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Research article

Contamination by microplastics and sorbed organic pollutants in the surface waters of the Tietê River, São Paulo-SP, Brazil

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ABSTRACT

Microplastics (MPs) are particles between 1 μm and 5 mm in size, originating mainly from poor solid waste and effluent management, that can reach water bodies from various sources. In freshwater environments, the occurrence, distribution, and characterization of this new class of pollutants are still little explored, especially in Brazil. The aim of this study was to assess the occurrence of MPs, as well as the presence and concentration of polychlorinated biphenyls (PCBs) sorbed to these particles in the surface waters of the Tietê River - SP. Surface water samples were collected in duplicate during the dry and wet seasons. The identification and characterization of the MPs was carried out through visual inspection and the chemical identity of the particles was verified using Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR). For the analysis of PCBs adsorbed to the MPs, the sample extracts were analyzed by gas chromatography coupled with mass spectrometry (GC-MS). The MPs were found in concentrations ranging from 6.67 to 1530 particles m^{-3}, with a predominance of the polymers polyethylene (PE, with 58.17 %) and polypropylene (PP, with 23.53 %). The main morphological categories identified were fragments (56.63 %), fibers (28.42 %), and transparent films (13.06 %). Higher abundances of PCBs were observed in the lower size range, between 0.106 and 0.35 mm. The total concentrations of PCBs in MPs ranged from 20.53 to 133.12 ng g^{-1}. The results obtained here are relevant for understanding the dynamics and level of contamination of MPs and organic pollutants sorbed to these particles in the Tietê River, as well as helping with mitigation measures for the restoration and preservation of this ecosystem.

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1. Introduction

When not disposed of properly, plastics can accumulate in aquatic and terrestrial ecosystems, where they are subject to degradation processes and progressively fragment into smaller and smaller particles [1]. Plastic particles in the size range between 5 mm and 1 μm are denominated microplastics (MPs) [2].

MPs have already been studied in different aquatic environments, especially marine environments (Koelmans et al., 2019 [3]). Approximately 70–80 % of the plastic waste that reaches marine ecosystems is transported by rivers, since many freshwater matrices are located close to waste disposal points and cross highly urbanized areas [4]. Among the sources of MPs in freshwater environments, industrial effluents, rainwater overflows, domestic effluent discharge, and wastewater treatment plant (WWTP) effluents are the main sources of MPs (Scherer et al., 2020 [5] [6]).

Due to their physicochemical properties, MPs can be considered potential vectors of organic pollutants, such as polycyclic aromatic hydrocarbons (PAHs), pesticides, and polychlorinated biphenyls (PCBs), since they have hydrophobic surfaces capable of sorbing these contaminants from water [7,8].

PCBs are considered as one of the synthetic organic contaminants with the greatest environmental persistence and, due to their ecotoxicological risks, are included in most environmental and human health surveillance programs around the world [9]. Although PCBs have been banned since 2001 by the Stockholm Convention [10] they still persist in the environment and can be found in all environmental compartments, especially in tissues and hydrophobic particles, such as MPs [11].

To date, studies on the occurrence of adsorbed MPs and PCBs have mainly focused on marine environments and beaches (Costigan et al., 2022 [12] [13]), highlighting the need for further investigations regarding these contaminants in freshwater ecosystems [14].

The Tietê is the Brazilian river with the highest levels of contamination due to the disorderly occupation of its banks and the dumping of domestic and industrial effluents in nature (including from pulp and paper mills, chemical plants, metallurgical factories, rubber industries, pesticide manufacturers, among others) [15]. Although it has high levels of anthropogenic contamination in some stretches, this body of water still presents considerable biodiversity and is a source of food for riverside communities located in some of the municipalities it drains. Despite its economic and social importance for the territory where it is located, there is only one study in the literature focusing on MPs in this river [16], which investigated the ingestion of MPs by fish (*Prochilodus lineatus*) native to this body of water.

Given this context, the aim of the current study was to investigate the abundance of MPs in the surface waters of the Tietê River and to evaluate the presence of PCBs sorbed to these particles, with a view to elucidating contamination by MPs and their potential to act as vectors in the transportation of organic pollutants in this important Brazilian river.

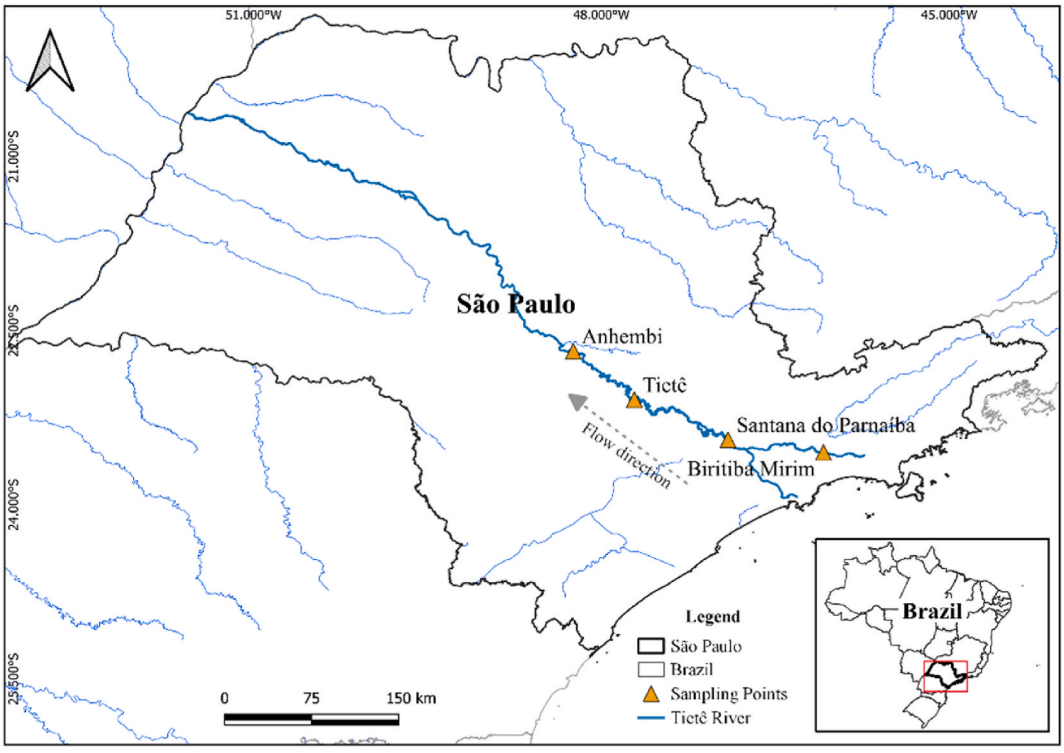

Fig. 1. Map of microplastic collection sites in the Tiête River, São Paulo, Brazil. P1 = Biritiba Mirim, P2 = Santana de Parnaíba, P3 = Tietê, and P4 = Anhembi.

2. Materials and methods

2.1. Study area

The Tietê River is the largest body of water in the state of São Paulo, Brazil. Along its 1171 km length, it drains 62 municipalities, including São Paulo, which is the most populous city in Latin America [17,18]. In the current research, 4 sampling points (P1, P2, P3, and P4) were established on the Tietê River, based on different degrees of water quality (CETESB, 2014), in order to allow comparison of the abundance of MPs at sites with contrasting characteristics (Fig. 1) (Supplementary Table 1).

Point P1, located in Biritiba Mirim, is in a region of intense agricultural activity, where the degradation of the Tietê water quality begins. Points P2 (Santana do Parnaíba) and P3 (Tietê) are located on the most polluted stretch of the river, in municipalities with high population densities and intense industrial activity. The last point, P4 (Anhembi), is located on the stretch where water quality improves consistently as a result of the self-depuration and dilution of contaminants.

2.2. Sampling

Four sampling campaigns were carried out (A, B, C, and D) in two different periods: Dry season (September–October 2020, Samples A and B) and wet season (March 2021, Samples C and D). No rainfall was recorded on the collection days (Supplementary Table 2). Simultaneous sampling was conducted for two purposes: I) Analysis of the occurrence of MPs in surface waters and II) Analysis of PCBs adsorbed on MPs in the surface waters of the Tietê River.

2.2.1. Analysis of the occurrence of microplastics in surface water

A 10 L aluminum bucket was used to collect surface water from the Tietê River (top 30 cm). In total, 200 L of water were collected at each point and transferred to a set of stainless steel sieves with openings of 0.106 mm and 5.6 mm. The material retained by the larger mesh sieve was discarded, and the remainder was dried in an oven at 60 °C and treated with 30 % hydrogen peroxide and ferrous sulfate heptahydrate at 75 °C to remove organic matter [19]. After oxidation, the samples were again transferred to the 0.106 mm steel sieve and dried at room temperature.

The samples were grouped by size using a stacked sieve scheme with openings of 0.106, 0.35, 0.5, 1.0, 2.0, and 5.6 mm, according to the methodology adapted from Desforges et al. [20]. The portions retained in each mesh were sorted and quantified manually, with the aid of stainless steel tweezers, a magnifying glass, and a stereoscopic microscope with 30× magnification. The MPs identified were categorized as fibers, fragments, pellets, microbeads, films, or styrofoam, according to the methodology adapted from Cable et al. [21] and GESAMP [22].

2.2.2. Analysis of polychlorinated biphenyls in microplastics from the Tietê river

The samples were collected using a nylon mesh net with an opening 0.6 m in diameter, 3 m long, and a mesh size of 0.3 mm, containing a cup with the same mesh. Sampling was carried out in the surface layer of water (top 30 cm), for 40 continuous minutes or until the net clogged. In the laboratory, all the contents collected were transferred to a set of stainless steel sieves with openings of 1 mm and 5.6 mm. Due to the excess of organic matter and the difficulty in cleaning the sample, a larger minimum size was established at this stage. The material retained by the larger mesh sieve was discarded, and the remainder was separated by density in NaCl solution (1.2 g cm^{-3}) and dried at room temperature [19]. Finally, the samples were sorted and categorized in accordance with the process detailed in the previous topic.

2.3. Characterization of the microplastics

The minimum amount of 10 % of each category of MPs was analyzed using Fourier transform infrared absorption spectrometry with attenuated total reflectance (FTIR-ATR by Bruker, model Vertex 70, USA). The absorption spectra were collected in the wavelength range from 4000 to 400 cm^{-1}, with a resolution of 4 cm^{-1} and 64 scans.

The data from each sampling point were processed using Microsoft Excel 2019 software and the graphs were created using GraphPad Prism software, version 8.0.1 for Windows, San Diego, CA, USA.

The spectra of MPs obtained in this study were compared with reference spectra described by Jung et al. [23]. To confirm the results of chemical identification, the online and free tool Open Specy (https://openanalysis.org/openspecy/) was used, which has open-access reference spectra. In this tool, the degree of correlation between the reference spectrum and the sample spectrum is measured using the Pearson correlation coefficient (r), which ranges from 0 to 1, indicating higher statistical correlation the closer it is to 1. To consider the identification result, only coefficients higher than 0.85 were accepted.

2.4. Quality control

The collected material was handled with care to avoid contamination, including washing with deionized water, drying with the opening down, and storage in containers covered with lids or aluminum foil. Samples were collected twice for each station. When handling the samples, the precautions taken included wearing cotton lab coats, masks made from natural fibers, and gloves. In addition, the analysis laboratory was kept closed to reduce draughts and the circulation of suspended particles.

2.5. Extraction of polychlorinated biphenyls from microplastics

We used 6 types of PCBs, containing 3 to 7 chlorine atoms in the mixture (2,4,4′-trichlorobiphenyl (PCB 28); 2,2′,5,5′-tetrachlorobiphenyl (PCB 52);2,2′,4,5,5′-pentachlorobiphenyl (PCB 101); 2,2′,4,4′,5,5′- hexachlorobiphenyl.(PCB 153); 2,2′,3,4,4′,5' -hexachlorobiphenyl (PCB 138); 2,2′,3,4,4′,5,5' -heptachlorobiphenyl (PCB 180)). These standards were obtained from AccuStandard (USA) with a purity of 99.99 %. According to Campanale et al. [24], these congeners were chosen as indicators of PCB contamination by the Stockholm Convention.

The US EPA method 8082A was adapted to extract PCBs from MPs. The MPs samples were ground in a cryogenic mill (Freezer Mill, 6870, Japan) and weighed in Falcon tubes. After adding 5 mL of ultrapure water and shaking manually for 30 s, 5 mL of n-hexane: ethyl acetate (1:1, v/v) were added and shaken for 1 min in a vortex with a porcelain stone. Next, 2 g of $Na2^{SO}4$ and 2 g of NaCl were added, before stirring again. After sonifying for 10 min and centrifuging at 5000 rpm for 5 min, 2 mL of the supernatant were collected in a Falcon tube $_4^{\text{containing clean-up (150 mg of PSA, 150 mg of C18, and 900 mg of MgSO}}$).

Except for the salts ($Na2^{SO}4$ and NaCl), porcelain stone, and water, the steps were repeated and the volume of supernatant collected became 5 mL. The tubes were shaken for 3 min and centrifuged at 5000 rpm for 5 min. The supernatants were transferred to 15 mL glass flasks and evaporated under a continuous flow of N_2 in a TurboVap. The extracts were then resuspended in 1 mL of n-hexane and analyzed by gas chromatography coupled to mass spectrometry (GC-MS). Due to the low mass of MPs collected (1.83 ± 0.78 g), only individual samples were obtained for each day, however, the chromatographic injections were carried out in triplicate per sample.

2.6. Chromatographic analysis

The chromatographic analyses were carried out on a gas chromatograph (Agilent Technologies, model 7820A) coupled to an electron impact (EI) mass spectrometer (Agilent Technologies, 5975C, USA), with an HP-5 ms fused silica capillary column (30 m × 0.25 mm x 0.25 μm). The carrier gas used was helium, at a constant rate of 1 mL min^{-1} and a pressure of 7.7 psi. The samples were injected by an automatic sampler in splitless mode, with 1 μL.

The temperatures of the GC injector and the MS transfer line were 280 and 330 °C, respectively. The oven temperature ramp was programmed as follows: 50 °C for 1 min, followed by an increase rate of 25 °C min^{-1} up to 200 °C, and then a rate of 10 °C min^{-1} up to 330 °C, with a final waiting time of 10 min, totaling 30 min of chromatographic running.

The MS operated in selective ion monitoring (SIM) mode (70eV), with source and quadrupole temperatures of 320 and 180, respectively. The solvent cut-off time was 8 min. The identification of PCBs was based on the retention time, mass spectra, and *m/z* ratios of each molecule. The curves used for the quantification of PCBs contained 5 concentrations (0.025; 0.05; 0.1; 0.25; and 0.5 μg mL^{-1}), and a correlation coefficient (r^2) greater than or equal to 0.995 was considered. Full details of the validation results can be found in the supplementary material (Supplementary Tables 3–10).

3. Results

3.1. Abundance, size and shape of microplastics

MPs were found at all the sites sampled, with average abundances of between 80 and 865 particles m^{-3} in the dry season and between 18.33 and 972.5 particles m^{-3} in the wet season (Table 1). It was not possible to observe a clear trend between the wet and dry seasons for the concentration of MPs at the four sampling points. The highest individual abundances of MPs were recorded during the wet season at points P2 and P3, with 1075 and 1530 particles $m^{-3,}$ respectively. On the other hand, the lowest concentrations were found at points P1 and P4, with values of 6.67 and 25 particles $m^{-3,}$ respectively.

In the current study, 1432 MPs particles were identified. In both periods (Dry and Wet Seasons), the most common category was the 0.106–0.35 mm range, representing 31 % and 56 % of abundance, respectively (Fig. 2-A). This range accounted for 44.62 % of total abundance in both periods. During the dry season, there was a lower abundance of MPs from 2 to 5.6 mm, while during the wet season, the lowest abundance was in the 1–2 mm range.

Table 1
Abundance of microplastics (particles m^{-3}), in the dry season (A = September and B = October 2020) and in the wet season (C and D = March 2021 sampling point in the Tiête River, São Paulo, Brazil. P1 = Biritiba Mirim, P2 = Santana de Parnaíba, P3 = Tietê and P4 = Anhembi.

Period	Sampling	Concentration (particles m^{-3})			
		P1	P2	P3	P4
Dry Season	A	156.67	900	210	120
	B	90	830	820	40
	Average	123.33	865	515	80
Wet Season	C	6.67	730	1530	40
	D	30	1075	415	25
	Average	18.33	902.5	972.5	32.5
Total average (± Standard deviation)		70.83 ± 67.13	883.75 ± 145.24	743.75 ± 582.23	56.25 ± 43.08

It can be seen that the total average number of MPs at all the collection points ranged from 56.25 ± 43.08 to 883.75 ± 145.24 particles m^{-3} in both the dry and wet seasons.

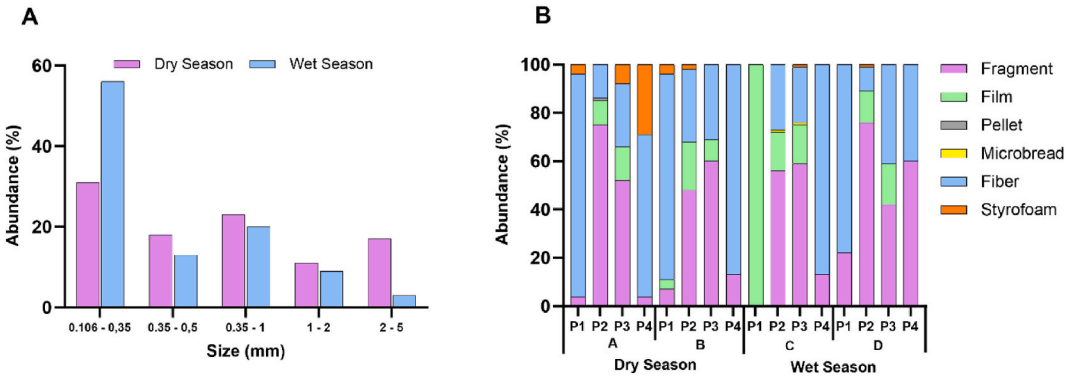

Fig. 2. Abundance of microplastics found in the Tietê River (São Paulo, Brazil) during the dry and wet seasons: A) size range; B) category. P1 =Biritiba Mirim, P2 = Santana de Parnaíba, P3 =Tietê, and P4 = Anhembi.

During the dry and wet seasons, the size range between 0.35 and 0.5 mm presented abundances of 18 % and 13 %, respectively. For MPs from 0.5 to 1 mm, the abundance was 23 % in the dry season and 20 % in the wet season. For particles between 1 and 2 mm, there was greater abundance in the dry season (11 %) than in the wet season (9 %). Finally, in the dry season, MPs between 2 and 5.6 mm accounted for 17 %, while in the wet season, this range had a lower occurrence, totaling 3 % of the particles. Analysis of the abundance of MPs in the surface waters of the Tietê River revealed several categories, including fibers, fragments, pellets, microbeads, films, and styrofoam (Fig. 3). Pellets were found only at P2 and microbeads only at P2 and P3.

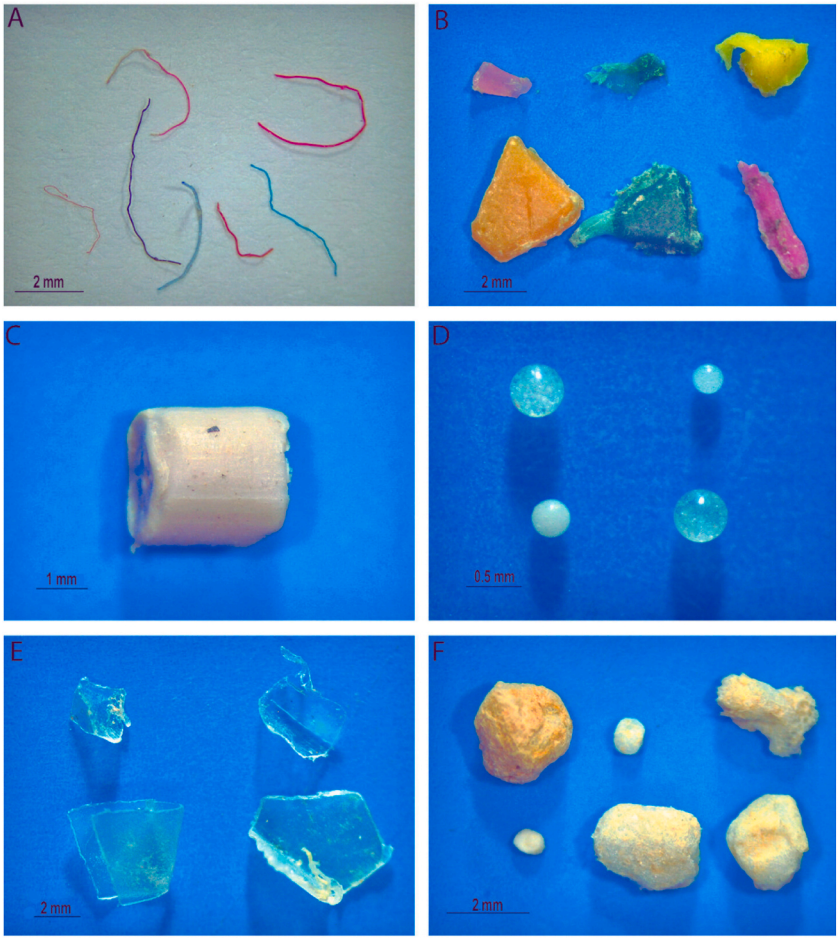

Fig. 3. Representative microplastic particles from the following categories: A) Fibers, B) Fragments, C) Pellets, D) Microbeads, E) Films, and F) Styrofoam found in the Tietê River, São Paulo, Brazil.

Considering all the MPs collected, the main categories in both periods were fragments (56.46 %), followed by fibers (28.42 %) and films (13.06 %) (Fig. 2- B). In the dry season, fragments accounted for 52.13 %, fibers for 34.35 %, and films for 10.94 % of the particles collected. In the wet season, fragments accounted for 32.70 %, fibers 12.64 %, and films 8.03 %. Styrofoam was present in both periods, with around 2.43 % in the dry season and 0.77 % in the wet season. The microbeads category was only found at P2 and P3 in the wet season, representing 0.52 % of the particles. Only one pellet-type MP was collected at P2 during the wet season (0.15 % of the particles).

During the dry season, fragments were the predominant category, while in the wet season there was a predominance of fibers. There were seasonal variations in the occurrence of the styrofoam, film, and microbead categories, which were more frequent in the dry season. The fragment category was subdivided by color (Fig. 1 of the supplementary material), with 63.02 % of the particles categorized as colored (yellow, blue, red, green, pink, or orange), 17.27 % as white, 10.10 % as black, and 9.61 % as transparent.

Finally, the concentration of PCBs was assessed in the MPs. A total of 419 particles between 2 and 5 mm in size were analyzed at points P2 and P3 (Fig. 2 of the supplementary material). Of these, four main morphological types were identified: fragments (65.87 %), pellets (24.11 %), films (5.49 %), and fibers (4.53 %). Styrofoam-type particles were not considered in this analysis.

3.2. Chemical characterization of microplastics

Despite their wide application in MPs studies, manual techniques for counting and classifying MPs have some limitations, such as variations between analysts due to the subjective nature of the analysis, potential errors in estimating concentration due to the small size of the particles and difficulty in distinguishing between different types of MPs through visual classification. Given this, representative MPs from each class were selected for confirmation of chemical identity through FTIR - ATR.

The results of the chemical characterization of the MPs collected in the surface waters of the Tietê River, through analysis of 159 particles using FTIR- ATR, revealed that the PE polymer was the most frequent, representing 58.17 % of the total particles analyzed. PP was identified in 23.53 % of the particles, followed by PET with 6.54 %, and PS and PU with 5.88 % each (Supplementary Figs. 3 and 5). Of these particles, two could not be identified (1.31 %), three contained a mixture of PP and PE polymers (1.96 %), and another three (1.96 %) were identified as rubber.

From the results, it was possible to observe the predominance of PE and PP polymers in the Tietê River, which are often used in polymeric materials, such as packaging, bags, and household utensils, among others (Li and Campos, 2020). The significant presence of PET should also be noted, as this polymer is commonly identified in packaging and bottles, which may suggest a specific source of contamination [25].

3.3. Sorption of polychlorinated biphenyls on microplastics

The results of the analysis of PCBs in MPs from the surface waters of the River Tietê are shown in Table 2. The table presents the values obtained for the quantification of the 6 PCBs congeners in MPs samples, categorized by collection point and period, covering both the dry and wet seasons. The individual average concentrations of PCBs were established in nanograms per gram (ng $g^{-1)}$ of sample. PCBs concentrations differed between the dry and wet seasons, as well as between the different samples collected at points P2 and P3.

The individual concentrations of PCBs ranged from 9.64 ± 0.24 to 53.01 ± 0.31 ng g^{-1}. PCBs 138 and 180 were found in all the

Table 2
Concentrations of polychlorinated biphenyls (PCBs) (ng g-1 sample) in microplastics collected during the dry season (A and B) and during the wet season (C and D) in the Tiête River, São Paulo, Brazil. ± represents the standard deviation of the mean (n = 3). P2 = Santana do Parnaíba and P3 = Tietê.

Congeneric	Collection point	Dry season (2020)		Wet season (2021)	
(ng g^{-1})		A	B	C	D
PCB 28	P2	n.d.	n.d.	n.d.	l.s.
	P3	n.d.	n.d.	n.d.	l.s.
PCB 52	P2	n.d.	n.d.	30.18 ± 0.22	l.s.
	P3	16.65 ± 0.28[a]	27.18 ± 1.21	30.54 ± 0.14	l.s.
PCB 101	P2	19.01 ± 0.72[a]	n.d.	20.48 ± 0.13	l.s.
	P3	12.91 ± 0.21[a]	15.58 ± 0.12[a]	10.79 ± 0.63[a]	l.s.
PCB 153	P2	13.11 ± 0.52[a]	n.d.	23.09 ± 0.16	l.s.
	P3	13.15 ± 0.10[a]	42.10 ± 0.31	30.74 ± 0.12	l.s.
PCB 138	P2	17.92 ± 0.13[a]	9.64 ± 0.24[a]	31.17 ± 0.39	l.s.
	P3	33.31 ± 0.82	53.01 ± 0.31	31.24 ± 0.33	l.s.
PCB 180	P2	19.02 ± 0.24[a]	10.89 ± 0.48[a]	28.20 ± 0.35	l.s.
	P3	19.05 ± 0.11[a]	33.29 ± 0.18	19.37 ± 0.84[a]	l.s.
Σ6 PCBs	P2	69.06	20.53	133.12	l.s.
	P3	95.07	129.06	122.68	l.s.

n.d. = Substance not detected or less than the LD (1.98–3.57 ng g^{-1}).
l.s. = Sample was lost during the cryogenic grinding process.
[a] = Substance quantified after fortification at 0.1 μg mL^{-1}

samples analyzed. All PCBs homologs were detected at P3, with the exception of PCB 28, in both sampling periods and at concentrations ranging from 10.79 ± 0.63 to 53.01 ± 0.31 ng g^{-1}.

The highest concentrations were found at P3 during the dry season, for PCB 138 (53.01 ± 0.31 ng g^{-1}), PCB 153 (42.10 ± 0.31 ng g^{-1}), and PCB 180 (33.29 ± 0.18 ng g^{-1}). The total concentrations of PCBs, i.e., considering the sum of the average of the individual concentrations of each congener, ranged from 20.53 to 133.12 ng g^{-1}, the highest of which was found at P2 during the wet season.

4. Discussion

4.1. Abundance, size, shape, and chemical characterization of microplastics

The results highlight the significant presence of MPs in the Tietê River during both the dry and wet seasons, in all the areas sampled. The lack of a clear relationship between seasons suggests that several factors, such as population density, urbanization, industry, climate, river hydrodynamics, and effluent disposal, have an influence on MPs concentrations in surface waters (Li and Campos, 2020). In Supplementary Fig. 4, it is possible to observe the sewage treatment frequencies in the collection municipalities, with Anhembi having the highest rate (75.12 %) and Santana do Parnaíba having the lowest rate (40.36 %), indicating a potential correlation with MPs concentrations.

In addition, larger MPs were found at sites P2 and P3 during the wet season, which may be associated with greater movement of plastic waste during this period, probably due to urban runoff and rainfall.

The identification of different categories of MPs indicates the heterogeneity of potential sources of contamination in the river. According to the data presented above, the fragments category was numerically dominant at almost all collection points, which is consistent with other data in the literature, where this category was the most abundant (Bertoldi et al., 2021; Koelmans et al., 2019 [26]). According to Fahrenfeld et al. [27], aspects such as morphology and the color of MPs can provide evidence about their origin in the environment. For example, MPs morphologies such as microbeads and fibers may be associated with wastewater discharge. On the other hand, fragments and films can be attributed to the continuous wear and tear of improperly disposed macroplastics (Zhou et al., 2018).

The high presence of colored fragments in the Tietê River suggests multiple sources of MPs in the collection areas [28]. The white fragments found may have originated either from white plastic objects or from the degradation of colored plastics in the environment, under the action of stressors, such as exposure to ultraviolet light. A previous study [29] revealed that MPs that were apparently white or colorless externally turned out after fragmentation to be colored internally. This highlights the possibility of dyes from microplastics leaching into the environment.

The frequency of black fragments suggests the presence of particles from the natural wear and tear of vehicle tires, which are easily washed away by rain through road runoff, and enter water bodies through city drainage systems [30].

High levels of fibers were identified at most of the sampling points, being the second most abundant category found in this study, suggesting that pollution from households may play a significant role in this body of water. These fibers come mainly from clothing made of synthetic materials and are transported to the aquatic environment through washing clothes, street runoff during rainfall, and airborne dust [31].

The presence of pellets at one of the collection sites may be related to the dispersion of polymeric materials into granules that are used as raw materials in industrial plants. In freshwater environments, pellets can reach the river through losses during the transportation process, whether by road or rain [32]. Although there is already more pressure on the industry to limite the use of microbeads in personal care items and cosmetics, it is still possible to find products containing these particles on the market [33]. In this way, microbeads end up reaching water bodies, mainly via domestic sewage (Jiang, 2018 [34]).

The categories of MPs found at P1 (fibers, fragments, and films) could be the result of both the degradation of macroplastics (present around the body of water, for example) and the discharge of sewage into the river or from upstream WWTPs. It should be noted that the Biritiba-Mirim WWTP is located upstream of P1 and its receiving body is the River Tietê.

Point P2 showed a high average concentration of MPs (883.75 ± 145.24 particles m^{-3}), possibly due to inadequate waste management, intense urban activities, and high population density, reaching 604.74 inhabitants m^{-2} (IBGE, 2010). The observation of various types of materials in the water suggests multiple sources of pollution, and the high population density is linked to increased consumption and disposal of plastics.

Collection point P3 had the highest concentration of MPs, reaching approximately 1530 particles m^{-3} during one of the sampling sessions in the wet season. The predominance of fragments and fibers was notable, and P3 was the only site where microbeads were observed. These high concentrations of MPs can be widely attributed to the absence of riparian forests, the high population density, and the dumping of untreated sewage near this point.

Finally, P4 presented one of the lowest concentrations of MPs among the collection points. The predominant category was fibers, followed by fragments and styrofoam. The site showed significant quantities of plastic materials on the river banks, possibly originating secondary MPs. Artisanal fishing is a crucial activity in the P4 area, where residents consume and sell various species of fish. The presence of styrofoam particles may be linked to the improper disposal of items such as the thermal boxes and styrofoam objects used to preserve fish.

The analysis of the MPs showed PE and PP to be the predominant polymers in the samples collected. This predominance can be attributed to the fact that the samples were collected on the surface of the river, as these polymers have a low density (0.89–0.97 g cm^{-3} for PE and 0.85–0.92 g cm^{-3} for PP), which makes it easier for them to remain in the upper part of the water column. In addition, these are the main polymers consumed globally and in Brazil [35]. In addition, the dynamics of the watercourse may allow denser

polymers, such as PET to be present in the surface layer of the water column.

The comparison of the data obtained in this study with other research conducted in Brazilian aquatic ecosystems reveals significant parameters and differences in the concentrations and compositions of MPs.

Rico et al. (2023) monitored MPs in the Amazon River, finding concentrations ranging from 5 to 152 particles m-3 in the Amazon River and its tributaries, and between 23 and 74,550 particles m-3 in urban streams. Remarkably, the measured concentrations in the Amazon are lower than those found in the Tietê River during both dry and wet seasons, with averages ranging from 70.83 to 883.75 particles m-3, indicating different contamination levels between these rivers, which can be explained by distinct hydrological characteristics and land use in the watersheds.

Queiroz et al. [36] found an average abundance of 3593 ± 2264 particles m-3, with higher values in the rainy season in the Amazon Continental Shelf. Conversely, abundance in The Sinos River, Southern Brazil, indicated an average of 330.2 particles L-1 in the raw river water and 105.8 particles L-1 in treated water [37]. Thus, it is possible to observe that the concentrations of MPs in the Tietê River are within the same order of magnitude as other areas studied in Brazil, highlighting widespread concern about MPs pollution, despite differences in sampling methods and measurement units among studies.

Besides abundance, the morphology and composition of MPs differ among studies. While fragments were the main morphologies found in the Tietê River, fibers were more predominant in the Amazon ([36]; Rico et al., 2023) and in The Sinos River [37]. These differences can be explained by distinct sources of pollution and transport processes of MPs in different freshwater environments.

In addition to physical risks to biota, such as obstruction of the gastrointestinal tract and injuries, MPs can act as vectors for metals and organic contaminants (Vo and Pham, 2021). Due to their physicochemical properties, PE, followed by PP and PS, are the polymers with the greatest affinity for organic molecules present in water (Wang et al., 2020). This means that these polymers can increase the risks associated with the transfer of organic contaminants to

Aquatic life, especially when they are ingested or absorbed (Yu et al., 2021).

Based on the review carried out by Shim et al. (2018), the relationship between the abundance of MPs and the size of the sampling mesh is inverse, indicating higher concentrations in smaller meshes. Although this study did not identify a correlation between size ranges and the number of MPs, it was found that the largest number of particles was in the smallest range (0.106–0.35 mm). Other research has also confirmed that the effects of MPs increase with decreasing particle size [38,39]. This is because the smaller the particle, the greater the surface area, and, therefore, the greater the ability to interact with pollutants and organisms present in the environment [40].

In Brazil, there are few studies on the occurrence of MPs in freshwater systems, and considering the potential risks of these contaminants, it is urgent that they are better regulated and that further studies like the current one are performed.

4.2. Sorption of polychlorinated biphenyls on microplastics

In this study, PCBs were selected due to their toxicity, historical presence in industrial activities, regulation, and potential impact on public health. It is important to note that hydrophobic particles such as MPs can easily adsorb PCBs on their surface, becoming potential vectors of these contaminants [11].

The results shown in Table 3 indicate significant contamination of the Tietê River by MPs and PCBs, related to the intense anthropogenic activity in the sampling region.

In the literature, the concentrations of PCBs found in environmental samples of MPs vary greatly, as shown in Table 3, and studies on the presence of PCBs in MPs collected from rivers are scarce. The concentrations of PCBs found in this study corroborate other findings reported in the literature for rivers and can be considered high, because high concentrations of organic matter and other natural particles (such as wood, biochar, coal, and sediments) compete in these environments, through hydrophobic interactions, for the sorption of organic contaminants [44].

Table 3
Concentrations of polychlorinated biphenyls (PCBs) in microplastics reported in other studies.

Source of samples	Morphological characteristics of the items	Analysis techniques	Concentration range ($ng\ g^{-1}$)	Reference
Tietê River, Brazil	Fragments, pellets, films, and fibers	Extraction by sonification and identification by GC-MS	20.53–133.12	This study
Ofanto River, Italy	Pellets and fragments	Extraction by ASE and identification by GC-MS	0.54–15.3	Campanale et al. [24]
Beaches on the Persian Gulf coast, Iran	Pellets	Extraction by immersion in hexane and identification by GC-MS	<10 - 624	Alidoust et al. [41]
Beaches on the coast of Portugal	Pellets	Extraction by immersion in hexane and identification by GC-MS	10–310	Mizukawa et al. [42]
Beaches on the coast of São Paulo, Brazil	Pellets	Extraction by soxhlet and identification by GC-MS	3.41–7554	Taniguchi et al. (2016)
Pearl River Delta, China	Fragments, pellets, styrofoam, films and fibers	Extraction by ASE and identification by GC-MS	1.86–456	Mai et al. [43]
Australia's coastal beaches	Pellets	Extraction by immersion in hexane and identification by GC-ECD	0.1–294	Yeo et al. (2015)

Despite the ban on the use of PCBs by the Stockholm Convention, Rios et al. (2007) [45] indicate that around half of all PCBs ever produced remain stored or in use. This significant stockpile can be released into the environment through leaks or improper disposal of electrical equipment, for example [46]. In addition, Eggleton and Thomas [47] highlight the continuous migration of PCBs present in contaminated soils and sediments to the aqueous phase. In this phase, these pollutants interact more prominently with biota and with natural organic particles (such as organic matter and coal) and synthetic particles (such as MPs), due to their hydrophobic nature [44].

The sampling points in this study are located on stretches of the Tietê River with different levels of water quality. Along the river, the degree of water quality is influenced by the characteristics of land use and occupation, the discharge of domestic and industrial effluents, and the de-characterization of the natural course due to rectification and canalization works [48].

The Tietê River runs through highly industrialized areas of the São Paulo metropolitan region, where the main sources and routes of PCBs into this water body may be present. Points P2 and P3 are located close to these sites, thus justifying the occurrence of PCBs in the MPs collected at these points.

The seasonal differences in PCBs concentrations may be associated with the climatic conditions of the region and the hydrological characteristics of the river. For example, during the dry season, the reduction in water flow can cause an increase in the concentration of PCBs, while the increase in water flow during the wet season can dilute the concentration of these contaminants.

In addition to MPs being capable of sorbing organic pollutants on their surfaces and facilitating their transport in aquatic matrices, the association between MPs and PCBs is concerning due to the synergistic, additive, or antagonistic effects they can have on aquatic biota [49], leading to changes in survival rates, growth, endocrine disruption, and inhibition of predatory performance (Bucci; Tulio; Rochman, 2020 [50]).

Considering the potential risks of MPs and PCBs to the biota, more studies are needed, focused on developing effective remediation alternatives, as well as on actions aimed at reducing the generation of plastic waste, raising awareness, and controlling sources of persistent organic pollutants in the environment.

5. Conclusion

The Tietê River is contaminated by MPs of different categories and synthetic polymers. Higher abundances of MPs were found at the sites with the highest population densities (P2 and P3). MPs from the fragment and fiber categories, in the size range between 0.106 and 0.35 mm, were dominant. Among the particles subjected to FTIR-ATR analysis, PE and PP types of polymers were most commonly identified in this study, which corroborates information in the scientific literature.

With the exception of PCB 28, all the other polychlorinated biphenyl congeners (PCB 52, PCB 101, PCB 153, PCB 138, and PCB 180) were found sorbed in the MPs samples analyzed. This study contributes to elucidating the current scenario of contamination by MPs and the transport of PCBs in the Tietê River through sorption by these particles. Given the importance and diverse uses of the Tietê River water along its course, the presence of adsorbed MPs and PCBs is worrisome and highlights the need for the development of more effective awareness strategies, improved solid waste management, and enhanced treatment of domestic and industrial effluents.

Data availability

Data will be made available on request.

Compliance with ethics requirements

This article does not contain any studies with human or animal subjects.

CRediT authorship contribution statement

Nicoli Gomes de Moraes: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Glaucia Peregrina Olivatto:** Writing – review & editing, Conceptualization. **Felipe Machado de Oliveira Lourenço:** Writing – review & editing. **Ana Laura Athayde Lourenço:** Writing – review & editing, Methodology. **Gustavo Munhoz Garcia:** Writing – review & editing, Methodology. **Rodrigo Floriano Pimpinato:** Validation, Methodology. **Valdemar Luiz Tornisielo:** Writing – review & editing, Supervision, Resources.

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.heliyon.2024.e36047.

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