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Dredging impacts on the toxicity and development of sediment quality values in a semi-arid region (Ceará state, NE Brazil)

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ABSTRACT

Sediment dredging impacts coastal environments by promoting the resuspension of fine particles and remobilization of contaminants that may trigger toxic effects. In this study, we evaluated the sediment quality in harbor areas of Mucuripe bay, a semi-arid ecosystem located in Ceará state (Brazil), which is subject to dredging activities. A sampling survey was conducted right after dredging operations and data compared to another survey performed prior dredging. Sediments were analyzed for fine particles, organic carbon, nutrients, metals, hydrocarbons, and tributyltin (TBT). Toxicity of whole-sediment and liquid phase exposures were also determined. The concentrations of Cd, Cr, Cu, and Zn decreased after dredging, which was confirmed by the geoaccumulation index. Levels of TBT dropped while phosphorus, aliphatic and polycyclic aromatic hydrocarbons increased. Toxic effects persisted, indicating a post-dredging recontamination combined with other sources such as urban runoff, wastewater discharges, harbor activities, and antifouling particles. Data from Mucuripe and Pecém harbors were compiled and site-specific sediment quality values (SQVs) were developed by using multivariate methods. The threshold values proposed by our study were lower and more effective to predict toxicity compared to international guidelines, indicating levels of contamination for this tropical region in which toxic effects may occur. Considering the large geographic area with different sediment characteristics of the Brazilian coast, this study represents a significant contribution to sediment toxicity assessment of dredging activities in semi-arid environments.

1. Introduction

Dredging materials are products of sediment excavation and removal from navigation channels of harbor zones, in order to facilitate the traffic of ships with different sizes and drafts. Hundreds of millions of tons of dredged sediments are disposed in coastal environments (e.g. shallow waters, estuaries, and embayments) every year, which make them as one of the most hazardous materials to threaten marine biota worldwide in terms of physical, chemical, and biological effects (Schipper et al., 2010; Manap and Voulvoulis, 2016).

The primary impact of sediment excavation is the resuspension of particles and the generation of turbid plumes that drift to adjacent habitats, changing the primary productivity (Fisher et al., 2015). Another plume can also be formed by the pumping of water excess inside the hopper cistern (known as overflow), allowing the dredge to be filled (Jones et al., 2016). Both processes combined can induce the recontamination by moving hazardous substances from the soft bottoms to the water column or favor them to sink on more recent sediment layers (Liu et al., 2016; Manap and Voulvoulis, 2016; Vagge et al., 2018). At the disposal site, the discharge of dredged materials can also inflict direct

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contamination (Cesar et al., 2014).

The chemicals frequently found in dredged sediments include trace metals, hydrocarbons, and biocides from antifouling systems such as tributyltin (TBT) (Castro et al., 2012; Moreira et al., 2017). These compounds are released from punctual and diffuse sources and deposited on the superficial layers posing risks of toxicity to benthic organisms (Burton and Johnston, 2010). Specific protocols for the evaluation, monitoring and management of dredged materials have been proposed, focusing on the chemistry-based criteria by means of environmental quality standards (EQS), sediment quality guidelines (SQGs) and sediment quality values (SQVs) (Burton, 2002; Choueri et al., 2009). SQGs are frameworks that include EQS for different groups of substances as a chemical-based metric, combined with the assessment of adverse biological effects (e.g. sediment toxicity) in a weight-of-evidence approach to estimate the impacts (Birch, 2018). SQVs are basically the EQS for sediments and are applied as a line of evidence in the SQGs. Whenever chemical concentrations in sediment samples are found above the first threshold value (Level 1), the toxicity may occur or not, and above the second value (Level 2), there will be a high probability to occur (Chapman and Mann, 1999; Bellas et al., 2011).

There are a variety of SQGs developed by different techniques that can be clustered into three main categories. The first one consists of Mechanistic Approaches which include equilibrium partitioning (EqP) to address bioavailability, chemical uptake and toxicity. The second group regards Empirical Approaches that are correlative studies matching chemical and toxicity data. The third group involves Sediment Quality Indices, which are based on geochemical information such as baseline concentrations and geoaccumulation indices (Birch, 2018). Environmental agencies from the USA (Long et al., 1995), Canada (EC, 2000), Europe (OSPAR 2008), and Australia (EA, 2002) have developed SQGs. However, for other countries, for instance Brazil, SQGs are based on those international values due to the lack of knowledge on sediment quality.

Tropical zones are biodiverse and in countries like Brazil, some regions differ from others in terms of climate, sedimentology, and productivity. In the Northeast region, for example, ocean waters are oligotrophic and benthic environments are marked by sandy deposits, rich in carbonates from biogenic origin and low contents of organic matter (Knoppers et al., 1999; Lacerda and Marins, 2006). Such characteristics contrast with other regions, such as the South and Southeast, where higher precipitation, colder waters, and elevated productivity predominate, in conjunction with sediment supply at the coastal zone (Dominguez, 2009). These features may result in distinct conditions of sediment contamination and adverse effects.

As for SQGs, procedures for disposal of dredged materials are established by the federal resolution CONAMA 454/12 (Brasil, 2012), which presents SQVs for metals, polycyclic aromatic hydrocarbons (PAHs), and TBT. However, previous studies have demonstrated that these threshold values are not effective to predict toxic effects because they are based on empirical SQVs adopted in USA and Canada, and did not consider differences of the Brazilian coast and its biodiversity (Abessa et al., 2006; Buruaem et al., 2012; Moreira et al., 2017). In this case, site-specific SQVs are more appropriate to properly address ecological risks (DelValls and Chapman, 1998). Choueri et al. (2009) developed site-specific SQVs for the Paranaguá and Santos Estuarine Systems (SE Brazil) by using an integrative approach. The values were more effective to predict effects in comparison to the SQVs set by the resolution CONAMA 454/12 (Moreira et al., 2019a). However, such SQV suitable for application in samples from estuarine systems of South and Southeast regions.

The state of Ceará has ecological and economic importance to the Brazilian Northeast region and its capital, Fortaleza, presents a population of more than 4 million inhabitants. Anthropogenic pressure has contaminated the coastal zone by multiple sources including urban runoff, sewage, industrial effluents discharges, and harbor activities (Cavalcante et al., 2009; Buruaem et al., 2012). Mucuripe and Pecém are the two main harbors of the state with a cargo volume of approximately 20 million tons in 2019 (ANTAQ 2020). Mucuripe has a siltation estimated in 610 000 m^3 (annual volume) and as a consequence, it is subjected to regular dredging activities for deepening and maintenance. Hence, the oceanic disposal of dredged material occurs at the western portion of Mucuripe bay in the area affected by the submarine outfall diffuser, 5 km towards the west and 3 km from the coast (Maia et al., 1998; Neto et al., 2018).

Sediment quality in harbor zones of Mucuripe bay and Pecém was previously assessed in terms of chemical characterization, toxicity and changes in benthic community structure (Moreira et al., 2017). In Mucuripe, higher contamination and toxicity were also observed during the resuspension caused by the hopper dredger, indicating the mobilization of contaminants (Moreira et al., 2019b). For both studies, the resolution CONAMA 454/12 failed to predict toxicity, confirming the importance of site-specific SQVs. Thus, we aimed to improve the knowledge of sediment quality in Brazil by addressing two main objectives. The first one is to evaluate the changes in sediment quality of Mucuripe bay after dredging operations by assessing contamination levels and toxicity, while the second one proposed a site-specific set of SQVs for the studied zone. Data obtained during different surveys were compiled from previous studies (Moreira et al., 2017, 2019b) and threshold values were produced matching levels of contamination and toxicity using a multivariate approach. Our results represent the first report of EQVs for semi-arid environments of the South Atlantic and will assist the proper evaluation of sediment quality and dredging management procedures in tropical regions such as Brazil.

2. Material and methods

2.1. Study strategy

The impacts of dredging on the sediment quality of Mucuripe bay were assessed from the results of two sampling surveys, the first one prior to dredging operations (survey 1), and the second one 4 years later, right after the dredging activities (survey 3). Data of survey 1 were obtained from a previously published study (Moreira et al., 2017) and included 10 sites sampled in August 2007: M1 and M2, situated in front of commercial docks, M3 to M5, close to fishing and tanker piers, M6 to M8, close to the access channel, and M9 and M10, at the unsheltered areas. Survey 3 was conducted for this study in August 2011 at the same locations of survey 1, except for M10 (M1b to M9b) (Fig. 1).

For the development of SQVs, data from Pecém harbor and Mucuripe bay (surveys 1, 2 and 3) were compiled. Results of Pecém were also obtained from a previous study and sediments were sampled in January 2008 (Moreira et al., 2017). The survey 2 involved two different periods of intense dredging in January 2011 (MD1 to MD3) and July 2011 (MD1′ to MD3′). This strategy was adopted because the excavation activities affected the whole area on a daily basis, limiting the sampling to the following 3 sites: MD1, in front of the commercial docks, MD2 close to the pier of the oil terminal, and MD3, at the entrance of the navigation channel. Two samples from survey 2 were included in the dataset to increase the number of samples and obtain a robust results, one from the cistern of the trailing suction hopper dredger (OF, overflow), and the other from a reference site at Requenguela beach, in the city of Icapuí (4°40′54.7″S, 37°20′13.9″W).

2.2. Sediment analysis

Surface sediment samples (upper 3 cm) were obtained after dredging (survey 3) using a van Veen grab (0.026 m^2). For each site, three subsamples were combined to obtain a composite sample, transported to the laboratory in coolers boxes and divided into 3 aliquots. The first one was designated for particle size and levels of nutrients and inorganic contaminants analysis, which was dried at room temperature by using a desiccator cabinet and stored in plastic containers. The second one, used

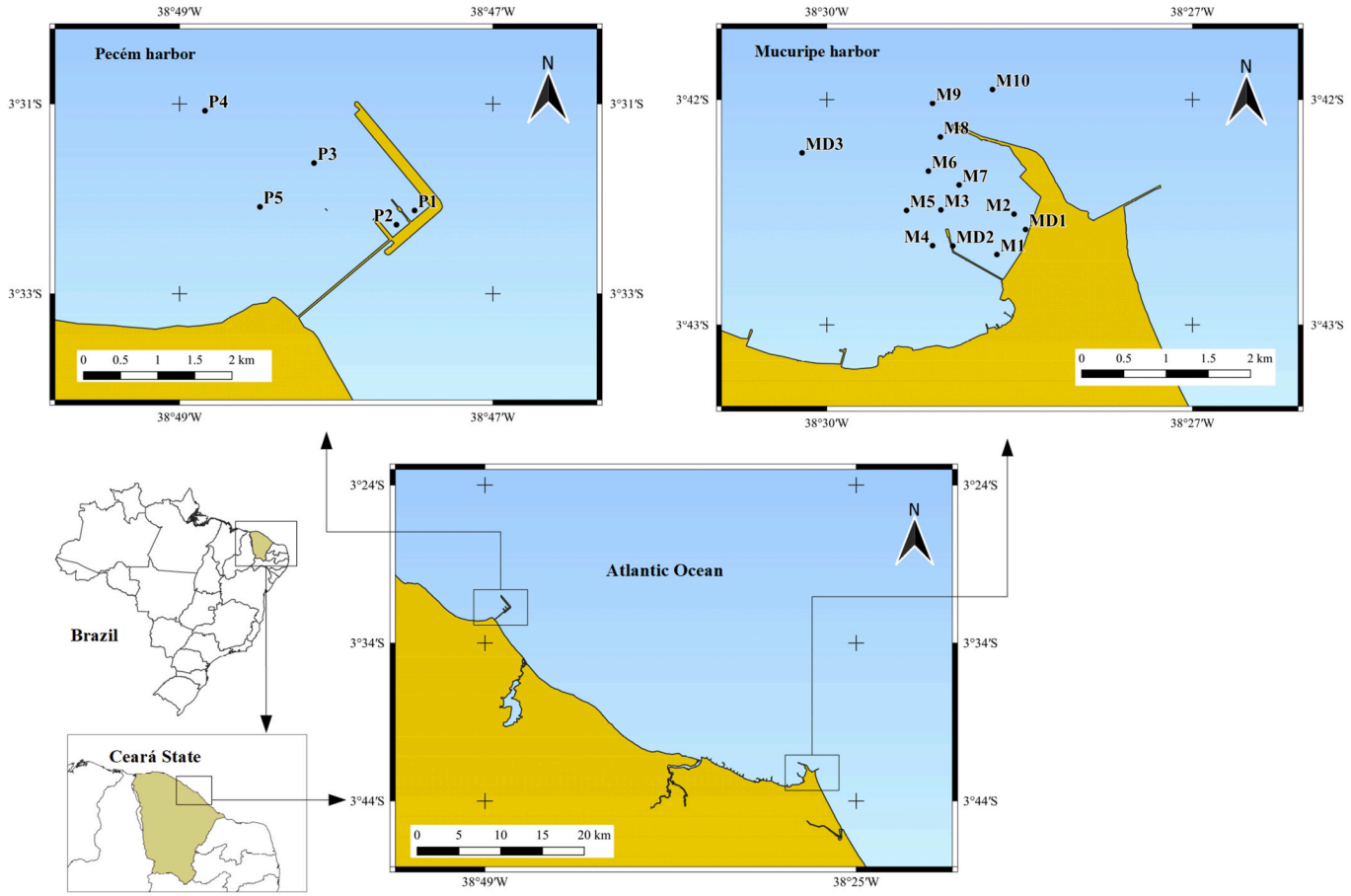

Fig. 1. Sites of sediment sampling in Mucuripe and Pecém harbors (P1 to P5). Survey 1 conducted prior to dredging (M1 to M10), survey 2 during intense dredging (MD1 to MD3), and survey 3 performed after dredging activities (M1b to M9b).

for the analysis of organic contaminants, was wrapped into precleaned aluminum foil and stored at −20 °C. The third aliquots for toxicity tests were stored at 4 °C.

For sediment analysis, the content of fines (%) was determined as a particle size metric. Nutrients included the levels in % of total organic carbon (TOC), total nitrogen (N), and phosphorus (P). Inorganic contaminants analysis were based on the quantification of major (Al and Fe) and trace metals (Cd, Cr, Cu, Hg, Ni, Pb, and Zn). Organic compounds included concentrations of 26 aliphatic hydrocarbons (AHs: n-alkanes C_{12} to C_{35}, resolved compounds, unresolved complex mixture (UCM), pristane and phytane, 33 polycyclic aromatic hydrocarbons (PAHs), and 26 linear alkylbenzenes (LABs). For these groups of hydrocarbons, results were expressed as the total sum of individual compounds. The organotin tributyltin (TBT) was also determined in sediment samples. Analytical procedures and protocols are described in previous studies (Castro et al., 2012; Buruaem et al., 2016; Moreira et al., 2017; Lacerda et al., 2019). Regarding the quality assurance (QA) and Quality control (QC), the results were expressed as average of 2 repeated measurements (coefficient of variation below 10%). The validation of analytical methods was checked by the analysis of surrogates, blank samples, and Certified Reference Materials (CRM) for metals (BCR® 667) hydrocarbons (NIST® SRM® 1944) and TBT (PACS -2/NRC - CNRC).

The toxicity was assessed for solid and liquid phases. The whole sediment (WS) exposure was employed as a solid-phase bioassay, according to the protocol described by the Brazilian National Standards Organization (ABNT) in the Brazilian National Standard (NBR) #15638 (ABNT, 2008). The mortality rate of the burrowing amphipod *Tiburonella viscana* after 10 days was used as the endpoint due to its sensitivity to a wide range of contaminants (Melo and Nipper, 2007). The liquid phase involved the analysis of elutriates (ELU), which are liquid extracts that simulate sediment resuspension and were prepared according to USEPA (2003). Samples were tested for the waterborne toxicity based on the embryo-larval development of the sea urchin *Lytechinus variegatus,* following the method described by ABNT in the NBR #15350 (ABNT, 2006).

For all treatments, negative controls were prepared. Sediments from a pristine location (amphipods sampling site) were used in the solid phase bioassays, and filtered and uncontaminated seawater was used in the liquid phase exposures. Salinity, temperature, and dissolved oxygen were checked during the experiments. Total ammonia concentration was measured in ELU chambers and unionized ammonia (NH_3) levels were estimated to assess its contribution to toxicity. The student's t-test was used to compare effects measured in each treatment and the respective controls. Samples that significantly differed were considered toxic.

2.3. Data interpretation and development of site-specific SQVs

Changes in sediment contamination over the 3 surveys were assessed by interpolating confidential intervals of concentrations measured for each survey and plotting as Box and Whisker charts. Sediment toxicity was evaluated qualitatively based on the results of toxic or nontoxic assignment of the student's t-test. The contamination status of samples based on major and trace metals was assessed by means of geo-accumulation Index (I_{geo}), following the calculations:

$$I_{geo} = \log 2(C_n / 1.5 * B_n)$$

where C refers to the concentration of the individual metal $_n$ measured in

the sample, B represents the background value or reference one for the respective metal, and the 1.5 factor corresponds to possible variations due to natural processes (Buruaem et al., 2012). After that, results are compared to a qualitative scale of contamination status (Table 2, Supplementary material) in which Igeo values above 1 represent signs of contamination. Reference values obtained by Aguiar et al. (2007) for sediment samples from the continental shelf of Ceará were adopted for all metals, except for Hg which were obtained from Ceará River Estuary (Marins et al., 2004), and Cd which was assessed in relation to background values determined for harbor areas of Santos Estuarine System (Luiz-Silva et al., 2006). For hydrocarbons, low molecular weight (LMW, 2 and 3 aromatic rings PAHs) and high molecular weight (HMW, 4 to 6 aromatic rings PAHs) were computed, the compounds benzo[a]anthracene (BaA), chrysene (Chry), Indeno[1,2,3-cd]pyrene (IP), and Benzo [ghi]perylene (BghiP) were selected and their origin assessed following the diagnostic ratios: $\sum$15 LMW/$\sum$18 HMW <1 pyrogenic and 1< petrogenic; IP/(IP + BghiP) < 0.2 indicate petrogenic, 0.2–0.5 Petroleum combustion and 0.5< Grass, wood and coal combustion; and (iii) BaA/(BaA + Chry) < 0.2 petrogenic, 0.2–0.35 coal combustion and 0.35< combustion (Yunker et al., 2002; Zhang et al., 2008).

Concerning the development of site-specific SQVs, we adopted the empirical approach proposed by DelValls and Chapman (1998) with modifications. Principal component analysis (PCA) was used to match concentrations of contaminants and biological effects. First, we constructed a matrix containing data from all surveys. Sediment analysis data included the % of fine particles, TOC, N, P, Al, and Fe, and concentrations of trace metal (Cd, Cr, Cu, Hg, Ni, Pb, and Zn), hydrocarbons (AHs, PAHs, and LABs) and TBT. Toxicity data consisted of adverse effects (% of mortality and abnormal larvae) of each sample corrected by their respective control, according to the survey. Data expressed in % were transformed into a linear scale using an arcsine transformation and, after that, the matrix was normalized by logarithmic function Log (x+1) to adjust skewed distributions. Then, a PCA was performed to extract up to 92% of the variance in the first 3 components. The cut-off values were set at |0.40| for factor loading to establish the significant correlations (Comrey and Lee, 1992).

To establish the threshold levels, we used the outcome provided by PCA ordination in which samples whose toxicity was correlated to concentrations of chemicals were separated from those that presented low contamination, or absent toxicity. In this case, higher scores for PC1 and PC2 determined the cluster. For each contaminant, the 95th percentiles of samples with minimal contamination were set as Level 1 (contamination not matched with toxicity), where below these values toxic effects are not expected. Similarly, the 95th percentiles of samples associated with PC1 and PC2 were set as Level 2, where toxic effects are expected to occur above them. When the Level 2 was determined below those for Level 1, we considered the value for Level 1 as the only threshold (e.g. for Cd, AHs, LABs, and TBT). The predictive performance of site-specific SQVs in comparison to SQGs of resolution # 454 was analyzed by means of sediment quality guideline quotients (SQGQs). In this approach, the concentrations measured in each sample were divided by their respective probable effect concentrations (Level 2) and, after that, the mean quotients were computed and samples classified according to Fairey et al. (2001):

(a) Minimal contamination with low or no toxicity: SQGQ values between 0 and 0.1;
(b) Moderate contamination that may produce toxicity: SQGQ values between 0.1 and 0.25;
(c) Strong contamination that causes toxicity: SQGQ values greater than 0.25.

Table 1
Results of particle size, chemical characteristics, and toxicity of sediments from Mucuripe Bay after dredging activities (survey 3). Values expressed as dry weight basis, except for toxicity. WS= Whole sediment toxicity (% of amphipod mortality); ELU = toxicity of elutriates (% of sea urchin abnormal larvae). *Toxic sample compared with respective control.

Site	Fines (%)	TOC (%)	N (%)	P (%)	Al (%)	Fe (%)	Hg (ng g^{-1})	Cd (µg g^{-1})	Cr (µg g^{-1})	Cu (µg g^{-1})	Ni (µg g^{-1})	Pb (µg g^{-1})	Zn (µg g^{-1})	AHs (µg g^{-1})	PAHs (ng g^{-1})	LABs (ng g^{-1})	TBT (ng Sn g^{-1})	Toxicity	
																		WS	ELU
M1b	90.18	1.91	0.86	2.50	2.03	1.39	79.90	0.17	13.96	20.44	15.25	21.37	27.71	219.26	566.48	42.32	15	*57 ± 15	*100 ± 0
M2b	68.89	1.38	0.68	2.98	1.89	1.25	50.10	0.18	11.73	16.86	11.52	65.89	25.05	586.45	4042.84	110.90	9.8	*60 ± 26	*100 ± 0
M3b	78.67	0.47	0.50	2.19	1.94	1.17	24.90	0.18	13.22	7.66	10.75	13.21	12.13	22.74	62.14	<0.85	33.6	*53 ± 21	*100 ± 0
M4b	88.18	0.93	0.60	2.68	1.89	1.47	28.80	0.55	13.55	12.10	12.77	17.04	16.54	22.96	100.54	8.68	16.4	*90 ± 17	*100 ± 0
M5b	95.36	1.53	0.61	2.81	2.09	1.64	38.20	0.28	4.31	9.91	14.49	20.53	18.60	27.96	152.06	11.10	11.4	*53 ± 15	*100 ± 0
M6b	73.02	0.16	0.39	2.31	0.83	0.91	15.50	0.23	<2.00	5.54	7.19	23.28	9.08	16.97	47.15	<0.85	52.6	*87 ± 23	*100 ± 0
M7b	56.86	0.57	0.19	2.70	1.02	1.01	24.40	0.14	15.81	7.42	8.60	24.51	11.17	8.95	29.97	3.87	39.6	37 ± 15	*100 ± 0
M8b	45.83	0.27	0.32	3.30	0.66	0.75	33.10	0.18	<2.00	6.64	6.73	10.54	8.71	1.44	35.90	<0.85	<2.0	27 ± 6	*100 ± 0
M9b	1.86	0.04	0.26	2.54	0.08	0.14	4.40	0.15	<2.00	3.18	2.29	8.42	1.03	0.95	<1.00	<0.85	<2.0	27 ± 12	22 ± 7

Table 2
Principal Components Analysis (PCA) results for particle size, chemical contamination, and toxicity of sediments collected in harbor areas of Ceará state (Surveys 1 to 3). Significant correlations marked in bold.

Variable	PC 1	PC 2	PC 3
% of fines	**0.80**	0.32	−0.45
TOC	**0.96**	−0.10	0.05
N	**0.97**	−0.01	−0.14
P	**0.85**	0.22	−0.40
Al	**0.95**	−0.29	0.04
Fe	**0.95**	−0.29	−0.04
Hg	**0.70**	0.44	−0.07
Cd	**0.74**	−0.38	−0.01
Cr	**0.67**	**−0.64**	0.34
Cu	**0.95**	−0.10	0.17
Ni	**0.90**	−0.40	0.13
Pb	**0.67**	**0.69**	0.07
Zn	**0.53**	**−0.73**	0.40
AH	**0.53**	**0.69**	0.48
PAHs	**0.50**	**0.65**	**0.54**
LABs	**0.55**	**0.69**	0.46
TBT	**0.71**	**−0.56**	0.01
WS	**0.82**	0.10	−0.38
ELU	**0.77**	0.32	−0.50
Eigenvalue	**11.54**	4.05	1.84
Variance	**60.76%**	21.34%	9.70%
Cumulative variance	**60.76%**	82.10%	91.80%

3. Results and discussion

3.1. Changes in sediment quality of mucuripe harbor after the dredging operations

Sediment transport along the Ceará coast occurs from East to West influenced by Trend winds and North Brazil Current (Dominguez, 2009). The main geochemical process controlling the distribution of contaminants in Mucuripe bay is related to siltation at the harbor zones. In this case, the suspended particulate matter is the main carrier (Dias et al., 2013; Lacerda et al., 2020) and seasonal variations are relevant for estuarine areas (Dias et al., 2016). Based on that, we hypothesized that the concentration of contaminants increases in harbor areas over time, and the intervention caused by the dredging can alter the sediment quality as discussed as follows.

3.1.1. Particle size, total organic carbon, and nutrients

Data on sediment quality in Mucuripe bay for survey 3 are presented in Table 1. Results for surveys 1 and 2 are compiled in the supplementary material (Table 1). Levels of mud prior to dredging (survey 1) ranged from 15 to 24%, with higher values observed in a M1, M3, M5 and M6. After dredging activities (survey 3), levels of fines increased in all samples (above 45%), except for the unsheltered site (M9), indicating the off-limits of the deposition zone produced by the harbor installations.

Outer shelf sediments of Ceará coast (40 m isobath) are characterized by coarser materials, contrasting with particles deposited at the shore line to inner shelf limits (20 m isobath), that are covered by sandy sediments and biodetritic gravels, with up to 2.5% of mud (Freire et al., 2004). The higher amounts of fines observed within harbor areas resulted from the siltation caused by the jetties (Maia et al., 1998; Neto et al., 2018). The increased levels of fines after dredging operations can be explained by the mixed effect of siltation and resuspension of particles caused by excavation procedures. The overflow pumped from the hopper presents high amounts of mud, boosting the spill and plume formation (Jones et al., 2016). As a consequence, the suspended particles are deposited within the harbor area at the navigation channel due to the sheltering effect produced by the jetties.

Contents of TOC were similar among surveys varying from 0.16 to 1.43% prior to dredging (survey 1) and from 0.04 to 1.91% after dredging (survey 3), with higher levels found at the sheltered sites (M1 to M7). Results are consistent with the distribution of Ceará state continental shelf, which is covered by carbonate-rich sediments originated from the biogenic origin (*Lithothamnium* sp., *Halimeda* sp., corals, bryozoans, and other organisms) (Knoppers et al., 1999; Marques et al., 2008). These deposits are characterized by levels of organic carbon up to 1.4% for areas with finer particles and 0.4% for areas with coarser sediments (Knoppers et al., 1999).

Although nutrients presented similar ranges of concentrations among surveys, there was a slight increase of N (based on the median values) and up to one order of magnitude increase of P in survey 3 compared to survey 1 (Fig. 2). The inputs of both nutrients along the northeast coast, including Ceará state, are originated from natural processes and multiple activities such as atmospheric deposition, domestic and industrial wastes, urban runoff, agriculture, and intensive shrimp farming (Lacerda 2006; Marins et al., 2011; Barcellos et al., 2019). Since the impacts in Mucuripe bay are produced by urban and industrial activities, the effects of dredging activities may be associated with the remobilization or representing past conditions related to urban runoff and wastewater discharges.

3.1.2. Sediment contamination by trace metals, hydrocarbons and tributyltin

For metals, concentrations were determined for Al (0.08–2.09%), Fe (0.14–1.64%), Hg (4.4–79.9 ng g^{-1}), Cd (0.14–0.55 μg g^{-1}), Cr (4.31–15.81 μg g^{-1}), Cu (3.18–20.44 μg g^{-1}), Ni (2.29–15.25 μg g^{-1}), Pb (8.42–65.89 μg g^{-1}), and Zn (1.03–27.71 μg g^{-1}). These levels were higher than those reported for shelf sediments of Ceará coast (Aguiar et al., 2014), and they also changed by dredging operations. Concentrations of Al, Fe, Cr, Cu, Hg, and Ni did not show any trends among surveys (Fig. 2), while decreases of Cd and Zn were seen from survey 1 to survey 3. On the other hand, the concentrations of Pb increased in survey 3. These changes are confirmed by the results of I_{geo}, which classified samples of survey 1 from uncontaminated to moderately contaminated for Al, Cr, and Ni, moderately to strongly contaminated for Cu, and strongly contaminated for Cd and Zn. In survey 3, concentrations of Pb and Cd were found as moderately contaminated levels, while Hg and Cu were classified as moderately to strongly contaminated. Levels of Fe were classified as uncontaminated in both surveys (Table 2 and of supplementary material, and Fig. 3).

The main sources of Cr, Cu, and Zn in Mucuripe bay were attributed to specific inputs from harbor activities and marinas in the vicinity (Moreira et al., 2017). It has been reported that these metals, along with Cd, Ni, Pb, and Sn are found in higher concentrations in sites impacted by antifouling particles, since they are constituents of the polymeric matrix of antifouling coatings (Soroldoni, 2010). Regarding the Hg distribution, Lacerda et al. (2019) reported higher concentrations in suspended particulate matter inside the harbor area compared to the unsheltered area, indicating a contribution of this component to Hg deposition.

Regarding hydrocarbons, results for AHs are shown in Table 1 and data of n-alkanes, resolved aliphatics, (UCM), and carbon preference index (CPI) are detailed in supplementary material (Tables 4 and 5). Levels of AHs increased in one order of magnitude in samples collected near the commercial docks (M1b and M2b) in survey 3 in relation to survey 1. Concentrations of n-alkanes were low in both surveys at levels of uncontaminated conditions (Volkman et al., 1992), with a slight increase observed after dredging (survey3) in M1 to M5. Values of CPI were similar among surveys (above 3), which indicates a biogenic source from terrestrial plant waxes (NRC, 1985). However, values below 1 (petroleum contribution) were found in M2b, which is also affected by the discharges of effluents from an oil refinery (Moreira et al., 2017). High levels of UCM were also corroborating the evidence of oil contamination inputs (Frysinger et al., 2003). The isoprenoid hydrocarbons pristane and phytane are originated from isoprenoidyl products in petroleum (Volkman et al., 1992) and uncontaminated samples

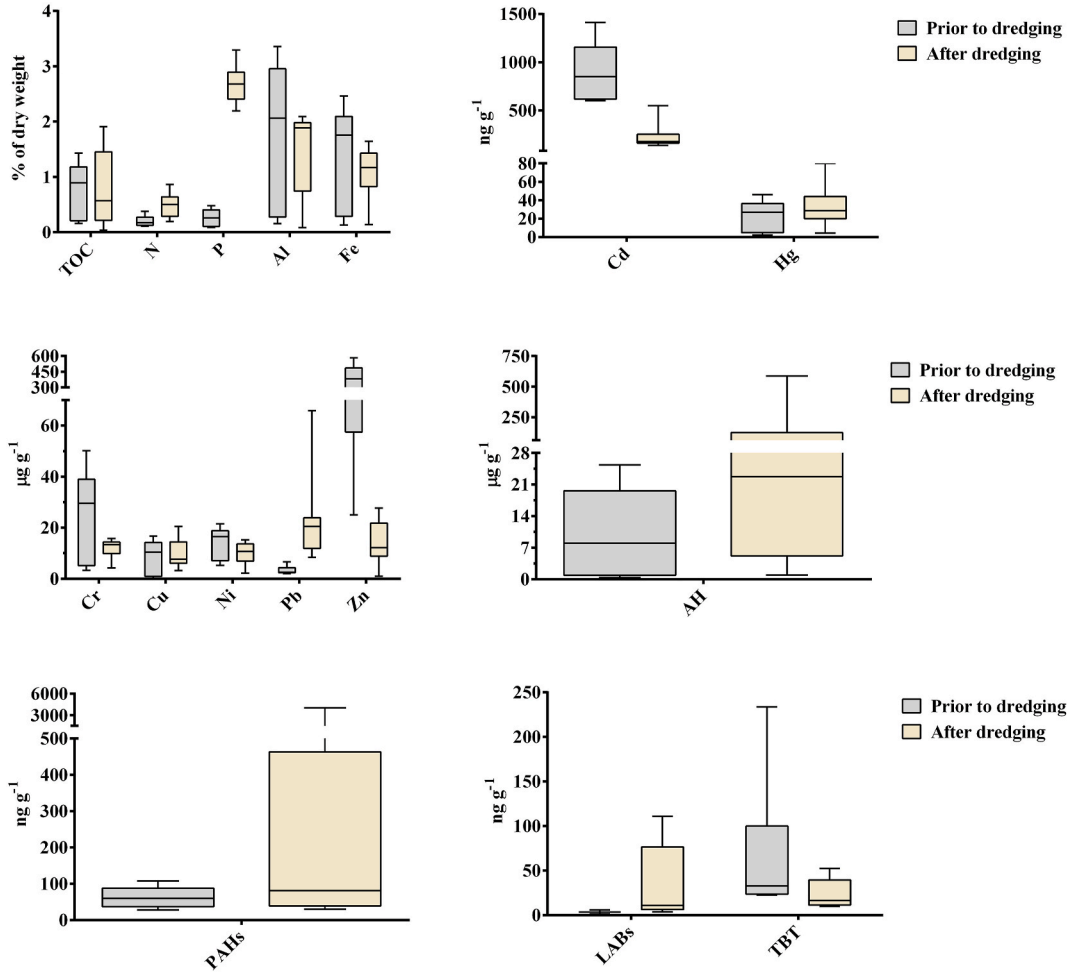

Fig. 2. Results of sediment chemistry observed in Mucuripe harbor prior to dredging (Survey 1), and after dredging activities (Survey 3). Values expressed as box-and-whisker plots of 95% confidence intervals of concentrations.

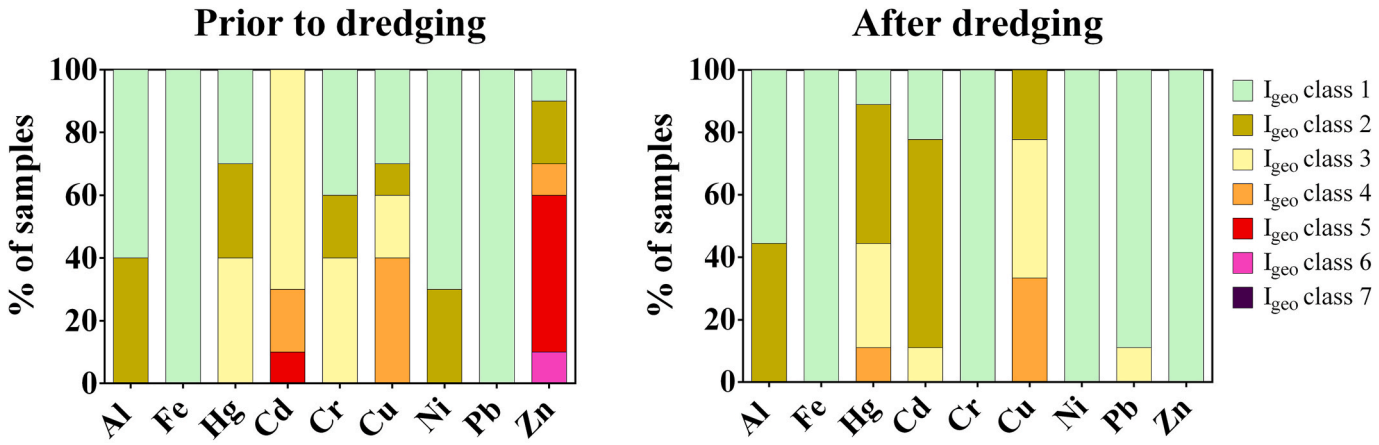

Fig. 3. Results of geoaccumulation index (I_{geo}) calculated for selected metals measured in sediments of Mucuripe harbor prior to dredging (Survey 1), and after dredging activities (Survey 3).

exhibit pristane/phytane between 3 and 5. Values reported prior and after dredging ranged from 1 to 1.5, and 0.8 to 1.57 respectively, indicating inputs of degraded petroleum (<1) and less degraded or fresh petroleum (>1) (Harji et al., 2008).

Distribution of PAHs was similar to AHs, with increased levels observed in survey 3 compared to survey 1, especially near commercial docks (M1b and M2b). The proportion of LMW and HMW PAHs were similar in both surveys, despite the change in total concentrations. A study on PAH concentrations during a dredging monitoring conducted in the Oil Port of Genoa-Multedo (Italy) revealed that 6 LMW-PAHs (2–3 rings) were predominant in water, while 6 HMW-PAHs (5–6 rings) were abundant in sediments, indicating a combined effect of dredging and physical-chemical properties of compounds on the distribution of PAHs (Vagge et al., 2018). For Mucuripe samples, the ratios ∑15 LMW/∑18

Table 3
Component scores based on PCA results matching particle size, chemical contamination and toxicity of samples collected in harbor areas of Ceará state (Surveys 1 to 3).

Sample	PC 1	PC 2	PC 3
Survey1 (prior to dredging)			
M1	−2.66	−0.11	0.37
M2	−2.65	−0.08	0.37
M3	−2.60	−0.16	0.36
M4	−2.70	−0.04	0.39
M5	−2.85	−0.01	0.34
M6	−2.76	−0.04	0.38
M7	−2.77	−0.05	0.37
M8	−3.01	0.02	0.35
M9	−2.70	0.04	0.19
M10	−1.24	−0.65	0.34
P1	5.15	−4.67	1.98
P2	4.55	−3.95	1.60
P3	4.53	−4.07	1.67
P4	2.68	−2.05	−0.29
P5	1.41	−1.56	0.10
Survey 2 (during intense dredging)			
Of	−2.65	0.36	0.25
Icapuí	−2.97	0.48	0.21
MD1	−2.71	0.39	0.26
MD2	−2.93	0.44	0.24
MD3	−3.00	0.53	0.23
MD1′	−2.70	0.43	0.29
MD2′	−3.05	0.55	0.22
MD3′	−3.02	0.55	0.22
Survey 3 (after dredging)			
M1b	5.87	2.47	−0.38
M2b	7.25	7.36	4.08
M3b	2.99	0.06	−1.99
M4b	4.14	0.47	−2.30
M5b	4.00	0.94	−2.02
M6b	2.67	0.48	−2.60
M7b	2.53	0.33	−1.64
M8b	1.56	0.88	−2.06
M9b	−0.36	0.65	−1.53

HMW were below 1 (Tables 6 and 7 supplementary material), indicating pyrogenic sources from the incomplete combustion of fossil fuels (Zhang et al., 2008). The cross plot of IP/(IP + BghiP) versus BaA/(BaA + Chry) ratio separated samples from survey 1, which presented a contribution of oil combustion, from survey 3 samples, characterized by compounds from mixed sources and biomass combustion (Fig. 4). These results confirm the effects of multiple sources of PAHs on the bay, such as the traffic of ships, combustion of fuel from other vehicles, combined with industrial activities, atmospheric inputs of urban dust, and runoff (Liu et al., 2005; Zhang et al., 2008). As for biomass signature found after dredging, it can be related to deposited materials in past conditions and remobilized by dredging as demonstrated by the results of sediment resuspension experiments conducted by Guigue et al. (2017).

Relevant concentrations of LABs were detected only in samples collected near commercial docks, with higher values reported in survey 3 compared to survey 1 (Tables 8 and 9 of supplementary material). Main compounds included LABs with 11–13 carbons in their compositions, in agreement with commercial detergent formulations (Raymundo and Preston, 1992), indicating inputs from domestic and industrial sewage.

Levels of TBT reduced in survey 3 (9.8–52 ng Sn g^{-1}) compared to survey 1 (22.6–233.8 ng Sn g^{-1}), confirming that the bay area is affected by antifouling compounds and their levels were also influenced by the removal of the most recent layer of contaminated sediments. TBT has been used as a biocide in antifouling paints since 1960′s (Almeida et al., 2007) and its environmental concentrations have reduced in coastal areas worldwide as a result of international restrictions (Bolam et al., 2014; Castro et al., 2018). Despite the efforts for minimizing its use in large commercial ships, TBT contamination remains relevant in coastal areas influenced by the presence of marinas, yards and docks for small vessels (Castro et al., 2012; Abreu et al., 2020), as in the case of the Mucuripe bay.

3.1.3. Sediment toxicity

Sediment toxicity results were also consistent among surveys, with toxicity reported in samples with higher levels of contamination. Samples collected after dredging operations (survey 3) exhibited significant mortality rates of amphipods in M1b to M6b (acute toxicity), and abnormal larvae development in all sites except M9b (chronic toxicity) (Table 1). For survey 1, prior dredging operations, the toxicity was also associated with changes in benthic macrofaunal communities' composition, highlighting the ecological relevance of such contamination (Moreira et al., 2017). During the intense dredging, sediments also induced alterations to other organisms such as the copepod *Tisbe biminiensis* in WS toxicity tests (reduction in fecundity), the mysid *Mysidopsis juniae* (lethality) and abnormal larvae development of the sea urchin *L. variegatus,* both exposed to the sediment-water interface (ISA) experiments (Moreira et al., 2019b). Despite the reduction in the concentrations of some chemicals caused by the dredging (e.g. Cd, Cr, Ni, Zn, and TBT), the post dredging condition caused toxicity in samples of survey 3 for WS and ELU. The biological effects of contamination vary according to the distribution of benthic species within layers of the sedimentary column, their interaction with particles and porewater (e.g., burrowing, tube building), and feeding behavior (e.g. filter-feeding, deposit-feeding) determining the main forms of exposure (Haukås et al., 2010; Coleman et al., 2014).

During sediment resuspension, relevant alterations in physical-chemical parameters occur, in particular, the redox potential. In this case, contaminants that were initially immobilized in the form of sulphide complexes and organic matter (Roberts, 2012), can be oxidized and became bioavailable. For metals, the resuspension of the anoxic layer causes the oxidation of sulfides and their remobilization to the dissolved phase, in rates that are influenced by the grain size, concentrations of acid volatile sulfides, and organic matter (Eggleton and Thomas, 2004; Cantwell et al., 2008). For organic compounds, desorption and transfer to the dissolved phase are also regulated by solubility properties and changes in environmental conditions such as pH (Goossens and Zwolsman, 1996).

Following the formation of the overflow plume, contaminants bound to particles can also become available and cause toxicity (Schipper et al., 2010; Roberts, 2012; Jones et al., 2016). During dredging activities in the Mucuripe bay, oysters and clams that were caged within the harbor zone and exposed to the overflow plume exhibited increased levels of metals (Cu, Ni, Pb, and Zn) and hydrocarbons (LABs and PAHs) in their tissues. Sublethal effects were also observed in gills and digestive glands (oxidative stress and DNA damage), indicating the potential risks produced by the overflow (Moreira et al., 2019c). Such findings were confirmed by the acute toxicity to the mysid *Mysidopsis juniae* (LC50 96h

Table 4
Site-specific sediment quality values (SQVs) proposed for coastal sediments of Ceará state based on the analysis of Mucuripe Bay and Pecém Harbor samples. Level 1 not determined (ND) for Cd, AHs, LABs, and TBT.

Threshold	Hg (ng g^{-1})	Cd (μg g^{-1})	Cr (μg g^{-1})	Cu (μg g^{-1})	Ni (μg g^{-1})	Pb (μg g^{-1})	Zn (μg g^{-1})	AHs (μg g^{-1})	PAHs (ng g^{-1})	LABs (ng g^{-1})	TBT (ng Sn g^{-1})
Level 1	42.11	ND	44.51	15.65	20.21	23.97	542.97	ND	925.66	ND	ND
Level 2	67.98	1.08	59.00	19.36	24.13	47.27	602.97	445.53	1957.02	83.47	189.60

Table 5
Compilation of sediment quality guidelines (SQGs) and sediment quality values (SQVs) adopted in different countries.

Location	Threshold	Cd (μg g^{-1})	Cr (μg g^{-1})	Cu (μg g^{-1})	Hg (μg g^{-1})	Ni (μg g^{-1})	Pb (μg g^{-1})	Zn (μg g^{-1})	ΣPAHs (ng g^{-1})	TBT (ng Sn g^{-1})	Reference
SQGs											
Australia	Level 1	1.5	80	65	0.15	21	50	200	4000[a]	5	EA (2002)
	Level 2	10	370	270	1	52	220	410	45000	70	
Brazil	Level 1	1.2	81	34	0.3	20.9	46.7	150	4000[a]	100[d]	Brasil (2012)
	Level 2	7.2	370	270	1	51.6	218	410	ND	1000[d]	
Canada	Level 1	0.67	52	19	0.13	15.9	30	120	1684[b]	ND	(EC, 2000; Macdonald
	Level 2	4.2	160	110	0.7	42.8	110	270	16770	ND	et al., 1996)
USA	Level 1	1.2	81	34	0.15	20.9	46.7	150	4022[a]	ND	Long et al. (1995)
	Level 2	9.6	370	270	0.71	51.6	218	410	44792	ND	
UK	Level 1	0.4	40	40	0.3	20	50	130	100	100[d]	OSPAR (2008)
	Level 2	5	400	400	3	200	500	800	ND	5000[d]	
SQVs											
EUA	Level 1	ND	110	68	37	88	49	156	ND	ND	DelValls and Chapman
	Level 2	ND	134	98	57	94	115	225	ND	ND	(1998)
Cádiz	Level 1	0.51	101.2	209	0.46	ND	260	513	ND	ND	DelValls and Chapman
	Level 2	0.96	283.9	979	0.57	ND	270	1310	ND	ND	(1998)
Cádiz	Level 1	0.65	ND	20.8	ND	8.9	21.6	138	97	ND	Choueri et al. (2009)
	Level 2	1.2	ND	169	ND	42.3	76	360	100	ND	
Santos	Level 1	ND	ND	ND	0.08	3.9	10.3	37.9	163	ND	Choueri et al. (2009)
	Level 2	0.75	65.8	69	0.32	21.2	22.1	110.4	950	ND	
Paranaguá	Level 1	ND	27.8	6.5	ND	10.98	17.6	26.9	20	ND	Choueri et al. (2009)
	Level 2	ND	48.8	6.5	ND	19.1	17.6	41.3	30	ND	
Ceará state	Level 1	ND	44.5	15.6	[c]0.042	20.2	24.0	543.0	925.7	ND	This Study
	Level 2	1.1	59.0	19.4	[c]0.068	24.1	47.3	603.0	1957.0	189.6	

ND = Not determined.
[a] = Σ16 PAHs: acenaphthene, acenaphthylene, anthracene, fluorene, indeno[1,2,3-c,d]pyrene, 2-methyl naphthalene, naphthalene, phenanthrene, benzo[a] anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, chrysene, dibenzo[a,h]anthracene, fluoranthene, and pyrene.
[b] = Σ19 PAHs: anthracene, benzo[a]anthracene, benzo[ghi]perylene, benzo[a]pyrene, chrysene, fluoranthene, indeno[1,2,3-cd]pyrene, pyrene, and phenanthrene.
[c] = concentrations are referred to fine particle fractions (<63 μm).
[d] = concentrations expressed as ng g^{-1}.

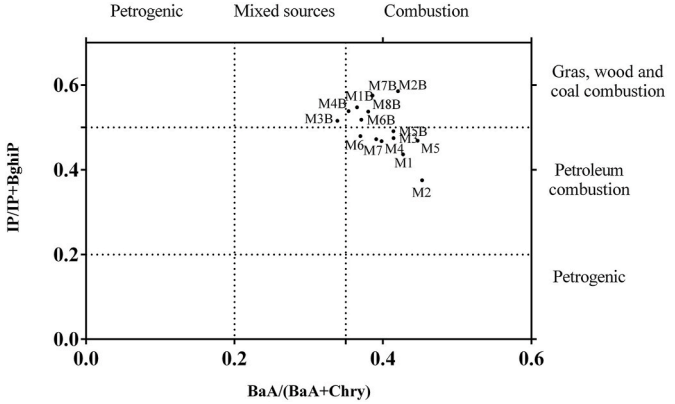

Fig. 4. Cross plots ratios of IP/(IP + BghiP) versus BaA/(BaA + Chry) for the sediments collected in Mucuripe harbor prior to dredging (M1 to M10), and after dredging activities (M1b to M9b). BaA: benzo[a]anthracene; Chry: chrysene; IP: Indeno[1,2,3-cd]pyrene; BghiP: Benzo[ghi]perylene.

of 66%) and chronic effects on *L. variegatus* larvae development (EC50 24h of 58%), determined in samples obtained directly in the dredger cistern (Moreira et al., 2019b). In survey 3 (post dredging), the increasing contamination (P, Cu, Ni, AHs and PAHs) can also be related to the deposition of the suspended particulate matter, as a result of the recontamination.

3.2. Site-specific sediment quality values (SQVs) for harbor areas of Ceará State

Results of PCA based on the results of all surveys including data of Pecém harbor are presented in Tables 2 and 3. PC1 computed for 60.76% of the variances and positive coefficients correlated % of fines, TOC, all the contaminants, and sediment toxicity, confirming the role of sediments as a sink of contaminants which can be associated with the toxicity observed. PC2 accounted for 23.76% of the variance and negative correlations were found for Cr, Zn, and TBT, while positive correlations were reported for Pb, AHs, PAHs, and LABs, indicating that these contaminants originated from different sources. PC3 accounted for 9.7% and only positive correlation for PAHs was found. The bidimensional ordination of PC1 and PC2 separated samples of surveys 1 and 2 from those of survey 3 and Pecém harbor area (Fig. 1 of supplementary material). Site-specific SQVs produced for metals, hydrocarbons and TBT are presented in Table 4. Both levels 1 and 2 were reported, except for Cd, AHs, LABs, and TBT, which presented only threshold level 2.

The predictive performance of SQVs in comparison to SQGs of resolution #454/12 was assessed through SQGQs. It is possible to observe that values adopted in Brazil fail to predict toxicity, as demonstrated by linear regressions of SQGQs versus acute toxicity of WS samples. In this case, toxic samples assigned as minimal (i.e. MD1, M3b to M6b) and moderate (i.e. MD1, M1b, M2b, P4, and P5) by the SQGs, changed to moderate (i.e. M3b and M6b) to strong contamination (i.e. MD1, M1 to M7, P1 to P4, M1b and M2b) by using SQVs (Fig. 5 A and B; C and D). Our findings corroborate with previous studies that reported such limitations of SQGs to predict toxicity (Choueri et al., 2009; Buruaem et al., 2012; Moreira et al., 2017).

A compilation of SQGs and SQVs adopted in different countries is given in Table 5. In general, SQGs adopted in Australia (EA, 2002), Brazil (Brasil, 2012), North America (Long et al., 1995; EC, 2008; Macdonald et al., 1996) and UK (OSPAR, 2008) are higher than SQVs proposed for San Francisco Bay and Bay of Cadiz (DelValls and Chapman, 1998) and estuarine systems of Santos and Paranaguá (Choueri et al., 2009). Such differences have been reported by other authors (Burton, 2002; Birch, 2018) and they were due to the respective methods of development.

SQGs were developed by empirical approaches based on total sediment concentrations, without considering changes in bioavailability such as equilibrium partitioning (EqP), which assumes that contaminant

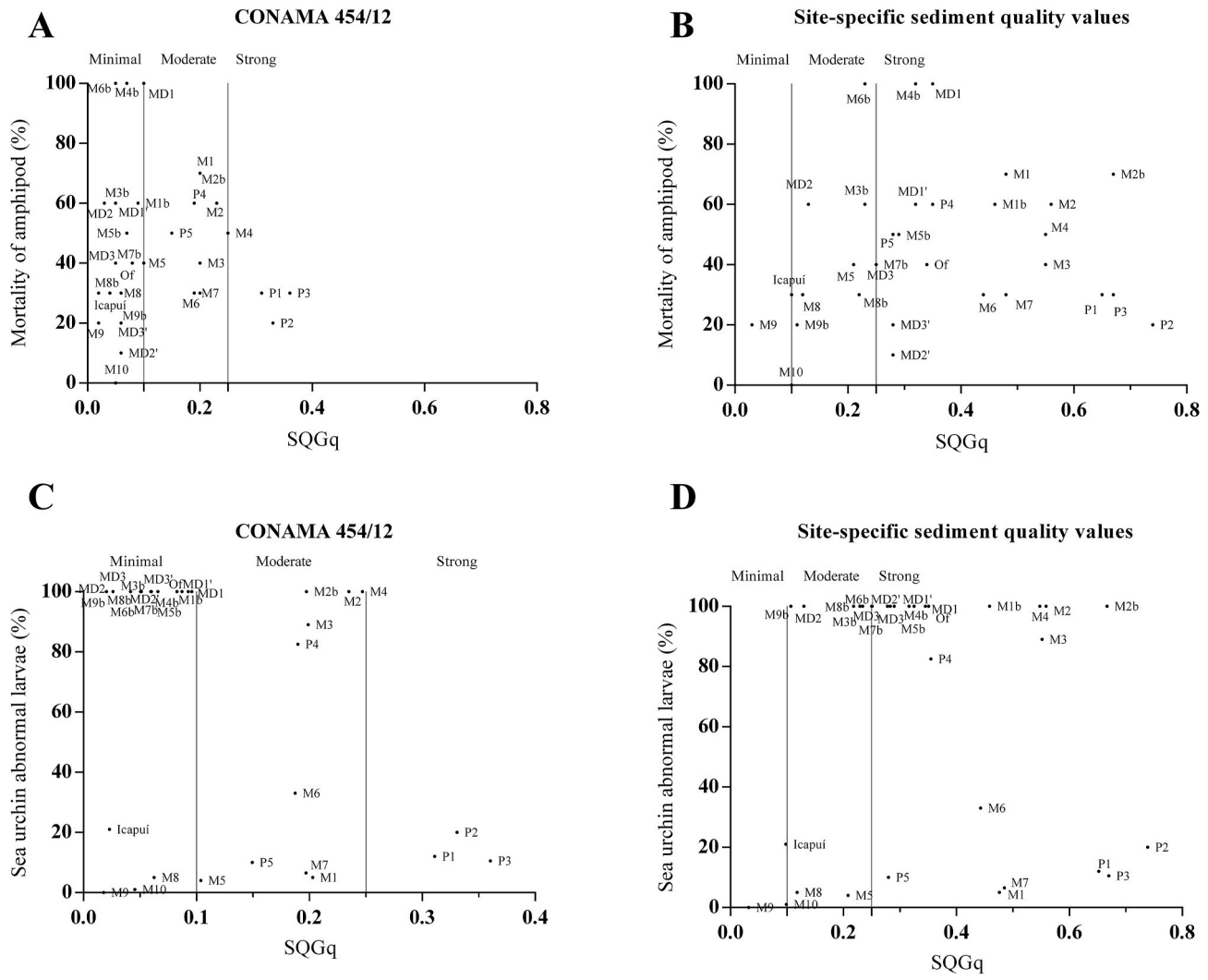

Fig. 5. Predictive performance of sediment quality guidelines of the resolution 454/12 and site-specific values (SQVs) assessed by means of sediment quality guideline quotients (SQGQs) in relation to whole-sediment toxicity of the amphipod *T. viscana* (A and B) and chronic toxicity of elutriates on sea urchin larvae *L. variegatus* (C and D).

exposure is driven by its pore water concentration or interactions with acid volatile sulfides (Burton, 2002). Birch (2018) revised 19 approaches used to develop EQS for sediment and pointed out the advantages of these values as a screening tool due to its broad application to a wide range of contaminants and sediment types. However, they need to be calibrated for site-specific assessments since some of these values lack toxicity data. Thus, SQGs are reliable and predictive if they can correctly predict toxicity and non-toxicity (Birch, 2018). In our study, site-specific SQVs were considered effective to predict effects. The SQGQ method indicated that toxic effects occur at strong contamination levels, and SQVs predicted 60.9% of WS toxicity, while SQGs adopted by the resolution 454/12 predicted only 4.3% (Fig. 5 A, B). For ELU, SQVs predicted correctly 77.3% of samples, contrasting with 4.5% of SQGs (Fig. 5 C, D). However, we pointed out that the effectiveness of EQS is also based on their ability to perform the similar predictions on an independent set of samples (Long and MacDonald, 1998).

4. Conclusions

In summary, we observed dredging-related changes in the sediment quality of harbor areas in Mucuripe bay. The concentration of metals (especially Cd, Cr, Cu, and Zn) decreased after the operations and the results were corroborated by the I_{geo} index, which assessed the enrichment of metals in relation to a baseline. Concentrations of TBT were also reduced, but results of nutrients and hydrocarbons revealed an increase in P, PAHs, and AHs as a result of remobilization. Hydrocarbons were originated mostly from pyrogenic processes, with contributions of mixed sources. These findings indicate the relevance of specific inputs from urban runoff, wastewater discharges, oil terminal effluents, harbor activities, and antifouling biocides on sediment contamination. Site-specific SQVs for the coastal zone of Ceará state, which is a typical semi-arid environment, were developed based on results from Mucuripe and Pecém harbors. The threshold values were more effective to predict toxicity compared to guidelines adopted in Brazil and other countries. In this sense, we recommend further investigations on the predictive ability of SQVs proposed for Ceará state in areas influenced not only by harbor activities but also other stressors.

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Credit author statement

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envres.2020.110525.

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