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Microplastics and microfibers in the Guajará Bay, Amazon delta: Potential sources and variability

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ABSTRACT

The role of Amazon on the transport and as a source of microplastics (MPs) to the ocean is uncertain. This study is an assessment on the distribution of MPs and microfibers (MFs) in a portion of the Amazon delta. Guajará bay is a potential source for surrounding waters, since a metropolis is located at the right margin. Surface water samples were collected during the dry and rainy season of 2014/2015 at six stations. MP and MF abundance ranged from 218 to 5529.98 (1565.01 ± 196.94) particles·m^{-3}. Transparent, white and blue particles were frequent. Higher values were detected on the right, urbanized margin of the bay ($p = 0.0124$). Most of the particles were anthropogenic cellulose fibers (68.8 %). Polyethylene terephthalate (52.9 %) and polyamide (34.4 %) were the dominant polymers. Our results indicate higher MP and MF abundances near to the potential source, the urban nucleus, and related to local hydrodynamic characteristics.

Plastics are omnipresent in our daily products, but since these materials are not properly disposed of or recycled, they reach the aquatic ecosystems. The plastics accumulated in the environment, coupled with their slow fragmentation, resulted in a widespread distribution of micro-sized plastic particles, even in relatively preserved (Morais et al., 2020) and remote areas (Bergmann et al., 2017). In addition, tropical environments are expected to be polluted by microplastics (MPs) since they have a higher rate of plastic degradation and consequent MP formation due to high temperatures, exposure to ultraviolet light and weathering rate (Arias-Villamizar and Vázquez-Morillas, 2018).

The continent is the main source of plastics, transported primarily by watersheds (Meijer et al., 2021) to the ocean. Amazon, the largest world's river basin, is the natural source of huge loads of sediments, associated to its massive freshwater discharge ($>5 \times 10^{12}$ m^3/y, Callède et al., 2010) and the largest contributor to solute release to the Atlantic (Nittrouer et al., 2021). Therefore, it is expected that the Amazon basin becomes a significant source of plastics due to the intense discharge and recent urbanization (Giarrizzo et al., 2019). In addition, Brazil is the 4th country in the world in terms of plastic waste production and is one of the least effective plastic recyclers (approximately 1.3 %), well below the global average (Kaza et al., 2018).

Guajará bay, located at the southern region of the Amazon delta, may be a potential source of MPs, since the second and fourth largest cities in the Amazon are located at the right margin (IBGE, 2021). Sewage collection and treatment are neglected at the Brazilian Amazon, being nearly or totally absent in most cities (Mansur et al., 2016). In addition, the Brazilian Amazon contributes around 6 million tonnes/year and 328 kg/capita/year of solid wastes, which lacks a significant recycling chain (ABRELPE, 2021).

Rivers are the main MP transport route to the ocean, and these particles can present long-term retention, putting at risk the freshwater biota (Yuan et al., 2022). The Amazon biome presents the greatest

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biodiversity hotspot of freshwater fishes on the planet (Tonella et al., 2023), which has been impacted by plastics already (Andrade et al., 2019), potentially leading to impacts on biodiversity. Despite the negative scenario, knowledge on MPs in the Amazon is still limited to a few published studies, most of them related to ingestion by animals (e.g. Dos Anjos Guimarães et al., 2023).

The investigation of MPs in continental and estuarine waters is necessary to understand the levels of pollution, and to define a baseline for future environmental mitigation measures. Here, we quantified MPs and MFs from surface waters of the Guajará bay, located in the complex Pará river estuarine system, Amazon delta, since the region is a possible source of MPs to adjacent areas.

Sampling took place at Guajará Bay and adjacent areas (Fig. 1), which is part of the Pará River estuarine system, on the southern portion of the Amazon delta (da Silva Gregório and Mendes, 2009), and downstream the Tocantins-Araguaia, Guamá and Capim rivers. While the right margin of the Guajará bay is heavily urbanized and represented by the urban nucleus of Belém metropolitan region, the left margin is composed of a vegetated island and several channels and supports a traditional community (Neu et al., 2018).

The estuarine system of the Pará River starts at das Bocas Bay and continues to the Atlantic Ocean, surrounding the south and east of the Marajó island, where it receives a fluvial contribution from the Amazon River through the Breves and Boiuçu channels (Callède et al., 2010). The estuary is influenced by three major river basins: the Amazon, the Araguaia-Tocantins, and the North Atlantic Coast basins. The system is strongly influenced by the proximity to the ocean. Semi-diurnal tides alter river hydrodynamics, increase the water's residence time and provide an enhanced connection between the riparian zone, floodplains, and the main river channel (Gagne-Maynard et al., 2017).

The region presents a strong rainfall seasonality, with a dry period from August to December, and a rainy period, from January to July. Based on the historical series (1987–2016), the average annual temperature is 26.7 ± 0.4 °C and the average annual precipitation is 3206 ± 131 mm (INMET, 2018).

The samples were collected during three monthly sampling campaigns in two seasonal periods: the dry (August to October 2014), and the rainy season (February to April 2015). Sampling was carried out in a 17 km long transect crossing the Furo Grande, the Guajará Bay and the Guamá River that bathe the city of Belém. A total of six sampling stations were established: three on the right margin (Belém city, P1–P3) and three on left margin (Onças Island, P4–P6). Additional data like limnological variables are available as supplementary material (Table S1).

Surface water samples (100 L) were collected using a 10 L aluminum bucket. The water was immediately filtered through a 50 μm mesh

Fig. 1. Sampling stations for microplastic and microfiber assessment in surface water of Guajará bay and adjacent areas, southern portion of the Amazon Delta, Brazil. Sampling stations P1 to P3 were defined as right margin (P1 is on the right margin of Guamá river, while P2 and P3 are on the right margin of Guajará bay), and P4 to P6 as the left margin.

stainless-steel sieve. The bucket and sieve were previously cleaned at each sampling station with distilled water. The retained material was transferred to clean, externally identified glass flasks containing pre-filtered solution of 40 % ethyl alcohol for preservation and were stored in the dark until laboratory analysis. The separation and characterization of potential MPs was performed in glass petri dishes, and the particles were analyzed individually using a stereomicroscope. MFs and MPs were counted and classified by size, type and color according to GESAMP (2019). The inferior size limit considered was 100 μm since the particles were mechanically separated under the stereomicroscope.

A maximum of 30 units from each type and color, in each season, were randomly selected, measured, photographed, and analyzed by Fourier-transform infrared (FTIR) spectroscopy using the Varian FT-IR 660 spectrometer in the mid-infrared region, with Agilent software for processing the spectra at the Multiuser Spectrometry Laboratory from the Institute of Chemistry, Universidade Federal Fluminense, Brazil. Each particle was analyzed individually and identified by comparing their infrared spectra with reference spectra published in the literature for plastic and cellulose polymers (Jung et al., 2018). The diagnostic absorption bands were compared to those of the literature references, and only spectra with a match percentage ≥ 60 % were accepted. In addition, the full spectral profile of each particle was compared to the references to confirm the assignments.

At the laboratory, standard control procedures were adopted, including the use of a white cotton coat, coverage of labware and samples, and avoidance of plastics (Prata et al., 2021). Cleaning was often carried out on site, and the materials were washed with milli-q water before and after use (Queiroz et al., 2022). Filtration and sample preparation were performed inside a laminar flow hood to minimize airborne contamination. Blank controls were prepared for the ethyl alcohol and milli-q water, with three replicates for each solution. The optical equipment was covered to avoid external contamination (Torre et al., 2016).

Abundance data (particles·m^{-3}) were provided as mean followed by the standard error. A permutational multivariate analysis of variance (PEMANOVA) was used to test for differences in particle composition between the river margins (two fixed levels: right and left margin), and seasons (wet and dry season) (Anderson et al., 2008). The abundance of the different MP (chips, fragments, and films) and MF types (plastic and cellulose fibers) were considered as independent variables. Statistical significance was tested using 9999 permutations of the residuals with a reduced model (Freedman and Lane, 1983) and the Type III (partial) sums of squares (Anderson et al., 2008). The PERMANOVA was run on a Euclidean similarity matrix, calculated from the $\log_{10}(x + 1)$ transformed abundance data. The differences between the significant treatments were visualized in a metric multidimensional scaling (MDS) ordination by the bootstrap averaging based on repeated resampling from the original dataset (Clarke and Gorley, 2015). The global relationships among spatio-temporal treatments variation in particle composition were assessed using the shade plot routine. All statistical analyses were performed in PRIMER v7 (Clarke and Gorley, 2015).

The MPs and MFs were recorded in all the 35 surface water samples (P3 from April 2015 was missing). The minimum values were observed at P2, 10/2014 (65 MPs·m^{-3} and 153 cellulose MFs·m^{-3}), and the maximum for P1, 09/2014 (1719 MPs·m^{-3} and 3811cellulose MFs·m^{-3}). Significant difference was not detected between seasons (1708.1 ± 249.3 and 1429.9 ± 305.6 particles·m^{-3} for the rainy and dry season, respectively), but the right, urbanized margin of Guajará bay showed higher particle abundance than the left margin (1907.8 ± 342.5 and 1239.4 ± 183.4 particles·m^{-3}, respectively).

The one-way PERMANOVA analysis demonstrated a significant difference among the sampling stations ($p = 0.0395$) and between the left and right margins ($p = 0.0124$) (Table 1). Differences were observed between P1 and all the left margin stations (P4–P6) and between P3 and stations P4 and P6 (Table S2), confirming the higher abundances of MPs and MFs in the right margin. However, the two-way PERMANOVA resulted in the absence of significance for the interaction between seasonality and river margin (F = 1.1729, $p = 0.2939$).

The MDS depicted the clear formation of two groups, one for each river margin. Most of the samples (~80 %) were included in these groups according to the 95 % confidence interval (Fig. 2). The shade-plot elucidated the distribution of the types of particles between seasons and margins, with higher values for all particle types occurring at the right margin. In addition, cellulose and plastic MFs were the most abundant particles and higher abundances of cellulose MFs were observed during the rainy season (Fig. 3).

Cellulose MFs were dominant, with abundances from 153 to 3811 particles·m^{-3} (1063.3 ± 135.1 particles·m^{-3}), followed by plastic MFs (64–1658; 450.6 ± 60.2 particles·m^{-3}). Fragments, chips and films were present in lower abundances (Fig. S1), or even absent from samples: fragments ranged from 0 to 54 particles·m^{-3} (23.3 ± 3.3 particles·m^{-3}), chips between 0 and 50 particles·m^{-3} (11.9 ± 2.6 particles·m^{-3}) and films between 0 and 30 particles·m^{-3} (5.4 ± 1.1 particles·m^{-3}) (Fig. S2).

Ten distinct colors were observed (Fig. 4, Fig. S1). Transparent MFs were widely common (85.61 %), in addition to blue (6.28 %), black (3.14 %), green (2.61 %), red (2.14 %), and yellow (<1 %) colors. A similar pattern occurred for the fragments, with dominance of transparent (58. 95 %), followed by white (22.4 %), green (8.01 %), brown (3.5 %) and smaller proportions of other colors. The green paint chips were the most common (37.21 %), followed by white (23.25 %), equal proportions of blue and yellow (16.28 %) and multicolored chips (6.98 %). Most films were transparent (65.53 %), followed by white (20.39 %), multicolored (7.28 %), blue (4.85 %) and finally by black and orange (<1 %).

Regarding the size, microplastics represented 94.97 % of the particles, followed by a reduced contribution of mesoplastics (5.03 %) (Fig. S1). The MFs presented a minimum of 200 μm and a maximum of 4.9 mm (2.396 ± 1.12 mm), while fragments were found in a size range between 100 μm and 3.9 mm (1.488 ± 1.191 mm). The chips were described between 100 μm and 3.8 mm (1.77 ± 1.07 mm), and the films presented a variation of 200 μm to 4.6 mm (1.19 ± 0.86 mm). Some MFs and films were also classified as mesoplastics, since they reached up to 10.9 mm and 9.6 mm, respectively. Anthropogenic cellulose MFs were the dominant particle type (68.8 %), followed by plastic MFs (28.61 %), fragments (1.47 %), chips (0.77 %) and films (0.35 %) (Fig. 4, Fig. S1).

Except for the cellulose microfibers, 10 types of polymers were identified: polyethylene terephthalate (PET, 52.86 %), polyamide (PA, 34.43 %), polypropylene (PP, 4.16 %), ethylene vinyl acetate (EVA, 2.72 %), polyvinyl chloride (PVC, 1.82 %), polyethylene (PE, 1.59 %), acrylonitrile butadiene styrene (ABS, 0.24 %), methyl polymethacrylate (PMMA, 0.18 %), and mixed cellulose (ABS, PVC and PET, 1.7 %). The most common polymers, PET and PA, were widely represented by plastic fibers. The fragments were the most heterogeneous regarding polymer composition, with PVC and PE as the most frequent, followed by PE, PVC, EVA, PMMA and mixed cellulose - ABS. The films were composed mainly of cellulose, in addition to EVA and PE, while all the analyzed chips were composed of EVA.

Table 1
Results from the one-way PERMANOVA for each factor, demonstrating the significant interactions between the sampling stations and river margins. Bold and italics: significant values ($p < 0.05$).

Factors/groups	Total sum of squares	Within-group sum of squares	F	p
Sampling months	45.34	39.18	0.9124	0.5211
Sampling stations	45.34	33.94	1.948	***0.0395***
Dry × rainy season	45.34	44.12	0.9176	0.4017
Left × right margin	45.34	39.98	4.429	***0.0124***

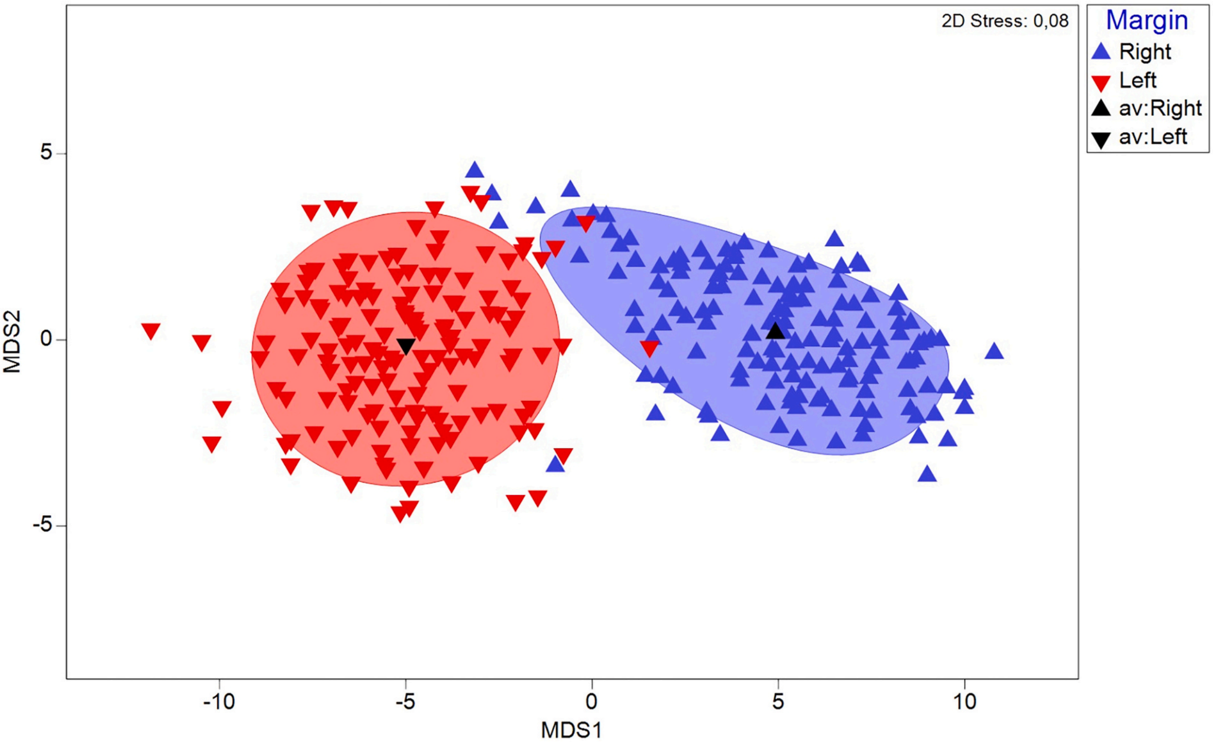

Fig. 2. Multidimensional scaling (MDS) ordination, depicting the formation of two groups of samples: the left and the right margins of Guajará bay.

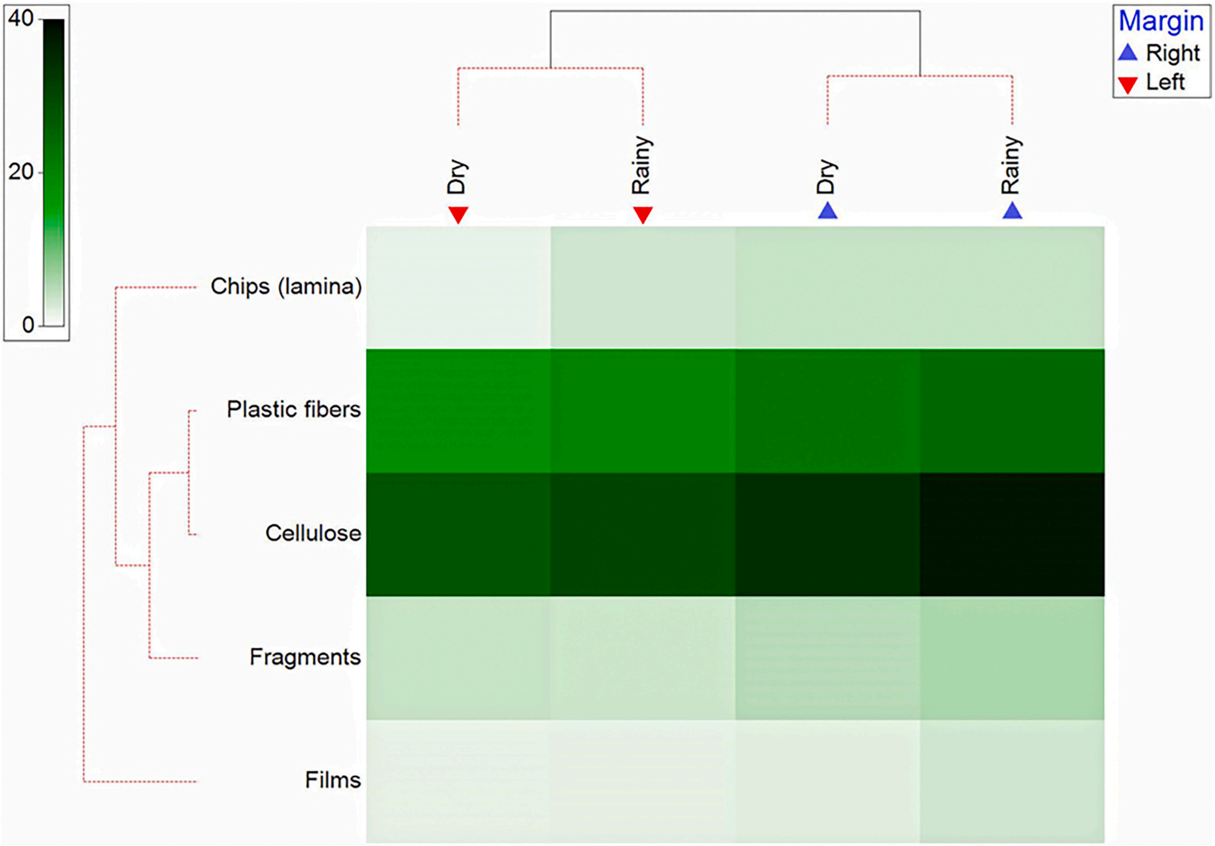

Fig. 3. Shade plot graphic illustrating the relationships among spatio-temporal treatments in particles types from surface water samples from Guajará bay. Shading intensity within the matrix indicates the relative abundance of each microplastic types (a legend is in the upper left of plot).

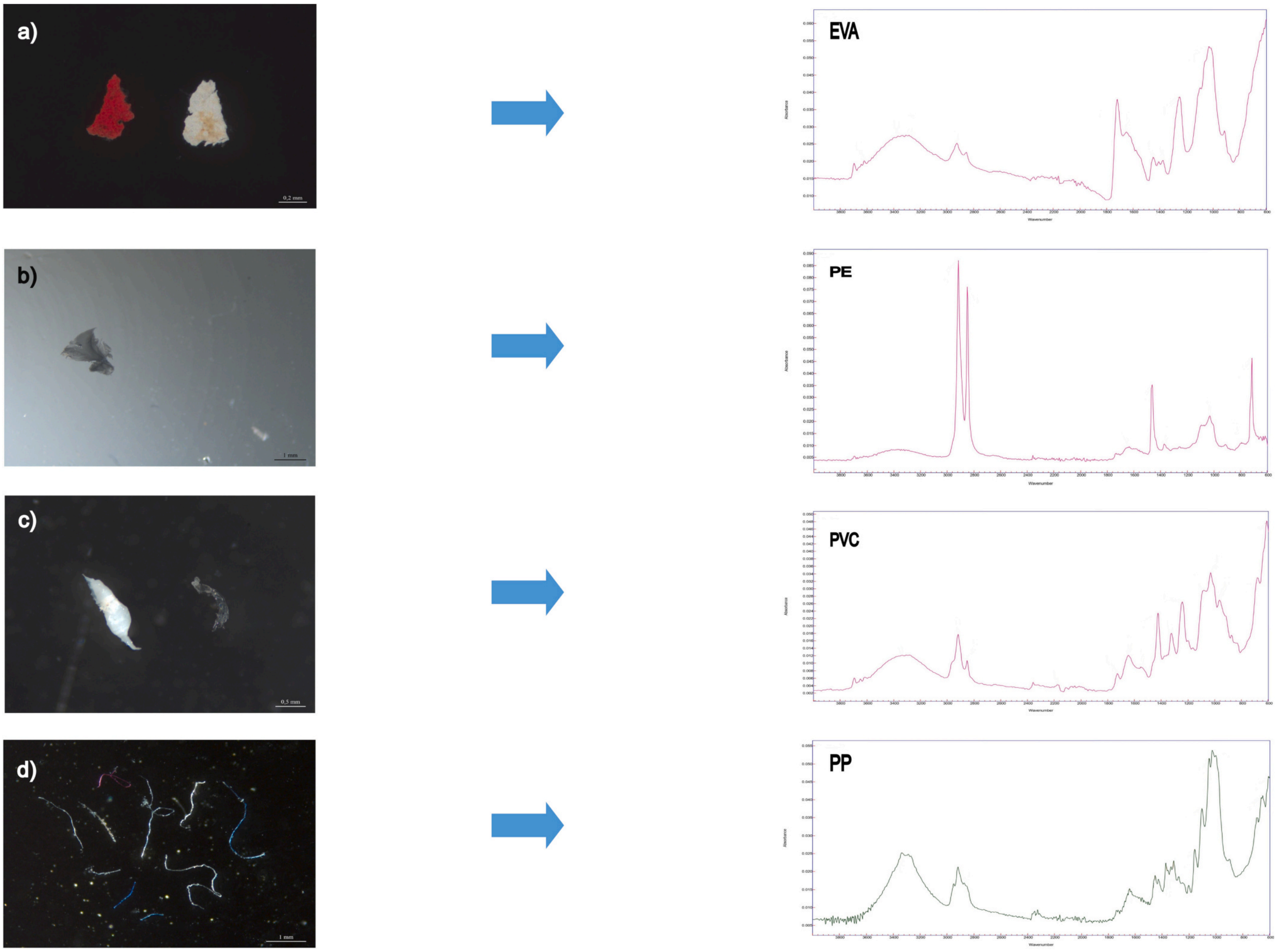

Fig. 4. Examples of microplastics and microfibers from Guajará bay, Brazilian Amazon. A) ethylene-vinyl acetate chips; B) polyethylene textile film; C) polyvinyl chloride microplastic fragments; D) textile, polypropylene fibers.

The Amazon is a continental sized area, but data on MPs are absent for most of it. Recently, the first paper on MPs in Guajará Bay analyzed six surface water samples and found abundances between 23 and 3095 particles·m^{-3} (Rico et al., 2023). If we excluded cellulose fibers from our study, the MPs abundance alone dropped to 65–1719 particles·m^{-3}, similar to the findings described by Rico et al. (2023).

The abundance described here is below those reported in some estuaries and bays located in Brazil and in the world (see Table S3), but these studies used mesh apertures from three to six times higher than ours and were generally conducted in mesohaline to euryhaline waters. On the other hand, the values described in Guajará bay were similar to an estuary in China (Wu et al., 2019). Values from two times to even one order of magnitude higher than ours were also observed in other estuaries (Table S3); however, these studies used a lower limit for mesh size like ours, ranging from 40 to 75 μm. The great variability on the estimation of MP abundances relies on the lack of methodological standardization, although the values described here were inside the range observed by the few authors in the same or in adjacent areas (Queiroz et al., 2022; Rico et al., 2023).

Despite the limited information on MPs in environmental matrices in the Amazon, the dominance of MFs in riverine water is a common finding (Forrest et al., 2020; Acharya et al., 2021). The dominance of anthropogenic MFs in the Amazon was described in the sediment (Gerolin et al., 2020), and in surface waters along the Amazon River (Rico et al., 2023), where 51 % of the particles were plastic fibers (cellulose fibers were not accounted), and on surface waters from the Amazon shelf, when 58 % of the particles were MFs during the rainy season (Queiroz et al., 2022). These MFs may result from the release from synthetic fabrics during the washing processes (Browne et al., 2011), reaching effluents and consequently the bay, since it drains a metropolitan area. Another possible source for plastic MFs is the fisheries industry, a main economic activity at the Brazilian Amazon coast (Jimenez et al., 2020). The disposal of fishing artifacts in the aquatic environment is a common practice and may be a source of nylon MFs (Martinelli Filho and Monteiro, 2019).

The most common polymers described here were also common in previous studies from the Amazon. PE, PVC, and PA were the most frequent MPs extracted from the stomach content of freshwater fishes from the Xingu River (Andrade et al., 2019). Nylon (PA) was also identified by the single publication on the ingestion of MPs by a freshwater invertebrate in the Amazon, the endemic shrimp *Macrobrachium amazonicum* (dos Anjos Guimarães et al., 2023). Both authors related the presence of PA to materials that are widely used in the fishing industry, which possibly contributes to the presence of these MPs in surface waters.

Even fewer studies determined the polymeric composition of MPs in environmental samples in the Brazilian Amazon. A recent study found PA, followed by PU, ABS, PET and EVA as the most abundant polymers in surface waters from the Amazon shelf (Queiroz et al., 2022). The assessment by Rico et al. (2023) was the first to describe polymeric composition of the MPs in surface water samples from the Amazon River basin. The authors also observed that PET was the most common polymer, followed by PP, PS and PE. Despite some variations on the FTIR techniques employed here and in Rico et al. (2023), the main difference is that PA was the second most common polymer here but absent from the previous study.

PA and PET tend to shrink due to their specific gravity (GESAMP, 2019); however, they were dominant polymers in this study. Some factors such as hydrodynamics may explain this pattern, since Guajará bay presents physical forces such as strong wind currents, and a voluminous water flow (Prestes et al., 2020). In addition, the low topography, the mesotidal regime and turbulence (da Silva Gregório and Mendes, 2009) would allow the constant resuspension of MPs and MFs in the water body.

Our study area, on the east portion of the Amazon delta, is influenced by the Tocantins basin and its mouth. The Tocantins basin is the most anthropogenically impacted in the Amazon biome, with 58 % of its area occupied by agricultural activity (Pelicice et al., 2021). Intense land change use, leading to development of agriculture, cattle farming and the fast growth of cities, took place on the margins of the river tributaries (Becker, 2016). Owing to the economic activities, settlement of middle-sized cities on the river margin, and lack of proper sewage and solid waste management, the river mouth is a possible source of MPs and MFs to the Amazon delta.

Guajará bay is wrongly attributed as a bay. In fact, it is part of the lower Pará river estuary (Barros et al., 2015). The strong water flow, tidal transport and turbulence are high variable (Prestes et al., 2020; da Silva Gregório and Mendes, 2009), and probably influence the distribution, dispersal, and fate of MPs and MFs. The hydrodynamic model by Barros et al. (2015) showed higher concentrations of contaminants associated to the right margin of the bay, due to a low velocity stream. This feature, in addition to the proximity of the potential MP/MF sources (Belém - Ananindeua metropolitan area) on the right margin, would explain our results.

Many questions remain regarding the sources and sinks of MPs, as well as their spatial and temporal variability in the Amazon delta. Rico et al. (2023) performed a single sampling in 2020, but even our study with six months of sampling was not sufficient to elucidate significant differences between the dry and rainy seasons. It is possible that the rainy period presents higher abundances of plastic particles due to the watershed transport, but a proper sampling design is still necessary to verify such hypothesis. Besides the Belém metropolitan area, the Tocantins basin and the Boiuçu and Breves channels are important regions to determine the sources of MPs and MFs in the delta and their possible transport to the inner adjacent shelf. The enormous delta and its complexity call for a more detailed study on the potential contributions of the tributaries and the transport of MPs and MFs to the adjacent Atlantic waters.

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CRediT authorship contribution statement

Luana Francy Oliveira Santos: Methodology, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization. **Vania Neu:** Project administration, Funding acquisition, Resources, Conceptualization, Methodology, Writing – review & editing. **Raqueline Cristina Pereira Monteiro:** Data curation, Investigation, Visualization, Formal analysis. **Vinicius Tavares Kütter:** Methodology, Writing – review & editing. **Leonardo Mario Siqueira Morais:** Supervision, Writing – original draft, Writing – review & editing. **Abilio Soares-Gomes:** Methodology, Formal analysis, Writing – review & editing. **David Chelazzi:** Data curation, Methodology, Formal analysis, Writing – review & editing. **Tommaso Giarrizzo:** Conceptualization, Methodology, Formal analysis, Investigation, Supervision, Writing – original draft, Writing – review & editing. **José Eduardo Martinelli Filho:** Project administration, Funding acquisition, Resources, Conceptualization, Methodology, Formal analysis, Writing – review & editing, Writing – original draft, Visualization, Supervision.

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

Data will be made available on request.

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