

## Effects of harbor activities on sediment quality in a semi-arid region in Brazil



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### ABSTRACT

Tropical marine environments are rich in biodiversity and the presence of harbor activities in these areas can harm the coastal ecosystems. In this study, we assessed sediment quality of two harbors from a tropical region in Brazil by applying multiple lines-of-evidence approach. This approach included the integration of results on: (1) grain size, organic matter, organic carbon, nitrogen, phosphorus, trace metals, polycyclic aromatic hydrocarbons, linear alkylbenzenes, and tributyltin; (2) acute toxicity of whole sediments and chronic toxicity of liquid phases; and (3) benthic community descriptors. Our results revealed that the main contaminants detected in sediments from Mucuripe and Pecém Harbors were chromium, copper, nitrogen, zinc, and tributyltin. These toxicants arise from typical harbor activities. However, the changes in benthic composition and structure appear to depend on a combination of physical impacts, such as the deposition of fine sediments and the toxic potential of contaminants, especially in Mucuripe. Thus, apart from toxicants physical processes are important in describing risks. This information may assist in management and conservation of marine coastal areas.

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### 1. Introduction

Both the development and operation of harbors have been associated with negative impacts on the surrounding coastal zone. Development and operation activities include the construction of jetties, changes in sediment transport, and pollution due to contaminant inputs from several diffuse sources, such as sewage, wastewater, petroleum and its derivatives, and compounds used in anti-fouling coatings (NRC, 1997). These sources are often associated with the input and spread of contaminants throughout the different environmental compartments, including waters, sediments, and biota.

Sediments require special attention because they present higher concentrations of contaminants than the water column. They act not only as a sink, but also as a secondary source of

contaminants for the biota, a situation which may lead to ecological risks to benthic communities (Burton and Johnston, 2010). In port zones, dredging operations have been frequently required to increase or maintain operational depth. This process generates ecological impacts in the dredged areas through sediment removal and resuspension and also affects disposal sites, which receive the dredged material (Torres et al., 2009).

In order to understand the effects of different stressors on biota, the use of different lines of evidence has been recommended in sediment quality assessment. These lines of evidence include chemical analyses combined with ecotoxicological and ecological approaches (Chapman and Hollert, 2006; McPherson et al., 2008; Arfaeina et al., 2016). Among these methods, the sediment quality triad, or SQT (Chapman, 1990; Long and Chapman, 1985), integrates evaluations of benthic community structures with sediment toxicity and chemistry in order to provide a better assessment of pollution-induced degradation than the use of these line of evidence (LOE) alone (McPherson et al., 2008).

Tropical regions in equatorial zones such as northeastern Brazil are very biodiverse environments (Stuart-Smith et al., 2013). In

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these coastal areas, the low productivity associated with sandy sediments rich in biogenic carbonates is often found (Eku and Knoppers, 1999; Lacerda and Marins, 2006). Therefore, anthropic impacts such as contamination may pose risks to the structure and functioning of these ecosystems.

Few studies have been performed on the quality of sediments from tropical areas, especially in equatorial and semi-arid regions. Therefore, studies focused on the associations between contamination levels and biological effects are necessary to assist in the management of the environmental effects that accumulate in dredged material. Brazil has recently expanded its industrial and harbor activities in underdeveloped regions, including the Northeast. The government considered these actions to be strategic, but they have also increased of anthropogenic pressures on coastal zones.

Economically, Ceará is one of the most important states in the Brazilian Northeast. The capital city of Fortaleza has more than 2.3 million inhabitants distributed over an area of 313 km<sup>2</sup>. The city's main anthropogenic impacts involve harbor activities, factories, and a petroleum refinery (Cavalcante et al., 2009; Buruaem et al., 2012). Mucuripe and Pecém are the state's two harbors. They ship most of the goods produced and traded in the region, a factor which makes these areas vulnerable to contaminants inputs from the port activities.

In light of these factors, the main goal of this study was to investigate the sediment quality of Mucuripe Harbor and Pecém Harbor, both of which are located in the Brazilian state of Ceará. The SQT approach was applied in order to achieve this goal. Three lines of evidence (chemical analyses, sediment toxicity, and benthic community descriptors) were combined in order to produce a diagnosis of sediment quality in this tropical region of the Brazilian coast, which was chosen because of its high ecological significance and because of the recent intensification of industrial and port-related activities.

## 2. Material and methods

### 2.1. Study area

The climate of the northeastern region of the Brazilian coast is largely influenced by the Intertropical Convergence Zone (ITCZ). However, it also depends of other phenomena, such as the El Niño-Southern Oscillation (ENSO). During El Niño periods, the weather becomes drier, while during La Niña periods, rainfall volumes often go above multi-annual averages for the region. In addition, the climate is marked by semi-arid conditions with limited ranges in temperature, with averages of 24 °C to 30 °C (Paula et al., 2013). There is a predominance of trade winds coming from the E-SE at an average speed of 4 m/s, a factor which determines sediment transport in this direction (Maia et al., 1998; Jimenez et al., 1999).

Sediments from the Ceará coast exhibit organogenic and terrigenous facies. The organogenic substrates are derived from *Lithothamnium* (Rodophyta) and *Halimeda* (Clorophyta) species of calcareous algae. Their presence contributes approximately 75–95% of calcium carbonate deposition, with organic matter ranging from 23% to 46%. Sediments from terrigenous facies are characterized by siliciclastic material, including quartz sand, feldspar, heavy minerals (smectite, kaolinite and illite), and clay (Lacerda and Marins, 2006; Marques et al., 2008). The texture of sediments in the outer shelf (40 m isobath) is marked by gravel, while the sediments from the inner shelf (below the 20 m isobath) are composed of sand with biodetritic gravels and low amounts of mud — mud levels have been found to be less than 2.5% (Freire et al., 2004).

Mucuripe Harbor is located within the Mucuripe Bay in the

Brazilian city of Fortaleza. Its infrastructure includes an access channel, anchorage areas, evolution basins, and a long jetty (1900 m long). Pecém Harbor is located in a port industrial complex approximately 60 km west from the capital. Pecém is an offshore terminal, which is considered to be technological advancement. It was constructed 2000 m away from the shoreline and connected to land by a bridge. Thus, the coastal currents are not completely affected by port facilities, since sediment transport occurs between the pillars of the port.

### 2.2. Sediment sampling and handling

In Mucuripe Harbor, sediment sampling was performed in August 2007 at 10 stations (Ms) from different areas. M1 and M2 were located in front of commercial docks, where the docking of ships and effluent discharges from a major oil refinery occur. M3, M4 and M5 were situated at fishing and tanker piers, where oil from the refinery is unloaded. M6 and M7 were placed at the access channel, and M8, M9, and M10 were established in unsheltered areas. In Pecém Harbor, sediments were collected from 5 stations (Ps) in January 2008. P1 and P2 were situated close to the docking piers, where steel products, bulk liquids, liquefied gases, and general cargo are shipped and docked. P3 was established at the access channel. P4 and P5 corresponded to unsheltered areas; P4 was located in a diffraction zone of waves and currents, while P5 was under the influence of the sediment transport that occurs between the pillars the port bridge (Fig. 1).

The sediments collected for toxicity tests were placed in refrigerated coolers and transported to the laboratory, where they were stored at 4 °C in dark conditions. For the benthic community analysis, three replicates were collected from each station using a Van Veen grab sampler (0.026 m<sup>2</sup>). Sediment samples were carefully washed and sieved through a 0.5-mm mesh. Next, the retained material was fixed using 4% buffered formalin. It was washed and then preserved in 70% ethanol. In the laboratory, biological material was sorted, identified, and quantified under a stereoscopic microscope Zeiss® model Stemi DV4 Stereo. For the chemical analyses, two aliquots were separated. The first aliquot was dried at room temperature by using a desiccator cabinet and packed in plastic containers for subsequent metal analyses. For the analysis of the physical and chemical parameters and the assessment of organic contaminants, the sediment samples were placed into pre-cleaned aluminum foil and stored at –20 °C.

### 2.3. Sediment analyses

Particle size distribution was measured using the wet sieving method to separate fine sediments (silt + clay), followed by dry sieving to separate gravel and sand fractions (McCave and Syvitski, 1991). Calcium carbonate contents (CaCO<sub>3</sub>) were estimated using digestion in HCl and gravimetry (Gross, 1971). After CaCO<sub>3</sub> removal, total organic carbon (TOC) levels were determined using a Shimadzu TOC-V TOC analyzer coupled with a SSM-5000A solid sample combustion unit. Organic matter content (OM) was determined using the method of ignition and gravimetry (Luczak et al., 1997). Nutrients, total nitrogen (N), and phosphorus (P) were estimated using extraction and spectrophotometry (Grasshoff et al., 1999).

The major metals (Al and Fe) and trace metals (Hg, Cd, Cr, Cu, Ni, Pb and Zn) were analyzed after extraction with an acid solution (9 ml of HNO<sub>3</sub> + 3 ml of HCl) according to the EPA 3051A protocol (USEPA, 1996). A high pressure microwave system was used (CEM Corporation, model MDS—2000). Lead, nitrogen, and mercury concentrations were measured using the flame mode of a Varian Spectr-AAS-220-FS fast-sequential atomic absorption spectroscope. Mercury concentrations were measured via cold vapor

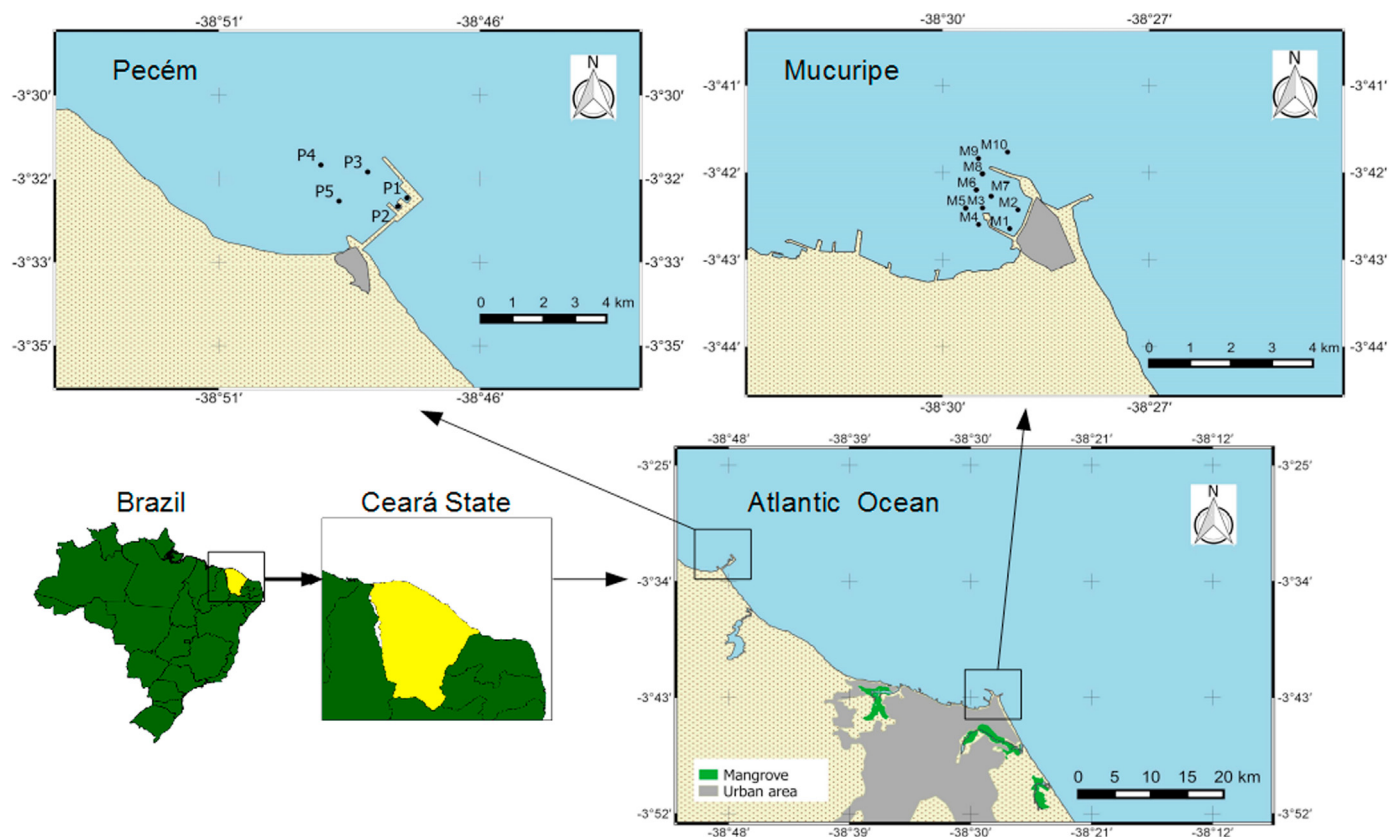


Fig. 1. Sediment sampling stations.

generation by coupling the spectrometer to a typical flow injection (FI) manifold for flow injection analysis. The methods were validated through an analysis of reference materials (SRM 2704, Buffalo River Sediment and SRM-1646a, Estuarine Sediments). A detailed interpretation of these results (recovery, limits of detection and quantification) are discussed in [Buruaem et al. \(2012\)](#).

Hydrocarbon analyses were performed on freeze-dried samples of sediments that had been extracted using a mixture of n-hexane and dichloromethane (1:1) in Soxhlet ([UNEP, 1991](#)). The extracts were then fractionated into F<sub>1</sub> including aliphatics (AHs) and linear alkylbenzenes (LABs) and F<sub>2</sub> for polycyclic aromatic hydrocarbons (PAHs) via silica gel-alumina column chromatography. They were then quantitatively analyzed using an Agilent 6890 gas chromatograph coupled to a 5973 N mass spectrometer (GC/MS). Certified standards from AccuStandard (USA) were used to build analytical curves and blanks, while reference material from National Institute of Standards and Technology - NIST (SRM 1944) was used for surrogates.

Tributyltin (TBT) concentrations were measured following the method provided by [Castro et al. \(2012\)](#). Briefly, 5g of dry sediments were spiked with 100 ng of tripropyltin as a surrogate standard, and the resulting product was added to 15 ml of tropolone solution in methanol and concentrated HCl. Samples were then ultra-sonicated and centrifuged, and the extract volume was reduced to be derivatized by pentylmagnesium bromide in a diethylether solution. After clean-up, the extracts were analyzed in a Perkin Elmer Clarus 500MS gas chromatograph (GC-MS). The tetrabutyltin solution (1000 ng Sn/ml) was added as an internal standard, and quality assurance and control was based on regular analyses of blanks, spiked matrices, and certified reference material (PACS-2/National Research Council of Canada, Ottawa, Canada). Sample recoveries were between 88.5% and 109%, and relative standard deviation (RSD) was below 20%.

#### 2.4. Comparison to Sediment Quality Guidelines (SQGs)

Considering the fact that the study areas are under the influence of dredging operations, the results of the chemical analyses were compared to threshold and probable effect levels (Levels 1 and 2, respectively) of the international criteria ([FDEP, 1994](#); [Long et al., 1995](#); [EC, 2008](#); [HPA, 2011](#)) adopted by the revised version of Brazilian Federal legislation for dredged sediments ([Brasil, 2012](#)). The results were also compared to threshold and probable effects (T and P, respectively) of site-specific sediment quality values (SQV) produced for the Santos Estuarine System by [Choueri et al. \(2009\)](#). Comparisons were based on the use and interpretation of sediment quality guideline quotients, or SQGQs ([Fairey et al., 2001](#)) for metals, PAHs, and TBT (only for SQGs); the concentrations were divided by their respective probable effect values. Based on the computation the mean quotients, samples were ranked according to the following contamination criterion:

- Minimal contamination. Uncontaminated sediments: SQGQ and SQVQ values between 0 and 0.1;
- Moderate contamination. Contamination levels may produce occasional toxicity: SQGQ and SQVQ values between 0.1 and 0.25;
- Strong contamination. Contamination levels will probably cause toxicity: SQGQ and SQVQ values greater than 0.25.

#### 2.5. Sediment toxicity

Sediment samples toxicity was assessed in order to determine the deleterious effects on the local biota. Four exposure pathways were analyzed, including whole sediment (WS) as a solid-phase bioassay, and pore water (PW), sediment-water interface (SWI), and elutriates (ELU) as liquid-phase bioassays.

The WS bioassay was performed according to the protocol described by Brazilian National Standards Organization (ABNT) in Brazilian National Standard (NBR) No. 15,638 (ABNT, 2008). The mortality rate of the burrowing amphipod *Tiburonella viscana* was used as an endpoint to assess the acute toxicity associated with whole sediment exposure.

PW was extracted from sediments using the suction method (Winger and Lasier, 1991). The PW tested for waterborne toxicity using the embryo-larval development of the sea urchin *Lytechinus variegatus* according to the protocol of the ABNT's NBR No. 15350 (ABNT, 2006) at 100%, 50% and 25% dilutions. Sea urchin spawning was induced by 9-volt electric shock and subsequent *in vitro* fertilization. The test was conducted by exposing approximately 400 embryos in each replicate, including a negative control (filtered seawater). After 24 h of exposure, embryos were analyzed microscopically for morphological anomalies and delayed development. The dilutions were used to interpret the data on the effects of unionized ammonia, which can contribute to the toxicity of pore water samples (Losso et al., 2007).

A sediment-water interface (SWI) test was performed following the method described by Anderson et al. (2001a) and adapted by Cesar et al. (2004) for small volumes. This treatment assesses the effects of compounds that are transferred from the upper sediment layers and which may affect organisms in the adjacent water column. In this procedure, the test system was set up in test tubes containing 1:4 sediment and water (v: v). After an equilibrium period, sea urchin embryos were exposed following the PW protocol described previously.

Elutriates (ELU) were prepared according to USEPA (2003a) recommendations. Sediment samples were mixed with diluted seawater at a ratio of 1:4 (v: v) in a jar test apparatus for 30 min and were incubated in 4 °C for sedimentation of suspended material. Next, aliquots of the supernatant were separated and tested for the toxicity based on the embryo-larval development of the sea urchin *L. variegatus*.

Negative controls were prepared for all of the treatments. Filtered and uncontaminated seawater was used in the PW tests, while sediments from a reference site (Engenho D'água Beach, located on the northern coast of São Paulo State) were used in the WS tests, as well in the remaining liquid phase tests (SWI and ELU). Salinity, temperature, and dissolved oxygen were controlled during the execution of tests. Total ammonia concentration was measured using the phenate method, Section 4500-NH<sub>3</sub>C (APHA, 1999). Unionized ammonia (NH<sub>3</sub>) levels were estimated using the model proposed by Whitfield (1974) in order to assess the contribution of ammonia to toxicity. NH<sub>3</sub> levels above 0.05 mg/L may contribute to the toxicity of PW samples that include *L. variegatus* (Abessa et al., 2008).

Student's *t*-test was used to compare responses from each sample and the respective controls; bioequivalence was used for the liquid phase treatments. Samples that differed significantly from the control were considered toxic. The linear interpolation method (Norberg-King, 1993) was applied to diluted PW samples in order to calculate the inhibitory concentration on 10% of embryos (IC<sub>10</sub>). The results were transformed into toxic units (TU = 100/IC<sub>10</sub>).

## 2.6. Macrobenthic infaunal analysis

Macrobenthic infaunal community descriptors were analyzed in both harbors as ecological indicators of sediment quality. Species were identified, and the number of species and specimens were estimated and expressed relative to 0.16 m<sup>2</sup>. Diversity was evaluated using richness (Margalef), evenness (J), and the Shannon–Wiener index (H' log<sub>2</sub>). The density of each major taxonomic group was also calculated (Mollusca, Crustacea, Polychaeta, and

Nematoda) and expressed as a percentage.

## 2.7. Integrated approach: Ratio-to-Mean Values (RTM) and multivariate analysis

This first integrative approach employed was an adaptation of the ratio-to-reference method (Long and Chapman, 1985) and the ratio-to-maximum value methods (DelValls and Chapman, 1998) proposed by Abessa et al. (2008) and later applied by Cesar et al. (2009). Using the data matrix, values obtained for each variable in each LOE were converted into non-dimensional values (ratio-to-mean-values, or RTMVs) which were each normalized by the arithmetic means calculated for all stations. Then, the RTMVs were combined through the calculation of a mean, thus producing a single new value for each LOE. Next, the values were plotted on three-axis graphs, and triangles were formed. The area of each triangle represented the site-specific RTM, which is interpreted as a degree of degradation. The second approach sought to integrate the results in a principal component analysis (PCA) through the simultaneous analysis of the physical and chemical matrix with the biological responses matrix in the same multidimensional space.

## 3. Results

### 3.1. Sediment properties, chemical contamination, toxicity and benthic community descriptors

The data on sediment properties and the results of the chemical analysis are summarized in Table 1. The sediments from Mucuripe and Pecém were composed mainly of sands; higher levels of fine particles (mud) occurred in the sheltered areas of both harbors, compositions which contrasted with the high levels of gravel in the sample from M10 (40.82%). Levels of calcium carbonates ranged from 3.20% to 37.40% in Mucuripe and from 2.40% to 31.70% in Pecém. Organic matter contents ranged from 0.13% to 16.22% in Mucuripe and from 2.49% to 14.87% in Pecém. Low levels of TOC and nutrients (N and P) were detected, and their spatial distributions were similar to those of OM and CaCO<sub>3</sub>, with higher values in the samples from M1 to M7 and in P1 to P3.

At sheltered stations (M1 to M7), the levels of major and trace elements were much higher (one order of magnitude) than at unsheltered stations (M8, M9 and M10). A similar pattern was found in Pecém, where levels were found to be higher than in Mucuripe. The concentrations of aliphatic hydrocarbons (AHs) ranged from 0.3 µg/g to 25.7 µg/g in Mucuripe and from 1.1 µg/g to 19.5 µg/g in Pecém, while levels of polycyclic aromatic hydrocarbons (PAHs) ranged from 36.7 ng/g to 107 ng/g in Mucuripe and from 31.9 ng/g to 292 ng/g in Pecém. Linear alkylbenzenes (LABs) were found only in M4 and M6 (1.3 ng/g and 5.8 ng/g, respectively). Meanwhile, in Pecém, they were found in P1 to P4 and ranged from 1.6 ng/g to 4 ng/g. TBT levels were detected in both harbors: TBT was present in M1 to M7 in Mucuripe and at all of the Pecém stations.

The results of toxicity bioassays are presented in Table 2. Samples from M1 to M8 (Mucuripe) induced significant amphipod mortality. Similarly, most sediments from Pecém were found to be toxic (with the exception of P2). All samples were toxic for PW at 100% and the calculated TU showed that M2, M3, M4, M8 and M10 were the most toxic, with values above 30 TU. NH<sub>3</sub> levels were found in all liquid phase exposures; the highest values (above 0.05 mg/L) were detected in PW samples. SWI toxicity was observed in M1 to M7 in Mucuripe and in all samples from Pecém. The elutriates induced effects in samples from M2, M3 and M4, as well as in samples from P2 and P4.

**Table 1**

Physical and chemical characteristics of sediments from Mucuripe Harbor and Pecém Harbor. Values reported on dry weight basis.

Station	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	P1	P2	P3	P4	P5
Depth (m)	7.4	15.0	15.4	14.7	10.7	14.6	9.6	9.6	11.9	12.6	16.7	16.4	17.5	17.2	15.1
Gravel (%)	0.00	0.00	0.00	0.00	0.10	0.00	0.00	0.00	0.00	40.8	0.00	0.00	0.00	0.00	0.00
Sand (%)	75.6	92.0	77.0	96.2	84.2	83.0	94.1	99.4	99.8	59.0	95.5	89.1	91.4	85.7	93.1
Mud (%)	24.0	7.86	22.7	2.42	15.5	16.7	5.45	0.59	0.08	0.00	4.42	10.9	8.17	14.3	6.89
CaCO <sub>3</sub> (%)	6.50	24.8	22.0	19.4	21.0	37.4	12.9	9.80	3.20	6.20	31.7	9.30	2.40	24.6	11.1
OM (%)	5.61	12.7	5.99	16.2	3.28	8.60	12.2	0.96	0.57	0.13	14.9	12.2	14.7	7.71	2.49
TOC (%)	0.94	1.43	0.96	1.40	0.44	1.11	0.85	0.16	0.18	0.22	1.35	1.14	1.39	0.78	0.51
N (%)	0.37	0.38	0.18	0.24	0.17	0.11	0.21	0.13	0.12	0.16	0.57	0.47	0.50	0.33	0.25
P (%)	0.23	0.25	0.42	0.40	0.48	0.35	0.27	0.11	0.09	0.10	0.93	0.76	0.80	0.68	0.43
Al (%)	2.90	2.42	2.49	3.13	0.87	3.36	1.70	0.30	0.16	0.20	4.46	3.56	4.09	1.56	0.83
Fe (%)	1.94	2.39	1.85	2.47	0.80	1.99	1.66	0.29	0.13	0.28	2.78	2.38	2.70	1.49	1.10
Hg (µg/g)	0.04	< 0.03	0.03	0.04	< 0.03	0.04	< 0.03	< 0.03	< 0.03	< 0.03	0.04	0.03	0.04	0.03	< 0.03
Cd (µg/g)	0.90	0.85	1.41	0.64	< 0.60	0.60	< 0.60	< 0.60	< 0.60	< 0.60	0.60	1.28	< 0.60	< 0.60	< 0.60
Cr (µg/g)	38.0	38.1	32.3	50.1	13.4	41.5	26.9	5.23	3.33	5.16	63.1	48.5	56.2	25.6	17.7
Cu (µg/g)	13.8	15.5	11.2	16.7	3.71	12.1	9.77	0.49	< 0.70	1.01	18.8	16.9	15.8	8.62	5.49
Ni (µg/g)	16.5	19.9	16.7	21.5	6.95	17.8	15.2	7.20	< 2.00	5.29	24.7	22.2	23.8	11.5	11.7
Pb (µg/g)	2.59	6.69	3.58	2.60	< 2.00	2.54	2.06	< 2.00	< 2.00	< 2.00	3.55	< 2.00	< 2.00	< 2.00	< 2.00
Zn (µg/g)	469	536	399	583	199	421	365	62.6	25.0	41.9	639	569	583	312	215
AHs (µg/g)	25.4	19.1	17.6	21.1	1.06	2.46	13.5	1.01	0.46	0.34	14.8	19.5	17.4	1.50	1.06
PAHs (ng/g)	77.9	107.8	59.6	87.9	27.9	36.7	43.4	< 1.00	< 1.00	< 1.00	144	292	77.2	31.9	182.8
LABs (ng/g)	< 0.85	< 0.85	< 0.85	5.84	< 0.85	1.35	< 0.85	< 0.85	< 0.85	< 0.85	3.20	2.90	3.98	1.59	< 0.85
TBT (ng/g)	23.6	40.6	100	26.8	33.0	22.6	233	< 2.00	< 2.00	< 2.00	73.0	53.0	49.2	45.0	31.0

**Table 2**

Sediment toxicity results and benthic community descriptive parameters in sediments from Mucuripe Harbor and Pecém Harbor.

Station	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	P1	P2	P3	P4	P5
WS	67 ± 25 <sup>*</sup>	67 ± 12 <sup>*</sup>	43 ± 15 <sup>*</sup>	50 ± 10 <sup>*</sup>	40 ± 10 <sup>*</sup>	30 ± 10 <sup>*</sup>	27 ± 6 <sup>*</sup>	27 ± 6 <sup>*</sup>	17 ± 15	7 ± 12	23 ± 12 <sup>*</sup>	17 ± 6	37 ± 12 <sup>*</sup>	60 ± 10 <sup>*</sup>	53 ± 25 <sup>*</sup>
PW (TU)	3.6 <sup>*</sup>	33.3 <sup>*</sup>	33.3 <sup>*</sup>	33.3 <sup>*</sup>	8.0 <sup>*</sup>	32.2 <sup>*</sup>	4.8 <sup>*</sup>	31.0 <sup>*</sup>	15.6 <sup>*</sup>	33.3 <sup>*</sup>	2.0 <sup>*</sup>	4.6 <sup>*</sup>	5.2 <sup>*</sup>	8.4 <sup>*</sup>	7.9 <sup>*</sup>
NH <sub>3</sub> in PW (mg/L)	<b>0.13</b>	<b>0.11</b>	<b>0.05</b>	<b>0.33</b>	<b>0.07</b>	<b>0.1</b>	<b>0.11</b>	<b>0.05</b>	<b>0.1</b>	<b>0.09</b>	<b>0.12</b>	<b>0.18</b>	<b>0.24</b>	<b>0.11</b>	<b>0.08</b>
SWI	78.2 ± *	90.5 ± *	92.0 ± *	97.5 ± *	75.7 ± *	96.7 ± *	82.2 ± *	23.5 ±	8.0 ±	8.5 ±	98.0 ± *	99.2 ± *	96.0 ± *	100.0 <sup>*</sup>	100.0 <sup>*</sup>
NH <sub>3</sub> in SWI	0.02	0.04	0.04	<b>0.08</b>	0.02	<b>0.05</b>	0.02	0.02	0.01	0.03	ND	0.01	ND	ND	ND
ELU	4 ± 3	100 ± *	89 ± 9 <sup>*</sup>	100 ± *	4 ± 3	33 ± 6 <sup>*</sup>	8 ± 7	5 ± 1	1 ± 1	1 ± 1	14 ± 5	18 ± 6 <sup>*</sup>	12 ± 5	83 ± 11 <sup>*</sup>	12 ± 10
NH <sub>3</sub> in ELU	ND	<b>0.05</b>	0.04	<b>0.08</b>	0.02	<b>0.07</b>	0.01	0.01	0	0	0.02	0.03	0.04	<b>0.05</b>	0.04
Species – 0.16 m <sup>2</sup>	9	8	9	9	19	5	14	11	3	1	9	11	8	8	7
Individuals – 0.16 m <sup>2</sup>	36	16	14	15	49	14	51	23	5	1	20	39	20	15	11
Richness – 0.16 m <sup>2</sup>	2.23	2.52	3.03	2.95	4.63	1.52	3.31	3.19	1.24	NC	2.67	2.73	2.34	2.58	2.5
Evenness – 0.16 m <sup>2</sup>	0.86	0.79	0.93	0.94	0.84	0.7	0.9	0.86	0.86	NC	0.73	0.68	0.73	0.85	0.97
Diversity – 0.16 m <sup>2</sup>	2.74	2.38	2.95	2.97	3.57	1.63	3.43	2.98	1.37	0	2.32	2.37	2.18	2.56	2.73
Mollusca (%)	6.25	5.56	16.7	0	10.6	20.4	3.51	8.54	25	0	11.1	65.1	70.6	56.1	61.1
Nematoda (%)	2.08	10.3	0	0	4.76	0	0	9.12	0	0	6.06	0	0	0	11.1
Echinodermata (%)	0	0	0	0	0	0	0	39.6	50	100	0	2.08	0	0	0
Polychaeta (%)	91.7	78.6	70	100	56.4	79.6	89.8	22.2	0	0	82.8	30.9	29.4	43.9	16.7
Crustacea (%)	0	5.56	13.3	0	28.1	0	6.67	20.6	25	0	0	1.85	0	0	11.1

WS=% of amphipod mortality; SWI and ELU=% of abnormal larvae; TU=toxic units. Levels of NH<sub>3</sub> above 0.05 mg/L are marked in bold.<sup>\*</sup> Toxic (p < 0.05).

As mentioned previously, the benthic community was evaluated as an ecological indicator of sediment quality. A total of 39 taxa were observed in Mucuripe (Appendix A and B). Polychaeta was the most abundant group from M1 to M7, followed by Mollusca, Crustacea, and Echinodermata (Table 2). In Pecém, 27 taxa were identified as Mollusca, which was the most abundant group in P2 to P4, followed by Polychaeta, Nematoda, and Crustacea. In both harbors, the numbers of species and specimens per station were low, with the highest values detected at M5, M7, M8, and P2. The highest diversity, evenness, and richness values were observed in Mucuripe sediments (M4, M5, M7 and M8); these values were lower in Pecém.

### 3.2. Comparison to sediment quality guidelines

Results of the chemical analyses were compared to the international benchmarks used in the Brazil's federal SQGs (Brasil, 2012), as well as to site-specific SQVs (Choueri et al., 2009) in order to classify samples according to their potential toxicity. This

comparison showed that SQGs were not appropriate for predicting toxicity because they indicated moderate contamination in M1 to M7 and P2 to P5, samples which exhibited high toxicity. The results were more consistent with the classification obtained by using site-specific values. This failure on the part of SQGs to predict effects has been reported in other studies (conducted in southeastern Brazil by Abessa et al., 2006; Abessa et al., 2008; Choueri et al., 2009; Buruaem et al., 2012). The consistency is our study reaffirms the need for the development of site-specific SQVs for tropical zones such the northeastern Brazil. (Table 3).

### 3.3. Integrated assessment

Overall, all the three lines of evidence (chemical makeup, toxicity, and benthos) influenced station rankings. High RTM values were calculated for samples with consistent alterations in each component (Fig. 2). Thus, the RTM indices indicated that the sheltered sites were the most degraded in terms of the sediment quality (M4, M2, M3, and M6 in Mucuripe and P1 to P4 in Pecém).

**Table 3**  
Comparison to Brazilian SQGs and site-specific SQVs for Mucuripe Harbor and Pecém Harbor based on the SQGq approach and the respective results on toxicity tests.

Site	CONAMA 454/12		Choueri et al. (2009)		Toxicity observed
	SQVq	Classification	SQGq	Classification	
M1	0.20	Moderate	0.91	Strong	WS, PW and SWI
M2	0.23	Moderate	1.02	Strong	WS, PW, SWI and ELU
M3	0.20	Moderate	0.90	Strong	WS, PW, SWI and ELU
M4	0.25	Moderate	1.05	Strong	WS, PW, SWI and ELU
M5	0.09	Moderate	0.42	Strong	WS, PW and SWI
M6	0.19	Moderate	0.81	Strong	WS, PW, SWI and ELU
M7	0.18	Moderate	0.70	Strong	WS, PW and SWI
M8	0.05	Minimal	0.25	Moderate	WS and PW
M9	0.03	Minimal	0.17	Moderate	PW
M10	0.04	Minimal	0.21	Moderate	PW
P1	0.28	Strong	1.16	Strong	WS, PW, SWI and ELU
P2	0.25	Moderate	1.13	Strong	PW, SWI ELU and SWI
P3	0.25	Moderate	1.06	Strong	WS, PW and SWI
P4	0.14	Moderate	0.61	Strong	WS, PW, SWI and ELU
P5	0.11	Moderate	0.48	Strong	WS, PW and SWI

\* CONAMA – Brazil's National Environment Council.

This finding allows us to state that proximity to the sources of contamination is relevant in predicting sediment quality.

PCA results are presented in Fig. 3 and Appendix C. The first three axes explained 80.20% of total variance. Adopting the cut-off value of 10.5, positive correlations to Axis 1 (PC1 > 0) explained 62.13% of variances and indicated that the occurrence of fine

sediments, organic enrichment (OM, TOC, N, and P), high levels of metals, hydrocarbons, and TBT correlated with sediment toxicity (WS, SWI, and ELU) and with richness, evenness, Polychaeta, and the number of individuals. Negative correlations with this axis (PC1 < 0) represented Echinodermata specimens and the deposition of coarse sediments. Axis 2 accounted for 10.36% of variances, and positive correlations (PC2 > 0) indicated high levels of lead, while negative correlations (PC2 < 0) represented depth zones with nitrogen and LABs. Axis 3 explained 7.76% of variance, and positive correlations (PC3 > 0) to it were associated with coarse sediments. Negative correlations (PC < 0) were found for sand, richness, evenness, diversity, and crustacea.

The two-dimensional ordination of Axes 1 and 2 is presented in Fig. 4; the findings corroborate the RTM results. Based on the correlations described above, Axis 1 separated the sites with a higher degree of sediment degradation (M1 to M4, M6, M7, and P1 to P4) from those which were found to have good indicators of environmental quality, such as low levels of contaminants and low toxicity (M5, M8 to M10, and P5). Axis 2 grouped the stations by harbor location.

#### 4. Discussion

##### 4.1. Physical and chemical characteristics of the sediments

The presence of sediments with higher percentages of fine particles (mud) and organic matter in deeper and sheltered areas may be associated with the creation of deposition zones in both



Fig. 2. Ratio-to-mean values calculated for sediment samples from Mucuripe Harbor and Pecém Harbor.

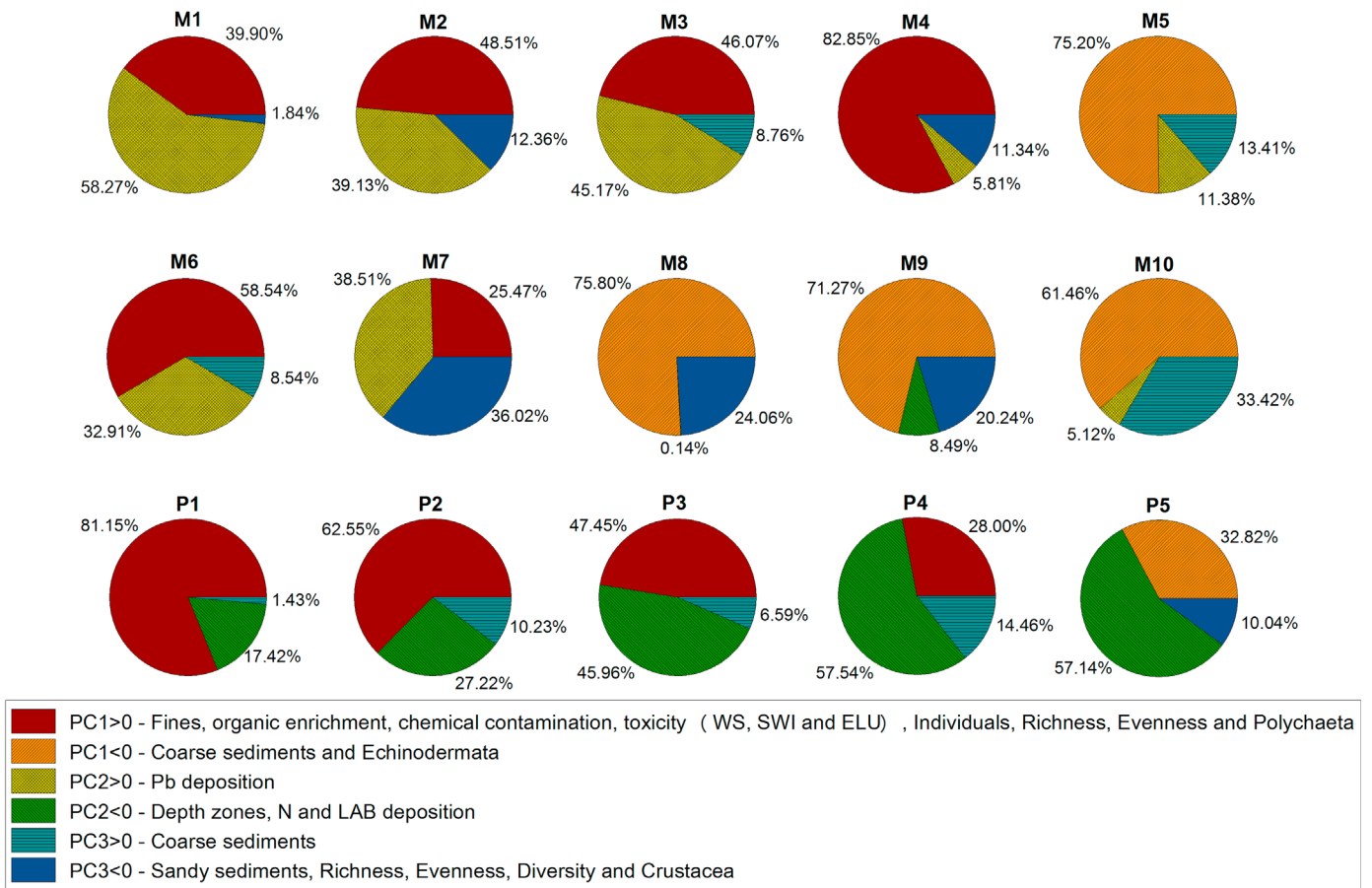


Fig. 3. PCA scores expressed as a percentage of variance for each sample from Mucuripe Harbor and Pecém Harbor.

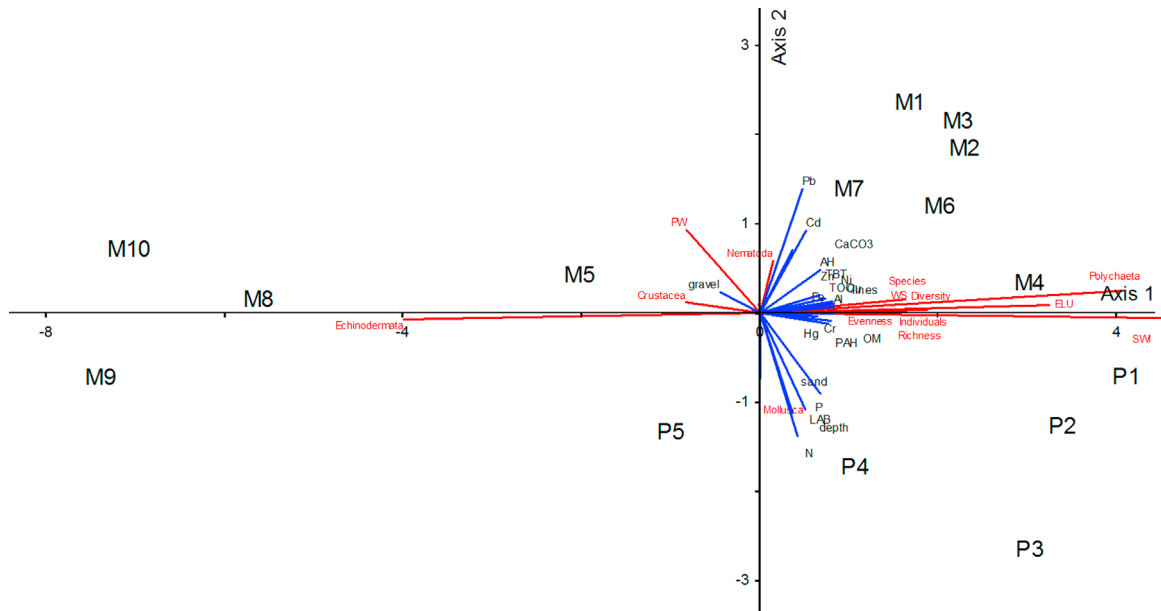


Fig. 4. Ordination results of the principal component analysis based on lines-of-evidence data.

ports due to the installation of port jetties. The construction of the jetty in Mucuripe affected sediment transport, and the current diffraction induced by the structure caused the siltation of the ocean bottom with fine particle sediments (Maia et al., 1998). In Pecém, similarities in jetty arrangement and direction of currents may result in a similar pattern of deposition.

These results are in accordance with the descriptions of the

typical characteristics of the region: carbonate fraction is biogenic, with levels from 0.2% to 95%, and organic matter contents range from 0.76% to 38.9% (Freire et al., 2004; Lacerda and Marins, 2006; Marques et al., 2008). Thus, our results corroborate the hypothesis that the presence of jetties in each harbor produces physical impacts on both local environments by changing the sediment type and favoring deposition in certain areas (shadow zones). In these

low energy places, fine particles, organic matter, and pollutants tend to deposit in sediment layers. Because punctual and diffuse sources (such as metals, hydrocarbons, and antifouling biocides) are known to contaminate harbor areas, depositional areas can also be expected to accumulate such contaminants (Casado-Martínez et al., 2006; Martínez-Lladó et al., 2007).

Analyzing the same samples used in this study, Buruaem et al. (2012) reported that aluminum, iron, mercury, chromium, copper, nitrogen, and zinc are continental in origin and are transported along the coast with other materials, including fine particles, which are all deposited in sheltered areas of both harbors. In the cases of aluminum, chromium, copper, nitrogen, and zinc, concentrations were considered to be influenced by human activity and were enriched when compared to sediment concentrations collected from the continental shelf of Ceará State (Aguiar et al., 2007). The enrichment of chromium, copper, nitrogen, and especially zinc may be associated with specific sources of pollution (harbor areas and marinas), since they are frequently associated with antifouling particles. Turner (2010) reported high levels of barium, chromium, copper, lead, tin, and zinc in zones subjected to the release of antifouling residues. The author stated that copper and zinc are elemental constituents of the polymeric matrix of these residues, as are significant amounts of other trace metals (such as Ba, Cd, Cr, Ni, Pb, and Sn). The author also reported that the presence of tin may reflect traces of former organotin formulations in the composite.

Meanwhile, AHs are important petroleum derivatives and are an indicator of the presence of petroleum or hydrocarbons (Volkmann et al., 1992). PAHs are released from natural and human activities and products, including coal and wood burning, oil combustion, industrial activities, effluents, and accidental fuel spills (USEPA, 2003b). Buruaem et al. (2016) analyzed the same sediment samples and stated that the contamination levels observed were lower than those of other industrialized areas of Brazil, such as Santos and Guanabara Bay (located in the southeastern region of the country). The analysis of n-alkanes also revealed the contribution of biogenic sources of AHs. By using diagnostic ratios, however, the authors have identified the origin of the PAHs as fuel combustion, and possibly shipping activities, thus confirming the presence of port activities as sources of contaminants in both harbors.

Meanwhile, the LABs that have been released into the environment as by-products of linear alkylbenzene sulfonate (LAS) detergents, and their environmental occurrence has been associated with sewage discharge and both domestic and industrial waste (Eganhouse, 1986). Buruaem et al. (2016), however, argue that the contribution of these sources to the quality of the sediments studied seems to be negligible; thus, the values reported herein can be considered baselines for samples collected in nearby harbor areas.

TBT was detected in lower concentrations than in other areas of Brazil and South America, including the Paranaguá Estuarine Complex 363–2796 ng/g, the Santos Bay 90–482 ng/g, and the San Vicente Bay in Chile 14–1560 ng/g (Castro et al., 2012; Santos et al., 2011). This chemical is introduced into the marine environment through its use as a biocide in TBT-based antifouling paints on hulls of ships, small boats, coastal and offshore structures, and ducts (Castro et al., 2012). In light of all of the results, it is possible to affirm that the sediment contamination in Mucuripe and Pecém reflects the characteristics that are typically associated with harbor activities.

#### 4.2. Biological effects and integrative assessment

The integrative result show that, metals, hydrocarbons, and TBT were correlated with geochemical carriers, which are considered

to be important binders of contaminants in sediments (Burgess and Kester, 2002; Mzoughi and Chouba, 2011). Whole sediment and the liquid phase (WSI and ELU) toxicity were also correlated, a finding which indicates that both the bioavailability and remobilization of contaminants into the aqueous phase may have occurred, thus leading to toxicity. In a WS experiment, amphipods were directly exposed to chemicals associated with sediment particles and those dissolved in PW. Toxicity was then induced through exposure via the corporal surface, the respiratory system, and feeding (Kennedy et al., 2009; Burton and Johnston, 2010).

All PW samples were toxic and exhibited high levels of NH<sub>3</sub>, including uncontaminated samples such as those from M8 to M10. In these cases, the test organisms were exposed to dissolved contaminants that may be absorbed via diffusion through the entire body surface. The organisms may also experience effects of confounding factors, such as unionized ammonia originating from natural sources and therefore contributing to toxicity (Chapman et al., 2002). However, contaminated sediments present high concentrations of ammonia as a result of human activities and, ammonia may thus be treated as a pollutant (Losso et al., 2007). Therefore, due to the biogenic characteristics of sediments, PW toxicity of the samples from M8 to M10 may be the result of natural levels of ammonia (Chapman et al., 2002; Abessa et al., 2008); for the remaining stations, however, toxicity may more likely be associated with sediment contamination.

According to Camargo and Alonso (2006), the interactions between ammonia and copper, zinc, chlorides, and phenols may result in synergistic effects that enhance the toxic effect. Studies using sediment toxicity identification evaluation (TIE) conducted by Araujo et al. (2013) and Camargo et al. (2014) revealed ammonia's contribution to the toxicity of WS and SWI in a study of the amphipod *Tiburonella viscana* and the undescribed benthic copepod *Nitocra* sp. by testing samples from Xixová-Japuí State Park, located in the Santos Bay, an area chronically infected by multiple sources of contamination.

Results from the SWI and ELU indicate a transfer of contaminants to the water column through diffusion and resuspension, a process which produces risks to planktonic and epibenthic organisms alike. Natural processes such as waves and tidal currents, as well as human dredging activities, can lead to the resuspension of sediments. Once in the water column, chemicals may be remobilized in the dissolved phase (Cantwell and Burgess, 2004).

Wauhob et al. (2007) observed the transfer of cadmium, copper, aluminum, and pesticides, a process which produced effects on the embryo-larval development of the sea urchin *Arbacia punctulata* and the copepod *Schizopera knabeni*. The specimens were exposed to sediment samples from the Bay of Corpus Christi, Texas (USA) in the testing of SWI chambers. Cesar et al. (2004) also reported a transfer of zinc, lead, aluminum, and iron, which had effects on the sea urchins *Arbacia lixula* and *Paracentrotus lividus* in the testing of sediments from Portman Bay (Spain). Torres et al. (2009) assessed the composition of the total suspended solids and dredge overflow waste pumped back into the aquatic ecosystem during dredging operations and reported an increase in mercury, lead, zinc, and PAHs levels. These findings indicate that toxic sediments from Mucuripe and Pecém may reflect risks associated with dredging operations.

TBT concentrations in the environment were also correlated with toxicity in both harbors. Castro et al. (2007) observed a high frequency of imposex in the gastropods *Stramonita haemastoma* and *Stramonita rustica* collected in Mucuripe and Pecém. This finding indicates the occurrence and bioavailability of organotin compounds and corroborates the evidence of impacts associated with antifouling particles. Perina et al. (2011) reported high TBT and triphenyltin (TPT) toxicity during embryo-larval development



of the sea urchin *L. variegatus*, the same species used as a model for liquid phase toxicity in our study. These findings suggest that organotin compounds, as well as other compounds found in antifouling particles, are chemicals of interest in both areas.

In their study on the benthic community, Rocha-Barreira et al. (2001) reported higher values of diversity and richness along the sand banks of Futuro Beach, a few meters upstream from the jetty of Mucuripe Harbor. Polychaeta and Mollusca were found to be the most abundant groups, followed by Crustacea and Echinodermata. The authors found the bivalve *D. striatus* and the spionid *S. lefebvrei* to be the most abundant species. They also reported the occurrence of the echinoid *M. quinquiesperforata*.

In our study, the opportunistic spionid *P. pinnata* and the shallow water clam *M. cleryana* were the most abundant species, particularly in the sheltered zones. *M. quinquiesperforata* occurred only in sandy sediments. This difference between the stations in terms of community composition is also an indicator of changes in sediment type. In Pecém, the assemblages differed from those found in Mucuripe, with a predominance of mollusks such as *N. semiornata*, *C. caribea*, and *Strigilla* sp. In this case, the jetty located offshore may produce a less severe deposition of materials than that which was observed in Mucuripe. An exception was the sample from P1, which was dominated by Polychaeta.

Sediment type and texture are considered major drivers of benthic communities. The heterogeneity of substrates produces a variety of habitats, a context which enables the settlement of different organisms and thus results in a diverse environment (Fresi et al., 1983). As discussed, sediment type in Mucuripe and Pecém is influenced by the structure of jetties, a fact which is corroborated by the high density of polychaetes in the deposition zones. Some species of this group can rapidly colonize stressed environments through either natural or anthropogenic means due to their short life cycle, high fertility rate, and reduced body size, all of which makes it easier for them to occupy new habitats (Ugland et al., 2008; Dauvin and Ruellet, 2009).

Some polychaetes found in this study, such as *Laeonereis acuta* and the spionids *Prionospio pinnata* and *Polydora* sp, are opportunistic and tolerant to pollution. These factors favor their proliferation and make them abundant and diverse in both contaminated or organically enriched areas (Borja et al., 2000; Long et al., 2001; Dauvin and Ruellet, 2009). Therefore, the correlations found between PCA results for sediment contamination, toxicity, number of individuals, richness, evenness and occurrence of Polychaeta indicate that the changes in the benthic community have resulted not only from changes in sediment type but also from the effects of pollution. Meanwhile echinoderms and amphipods are sensitive to contaminants and are the first to disappear in polluted areas; they are therefore considered good indicators of environmental quality (Borja et al., 2000; Dauvin and

Ruellet, 2007). Evidence of such responses is reflected in the negative correlations of Axes 1 and 3, which were more relevant to the contribution of the variances of sites M5, M7 to M10, and P5 (Fig. 4).

## 5. Conclusion

The occurrence of deposition zones generated by jetties in both harbors, but more significantly in Mucuripe, provides the primary and main evidence of impacts on sediment quality. In addition to the changes to sediment texture, these zones also induce the accumulation of contaminants in sediments at levels capable of causing toxicity for the aquatic biota. The main contaminants in both ports were found to be chromium, copper, nitrogen, zinc, and TBT. These contaminants are from sources typical of harbor activities. This situation represents a scenario in which benthic organisms are exposed to a cocktail of diverse contaminants. The results of ecotoxicity bioassays and benthic community structure revealed biological effects that are themselves a result of sediment contamination. However, the changes to benthic composition and structure appear to depend of a combination of physical impacts, including the deposition of fine sediments and the toxic effects of contaminants, especially in Mucuripe.

Due to the high deposition of fine particles sediments the concerned areas are subject to dredging and ocean disposal. In such case we recommend the assessment and use of available techniques for remediation of contaminated sediments. Mulligan et al. (2001) and Peng et al. (2009) presented different methods similar to those applied for soil that can be used to mitigate the impacts of sediment contamination. Thus, our results have provided important details for the understanding of sediment quality of tropical environments. The application of SQG as management and conservation instrument has failed to predict toxicity, which reaffirms the need for the development of site-specific SQVs for tropical environments.

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## Appendix A. Benthic infauna species of Mucuripe

Specie	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10
<i>Corbula caribea</i>	0	0	0	0	0	0	0	1	0	0
<i>Abra aequalis</i>	0	0	0	0	0	0	0	0	1	0
<i>Macoma cleryana</i>	3	0	2	0	8	2	2	0	0	0
<i>Lucina pectinata</i>	0	1	0	0	0	0	0	0	0	0
<i>Cerithiopsis latum</i>	0	0	0	0	0	0	0	1	0	0
<i>Mellita quinquiesperforata</i>	0	0	0	0	0	0	0	8	3	1
<i>Melinna cristata</i>	0	0	0	1	0	0	0	0	0	0
<i>Aphelochaeta</i> sp	0	0	0	1	0	0	0	0	0	0
<i>Chaetozone</i>	0	0	0	0	0	0	2	0	0	0

<i>Timarete</i>	0	0	0	0	1	0	8	0	0	0
<i>Cossura candida</i>	0	1	1	0	0	0	1	0	0	0
<i>Grubeulepis fimbriata</i>	0	0	0	0	1	1	0	0	0	0
<i>Pareulepis multibranchiata</i>	0	0	0	0	0	0	1	0	0	0
Eunicidae	0	0	0	0	0	0	0	1	0	0
<i>Glycera lapidum</i>	4	0	1	1	2	0	8	0	0	0
Glycinde	0	0	0	0	0	1	0	0	0	0
<i>Magelona papillicornis</i>	0	0	0	1	1	0	5	0	0	0
<i>Magelona posterolongata</i>	11	0	4	0	2	9	5	0	0	0
<i>Ceratocephale</i>	0	1	0	0	0	0	0	0	0	0
<i>Laeonereis acuta</i>	4	0	0	2	1	0	6	0	0	0
<i>Scoloplos (Leodamas) sp</i>	3	0	0	0	1	0	1	0	0	0
Paraonidae	0	0	0	0	0	0	2	0	0	0
<i>Eulalia sp</i>	1	0	0	0	0	0	0	0	0	0
Sabellidae	0	0	0	0	1	0	0	0	0	0
<i>Sthenolepis grubei</i>	0	0	1	2	0	0	0	1	0	0
<i>Polydora</i>	0	0	0	3	2	0	0	0	0	0
<i>Prionospio pinnata</i>	8	8	1	4	0	0	2	1	0	0
<i>Prionospio cf. pygmaea</i>	0	0	0	0	1	0	7	1	0	0
<i>Prionospio sp</i>	0	0	2	0	0	1	0	0	0	0
<i>Sternaspis capillata</i>	0	1	0	0	0	0	0	0	0	0
<i>Nicolea sp</i>	0	0	0	0	3	0	0	0	0	0
Terebellidae	1	0	0	0	0	0	0	0	0	0
<i>Xiphopenaeus kroyeri</i>	0	0	0	0	1	0	0	1	0	0
Cumacea	0	0	0	0	1	0	0	0	0	0
<i>Psammodallapsea</i>	0	0	0	0	2	0	0	2	0	0
Tanaidacea	0	0	1	0	0	0	0	0	0	0
<i>Ampelisca sp.</i>	0	1	1	0	0	0	1	3	1	0
<i>Jassa sp.</i>	0	0	0	0	4	0	0	0	0	0
<i>Erichthonius sp.</i>	0	1	0	0	14	0	1	0	0	0

## Appendix B. Benthic infauna species of Pecém

Specie	P1	P2	P3	P4	P5
<i>Nucula semiornata</i>	0	21	11	3	1
<i>Corbula caribea</i>	0	0	0	1	2
<i>Donax striatus</i>	0	0	1	0	0
<i>Ctena pectinella</i>	0	0	0	1	0
<i>Abra aequalis</i>	1	0	0	0	0
<i>Strigilla sp</i>	0	2	0	0	2
<i>Tellina trinitatis</i>	0	0	0	1	0
<i>Lucina pectinata</i>	0	0	1	0	0
<i>Paradentalium disparile</i>	0	0	1	1	0
<i>Graptacme eborea</i>	0	0	0	1	0
<i>Ophionereis reticulata</i>	0	1	0	0	0
<i>Notomastus sp</i>	0	2	0	0	0
<i>Chaetozone gracilis</i>	1	2	0	0	0
<i>Timarete</i>	0	0	0	0	2
<i>Grubeulepis fimbriata</i>	0	1	0	6	0
<i>Glycera lapidum</i>	1	0	0	0	0
<i>Glycinde multidentis</i>	1	1	0	0	0
<i>Magelona papillicornis</i>	0	0	3	0	0
<i>Ceratocephale</i>	0	0	1	1	0
<i>Laeonereis acuta</i>	0	1	0	0	0
<i>Scoloplos (Leodamas) sp</i>	11	6	1	0	0
Paraonidae	1	0	0	0	0
<i>Sigambra grubii</i>	1	0	1	0	0
<i>Sthenolepis grubei</i>	1	1	0	0	0
<i>Autolytus sp</i>	0	0	0	0	1
<i>Pinnixa sp</i>	0	1	0	0	0
<i>Excirrolana brasiliensis</i>	0	0	0	0	1



1-C <sub>14</sub> -LAB	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	< 0.85	17	82
1-C <sub>12</sub> -LAB recovery %	94	101	92	101	89	94	97	88	82	95	96	91	94	89	90			

### Appendix E. Concentrations of aliphatic hydrocarbons in sediment samples from Mucuripe and Pecém harbors ( $\mu\text{g g}^{-1}$ )

Compound	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	P1	P2	P3	P4	P5	Preci sion%	Reco very %
n-C <sub>12</sub>	< 0.003	< 0.003	< 0.003	0.005	< 0.003	0.006	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	5.4	70
n-C <sub>13</sub>	0.003	< 0.001	< 0.001	0.005	0.002	< 0.001	0.003	0.001	0.002	< 0.001	< 0.001	< 0.001	0.003	< 0.001	0.003	6.3	73
n-C <sub>14</sub>	0.004	0.003	0.007	0.018	0.006	0.007	0.010	0.004	0.004	0.004	0.006	0.007	0.007	0.010	0.005	4.0	88
n-C <sub>15</sub>	0.007	0.008	0.019	0.030	0.017	0.028	0.020	0.007	0.006	0.010	0.021	0.010	0.016	0.024	0.011	5.3	86
n-C <sub>16</sub>	0.004	< 0.001	0.008	0.031	0.010	0.017	0.013	0.007	0.003	0.020	0.010	0.002	0.006	0.012	0.003	2.7	96
n-C <sub>17</sub>	0.027	0.026	0.060	0.076	0.027	0.081	0.037	0.022	0.011	0.025	0.035	0.025	0.048	0.030	0.018	2.9	91
Pristane	0.011	0.012	0.023	0.036	0.012	0.021	0.014	0.013	< 0.008	0.015	0.012	0.010	0.014	0.013	0.009	6.0	87
n-C <sub>18</sub>	0.009	0.006	0.025	0.046	0.017	0.035	0.022	0.020	0.016	0.018	0.006	0.007	0.008	0.009	0.006	6.9	93
Phytane	0.010	0.010	0.015	0.024	0.010	0.015	0.010	0.009	0.008	0.009	0.009	0.011	0.015	0.008	0.013	8.8	78
n-C <sub>19</sub>	0.020	0.020	0.024	0.035	0.011	0.029	0.018	0.009	0.007	0.007	0.013	0.013	0.018	0.013	0.007	5.5	82
n-C <sub>20</sub>	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	0.010	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	6.0	91
n-C <sub>21</sub>	0.029	0.035	0.038	0.050	0.014	0.036	0.026	< 0.011	< 0.011	< 0.011	0.019	0.039	0.042	0.023	0.013	1.8	93
n-C <sub>22</sub>	0.015	0.013	0.016	0.033	0.009	0.021	0.013	0.012	0.008	0.005	0.011	0.011	0.017	0.009	0.007	2.0	94
n-C <sub>23</sub>	0.040	0.031	0.035	0.054	0.016	0.044	0.026	0.012	0.013	0.009	0.028	0.028	0.027	0.021	0.017	11	90
n-C <sub>24</sub>	0.016	0.016	0.020	0.029	0.011	0.028	0.017	0.017	0.019	0.015	0.012	0.021	0.024	0.020	0.011	13	91
n-C <sub>25</sub>	0.039	< 0.027	0.052	0.069	< 0.027	0.073	0.029	< 0.027	< 0.027	< 0.027	0.027	0.030	0.033	0.030	< 0.027	8.7	92
n-C <sub>26</sub>	0.014	0.016	0.020	0.031	0.008	0.019	0.013	0.007	0.011	< 0.007	0.016	0.021	0.033	0.029	0.013	6.0	88
n-C <sub>27</sub>	0.086	0.049	0.082	0.102	< 0.034	0.102	0.053	< 0.034	< 0.034	< 0.034	0.050	0.063	0.068	0.066	0.036	11	87
n-C <sub>28</sub>	< 0.034	< 0.034	< 0.034	< 0.034	< 0.034	0.038	< 0.034	< 0.034	< 0.034	< 0.034	0.035	< 0.034	0.049	< 0.034	< 0.034	6.0	89
n-C <sub>29</sub>	0.112	0.084	0.153	0.199	0.029	0.224	0.082	< 0.028	< 0.028	< 0.028	0.080	0.138	0.125	0.088	0.054	12	102
n-C <sub>30</sub>	0.113	0.057	0.058	0.082	< 0.028	0.052	0.044	< 0.028	< 0.028	< 0.028	0.075	0.068	0.100	0.048	0.044	8.5	96
n-C <sub>31</sub>	0.111	0.074	0.113	0.158	< 0.026	0.114	0.091	< 0.026	< 0.026	< 0.026	0.080	0.122	0.127	0.075	0.048	10	100
n-C <sub>32</sub>	< 0.026	< 0.026	< 0.026	0.031	< 0.026	< 0.026	< 0.026	< 0.026	< 0.026	< 0.026	0.047	0.047	0.042	0.026	< 0.026	5.6	96
n-C <sub>33</sub>	0.077	0.050	0.081	0.114	0.020	0.083	0.082	< 0.012	< 0.012	< 0.012	0.068	0.098	0.099	0.058	0.043	6.3	91
n-C <sub>34</sub>	0.024	0.015	0.021	0.020	< 0.012	0.012	0.013	< 0.012	< 0.012	< 0.012	0.023	0.026	0.028	0.023	0.016	10	87
n-C <sub>35</sub>	0.046	0.047	0.053	0.069	< 0.012	0.033	0.047	< 0.012	< 0.012	< 0.012	0.044	0.066	0.063	0.034	0.026	12	91
n-hexadecene recovery %	96	102	80	97	101	95	93	99	85	93	94	92	95	88	91		

### Appendix F. Concentrations of polycyclic aromatic hydrocarbons (PAH) in sediment samples of Mucuripe harbor ( $\text{ng g}^{-1}$ )

Compound	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10
Naphthalene	1.78	4.41	1.75	4.18	< 1.60	2.20	< 1.60	< 1.60	< 1.60	< 1.60
Methylnaphthalenes	1.68	6.75	< 1.30	3.93	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Biphenyl	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Ethyl-naphthalenes	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60
Dimethylnaphthalenes	2.72	10.6	< 2.60	6.87	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60
Acenaphthylene	< 3.70	< 3.70	< 3.70	< 3.70	< 3.70	< 3.70	< 3.70	< 3.70	< 3.70	< 3.70
Acenaphthene	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Trimethylnaphthalenes	1.58	3.83	< 1.30	3.20	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Fluorene	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Dibenzothiophene	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Phenanthrene	< 2.60	3.12	2.90	3.41	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60
Anthracene	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10
Methylphenanthrenes	3.56	6.99	3.08	4.42	< 2.20	2.68	3.10	< 2.20	< 2.20	< 2.20
Fluoranthene	6.64	5.90	7.97	8.00	3.51	4.15	2.80	< 1.30	< 1.30	< 1.30
Pyrene	6.11	6.62	5.98	6.36	2.85	3.30	2.44	< 1.30	< 1.30	< 1.30
Methylfluoranthenes	< 1.30	3.75	2.49	2.90	1.32	1.72	1.69	< 1.30	< 1.30	< 1.30

Retene	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Methylpyrenes	1.88	4.86	1.55	2.25	< 1.30	< 1.30	2.23	< 1.30	< 1.30	< 1.30
Benzo(c)phenanthrene	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20
Benzo(a)anthracene	3.57	4.16	3.07	3.20	2.14	1.73	1.27	< 1.20	< 1.20	< 1.20
Chrysene	4.79	5.03	4.34	4.84	2.65	2.95	1.98	< 1.20	< 1.20	< 1.20
Methylchrysene	4.57	8.82	2.79	4.26	1.48	2.76	2.01	< 1.20	< 1.20	< 1.20
Benzo(b)fluoranthene	3.44	3.12	2.59	3.02	1.99	2.09	1.43	< 1.30	< 1.30	< 1.30
Benzo(j)fluoranthene	2.81	1.97	1.68	2.04	1.35	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Benzo(k)fluoranthene	2.17	1.90	1.43	1.80	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
Benzo(e)pyrene	4.71	4.38	3.20	4.01	2.14	2.31	1.64	< 1.30	< 1.30	< 1.30
Benzo(a)pyrene	4.80	5.47	3.60	4.11	2.45	2.54	1.89	< 1.10	< 1.10	< 1.10
Perylene	7.49	3.7	4.24	6.01	1.31	3.88	17.6	< 1.20	< 1.20	< 1.20
Indeno[1,2,3-c,d]pyrene	4.54	3.39	3.32	4.26	2.23	2.12	1.61	< 1.00	< 1.00	< 1.00
Dibenzo(a,h)anthracene	1.14	1.35	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00
Benzo(b)chrysene	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10	< 1.10
Benzo(ghi)perylene	5.86	5.64	3.67	4.85	2.53	2.30	1.80	< 1.20	< 1.20	< 1.20
Coronene	2.03	2.05	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20	< 1.20
Naphthalene-d <sub>8</sub> recovery %	90	90	93	90	89	90	88	65	71	86
Acenafteno-d <sub>10</sub> recovery %	87	86	89	87	85	86	84	74	78	83
Phenanthrene-d <sub>10</sub> recovery %	89	88	91	88	87	88	86	86	90	85
Chrysene-d <sub>12</sub> recovery %	96	95	99	96	94	96	93	93	98	102
Perylene-d <sub>12</sub> recovery %	84	87	87	86	85	81	81	81	80	87

#### Appendix G. Concentrations of polycyclic aromatic hydrocarbons (PAH) in sediment samples of Pecém harbor (ng g<sup>-1</sup>)

Compound	P1	P2	P3	P4	P5	Precision %	Recovery %
Naphthalene	10.3	3.54	2.27	1.89	18.6	19	71
Methylnaphthalenes	2.60	1.57	1.37	1.47	2.57	17	74
Biphenyl	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	18	75
Ethyl-naphthalenes	< 2.60	< 2.60	< 2.60	< 2.60	< 2.60	10	76
Dimethylnaphthalenes	5.44	< 2.60	2.80	2.99	2.92	6.7	76
Acenaphthylene	< 3.70	< 3.70	< 3.70	< 3.70	< 3.70	4.1	77
Acenaphthene	< 1.30	< 1.30	< 1.30	< 1.30	1.44	4.4	82
Trimethylnaphthalenes	3.35	< 1.30	1.96	< 1.30	1.46	15	77
Fluorene	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	1.6	78
Dibenzothiophene	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	8.4	94
Phenanthrene	6.01	11.85	3.50	< 2.60	9.21	5.0	102
Anthracene	< 1.10	1.94	< 1.10	< 1.10	2.19	5.5	101
Methylphenanthrenes	3.34	5.89	2.81	< 2.20	4.60	5.4	95
Fluoranthene	18.4	40.1	10.3	3.84	25.3	6.0	87
Pyrene	14.3	29.2	7.58	2.92	17.2	8.6	88
Methylfluoranthenes	4.51	7.73	3.04	1.38	7.50	7.6	82
Retene	< 1.30	< 1.30	< 1.30	< 1.30	< 1.30	9.0	92
Methylpyrenes	2.64	4.00	1.77	< 1.30	3.07	1.7	99
Benzo(c)phenanthrene	1.21	2.59	< 1.20	< 1.20	1.65	6.5	96
Benzo(a)anthracene	6.77	16.7	3.58	1.68	10.7	4.3	86
Chrysene	8.76	24.5	5.00	2.48	11.6	3.7	94
Methylchrysene	3.47	9.27	2.44	1.41	4.95	4.4	92
Benzo(b)fluoranthene	6.79	14.6	3.76	1.70	7.71	9.9	71
Benzo(j)fluoranthene	3.49	16.4	3.42	< 1.30	6.27	10	73
Benzo(k)fluoranthene	5.04	14.2	2.75	< 1.30	5.55	11	75
Benzo(e)pyrene	6.70	17.9	3.77	2.00	7.84	7.7	94
Benzo(a)pyrene	7.89	23.6	4.38	2.21	10.37	9.8	89
Perylene	3.58	9.03	3.22	2.01	3.96	12	85
Indeno[1,2,3-c,d]pyrene	6.37	16.4	3.62	1.81	7.18	14	79
Dibenzo(a,h)anthracene	4.76	3.06	< 1.00	< 1.00	1.63	11	83
Benzo(b)chrysene	< 1.10	1.29	< 1.10	< 1.10	< 1.10	15	81
Benzo(ghi)perylene	6.92	16.7	3.91	2.15	7.49	16	79
Coronene	1.30	< 1.20	< 1.20	< 1.20	< 1.20	19	77
Naphthalene-d <sub>8</sub> recovery %	91	84	87	86	85		
Acenafteno-d <sub>10</sub> recovery %	87	81	83	82	82		

Phenanthrene-d <sub>10</sub> recovery %	89	83	85	84	84
Chrysene-d <sub>12</sub> recovery %	97	99	102	101	100
Perylene-d <sub>12</sub> recovery %	84	98	90	87	89

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