Science of the Total Environment 905 (2023) 167704

Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Brominated flame retardants and natural organobrominated compounds in a vulnerable delphinid species along the Brazilian coast

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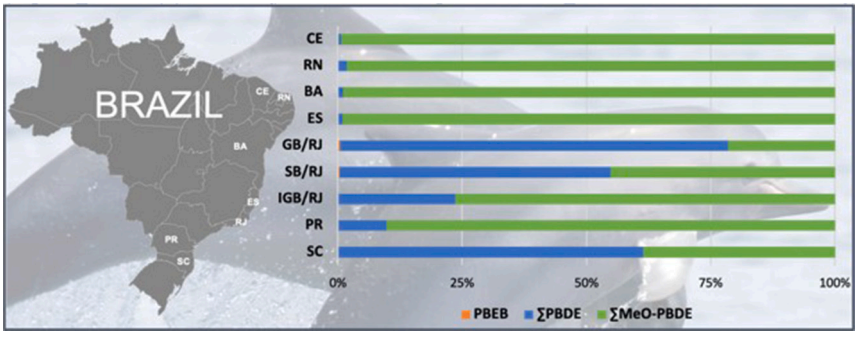
HIGHLIGHTS

- BFRs and MeO-BDEs were detected in Guiana dolphins along the entire Brazilian coast.
- Emerging BFRs were detected in 16 % of samples in SE Brazil, a chemical pollution hotspot.
- Concentrations of organobrominated compounds varied significantly among sampling sites.
- Organobrominated compounds can be used to differentiate ecological populations.
- Conservation plans for Guiana dolphins should consider specific demographic threats.

GRAPHICAL ABSTRACT



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https://doi.org/10.1016/j.scitotenv.2023.167704
Received 9 February 2023; Received in revised form 26 September 2023; Accepted 7 October 2023
Available online 10 October 2023

ARTICLE INFO

Editor: Susanne Brander

Keywords:
Sotalia guianensis
Polybrominated diphenyl ethers
Marine mammals
Methoxylated polybrominated diphenyl ethers
Cetaceans

ABSTRACT

Guiana dolphins, *Sotalia guianensis*, are vulnerable to extinction along their distribution on the Brazilian coast and assessing chemical pollution is of utmost importance for their conservation. For this study, 51 carcasses of Guiana dolphins were sampled across the Brazilian coast to investigate legacy and emerging brominated flame retardants (BFRs) as well as the naturally-produced MeO-BDEs. PBDEs and MeO-BDEs were detected in all samples analyzed, whereas emerging BFRs were detected in 16 % of the samples, all in Rio de Janeiro state. PBDE concentrations varied between 2.24 and 799 ng.g^{-1} lipid weight (lw), emerging BFRs between 0.12 and 1.51 ng.g^{-1} lw and MeO-BDEs between 3.82 and 10,247 ng.g^{-1} lw. Concentrations of legacy and emerging BFRs and natural compounds varied considerably according to the sampling site and reflected both the local anthropogenic impact of the region and the diversity/mass of biosynthesizers. The PBDE concentrations are lower than what was found for delphinids in the Northern Hemisphere around the same sampling period and most sampling sites presented mean concentrations lower than the limits for endocrine disruption known to date for marine mammals of 460 ng.g^{-1} lw, except for sampled from Santa Catarina state, in Southern Brazil. Conversely, MeO-BDE concentrations are higher than those of the Northern Hemisphere, particularly close to the Abrolhos Bans and Royal Charlotte formation, that are hotspots for biodiversity. Despite the elevated concentrations reported for this group, there is not much information regarding the effects of such elevated concentrations for these marine mammals. The distinct patterns observed along the Brazilian coast show that organobrominated compounds can be used to identify the ecological segregation of delphinids and that conservation actions should be planned considering the local threats.

1. Introduction

The expansion of human populations triggers the development of new substances and compounds to support global growth (UNEP, 2019). Among these new compounds, brominated flame retardants (BFRs) were extensively applied to limit fire propagation in several matrices. The main anthropogenic BFRs, the polybrominated diphenyl ethers (PBDEs), is present in foam, plastic, textile, and electronic boards (Eljarrat and Barceló, 2011). However, their wide application and physical-chemical properties led to the accumulation of PBDEs in several abiotic compartments as well as their bioaccumulation in biotic samples, raising concern regarding their toxic effects (de Wit, 2002). Currently, BFRs have been detected in the most diverse ecosystems worldwide (Barón et al., 2015; Covaci et al., 2008; Kalantzi et al., 2009).

The chemical pollution by BFRs in the coastal marine environment is highlighted when the transference of pollutants throughout the food chain is considered (de Lacerda and Malm, 2008; Losada et al., 2009). In these cases, elevated concentrations of such compounds can be found in apex predators because of biomagnification (Losada et al., 2009; Weijs et al., 2009), inducing not only individual-level effects but also population-level consequences. Furthermore, some studies reported the presence of organobrominated compounds from natural sources (Covaci et al., 2008; Vetter et al., 2001, 2002), being the most usual methoxylated BDEs (MeO-BDEs). Their synthesis has been linked to sponges, algae, and associated organisms such as cyanobacteria (Malmvärn et al., 2005; Vetter et al., 2002), and elevated concentrations of these compounds, comparable to BFR concentrations, were already reported in marine mammals (Alonso et al., 2014).

To monitor micropollutants and biosynthesized compounds, sentinel species can be used, and marine mammals are good sentinels of the aquatic environment (Bossart, 2011). These species are alert to trends and impacts of these compounds in the ecosystem and their effects on human health, providing relevant insights regarding environmental alterations in spatial, temporal, and trophic scales (Tabor and Aguirre, 2004). Furthermore, their high trophic level, longevity, and phylogenetic proximity to humans allow the integration of the contamination profile in the medium and long-term due to chronic exposure via diet and, hence, to observe the long-lasting impact at a population level (Bossart, 2011).

The Guiana dolphin (*Sotalia guianensis*) is a small delphinid endemic to the Western Atlantic Ocean, recorded from Honduras to Santa Catarina, in Brazil (Flores et al., 2018; Jefferson et al., 2015). Field surveys show that the species populations present distinct patterns of residence and site fidelity (Azevedo et al., 2004). These odontocetes can be opportunistic foragers, preying on organisms from different habits and trophic levels (Bisi et al., 2013; di Beneditto and Siciliano, 2007). Young teleost fish species are the main prey in Guiana dolphins' diet, including cephalopods and crustaceans (di Beneditto and Siciliano, 2007). Their feeding habits are directly associated with the patterns and levels of accumulation of lipophilic compounds, e.g., the organobrominated compounds, because it is their key exposure pathway.

The species is classified globally as "Near Threatened" (Secchi et al., 2018) and as "Vulnerable" by the Brazilian government. The coastal habits of Guiana dolphins allow direct and indirect interactions with human populations, posing a threat to their conservation. Incidental captures in fishing gears reported along their entire distribution, diseases, ship traffic, sound pollution, and chemical pollution by trace-elements, organic matter, and persistent organic pollutants (POPs) may be driving the decline in Guiana dolphin populations in areas highly impacted by anthropogenic activities (Azevedo et al., 2017; Meirelles et al., 2022).

The detection of BFRs in cetaceans from the Northern Hemisphere, where these compounds were originally produced, has been described for several species. On the other hand, these reports are still scarce in the Southern Hemisphere (Alonso et al., 2012), where BFR formulations were not produced, but were imported and applied to a wide variety of products. Thus, the present study aimed to evaluate the bioaccumulation of organobrominated compounds from anthropogenic sources including the legacy PBDEs as well as the emerging BFRs (pentabromoethylbenzene (PBEB), hexabromobenzene (HBB), and decabromodiphenylethane (DBDPE)), and natural sources (MeO-BDEs) in Guiana dolphins along the Brazilian coast.

2. Material & methods

2.1. Study area

The present study was performed along the Brazilian distribution of the Guiana dolphin, in the Southwestern Atlantic Ocean (SWAO). Sampling sites included three coastal regions of Brazil: the northeastern (NE), the southeastern (SE), and the southern (S) regions. The Brazilian coast extends for 8500 km and its regions present different patterns of anthropogenic impact, oceanographic influences, and natural resources (Silveira et al., 2000; Vidal et al., 2020). Comparatively, there are fewer industrial poles in the NE than SE and S regions, and a higher influence of the greatest rhodolith beds in the world and the Abrolhos bank (Amado-Filho et al., 2012). The SE region comprises the largest urban and industrial poles in the country, and it is considered the most

anthropogenically impacted among the study sites. The S region is undergoing an industrial expansion and already hosts large and important industrial poles (CNI, 2017). The anthropogenic growth in a developing country allows these contaminants reach the marine ecosystem and elevated concentrations of POPs have been recurrently reported in local marine species (Lailson-Brito et al., 2012; de Oliveira-Ferreira et al., 2021; Santos-Neto et al., 2014; Yogui et al., 2010).

2.2. Sampling

To assess the concentrations and relative composition of organobrominated compounds in the subcutaneous adipose tissue of Guiana dolphins, 51 individuals stranded or incidentally captured were collected in seven states of three Brazilian regions. In the NE region, samples were collected in Ceará (CE; n = 4), Rio Grande do Norte (RN; n = 4), and Bahia (BA, n = 6); in the SE region, samples were obtained from Espírito Santo (ES; n = 4), and Rio de Janeiro (RJ; n = 22); and in the S region, samples were obtained from Paraná (PR; n = 5) and Santa Catarina (SC; n = 6). The samples collected in RJ were divided between three coastal bays (Guanabara Bay, Sepetiba Bay, and Ilha Grande Bay) since there is genetic and ecological evidence of distinct populations occupying those bays (Andrade et al., 2015; Bisi et al., 2013; Lailson-Brito et al., 2010). The sampling was performed between 2002 and 2010 (Supplementary Information, Table S1). Further information regarding the sampling of individuals can be assessed in Vidal et al. (2020).

Guiana dolphins' carcasses were measured, photographed, and necropsied according to specific guidelines (Geraci and Lounsbury, 2005). The subcutaneous adipose tissue was collected from the left side of the individuals, below the dorsal fin. Samples were wrapped in aluminum foil and frozen at −20 °C until analysis. Biological parameters, such as sex and total length were accessed during the necropsy (Supplementary Information, Table S1).

2.3. Chemical analyses

The chemical analyses were performed at the Environmental and Water Chemistry for Human Health laboratories, in the Department of Environmental Chemistry from IDAEA-CSIC, Barcelona, Spain. The analytical procedure was adapted from Eljarrat et al. (2004) and Guerra et al. (2010). The analytes included in the present study were tri-BDE-28 (2,4,4′-Tribromodiphenyl ether), tetra-BDE-47 (2,2′4,4′-Tetrabromodi phenyl ether), penta-BDE-99 (2,2′,4,4′,5-Pentabromodiphenyl ether), penta-BDE-100 (2,2′,4,4′,6-Pentabromodiphenyl ether), hexaBDE-153 (2,2′,4,4′,5,5′-Hexabromodiphenyl ether), hexaBDE-154 (2,2′,4,4′,5,6′-Hexabromodiphenyl ether), hepta-BDE-183 (2,2′,3,4,4′,5′,6-Heptabromodiphenyl ether), deca-BDE-209 (2,2′,3,3′,4,4′,5,5′,6,6′-Decabromodi phenyl ether), PBEB (Pentabromoethylbenzene), HBB (Hexabromo benzene), DBDPE (Decabromodiphenyl Ethane), 5-MeO-BDE-47 (2,2′,4,4′-Tetrabromo-5-methoxydiphenyl ether), 6-MeO-BDE-47 (2,2′,4,4′-Tetrabromo-6-methoxydiphenyl ether), 4-MeO-BDE-49 (2,2′,4′,5-Tetrabromo-4-methoxydiphenyl ether), 2-MeO-BDE-68 (2′,3,4′,5-Tetrabromo-2-methoxydiphenyl ether), 5-MeO-BDE-99 (2,2′,4,4′,5-Penta bromo-5′-methoxydiphenyl ether), 5-MeO-BDE-100 (2,2′,4,4′,6′-Pentabromo-5-methoxydiphenyl ether), 4-MeO-BDE-101 (2,2′,4,5,5′-Pentabromo-4′-methoxydiphenyl ether) and 4-MeO-BDE-103 (2,2′,4′,5,6′-Pentabromo-4-methoxydiphenyl ether).

Briefly, 1 g of the subcutaneous adipose tissue was ground and spiked with internal standards (BDE-181 and $^{13}C_{12}$-BDE-209), then added to the extraction cell with a cellulose filter and Hydromatrix. The tissue was extracted via the ASE system with an organic mixture of dichloromethane: n-hexane (1:1, v/v). The extraction cell was heated at 100 °C for 10 min at a pressure of 1500 psi. The lipidic content of each sample was measured gravimetrically after extraction, evaporating the organic solvents, and weighting the lipids in the analysis's vials. Samples were then treated with 20 mL of sulfuric acid, following active homogenization, and centrifugation at 3000 rpm until the complete separation between the acidic and organic phases. This step was repeated 2–3 times. To complete the sample purification, a solid-phase extraction with a semi-automatic Baker system, using neutral aluminum oxide cartridges was performed. Purified extracts were eluted in 20 mL of organic mixture dichloromethane: n-hexane (2:1, v/v). The final volume was reduced, and the purified extracts were resuspended in 30 μL of toluene.

Organobrominated compounds were detected in a gas chromatograph, coupled to a mass spectrometer detector in negative chemical ionization mode (GC-NCI-MS) connected to an automatic injector (Agilent Technologies models 6890, 5973, 5975C XL, and 7683B). A silica capillary column DB5-MS (Agilent Technologies, 30 m × 0,25 mm × 0,25 μm) was used for the chromatographic run. Ammonia was the reagent gas used at a 3.0 mL/min flow. Due to the thermal instability of decabrominated compounds (BDE-209 and DBDPE), the extracts were reinjected in a smaller DB5-MS column (Agilent Technologies, 15 m × 0,25 mm × 0,1 μm). Aliquots of 2 μL were injected in splitless mode at 275 °C. Ultra-pure helium (99,999 % pure) was used as carrier gas at a constant pressure of 10 psi. Interface and source temperatures were 250 °C.

Analyses were conducted by monitoring the $m/z = 79$ and 81 [Br^-] for all analytes, except for deca-BDE-209 and $^{13}C_{12}$-BDE-209, for which $m/z = 487$ and 489, and $m/z = 497$ and 498 were monitored, respectively. The identification of analytes was done in the ChemStation Data Analysis software and followed the criteria of not exceeding ±1 s of retention time observed in the calibration curve standard, and the ratio between monitored ions was <15 % of the expected value. The quantification of analytes was based on internal standards. BDE-181 was used for all PBDE and MeO-BDE congeners, and emerging BFRs, while $^{13}C_{12}$-BDE-209 was used for quantification of BDE-209 and DBDPE.

2.4. Quality assurance/quality control

To minimize the presence of interferents in the chemical analysis, highly pure reagents were used. Standard solutions were purchased from Cambridge Isotope Laboratories (Wisconsin, USA) and Wellington Laboratories Inc. (Ontario, Canada). Moreover, one analytical blank was added to every extraction batch. Only BDE-47 was detected in a few blanks at low levels, and the detected values were deducted from samples in the batch.

Calibration curves were constructed using the standard solutions, and the correlation coefficient for the curves was ≥0.995. To ensure the quality and reproducibility of the procedure, recoveries were calculated by spiked samples with standard solutions analyzed in replicates. Analytes' recovery ranged between 90 % and 120 %, being acceptable (Supplementary Information, Table S2). The method's limit of detection and quantification (LOD and LOQ, respectively) were calculated based on the signal-to-noise ratio. The LOD was calculated as three times the level of noise in chromatograms, while the LOQ was considered ten times de level of noise (Supplementary Information, Table S2). LODs ranged between 0.17 and 3.6 ng g^{-1} lipid weight (lw), whereas LOQs were between 0.58 and 12 ng g^{-1} lw.

2.5. Statistical analyses

The concentrations of organobrominated compounds were expressed in ng g^{-1} lw. Statistical treatment of the dataset was done in the STATISTICA 7.0® software. Descriptive stats were used to determine median, mean, minimum, maximum, and dispersion values.

A detrended correspondence analysis (DCA) was used to identify possible patterns of organobrominated compounds in the distinct localities. A PERMANOVA was chosen to test specific differences between sampling sites. Bray-Curtis dissimilarity was used as the basis for all data analysis, given the several values below LOD. When significant differences were detected, post-hoc pairs of 4999 permutations were tested.

3. Results & discussion

3.1. BFR profile

Table 1 summarizes the concentration levels of organobromine compounds obtained in Guiana dolphins (for individual sample results, see Supplementary Table S3). PBDEs were detected in all samples analyzed in the present study, while the emerging BFR PBEB was the only one detected among the emerging contaminants and present in 16 % of the samples (all of which were from RJ). PBDEs concentration in Guiana dolphins along the Brazilian coast varied between 2.24 and 799 ng.g^{-1} lw, and emerging BFRs between 0.12 and 1.5 ng.g^{-1} lw.

Among PBDEs, BDE-47 was the predominant congener in the contamination profile of Guiana dolphins from all nine localities (Fig. 1), representing between 48 % (GB/RJ) and 100 % (CE) of total PBDEs. The same pattern was described for other cetacean species in the Southwestern Atlantic Ocean (SWAO) (Alonso et al., 2012; Barbosa et al., 2018; Dorneles et al., 2010; Oliveira-Ferreira et al., 2022, 2023; Yogui et al., 2011a). On the other hand, BDE-183 was not detected in 98 % of the data set, only in individuals from GB/RJ and SB/RJ. BDE-209 was observed in individuals from GB/RJ, ES, BA, RN, and SC, but at a low frequency (Fig. 1).

The profile observed in Guiana dolphins is a portrait of the abundant use of PBDE formulations, particularly Penta-BDE, the ongoing debromination process these congeners are exposed to in the ecosystem, the distinct features of each environment, and the species metabolism of contaminants. Penta-, Octa-, and Deca-BDE formulations were the most commercialized worldwide (de Wit, 2002). BDE-47 was the main congener in Penta-BDE formulations (38–42 %) and it was indiscriminately applied for decades especially in furniture and foam (la Guardia et al., 2006). BDE-183 (42 %) and BDE-209 (92–97 %) were the main congeners in Octa- and Deca-BDE formulations, respectively, abundantly used in plastic and electronic devices (la Guardia et al., 2006). However, contrasting with the dominance of BDE-47, the latter congeners were detected at a very low frequency in the dataset, not reflecting their predominance in the commercialized formulations. BDE-183 can undergo debromination in the environment originating BDE-154, for example, which was detected in 11 % of samples from RJ. Also, BDE-209 is likely to be debrominated into smaller congeners (including BDE-47) due to photodegradation (Cheng et al., 2021; Shih and Wang, 2009). Despite present in approximately 80 % of all products manufactured globally, BDE-209 has been found in lower concentrations in environmental samples (Mcdonald, 2002). PBDEs highly brominated tend to present lower biomagnification factors than the less brominated congeners, which may affect their bioaccumulation in these apex predators (Losada et al., 2009). The biotransformation of BDE-99 into BDE-47 may also be a factor contributing to the predominance of this congener in Guiana dolphins from the SWAO (Stapleton et al., 2004, 2009). Nevertheless, when in the environment, these compounds may be degraded into metabolites with increased toxicity, bioaccumulation potential, and facilitated atmospheric transport (Ikonomou et al., 2002).

Local environmental conditions and distinct anthropogenic impact can also influence the PBDE congeners profile because of atmospheric and oceanographic features, the incidence of sunlight, organic matter input, and local trophic web structure, hence trophic transference of contaminants (Barón et al., 2015; Cheng et al., 2021; de Wit, 2002). Individuals from RJ, for example, showed a wider variety of PBDE congeners, and dolphins from the GB/RJ presented all 8 congeners analyzed in this study. The Guanabara Bay is one of the most degraded coastal bays in the Southern Hemisphere, highly eutrophic and with increased exposure to anthropogenic impact (Kjerfve et al., 1997; Soares-Gomes et al., 2016). The influence of these activities in the surroundings of the GB is likely to impact the observed profile.

Comparatively, PBDE concentrations were higher in cetaceans from the northern hemisphere around the same sampling period than in Guiana dolphins from the Brazilian coast (Fig. 4). Bottlenose dolphins (*Tursiops truncatus*) from the North Atlantic Ocean presented concentrations 10 to 20 times higher than the ones found in the present study, with mean of 1190 ng.g^{-1} lw concentration, and reaching until 6560 ng.g^{-1} lw depending on the sampling site (Kucklick et al., 2011; Yordy et al., 2010a, 2010b). In the North Sea, harbor porpoises (*Phocoena phocoena*) also presented concentrations 5 to 10 times higher than the Guiana dolphins from the South Atlantic Ocean, as mean concentrations in varied between 462 in neonates until 1194 ng.g^{-1} lw in adults (Weijs et al., 2010a). Except for Guiana dolphins from SC, the PBDEs are also up to 10 times higher in common dolphins (*Delphinus delphis*) – 199 ng.g^{-1} lw, bottlenose dolphins (*Tursiops truncatus*) – 1184 ng.g^{-1} lw, and pilot whales (*Globicephala melas*) – 240 ng.g^{-1} lw, found in southern European waters, in the Strait of Gibraltar (Barón et al., 2015). The production of these compounds was concentrated in Northern Hemisphere countries, which could influence the higher concentrations found in their sentinel species. Despite the differences in concentration levels between hemispheres, the contamination profile is very similar: BDE-47 is the main congener often detected in marine mammals; and the lower concentrations and detection frequency of congeners such as BDE-183 and BDE-209 (Law et al., 2010; Losada et al., 2009; Weijs et al., 2010a).

On the other hand, Guiana dolphins from São Paulo state, sampled between 1996 and 2003, presented PBDE levels in the same order of magnitude as the individuals herein analyzed (Yogui et al., 2011b). Dorneles et al. (2010) reported that hepatic concentrations of PBDE in Guiana dolphins from RJ sampled between 1994 and 2006 trended to increase and, while those concentrations were higher than the ones detected in the present study, it is important to highlight that the compounds were accessed in different matrices and different sampling periods (Dorneles et al., 2010). Recently, a temporal trend of decline was described for franciscana dolphins (*Pontoporia blainvillei*) between 2003 and 2019 associated with the prohibition of production and use of PBDEs in the Northern Hemisphere in 2009 (Oliveira-Ferreira et al.,

Table 1
ΣPBDE, ΣEmerging BFR and ΣMeO-BDE concentrations (ng.g^{-1} lw) in Guiana dolphins from the Brazilian coast: (CE) Ceará; (RN) Rio Grande do Norte; (BA) Bahia; (ES) Espírito Santo; (GB/RJ) Guanabara Bay, Rio de Janeiro; (SB/RJ) Sepetiba Bay, Rio de Janeiro; (IGB/RJ) Ilha Grande Bay, Rio de Janeiro; (PR) Paraná; and (SC) Santa Catarina.

	n	∑PBDEs			∑Emerging BFRs			∑MeO-BDEs		
		Mean ± SD	Median	Min - Max	Mean ± SD	Median	Min - Max	Mean ± SD	Median	Min - Max
CE	4	40.1 ± 58.2	15.5	4.93–129	<0.18	<0.18	<0.18	2277 ± 1695	2387	447.8–3884
RN	4	74.7 ± 51.2	68.0	21.6–141	<0.18	<0.18	<0.18	3473 ± 1403	3935	1470–4551
BA	6	63.3 ± 53.4	62.5	10.6–127	<0.18	<0.18	<0.18	6294 ± 3089	6306	1705–10,248
ES	4	15.6 ± 11.2	16.2	2.24–28.0	<0.18	<0.18	<0.18	2815 ± 3101	1766	368–7359
GB/RJ	9	170 ± 245	86.0	23.7–799	0.45 ± 0.17	0.49	<0.18–0.62	88.5 ± 144	23.7	3.82–443
SB/RJ	1	80.4 ± 60.5	56.4	16.5–197	0.60 ± 0.65	0.38	<0.18–1.51	88.3 ± 121	46.7	13.0–421
IGB/RJ	2	64.9 ± 55.3	64.9	25.8–104	<0.18	<0.18	<0.18	211 ± 177	211	85.5–336
PR	5	29.0 ± 24.0	26.1	3.59–60.7	<0.18	<0.18	<0.18	253 ± 127	242	128–400
SC	6	453 ± 246	509	148–713	<0.18	<0.18	<0.18	472 ± 311	320	190–910

(ΣPBDEs) Sum of PBDEs; **(ΣEmerging BFRs)** PBEB; **(ΣMeO-BDEs)** Sum of MeO-BDEs; **(SD)** Standard Deviation; **(Min)** Minimum; **(Max)** Maximum.

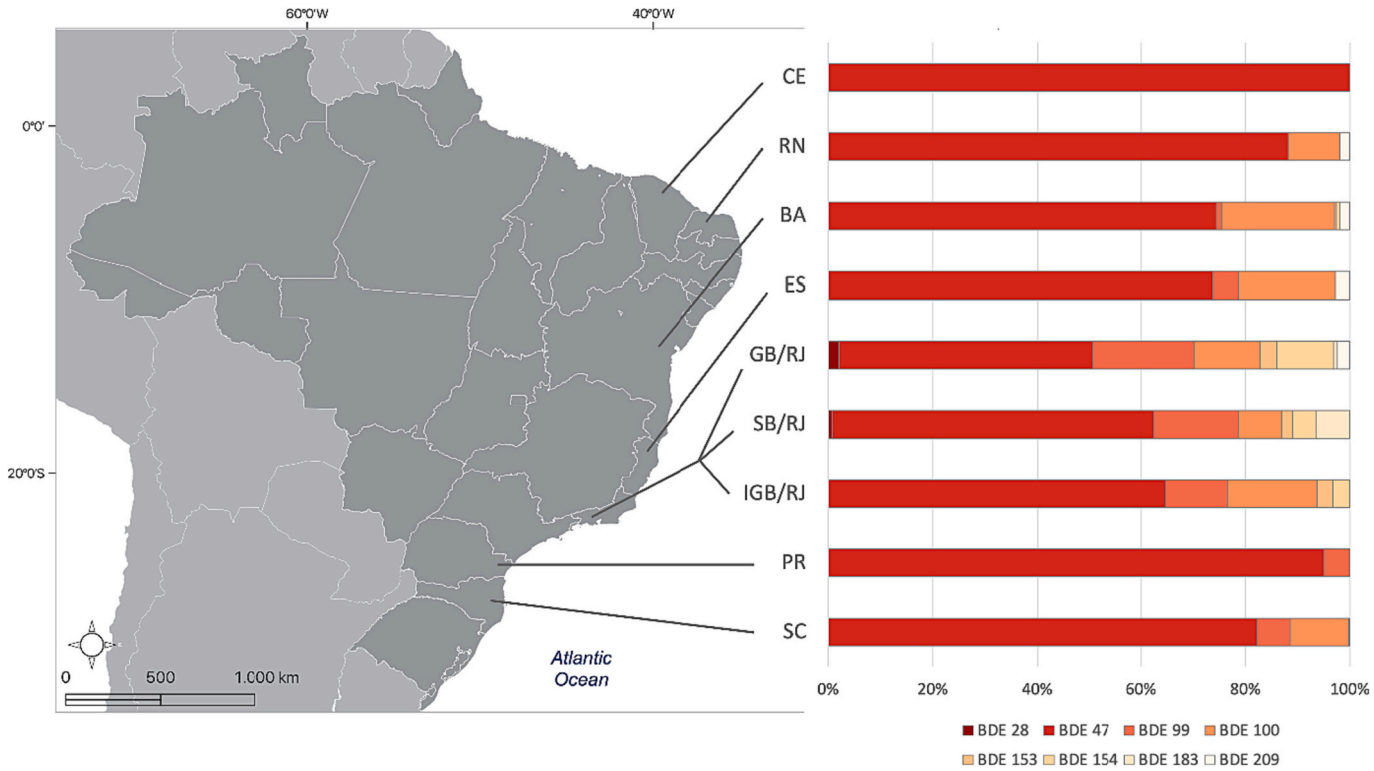

Fig. 1. PBDE congener profiles in Guiana dolphins from the Brazilian coast: (CE) Ceará; (RN) Rio Grande do Norte; (BA) Bahia; (ES) Espírito Santo; (GB/RJ) Guanabara Bay, Rio de Janeiro; (SB/RJ) Sepetiba Bay, Rio de Janeiro; (IGB/RJ) Ilha Grande Bay, Rio de Janeiro; (PR) Paraná; and (SC) Santa Catarina.

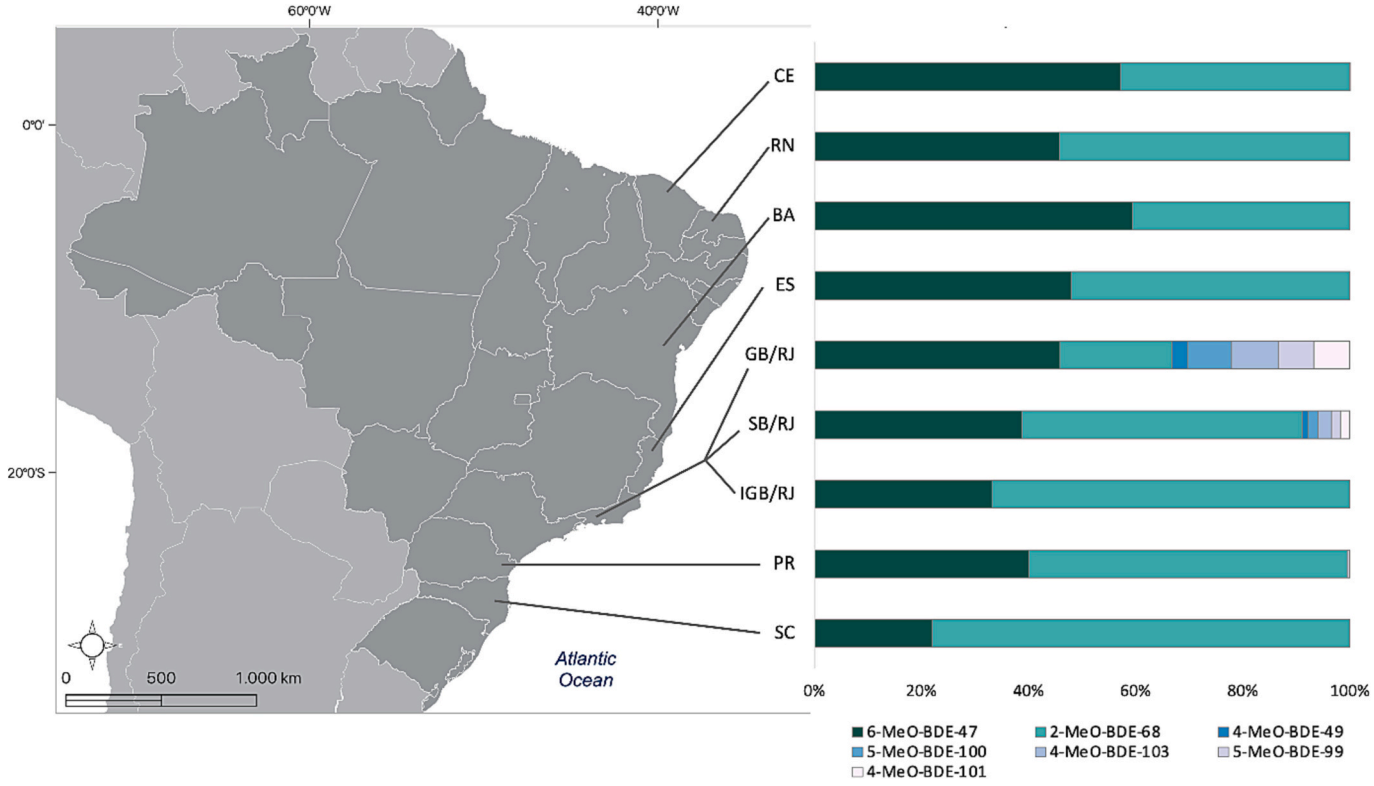

Fig. 2. MeO-BDE congener profiles in Guiana dolphins, *Sotalia guianensis*, from the following locations in the Brazilian coast: (CE) Ceará; (RN) Rio Grande do Norte; (BA) Bahia; (ES) Espírito Santo; (GB/RJ) Guanabara Bay, Rio de Janeiro; (SB/RJ) Sepetiba Bay, Rio de Janeiro; (IGB/RJ) Ilha Grande Bay, Rio de Janeiro; (PR) Paraná; and (SC) Santa Catarina.

2022). The restriction is also reflected in the decrease in muscle concentrations of PBDEs in striped dolphins (*Stenella coeruleoalba*) from the Mediterranean Sea (Aznar-Alemany et al., 2021) (Fig. 4).

Regarding the emerging BFR, PBEB was detected in 8 samples from RJ (GB/RJ and SB/RJ), in concentrations varying from 0.1 to 1.5 ng.g^{-1} lw (Supplementary Information, Table S3). PBEB has been recently reported in environmental samples. In cetaceans from the Brazilian coast, they were observed below the limit of quantification in franciscana dolphins (Alonso et al., 2012; Oliveira-Ferreira et al., 2022). Low concentrations of this compound were detected in polar bears (*Ursus maritimus*) from the Arctic (McKinney et al., 2011) and in other marine vertebrates like fishes and sharks (Houde et al., 2014; Strid et al., 2013). This emerging contaminant was used between 1970 and 1980 in the USA but given the demands for toxicological data regarding this BFR by the US Environmental Protection Agency (US-EPA), its production reduced in the following years (Gauthier et al., 2009). To the best of our knowledge, there are no records of PBEB being produced in Brazil and no information regarding its importation and/or use in the country. HBB and DBDPE were not detected in any sample of this dataset. Unlike their legacy BFR counterparts, the emerging PBEB, HBB, and DBDPE were not found to biomagnify in marine mammals' trophic webs from the northern hemisphere (Barón et al., 2015), and this could account for the low frequency at which they are detected in these cetaceans or undetected above the LOQ.

3.2. MeO-BDEs profile

MeO-BDEs were detected in all samples herein assessed. Concentrations in the blubber of Guiana dolphins ranged from 3.82 ng.g^{-1} lw in the GB/RJ and 10,247 ng.g^{-1} lw in BA. ng.g^{-1} lw (Table 1).

The profile of the biosynthesized MeO-BDEs in Guiana dolphins was dominated by 6-MeO-BDE-47 and 2′-MeO-BDE-68 (Fig. 2), but all 8 MeO-BDEs analyzed were detected in individuals from the Brazilian coast (Supplementary Information, Table S4). The origin of MeO-BDEs has been associated with primary producers like the red algae *Ceramium tenuicorne* and *Pollysiphona fucoides*, the brown algae *Pilayella littoralis*, the aquatic sponge *Ephydatia fluviatillis*, and the cyanobacteria *Aphanizomenon flosaquae* and *Nodularia sprumigena* (Bowden et al., 2000; Haraguchi et al., 2010; Malmvärn et al., 2005, 2008). While 2′-MeO-BDE-68 appears to be mainly synthesized by sponges and/or associated organisms, the production of 6-MeO-BDE-47 is mostly associated with algae and/or associated organisms (Malmvärn et al., 2005; Vetter et al., 2002).

The same bioaccumulation profile was reported for marine mammals and other marine vertebrates around the world (Rotander et al., 2012; Vetter et al., 2001, 2002). These natural compounds were found to biomagnify in trophic webs from both the Northern and Southern Hemisphere, resembling their anthropogenic analogs, the PBDEs (Barón et al., 2015; Losada et al., 2009). MeO-BDEs were present in elevated concentrations in Guiana dolphins from the Brazilian coast, especially in individuals from the NE region and ES state, areas influenced by the Abrolhos Bank. These concentrations were one order of magnitude lower than the mean detected in spinner dolphins (*Stenella longirostris*) and bottlenose dophins from Tanzania, 74,000 and 62,000 ng.g^{-1} lw, respectively (Mwevura et al., 2010). These are the highest blubber concentrations of MeO-BDEs in dolphins reported to date, in the Indian Ocean, a region characterized by its increased biodiversity (Mwevura et al., 2010). Conversely, Guiana dolphins from the most degraded SE and S regions of Brazil presented lower concentrations of MeO-BDEs, comparable with cetaceans from the Northern Hemisphere, such as the harbor porpoises from the North Sea – with mean 144 ng.g^{-1} lw (Weijs et al., 2010b), but also similar to concentrations found in delphinids from eastern South Africa, in the Indian Ocean, with mean 114 ng.g^{-1} lw (Aznar-Alemany et al., 2019) (Fig. 4).

3.3. Differences between localities

The highest median concentration of PBDEs in Guiana dolphins along the Brazilian coast was found in individuals collected in SC (509 ng.g^{-1} lw) and the lowest for CE and ES (16 ng.g^{-1} lw) (Table 1). SC, in southern Brazil, presents an increased influence on industrial activities, from the electroplating, textile, pharmaceutical, and metal-mechanics sectors. Furthermore, Babitonga Bay, where some individuals from the SC were sampled, receives de apport of four municipalities, including Joinville, which is the greatest urban center of the state, with approximately 412,000 habitants, but with only 10 % of domestic sewage treated (IBGE, 2011).

As for MeO-BDEs, the highest median concentration was reported for animals from BA (6305 ng.g^{-1} lw) in NE Brazil, and the lowest for GB/RJ (24 ng.g^{-1} lw) in SE Brazil. The Abrolhos bank, in the southern portion of BA, is considered the greatest and most biodiverse coral reef from the SWAO (Dutra et al., 2005). Furthermore, it holds the largest rhodolith bed in the world, with 21,000 km^2, representing a habitat for several vertebrate and invertebrate species and as a substrate for the fixation of innumerous algae (Amado-Filho et al., 2017; Moura et al., 2021). While the NE region and the ES state had an expressive contribution of MeO-BDEs, probably influenced by the increased local biodiversity - ES also suffers influence from the Abrolhos bank - the SE and S regions presented an elevated influence of anthropogenic compounds. As previously mentioned, Guanabara Bay is heavily impacted by anthropogenic activities, which may influence the low availability of natural compounds. Also, this coastal bay presents less complex trophic interactions when compared to the adjacent SB/RJ and IGB/RJ, which could be a reflection of local biodiversity loss (Bisi et al., 2013). Hence, it is likely that the biodiversity and abundance of primary producers biosynthesizing MeO-BDEs are compromised when compared to other localities of the Brazilian coast.

The highest concentrations of natural compounds worldwide seem to be associated with tropical regions in the Southern Hemisphere, due to their more intense production and/or availability, as well as to the distinct environmental degradation degree (Alonso et al., 2014; Dorneles et al., 2010). On a smaller scale, this pattern can also be observed along the Brazilian coast. The concentrations of MeO-BDEs in Guiana dolphins from RJ were similar to harbor porpoises (*Phocoena phocoena*) from a heavily impacted region of the Black Sea, at 47 ng.g^{-1} lw (Weijs et al., 2010a). On the other hand, concentrations found in ES were similar to more preserved environments and with increased biodiversity like in the melon-headed dolphin (*Peponocephala electra*) from Australia, of 2020 ng.g^{-1} lw (Melcher et al., 2005).

Looking even closer to the coastal bays of RJ, the pattern repeats itself. Lower MeO-BDE concentrations were detected in GB/RJ and SB/RJ, the most degraded bays, when compared to IGB/RJ, the most preserved and protected of the three, and with increased biodiversity.

The relative contribution of POPs has been repeatedly used to distinguish cetacean aggregations within a geographic range (Borrell et al., 2006; Bruhn U et al., 1999; Hobbs et al., 2001; Lailson-Brito et al., 2012; de Oliveira-Ferreira et al., 2021; Santos-Neto et al., 2014). The detrended correspondence analysis (DCA) pointed to a differentiation between Guiana dolphins from the nine localities considered in this study ($p = 0.0009$; Fig. 3) and PERMANOVA indicated a significant difference among all of them in canonical axis 1 ($p < 0.0001$) and 2 (p-0.0001) (Supplementary Information, Tables S5, and S6). 2′-MeO-BDE-68 and 6-MeO-BDE-47 were the compounds forcing the separations between groups in axis 1 positively, whereas BDE-154, BDE-153, 4-MeO-BDE-103, 4-MeO-BDE-101, BDE-99, 5-MeO-BDE-99, and BDE-100 – negatively. In canonical axis 2, no compound had an influence >0.2 to force the separation positively, and BDE-154, 5-MeO-BDE-99, and 4-MeO-BDE-101 forced the separation negatively (Fig. 3).

The naturally-produced 6-MeO-BDE-47 and 2′-MeO-BDE-68 were the main compounds separating the CE, RN, BA, and ES from the other localities, once their median concentrations were higher in the NE states

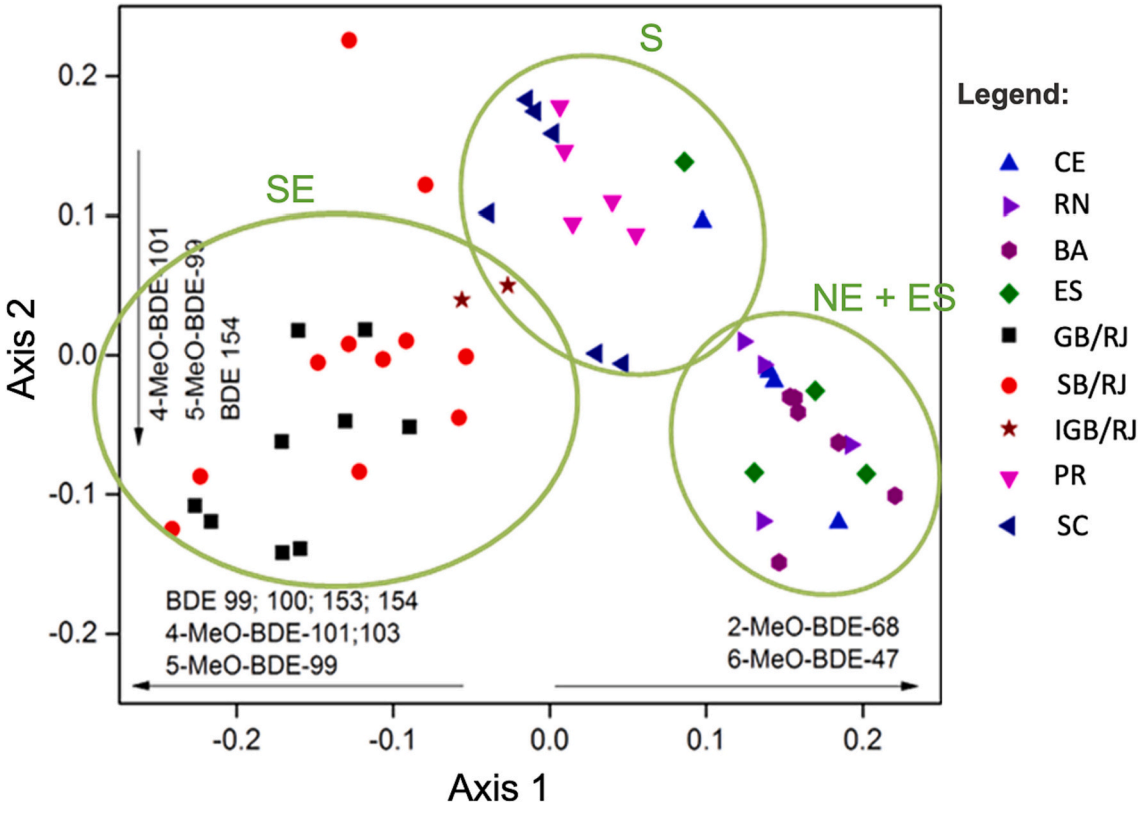

Fig. 3. Graphical representation of the detrended correspondence analysis (DCA) based on the concentrations of organobrominated compounds found in Guiana dolphins from the Brazilian coast: (CE) Ceará; (RN) Rio Grande do Norte; (BA) Bahia; (ES) Espírito Santo; (GB/RJ) Guanabara Bay, Rio de Janeiro; (SB/RJ) Sepetiba Bay, Rio de Janeiro; (IGB/RJ) Ilha Grande Bay, Rio de Janeiro; (PR) Paraná; and (SC) Santa Catarina.

plus ES. On the other hand, BDE-154, BDE-153, BDE-99, BDE-100, 4-MeO-BDE-103, 4-MeO-BDE-101, and 5-MeO-BDE-99 were important for the separation of GB/RJ, SB/RJ, and IGB/RJ. On axis 2, BDE-154, 5-MeO-BDE-99, and 4-MeO-BDE-101 forced the separation of PR and SC.

Hence, three main groups are observed in the graphical representation of the canonical variables (Fig. 3). The first includes the coastal bays of RJ (GB/RJ, SB/RJ, and IGB/RJ) in the SE region; the second comprehends the southern (S) Brazilian states (PR and SC); and NE Brazilian states (CE, RN, and BA) and ES forms the third group (NE + ES). PERMANOVA indicated differences in the organobrominated compounds profile of the three main groups in both axis 1 ($p < 0.0001$) and 2 ($p < 0.0001$).

The profile of BFRs and natural compounds in each group reflects the distinct anthropogenic pressures, local biodiversity, the bioavailability of these compounds for incorporation in trophic webs, and the physiological features of Guiana dolphins. It is important to highlight that the highest concentration observed in the study (799 ng.g^{-1} lw) was found in an individual from Guanabara Bay (GB/RJ). This bay is considered the most impacted estuary in the country. Its drainage basin counts 14 million habitants and over 12,000 industries of variate sorts (Kjerfve et al., 1997). A few other studies approached the contamination by organic pollutants in this bay and, repeatedly, the concentrations reported for marine fauna are found to be as high as heavily degraded areas from the Northern Hemisphere (Lailson-Brito et al., 2010; de Oliveira-Ferreira et al., 2021). Hence, marine mammals residing and/or using the bay are under exposure to multiple stressors. A decline of 37 % in the Guiana dolphins' population from the GB/RJ was reported in only 15 years, and current estimates are of <40 individuals (Azevedo et al., 2017). The elevated concentrations of PBDEs in this population could add to the risk of local extinction. Additionally, a population of rough-toothed dolphins (*Steno bredanensis*) inhabiting the same region was predicted to collapse within a century due to heavy organochlorine contamination (de Oliveira-Ferreira et al., 2021). Guiana dolphins from GB/RJ may be exposed to the same risk (Lailson-Brito et al., 2010) since there are food items common to both species' diets and carbon depletion in both delphinids suggests the same foraging grounds (Bisi et al., 2013). Cetaceans are susceptible to a contaminants cocktail, especially in coastal areas, and the effects of their mixture may be detrimental to an already declining population. Toxic effects of PBDEs in combination with organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) in the immune and endocrine systems were already reported for marine mammals (Das et al., 2006; Desforges et al., 2017).

While no thresholds for endocrine disruption were established for marine mammals thus far, Hall et al. (2003) pointed out that juvenile grey seals (*Halichoerus grypus*) presented a positive correlation between alteration in thyroid hormone levels and increase in PBDE blubber concentrations. The median PBDE concentration found in the juvenile pinnipeds was 460 ng.g^{-1} lw, ranging from 64 to 1500 ng.g^{-1} lw (Hall et al., 2003). Most individuals from this study presented values lower than the median observed for grey seals, but some dolphins from SC and GB/RJ were above it, raising the concern about its effect at an individual and population level.

Also in the SE region, Sepetiba Bay is under industrial expansion and counts 1.4 million inhabitants in its drainage basin, receiving approximately 70 tons of untreated sewage (IBGE, 2011). It also receives the industrial effluents of over 100 industries, which constitute one of the biggest industrial poles in RJ (IBGE, 2011). On the other hand, Ilha Grande Bay is the most preserved of the three coastal bays and with lower demographic density than the two previously mentioned (Costa, 1998). The bay presents singular biodiversity, rich in fauna and flora (Brandini, 1997; Costa, 1998; Lana et al., 1996). The upsurge of the cold and nutrient-rich South Atlantic Central Water (SACW) directly influences species abundance and diversity. Due to the scenic beauty and natural richness, IGB has 11 protected areas, and it is less affected by

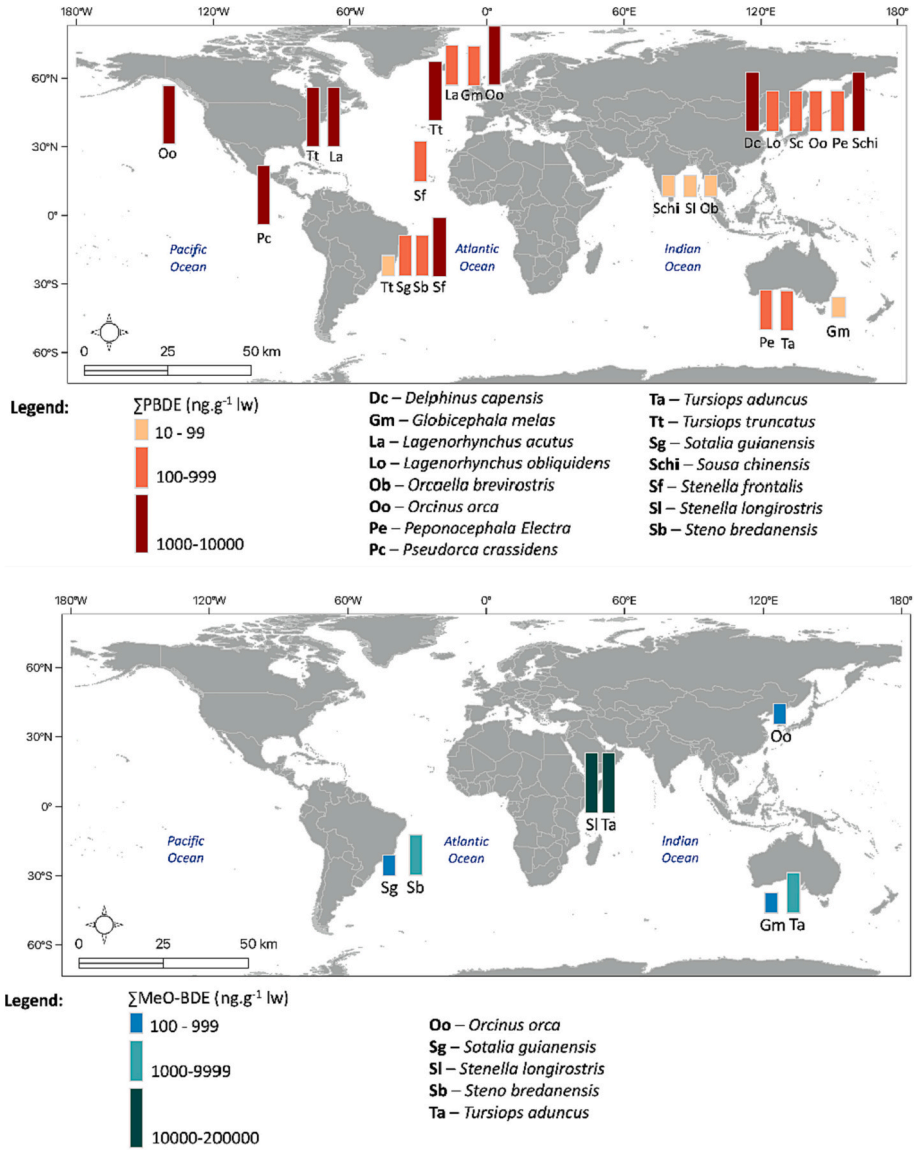

Fig. 4. ∑PBDEs and ∑ MeO-BDEs in males delphinids throughout the world.
The bars are proportional and references used can be found in the supplementary material (see Table S7).

BFRs contamination than GB/RJ and SB/RJ, and more influenced by MeO-BDEs.

In northeastern Brazil, the anthropogenic pressure is reduced when compared to southeastern Brazil and it is shown in the differences between PBDEs and MeO-BDEs. Furthermore, the sites where Guiana dolphins were sampled in the NE region are distant from great urban centers. Individuals from the ES, located in southeastern Brazil, were grouped with individuals from the NE region. While the southern portion of ES presents important urban agglomerates (IBGE, 2011), the samples obtained for the present study are from less impacted areas (the municipalities of Linhares and Guarapari). The incorporation of ES to the NE region was likely caused by the increased local influence of Abrolhos Bank in the species trophic web, increasing the MeO-BDE concentrations.

In the southern states, PR and SC, the dolphins were sampled from Paranaguá Bay and Babitonga Bay, respectively. The area around 1400 km^2 of Babitonga Bay is under the influence of untreated domestic and industrial sewage discharges, overfishing, and silting (Bastos, 2006; Cremer, 2006; Oliveira et al., 2006). Furthermore, Babitonga Bay also presents the São Francisco do Sul Harbor, one of the most important ports of S Brazil, and represents a constant threat due to the dredging, water contamination, and the risk of ship incidents (Gerhardinger et al., 2021). The elevated concentrations of PBDEs found in Guiana dolphins from the area are of particular concern for their conservation. The estuarine complex of Paranaguá Bay, in PR, hosts state and federal protected areas and its ecological importance is also given the diversity of ecosystems found in the complex (Lana, 1986). Furthermore, it holds a significant role in the social-economic development of PR, e.g., harbors (Paranaguá Harbor and Antonina Harbor), touristic and fishery activities, and is also affected by ship traffic, and chemical pollution (Lana et al., 2000; Santos et al., 2009).

Hence, local environmental characteristics and anthropogenic impact are not homogeneous throughout the Brazilian distribution of the Guiana dolphin, which is an important sentinel of environmental health. The distinct contamination profile detected in the localities suggests different trophic webs, and consequently, different ecological populations. Their identification using ecological markers, such as stable isotope analyses, other organohalogen pollutants, and genetic markers, is important while assessing the regional threats that populations may be undergoing and considering specific conservation efforts. The case of the Guanabara Bay population is of particular concern due to the high concentrations of PBDE, the indication of habitat degradation by the low

concentrations of MeO-BDEs, and the extremely small population size (Azevedo et al., 2017), being on the edge of local extinction.

4. Conclusion

The profile of BFR observed in Guiana dolphins along the Brazilian coast corroborates the predominant use of the Penta-BDE mixture, in which BDE-47 was the main compound in all localities. Furthermore, the PBDE concentrations found in Guiana dolphins from the different localities are a reflex of environmental characteristics, e.g., degree of degradation, once it influences the bioavailability of the compounds, along with the ecological and physiological features of the species, given the incorporation and excretion of these compounds. The highest median concentrations reported for PBDEs in the present study were in individuals from SC and GB/RJ. These concentrations are comparable to studies from highly degraded environments in the Northern Hemisphere. Among the BFRs of emerging concern (HBB, DBDPE, and PBEB), only PBEB was detected in Guiana dolphins.

Among the biosynthesized compounds investigated, 6-MeO-BDE-47 and 2′-MeO-BDE-68 were the predominant in all nine localities. Individuals from BA presented the highest concentrations, probably because of the influence of the increased biodiversity in Abrolhos bank, while the lowest was reported for GB/RJ, one of the most degraded environments on the Brazilian coast.

The profile of anthropogenic and natural organobrominated compounds can be used to distinguish ecological populations of coastal odontocetes since it reflects the local anthropogenic pressure and the distribution pattern of marine flora and fauna along the coast. The Northeastern states of Brazil (CE, RN, and BA), with ES, PR, and IGB/RJ presented an increased contribution of natural compounds, and are biodiversity-rich, whereas GB/RJ, SB/RJ, and SC presented the highest contribution of BFRs.

PBDEs are widely used as flame retardants all over the world, being currently detected in several biotic and abiotic compartments. The continued use of PBDEs in consumer products, especially the widespread use of penta-BDE, whose congeners are highly bioaccumulative, suggests that tissue concentrations among some populations of marine mammals and humans will continue to increase. Studies that have addressed the toxicology of PBDEs suggest that PBDE congeners likely to bioaccumulate (i.e., those observed in human tissues and other biota) have the propensity to disrupt thyroid hormones, cause neurobehavioral deficits, and possibly cause cancer.

Considering the vulnerability of the species and the specific characteristics of each population of Guiana dolphins, it is important that the data presented in this study be analyzed with the information on chemical pollution from each location, since it is necessary to consider the synergistic effects of the chemical compounds. In addition, it is of paramount importance that populations are constantly monitored to assess concentrations of persistent organic compounds, in addition to assessing the health of individuals, and potential ecological effects for the population, such as non-sustainability.

Funding sources

IDAEA-CSIC is a Center of Excellence Severo Ochoa (Spanish Ministry of Science and Innovation, MCIN/AEI/10.13039/501100011033, Project CEX2018-000794-S), and this project supported the present study. The authors thank the Rio de Janeiro State Government Research Agency (FAPERJ) and the Generalitat de Catalunya (Consolidated Research Group 2021SGR01150) for financing part of this study.

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Lara Gama Vidal: Formal analysis; Data curation; Investigation; Roles/Writing - original draft.

Nara de Oliveira-Ferreira: Roles/Writing - original draft; Writing - review & editing.

João Paulo Machado Torres: Funding acquisition; Resources; Validation.

Alexandre de Freitas Azevedo: Funding acquisition; Project administration.

Ana Carolina O. Meirelles: Data curation.

Leonardo Flach: Data curation; Investigation.

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Ana Bernadete L. Fragoso: Data curation.

Flávio J. Lima Silva: Data curation; Investigation.

Vítor Luz Carvalho: Data curation.

Milton Marcondes: Data curation.

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José Laison-Brito: Conceptualization; Funding acquisition; Investigation; Methodology; Project administration; Resources; Supervision.

Ethel Eljarrat: Funding acquisition; Investigation; Methodology; Project administration; Resources; Supervision; Validation.

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.

Data availability

No data was used for the research described in the article.

Acknowledgments

A.F. Azevedo and J. Lailson-Brito thank the National Council for Scientific and Technological Development (CNPq) for grants PQ-1B and 1D, respectively; and UERJ (Prociência). We thank the students from Aquatic Mammal and Bioindicator Lab (UERJ - Brazil), Environmental Chemistry Lab (CSIC - Spain) and Radioisotope Lab (UFRJ - Brazil). L.G. Vidal thanks the Coordination for the Improvement of Higher Education Personnel (CAPES - Finance Code 001) for providing her PhD grant. The authors thank the ICMBio - Estação Ecológica de Tamoios (ESEC Tamoios) and APA de Guapi-Mirim/ESEC da Guanabara for supporting the collection of carcasses in Ilha Grande Bay.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2023.167704.

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