are presented in Schettini et al. (2017) and Dominguez et al. (2017). The main aspects are: (1) the shelf is dominated by high salinity ( $s > 36.5 \text{ g kg}^{-1}$ ) and high temperature ( $t > 26 \text{ }^\circ\text{C}$ ) Tropical Water originated by the southern branch of the Atlantic South Equatorial Current, which bifurcates south of the study area; (2) even during the winter, when the local river contribution is maximum, fresh water influence is very low and limited to the inner shelf (Figure 2a); (3) The horizontal temperature distribution is relatively



**Figure 2.** Distribution of (a) salinity ( $\text{g kg}^{-1}$ ), (b) temperature ( $^\circ\text{C}$ ), (c) chlorophyll ( $\mu\text{g L}^{-1}$ ), (d) turbidity (NTU), (e) oxygen saturation (%), and (f) DDPH expressed as chrysene equivalents ( $\mu\text{g L}^{-1}$ ) on surface (top 1.5 m) waters from Pernambuco continental shelf during (W) winter (Aug 2013) and (S) summer (Jan 2014). Dots indicate sampling stations and dashed line represents isobaths.

monotonous with warmer waters at the inner shelf. The winter water temperature was  $\sim 2$  °C cooler than in summer (26 °C and 28 °C, respectively) (Figure 2b).

Chlorophyll, turbidity, oxygen saturation and DDPH have not been described for the region yet. Chlorophyll mean values were 0.6 and 0.7  $\mu\text{g L}^{-1}$  during winter and summer, respectively (Table 1). The chlorophyll spatial distributions (Figure 2c) present a mild gradient towards the shore, and some patches of higher chlorophyll especially during summer. One patch is adjacent to the Capibaribe estuary mouth and another is located to the north. Both presented values of  $\sim 5 \mu\text{g L}^{-1}$ . Turbidity was higher during winter than summer, which can be attributed to the increased continental runoff during the rainy season (winter), potentially delivering more fine sediment to the shelf, and also due to the higher wind action expected during the winter (Figure 2d). The winter time distribution does not show any particular pattern, though the higher values were recorded in the north. During summer there is a mild gradient towards the shore. Oxygen saturation was high ( $> 95\%$ ) in both seasons. However, the oxygen saturation (Figure 2e) was lower near the Capibaribe estuary mouth. During winter, there is a tongue of lower oxygen saturation from the Capibaribe mouth influence extending northwards along the inner shelf. During summer, the patch of lower oxygen saturation advances to the middle shelf, but it is shorter northwards. These distributions are likely related with the current regime (Schettini et al., 2017; Domingues et al., 2017). Currents are stronger and predominantly northwards during the winter. During the summer the currents are

sluggish, with major importance to the tides that allow the cross-shelf transport.

The summer chlorophyll levels and oxygen saturation in both seasons indicate the influence of the Capibaribe estuary waters on the adjacent shelf, mainly in terms of inorganic nutrients and organic matter contribution. This influence was not observed in the DDPH distribution. Figure 2f presents the spatial distribution of DDPH expressed in chrysene equivalents, nearly identical to the Carmópolis distribution. What catches the eye at first glance are the highest values off Suape in both periods, suggesting that large ship navigation activity in the vicinity of Suape Port is likely the main source of petroleum hydrocarbons. In fact, such contribution has already been reported elsewhere (Lemos et al., 2014; Zanardi-Lamardo et al., 2018). Interestingly, higher concentrations also occurred at outer shelf stations during winter. A second important pattern, in the summer, is the occurrence of a patch of higher concentrations on the outer shelf in the north. During the summer, the prevalent current is southwards; probably these compounds came from some source other than Suape that was located north of the studied area. Although small differences could be observed among stations, the DDPH concentrations represent a well mixed aquatic system. Concentrations were low when compared to other studies (Bicego et al., 2009; Li et al., 2010; Maciel et al., 2015; Arruda-Santos et al., 2018) and even compared to the proposed baseline for open water of the Southwest Atlantic (0.31  $\mu\text{g L}^{-1}$  oil Carmópolis equivalents at Bicego et al., 2002). Based on that, the authors suggest that the median concentration (including both winter and summer) of 0.02

**Table 1.** Summary of basic statistics (mean, median, standard deviation and 10% and 90% percentiles,  $n=36$ ) for salinity ( $\text{g kg}^{-1}$ ), temperature (°C), chlorophyll ( $\mu\text{g L}^{-1}$ ), turbidity (NTU), oxygen saturation (%), DDPH expressed as Carmópolis ( $\mu\text{g L}^{-1}$ ) and chrysene ( $\mu\text{g L}^{-1}$ ) equivalents, for near surface water on the Pernambuco continental shelf waters during winter 2013 and summer 2014 (winter/summer).

	Mean	Median	Standard Deviation	Percentile 10%	Percentile 90%
Salinity ( $\text{g kg}^{-1}$ )	36.87/36.58	37.09/36.63	0.48/0.18	36.17/36.46	37.25/36.68
Temperature (°C)	26.67/27.92	26.63/27.85	0.26/0.33	26.35/27.56	27.05/28.4
Chlorophyll ( $\mu\text{g L}^{-1}$ )	0.58/0.7	0.36/0.32	0.51/1.36	0.22/0.24	1.19/0.68
Turbidity (NTU)	9.58/7.78	8.53/7.54	3.84/1.21	5.88/6.9	15.28/9.38
Oxygen Saturation (%)	97.54/96.79	97.74/97.29	2.77/2.1	94.63/94.13	100.7/98.14
Carmópolis ( $\mu\text{g L}^{-1}$ )	0.09/0.05	0.07/0.04	0.07/0.05	0.04/0.00	0.16/0.11
Chrysene ( $\mu\text{g L}^{-1}$ )	0.03/0.03	0.02/0.02	0.03/0.02	0.01/0.01	0.06/0.06

$\mu\text{g L}^{-1}$  chrysene equivalents or 0.06 Carmópolis oil equivalents be considered as the baseline concentration for non-polluted coastal water for the tropical western Atlantic Ocean margin. A set of data reported prior to possible accidents is rarely available for comparison and / or recovery assessment. Therefore, this information will be very important for upcoming PAH in water studies, mainly those related to the mysterious oil spill that reached the Brazilian coast, primarily along the coast of the Northeast Region.

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## AUTHOR CONTRIBUTIONS

E.Z.L.: Conceptualization; Resources; Methodology; Supervision; Funding acquisition; Writing - original draft; Writing - review & editing.

C.A.F.S.: Conceptualization; Resources; Methodology; Supervision; Funding acquisition; Writing - original draft; Writing - review & editing.

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