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Microplastic distribution in the surface water and sediment of the Ergene River

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ABSTRACT

Rivers are major transport pathways for microplastics to reach the oceans. Although gained much attention over the last few years, there is still a relatively lack of knowledge on microplastics in rivers. This study aims to investigate (i) spatiotemporal distribution of microplastics in an industrially polluted river, (ii) the relationship of microplastic abundance with river's morphological and hydrodynamic characteristics (iii) the potential sources of microplastics inferred from the particle characteristics including shape, size, color and type. To achieve these aims, water and sediment samples were collected from six sites upstream of the Ergene River in May 2019 and Sep 2020. According to the results, surface water had an average concentration of 4.65 ± 2.06 and 6.90 ± 5.16 items L^{-1} (mean ± standard deviation, $n = 12$), respectively for the May 2019 and September 2020 periods, whereas 97.90 ± 71.72 and 277.76 ± 207.21 items kg^{-1} ($n = 18$) were observed for the sediment compartment, respectively. Microplastic levels in water correlated positively with stream depth but negatively with channel width. Fibers were the dominating shape both in water (88%) and sediment (70%) and majority of the particles were black (49% in water and 39% in sediment) and blue (25% in water and 18% in sediment). According to Raman spectroscopic analysis, polyethylene terephthalate (PET, 28%) and polyamide (PA, 27%) were dominating polymers in water, while polystyrene (PS, 56%) were dominant in sediment. Compared to many other rivers, the Ergene River had excessive levels of microplastics. The research indicated that textile industries and effluents from organized industrial zones were the foremost contributor of microplastics in the river.

1. Introduction

Beginning in the 1950s, plastic production has increased dramatically over the last decades in line with expanding population and increasing consumption. Mass production of plastics almost reached 370 million tonnes globally in 2020 (PlasticsEurope, 2021), however, more than half of this production is leaking and littering the environment (UNEP, 2021). Therefore, approximately 60–80% of anthropogenic litter in the environment is plastic(Derraik, 2002). Microplastic pollution has become a global concern, particularly in the last decade, due to its rapid increase and the potential hazards it poses to the environment. The plastic materials are mostly nondegradable or have very slow degradation rates, resulting in accumulation in various environmental compartments, in the form of microplastics -tiny plastic particles smaller than 5 mm in size. Microplastics may originate from primary or secondary sources. Primary sources mainly include cosmetic and medical products that constitute plastic pellets (Guerranti et al., 2019), whilst secondary microplastics are derived from the fragmentation of larger plastic items as a result of physical, chemical and biological processes (Cole et al., 2011). The majority of microplastics in the environment is originated from secondary sources (An et al., 2020).

Since oceans are accepted as ultimate sinks for microplastics, scientific research has generally focused on the occurrence, abundance and transport of microplastics in marine environments. However, approximately 80% of plastics in the marine environment originated from land-based sources whereby rivers act as the major transport pathways (Bowmer and Kershaw, 2010; Andrady, 2011). For example, González-Fernández et al. (2021) estimated that 307–925 million macro floating litter items are released annually from European rivers into the ocean of which 84% was plastics. Özgüler et al. (2022) calculated the total microplastics load from only ten small/large volume local rivers to Mersin Bay (the north-eastern Mediterranean) as high as 2216 billion items per year. It is worth noting that, some of the microplastics in the riverine environment may get deposited onto the bottom sediments.

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Therefore, besides being transport routes, rivers can also be hotspots due to the accumulation of microplastics in these environments.

Major sources of riverine microplastics can be listed as wastewater treatment plants (WWTPs) (Browne et al., 2011; Vermaire et al., 2017), the textile industry (Deng et al., 2020), cosmetics and medical products, fishing activities and anthropogenic litter (Guerranti et al., 2019; Daniel et al., 2020; Bashir et al., 2021), followed by agricultural plastics and tire and road wear particles (TRWP) (Nizzetto et al., 2016). Macro and micro-plastics may be introduced to rivers by the effluents of WWTPs, surface runoff, wind dispersal and atmospheric deposition (Dris et al., 2016; Napper and Thompson, 2016; Horton et al., 2017).

Microplastics have been reported in many global rivers, such as the Danube, Austria (Lechner et al., 2014); Seine, France (Gasperi et al., 2014); Rhine, Germany (Klein et al., 2015; Mani et al., 2015); Thames, UK (Horton et al., 2017); Ottawa, Canada (Vermaire et al., 2017); Saigon, Vietnam (Lahens et al., 2018); Nakdong, South Korea (Eo et al., 2019); Ganges, India (Napper et al., 2021; Singh et al., 2021); and others such as, Maozhou, Manas and Fenghua in China (Wu et al., 2020; Wang et al., 2021). Most of these studies were conducted with great volumes of river samples collected via manta trawls or plankton nets, typically with 100 μm or 330 μm pore sizes, due to relatively low concentrations of microplastics in the studied compartment. Recent studies have started to collect bulk samples (Wang et al., 2017, 2021; Eo et al., 2019), whi ch may provide a more straightforward investigation of smaller microplastics as tiny as 10 μm (Wu et al., 2020). However, knowledge of the occurrence of smaller spectrum of microplastics in aquatic environments is still limited, yet, research indicates that the adverse effects of microplastics increase with the decreased size of the plastic particles (Browne et al., 2008; Ma et al., 2016; Chen et al., 2017). This study aims to investigate the occurrence of microplastics in an industrially polluted river and determine the distribution of microplastics larger than 45 μm in size, in two river compartments; surface water and sediment. For this purpose, the Ergene River's upstream tributaries, Çorlu and Ergene, were chosen as the study site, mainly because of the heavy industrialization located in this area. Ergene River is one of the most polluted rivers in Turkey, receiving significant amount of industrial waste due to lack of inspection and inadequate treatment. Nevertheless, the Ergene River Basin is still an important agricultural area, where significant portion of rice, sunflower and wheat production of Turkey is carried out (Tokatlı and Varol, 2021). The use of the river water for irrigation of agricultural lands in the basin has become a potential public health issue, which makes the study site a hotspot for microplastic research. This research is the first study on the occurrence and identification of microplastics in the Ergene River.

2. Materials and methods

2.1. Study area and sample collection

Ergene River is located in the northwestern region of Turkey. The river rises from the Istranca Mountains in the northeastern part of the basin and flows for 283 km through the northwest-southwest direction, with a maximum depth of 6.8 m, before reaching the Aegean Sea. Significant amount of waste originating from industry and unplanned urbanization threaten the water resources feeding the Ergene River. Çorlu and Ergene tributaries are approximately in 60 km length and join the main stream from the eastern side of the river. There are numerous Organized Industrial Zones (OIZs) within the basin, being especially intensive in the sub-basins of the Çorlu and Ergene tributaries (Fig. 1a), which makes the area one of the most prone regions to contamination.

River samples were collected from the surface water and sediment in dry periods, in May 2019 and Sep 2020. The sampling was performed once in six stations including the Çorlu and Ergene tributaries and on the mainstream where the tributaries met (Fig. 1b). The coordinates of the sampling sites are given in Supplementary Information (Table S1). Water sampling was carried out using stainless-steel buckets and collecting samples into glass bottles. 15 L of surface water samples (0–15 cm depth) were collected in duplicates from each sampling site and preserved at +4 °C prior to analysis. Sediment samples were collected using a Van Veen grab sampler, which were then transferred to aluminium foil containers and covered tightly. Three replicates were collected for sediment samples and preserved at −20 °C in the freezer before extraction. Additionally, the parameters including flow rate (m s^{-1}), stream depth (m) and channel width (m) of the river were measured using Acoustic Doppler Current Profiler (ADCP) instrument. After measuring the river's hydrodynamical and morphological parameters, the evaluation was made according to four consistent measurements with an error rate of less than 5% for each location. Meteorological data during the sampling periods, and on-site measurements are given in Table S2.

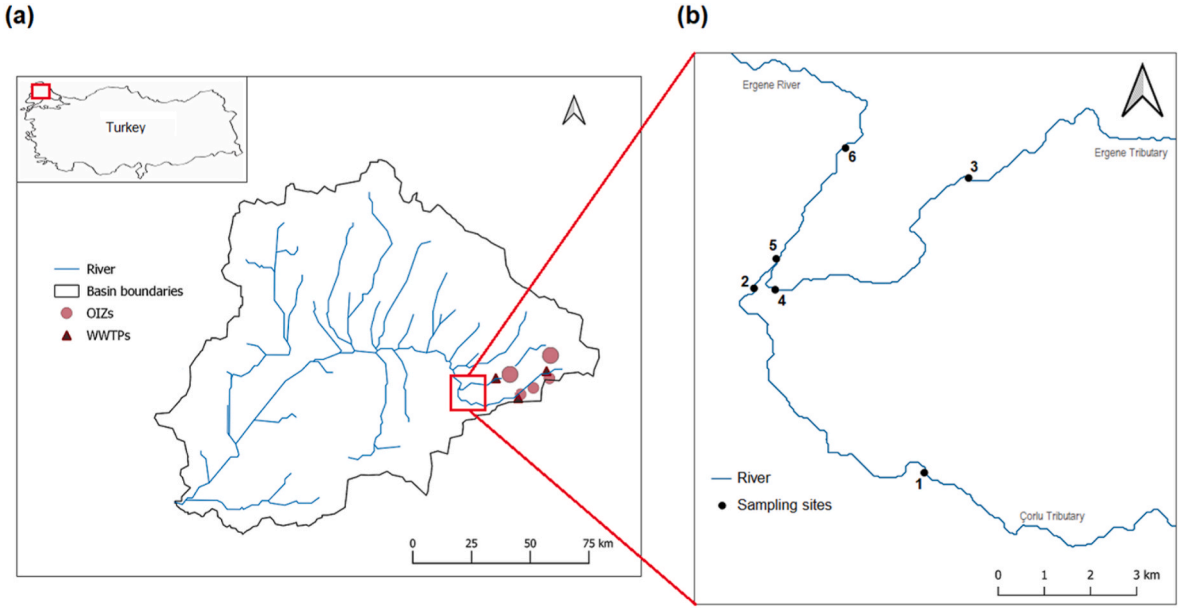

Fig. 1. Map of the study area: (a) Ergene River Basin and location of the OIZs and WWTPs within the study site. (b) Sampling sites.

2.2. Extraction of microplastics

The organic matter in the water samples was removed prior to microscopic observation by a modified version of the wet peroxide oxidation method developed by Masura et al. (2015). Firstly, the supernatant of water samples was filtered through a plankton mesh with a 45 μm pore size, previously checked for airborne contamination. On the other hand, organic matter that had settled at the bottom of the bottle was transferred to a beaker and digested adding 35% hydrogen peroxide (H_2O_2) solution. Depending on the amount of organic matter, H_2O_2 was added gradually at certain time intervals and the covered beakers were kept at 50^{O}C in the water bath until the organic matter disappeared. In case of any organic matter remained in the sample, the process was repeated so as not to exceed seven days (Nuelle et al., 2014). Finally, the digested aliquot was filtered through a 45 μm plankton mesh. For sediment samples, microplastics were extracted from each sediment replicate through the density separation method developed by Thompson et al. (2004). After homogenization, a 50 mL aliquot of each sediment replicate was weighed and stirred for 1.5 min in a glass beaker with 500 mL of concentrated NaCl solution (1.2 kg L^{-1}), which subsequently was allowed to settle for 1 h. The supernatant liquid including extracted particles from the sediment was filtered through a 45 μm plankton mesh. This procedure was repeated two times for three replicates of sediment samples.

2.3. Quality assurance and control

All laboratory work was carried out in a fume hood to prevent airborne contamination. The interior of the fume hood was cleaned with deionized water and ethyl alcohol before use. All laboratory glassware and equipment used were rinsed with tap water thoroughly followed by a final rinse using ethyl alcohol and deionized water. Laboratory glassware and sample containers were covered with aluminium foil. Possible sources of microplastic contamination were prevented by wearing nitrile gloves, cotton clothes and a lab coat during the study. Before filtration, plankton meshes were checked for probable airborne contamination under a stereo-microscope and cleaned by removing particles via forceps in case any airborne particle was detected. Control samples were produced to determine probable contamination. For this purpose, with each sample being examined, a 500 mL glass beaker filled with deionized water was placed in the fume hood, which was examined and analyzed under the stereo-microscope after going through the same processes. In case of any microplastics exist in the control samples, the number of particles detected in the blanks were subtracted from the number of particles in the river samples.

2.4. Identification and characterization of microplastics

Microplastic particles were visually identified under a stereo-microscope (Olympus SZX 16, 30x) and transferred to a clean cellulose filter paper (Whatman filter paper with 4–7 μm pore size), previously checked for airborne contamination. After photographing microplastics, ImageJ software was used to count the plastic particles and determine the length of each particle individually. Microplastics identified were categorized according to their colors and shapes as fiber, hard fragment, soft fragment, pellet, foam, rubber and others. The differentiation between hard and soft plastics is significant due to their distinct properties, applications, and recycling capabilities. Such differentiation could provide additional information on the sources of microplastics. Hard and soft fragments were distinguished visually or by applying slight pressure with the tip of the forceps. After visual inspection, particles were preserved in closed Petri dishes until polymer characterization.

Extracted particles were analyzed by Raman Spectroscopy (Renishaw Invia) to identify polymer types using 532 nm or 785 nm laser, laser power varying from 0.5 mW to 10 mW, with a 20x objective. Integration time and scan accumulations varied depending on the samples. The background interference was checked in each spectrum and as needed, background subtraction was applied to increase the spectrum quality. Spectra were recorded between 100 and 3200 cm^{-1} Raman shift and then corrected using Spectragryph Software (Menges, 2020). Finally, Wiley KnowItAll ID Expert Software was used to match the spectra with the reference polymer spectra found in the library. The spectra that have a match above 60% with the reference spectra was considered.

2.5. Statistical analyses

Statistical analyses were conducted with SPSS 16 (SPSS Inc., 2007) defining statistical significance as $p < 0.05$. The microplastic concentration dataset was transformed into the logarithmic form since the Kolmogorov-Smirnov normality test revealed that microplastic concentrations in water and sediment samples were not normally distributed ($p < 0.05$), nevertheless normality test of the transformed data met the value of $p > 0.05$. The differences in microplastic concentrations between the spring (May 2019) and fall (September 2020) seasons were examined both for water and sediment samples by running an independent *t*-test. Moreover, one-way analysis of variance (ANOVA) with Tukey's post hoc analysis was used to understand whether there is a significant spatial distribution between sampling stations. Finally, Pearson correlation tests were conducted to observe the relationship between environmental variables including flow rate, stream depth and channel width together with microplastic abundance in the river. The strength of correlation was determined using five scales from 'very weak' to 'very strong', given in Table S3.

3. Results and discussion

3.1. Microplastic abundance in water and sediment

A total of 761 and 1351 microplastic particles were extracted from the samples collected in May and September, respectively. Water samples had average concentrations of 4.65 ± 2.06 and 6.90 ± 5.16 items L^{-1} (mean ± standard deviation, $n = 12$), and sediment samples' average concentrations were 97.90 ± 71.72 and 277.76 ± 207.21 items kg^{-1} ($n = 18$) for the May 2019 and September 2020 periods, respectively. Both water and sediment samples collected at the end of the dry season (September) include higher concentrations of microplastic in total than the samples collected in the wet season (May). Several studies in the literature also reported higher microplastic abundance in rivers during dry seasons (Nel et al., 2018; Wu et al., 2020; Wicaksono et al., 2021), which was generally associated with decreased river flux that is limiting the dilution of microplastic concentration in rivers (Yan et al., 2019; Wu et al., 2020). In this study, the temporal variation of microplastic abundance was more dramatic for sediment samples than those for water samples. A statistically significant difference in the number of microplastics between the two periods was found for sediment ($F = 14.63$, $df = 20.19$, $t = -2.44$, $p = 0.02 < 0.05$), whilst statistical analysis did not indicate a significant temporal difference for riverine water ($F = 4.03$, $df = 22$, $t = -1.19$, $p = 0.25 > 0.05$). These outcomes can be attributed to variation in waste management, change in microplastic characteristics, such as polymer density and particle size, and associated settling and accumulation of these particles in sediment during the dry season. On the other hand, the sampling was done in the wet and at the end of the dry seasons, and no major differences were observed in river morphology and flow rates between two periods (Table S2), which could explain the relatively less temporal variation of microplastic abundance in water samples. Moreover, particular sampling sites (2 and 4) had an inverse manner compared to the other sites, where microplastic abundance was higher in May than those in September (Table 1). These variations can be as a result of differences in meteorological conditions between two sampling periods. Although both sampling were performed

Table 1
Microplastic concentrations in water and sediment samples.

Site	Water		Sediment	
	Mean ± SD[a] (items L^{-1})		Mean ± SD[a] (items kg^{-1})	
	May 20, 2019	Sep 30, 2020	May 20, 2019	Sep 30, 2020
1	1.60 ± 0.99	3.49 ± 0.71	57.18 ± 14.48	448.92 ± 66.66
2	6.60 ± 5.20	3.96 ± 0.41	57.99 ± 48.06	107.17 ± 40.04
3	7.44 ± 4.51	17.51 ± 2.16	62.76 ± 46.08	125.73 ± 92.64
4	3.32 ± 2.24	2.70 ± 0.27	45.02 ± 18.57	276.80 ± 400.44
5	3.32 ± 1.88	4.69 ± 1.95	250.64 ± 137.32	67.17 ± 22.89
6	5.60 ± 2.11	9.04 ± 5.68	113.81 ± 40.66	640.76 ± 444.63

[a] Standard Deviation.

in dry periods, wind speed on the day of sampling in September (mean = 17 km h^{-1}) was slightly higher than in May (mean = 14.83 km h^{-1}), whilst the wind directions were totally different (Table S2). A recent study by Leads et al. (2023) indicated that wind direction had a great effect on microplastic abundance and temporal variability.

Microplastic abundance data of all sites are given in Table 1. A significant spatial variation was found for water samples, particularly between Site 3 (upstream of the Ergene River tributary) and the other sampling sites in September ($F = 4.37$, $df = 23$, $p = 0.009 < 0.05$). These results are expected, considering a variety of environmental factors in the study area, especially due to different land uses (e.g. industries, agriculture, and farmland) and associated wastewater effluents (Baldwin et al., 2016; Kapp and Yeatman, 2018). Several studies indicated the relationship between increased microplastic concentrations and industrial areas (Alam et al., 2019; Cordova et al., 2022; Wong et al., 2020). Site 3 had the highest amount of microplastics in both seasons, probably depending upon the surrounding land use (residential area) and proximity to WWTPs and OIZs, in which textile industries heavily exist. Besides, both seasons had similar trends in the microplastic distribution along the river, exhibiting an increase in the abundance of microplastics along the Çorlu Tributary from Site 1 to Site 2, while the number of particles suddenly decreased from Site 3 to Site 4 along the Ergene River Tributary. Microplastic levels continued to decline at Site 5, the downstream of the junction, where the Çorlu Tributary meets the mainstream and the river reaches its maximum width amongst the sampling sites, but then increased again at Site 6. On the other hand, sediment samples did not illustrate a statistically significant spatial distribution ($F = 1.40$, $df = 35$, $p = 0.25 > 0.05$).

Microplastic abundance data obtained from this study were compared with some of the other studies conducted in different rivers of the world (Table 2). Compared to many other rivers, the Ergene River had excessive levels of microplastics. Average concentrations in the surface water were much higher than those in other rivers including eight rivers of Mersin, Turkey (Özgüler et al., 2022); European rivers (Dris et al., 2018; Rodrigues et al., 2018) and Ottawa River, Canada (Vermaire et al., 2017), but considerably lower than the concentrations in the Pearl River, China (Yan et al., 2019). Compared to other east Asian rivers, such as Qiantang (Zhao et al., 2020), Yangtze (Wang et al., 2017) and Nakdong (Eo et al., 2019), the abundance of microplastics were higher in the Ergene River (Table 2). However, it should be noted that the differences in sampling and analysis methods hinder the comparability of our results with the literature data. For example, microplastic levels for both water and sediment in our study were comparable to the Ciwalengke River, Indonesia (Alam et al., 2019), which receives industrial wastewater discharge similar to the Ergene River. Yet, unlike this study, the authors performed the analysis with a 1.2 μm plankton mesh, which hampered the exact comparison of the results. Microplastic levels in the sediment samples of this study were comparatively lower than other rivers including Nakdong (Eo et al., 2019), Pearl (Lin et al., 2018), Rhine (Klein et al., 2015) and Shanghai rivers (Peng et al., 2018), but comparable to the Antua (Rodrigues et al., 2018) and Ottawa Rivers (Vermaire et al., 2017). According to these results, river hydrodynamics can have an effect on relatively low concentrations of microplastics in river sediments due to resuspension of the particles.

Table 2
Comparison of microplastic concentrations in this study with other rivers.

Rivers	Mesh Size (μm)	Mean ± SD		Reference
		Water (items L^{-1})	Sediment (items kg^{-1})	
Ergene River, Turkey	45	4.65 ± 2.06 to 6.90 ± 5.16	97.90 ± 71.72 to 277.76 ± 207.21	This study
Mersin Rivers, Turkey	26	0.29 ± 0.06	–	Özgüler et al. (2022)
Seine River, France	80	0.11 ± 0.09	–	Dris et al. (2018)
Pearl River, China	50	8.9 to 19.86	–	Yan et al. (2019)
Qiantang River, China	45	1.18 ± 0.27	–	Zhao et al. (2020)
Yangtze River, China	50	2.52 ± 0.91	–	Wang et al. (2017)
Ciwalengke River, Indonesia	1.2	5.85 ± 3.28	303 ± 159	Alam et al. (2019)
Nakdong River, South Korea	20	0.29 ± 0.08 to 4.76 ± 5.24	1970 ± 62	Eo et al. (2019)
Antua River, Portugal	55	0.31	318.67	Rodrigues et al. (2018)
Ottawa River, Canada	100	1.35×10^{-3}	220	Vermaire et al. (2017)
Shanghai rivers, China	1	–	802 ± 594	Peng et al. (2018)
Pearl River, China	20	–	1669	Lin et al. (2018)
Rhine River, Germany	63	–	861	Klein et al. (2015)

3.2. Microplastic abundance and river characteristics

Currently, there is little knowledge on the relationship of microplastic abundance with river morphology and hydrodynamics. In this study, Pearson correlation tests were conducted to determine if there was a relationship between microplastic levels and the river's hydrodynamical variables including flow rate, stream depth and channel width. The test results are given in Table 3. For water samples, results showed a moderately positive correlation between the number of microplastics and stream depth. This can be explained by the different flow regimes in the river and associated dispersion of microplastics. Bed shear stress is higher in deep water, which generally results in elevated levels of turbulence that can increase the transport of sediment particles (Richards, 1982; Besseling et al., 2017). Indeed, once bed shear stress exceeds the critical shear stress, sediment particles begin to be resuspended (Waldschläger and Schüttrumpf, 2019). He et al. (2021) also indicated the lower deposition of microplastics onto sediment in the

Table 3
Pearson Correlation coefficients (r) between river hydrodynamical variables and microplastic abundance.

Hydrodynamical Parameters	Pearson's r	F	df	p
Water	items L^{-1}			
Flow rate (m s^{-1})	0.17	0.59	19	0.45
Stream depth (m)	0.51*	6.28	19	0.02*
Channel width (m)	−0.45*	4.54	19	0.04*
Sediment	**items kg^{-1}**	***F***	***df***	***p***
Flow rate (m s^{-1})	0.21	1.35	29	0.26
Stream depth (m)	−0.19	1.00	29	0.33
Channel width (m)	0.13	0.52	29	0.48

* indicates significance with $p < 0.05$.

Brisbane River mouth (Australia) could be due to the higher bed shear stress and dispersion of particles in that location, where water depth was much higher than in river upstream. Moreover, dispersion of particles increases with expanding channel width, which may cause dilution of microplastic concentrations. On the other hand, a moderately negative correlation was defined between the microplastic abundance and channel width, as a result of Pearson correlation tests. Coherently, it was expected to find a relationship between river morphological variables and sediment microplastic levels; yet, no significant correlations between these variables and sediment microplastic levels were observed. This may be due to the fact that ANOVA test did not define a statistically significant spatial distribution for the sediment samples. Likewise, statistical analyses implemented by Nel et al. (2018) did not find a significant relationship of sediment microplastic concentrations with stream depth and channel width.

Previous studies demonstrated a considerable relationship between microplastic abundance and flow rate (Kapp and Yeatman, 2018; Nel et al., 2018). However, in this study, statistical analyses did not exhibit a meaningful correlation between flow rate and microplastic abundance, either for water or sediment (Table 3). This may be because no significant spatiotemporal changes were observed in the flow rates of six different sampling locations. Hence, the longer-term continuous sampling and measurements are required to monitor remarkable changes in river flow and exactly understand whether flow rate influences microplastic levels in the Ergene River.

3.3. Shape, size, color, and type of microplastics

Particle density, shape and size distribution are dynamically changing parameters that strongly affect the sinking velocity, thereby the fate of microplastics (Enders et al., 2015; Kooi et al., 2016; Kowalski et al., 2016). Here, microplastic particles extracted were categorized according to their shapes as fiber, hard fragment, soft fragment, pellet, foam and other (rubber, film, etc.), which are shown in Fig. 2.

Different sources of microplastics cause them to occur in various shapes in the environment (Klein et al., 2015). In total, fibers (88%) were dominant in water samples, followed by soft fragments (4%), hard fragments (4%), pellets (2%), foams (1%), rubbers (<1%) and other (<1%). Plastic particles that could not precisely be identified were categorized as 'Other'. Although fibers were dominating shape also in the sediment samples (70%), hard fragments had a considerable proportion (23%) in sediment. Furthermore, a small number of soft fragments (5%), pellets (2%) and rubbers (<1%) were detected in the sediment samples. The spatiotemporal distribution of microplastic shapes in water and sediment is given in detail in Fig. 3a and b.

To date, fibers have generally been the dominantly found microplastic shape in aquatic environments (Browne et al., 2011), most likely due to the continual abrasion of synthetic textiles and laundry wastewater release (Napper and Thompson, 2016), as well as atmospheric fallout (Dris et al., 2016). Textile industries have also been reported as a significant point source of synthetic fibers (Deng et al., 2020; Cordova et al., 2022). From this viewpoint, fiber levels detected in water and sediment samples were not surprising, as, in our study area, textile industries and spinning mills were intensively active, especially within the Çorlu District and the north-eastern part of the Ergene sub-basin (upstream of Site 3). Besides domestic sewage, the river has been receiving a considerable amount of industrial wastewater from these regions for many years. As discussed in Section 3.1, notably higher amounts of microplastics were detected in water samples of Site 3, the closest location to the aforementioned industrial zones, in which the number of fibers was also extremely higher, compared to the samples of the other sites. Indeed, almost no other shape of microplastics than fiber was detected in the water and sediment samples from Site 3. The proportion of fibers was found higher both in the samples in the fall than those in the spring. This can be attributed to the differences in meteorological conditions, such as higher wind speed and altered wind direction on the day of sampling in September, discussed in Section 3.1. Indeed, Bullard et al. (2021) suggested that microplastic enrichment by wind was higher for fibers than pellet shaped particles, which could explain the increased number of fibers in the samples collected in the fall. Also, in this study, the second sampling (Sep 30, 2020) was carried out during the Covid-19 pandemic. Recent studies identified disposable face masks as a potential source of synthetic fibers in the environment (Fadare and Okoffo, 2020; Shen et al., 2021). Thus, an increase in fiber levels may be due to changing habits such as frequent cleaning and laundry and the use of single-use face masks during the Covid-19 pandemic.

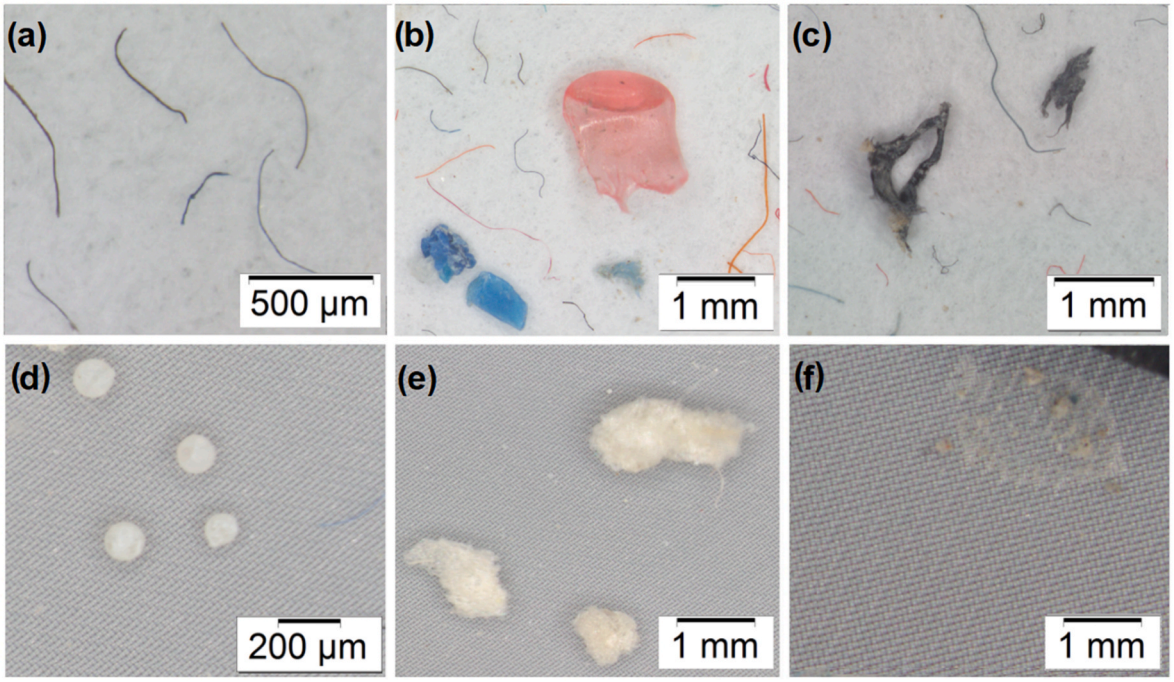

Fig. 2. Microscopic images of microplastics: (a) fibers, (b) hard fragments and fibers, (c) soft fragments and fibers, (d) pellets and fibers, (e) foams, and (f) rubber.

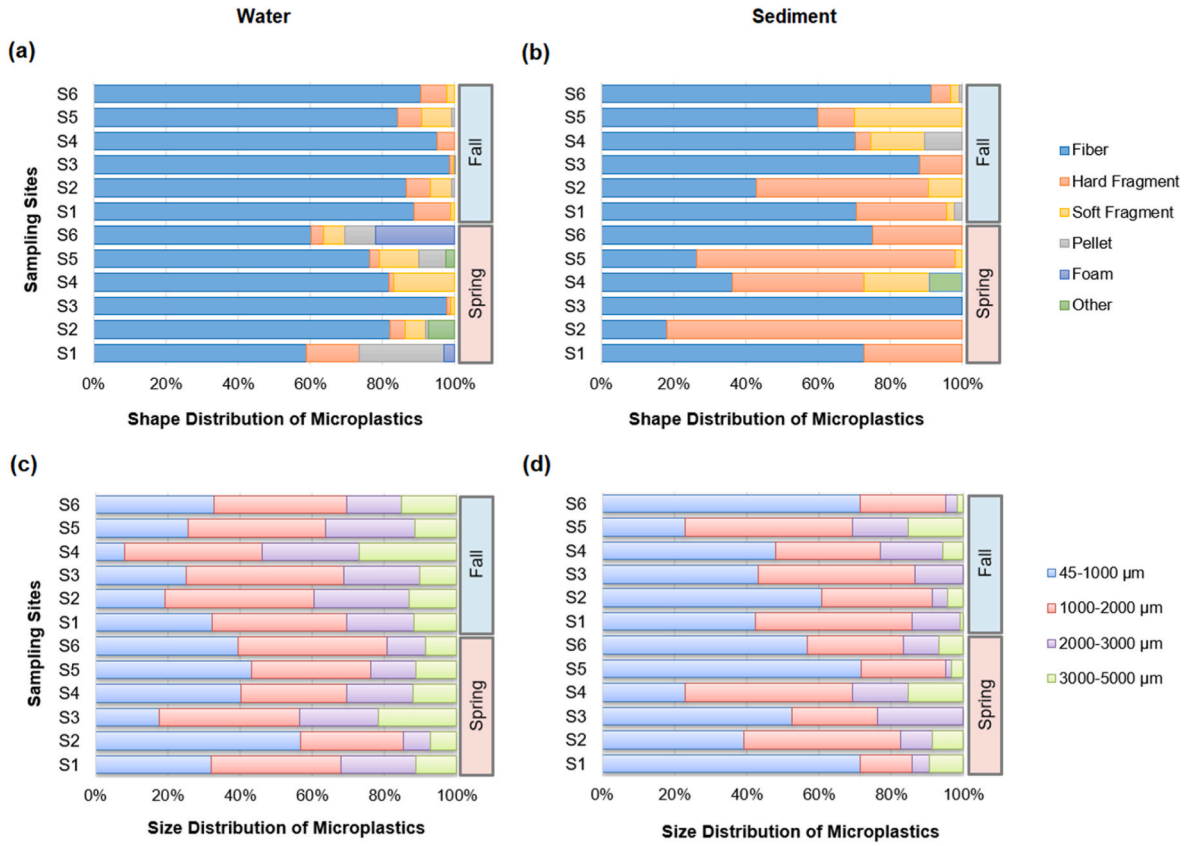

Fig. 3. Percentage distribution of microplastics by (a) shape in water samples, (b) shape in sediment samples, (c) size in water samples and (d) size in sediment samples.

Fibers were generally followed by hard or soft fragments in all samples, yet their abundance and predominance changed from site to site. Fragments detected in the samples revealed the degradation of plastic debris originated from secondary sources that mostly include anthropogenic activities, such as littering. Waste from various industries, such as plastic, machinery and automotive can be a potential source of hard fragments in the study area. Previous studies suggested that agricultural activities were also a substantial source of microplastics due to the use of plastic products, such as nylon mulch in crop growth (Nizzetto et al., 2016; Rodríguez-Seijo and Pereira, 2019). While the surrounding of Sites 1 and 3 mostly include industrial and residential areas, downstream of the river (Site 2 and from Site 4 to 6) is surrounded by farms and cultivated lands. Thus, some of the soft fragments (nylon particles and polyethylene greenhouse coverings) observed in these sites can be attributed to agricultural activities in the neighboring area. On the other hand, relatively low numbers of foam and rubber were detected in the samples. Foams were observed only in the water samples of two sites (Sites 1 and 6), whilst only one rubber was observed in all samples, which was detected in the sediment sample of Site 4. A small number of pellets were detected both in water and sediment samples. The existence of pellets is an indicator of primary source microplastics originating from cosmetic and medical products (Zitko and Hanlon, 1991; Patel et al., 2009). Primary microplastics most likely enter the aquatic environment through domestic wastewater discharge or spillage of plastic pellets used in industries (Gregory, 1996). Ergene River receives both domestic sewage and wastewater from Çorlu and Ergene OIZs near the study area, where several plastic and cosmetic industries, probably using plastic pellets in production, are located. However, a predominance of fibers and fragments suggests that microplastic pollution in the Ergene River and Çorlu Stream mostly originates from secondary source microplastics as a result of the abrasion of textiles and fragmentation of larger plastic items.

Here, microplastics were also categorized according to their sizes. Being different from previous studies (Wang et al., 2017, 2021; Wu et al., 2020), a higher a bundance of medium-sized microplastics ranging between 1000 and 2000 µm (38%) was observed in the water samples, which was followed by small-sized microplastics between 45 and 1000 µm (31%) and larger particles between 2000 and 3000 µm (18%) and 3000–5000 µm (13%), respectively. A similar outcome was observed in the Netravathi River, India, where the authors attributed their results to the shorter residence time of plastics and associated lower degradation, due to high flow rates (Amrutha and Warrier, 2020). In our study area, the mean flow rate of the river ranged from 0.258 to 0.748 m s^{-1} (Table S2). Therefore, the predominance of medium-sized particles in water may depend on other factors such as the source or characteristics of the plastic materials. Heat, sunlight and well-aerated conditions are ideal for the fragmentation of plastics (Harshvardhan and Jha, 2013). However, the Ergene River has very low dissolved oxygen values and dark color because of heavy industrial pollution for many years. In field measurements, it was observed that dissolved oxygen levels may decrease up to 0.1 mg L^{-1} (Table S2). This may slow down the fragmentation process and thus, influence the size of the particles, both due to decreased light penetration and the presence of anoxic conditions. Moreover, as previously mentioned, effluents of WWTPs can be considered the major sources of microplastics along the upstream of sampling sites. Although characteristics of microplastics in WWTPs may vary by location, recent studies indicated that the average particle size increased from influent (1111 µm–1135 µm) to effluent (1221 µm–1309 µm) of WWTPs, probably due to more effective removal of smaller particles in the biological treatment step (Akarsu et al., 2020; Vardar et al., 2021). Therefore, it becomes quite likely that particle size distribution in the river would depend on the properties of microplastics in WWTPs' effluents. However, though WWTPs located near our study area have similar treatment technologies to the above-mentioned plants,

it is not possible to obtain a precise outcome with the data produced in this study. To understand these complex factors, future work should undertake a more intensive sampling scheme to determine the characteristics of microplastics in wastewater effluents and monitor the source-to-sink variation of particle sizes along the Ergene River. On the other hand, the majority of the microplastics in sediment samples were surprisingly smaller than those detected in water samples. In total, the dominant size range was found to be between 45 and 1000 μm (55%), followed by 1000–2000 μm (32%), 2000–3000 μm (9%) and 3000–5000 μm (4%). These results for sediment samples are congruent with the previous studies (Li et al., 2020; Wu et al., 2020). However, here, the difference between the two compartments suggests higher fragmentation of microplastics in sediments than those on water surfaces. This may be because of the higher residence time of the microplastics deposited and buried deeper in the sediments allowing for fragmentation. Additionally, smaller-sized microplastics are more convenient both for homo- and hetero-aggregation than those with larger sizes, due to their lessened stabilities and adsorption on the surface of large suspended solids (Besseling et al., 2017; Li et al., 2019; Song et al., 2019). It is known that aggregation can increase the size and density of microplastics, which can accelerate the deposition of particles onto sediments (Ballent et al., 2016). Research also revealed that deposition as a result of aggregation can cause a decrease in the fraction of small-sized microplastics (<1000 μm) in the surface water (Cózar et al., 2011; Yan et al., 2021). Therefore, aggregation of small microplastics with each other or suspended solids may cause small-sized particles to be dominant in the sediment samples of the Ergene River.

Spatiotemporal distribution of particle sizes is given in detail in Fig. 3c and d. A slight increase in the proportion of larger particles (>1000 μm) was observed in the water samples collected in the fall, whilst no significant temporal variation was observed in the sediment samples. This may be due to the temporal variations in the characteristics of plastic materials and environmental factors, such as flow regime, sunlight, temperature and dissolved oxygen levels, which govern the fragmentation processes of microplastics.

The colors of microplastics can offer information on potential sources and weathering process of particles (Wicaksono et al., 2021). Identification of microplastic color is also important for understanding their bioavailability, since aquatic organisms may accidently ingest colored microplastics during feeding, due to their resemblance to their prey. Previous studies suggested that light-colored microplastics, such as white, transparent and blue could easily be misidentified and ingested by fish (Romeo et al., 2015; Ory et al., 2017). In this study, different colors of particles were observed in the overall samples, yet they were not equally distributed. Most of the particles were black (49%) and blue (25%) in the water samples, followed by red (14%), white (5%), brown (2%), transparent (1%), green (1%), orange (1%), purple (1%), pink (<1%), grey (<1%) and yellow (<1%). Likewise, dominant particle colors detected in the sediment samples were black (39%), blue (18%), transparent (15%) and red (14%). Besides, white (5%), green (3%), brown (2%), pink (1%), purple (1%), grey (1%) and yellow (1%) particles were also observed in the sediment samples. As mentioned above, the proportion of fibers was quite high in both seasons, which may affect the color diversity in the samples to some extent causing some colors to be similar and dominant (Wang et al., 2021). Therefore, color cannot solely be sufficient to identify potential sources of microplastics. Considering the industrial activities in the study area, it is quite likely that the main sources of colored fibers are textiles and waste from spinning mills. Other colored microplastics can be originated from plastic production factories, paints and packaging waste. White and transparent microplastics are generally associated with plastic bags and food packaging materials. Besides, microplastic colors are faded by environmental factors, which results in the occurrence of light-colored particles. In this study, the distribution of microplastic colors is similar both in water and sediment samples, yet a higher proportion of transparent particles in sediment samples is most likely due to a higher abundance of transparent hard fragments in sediment, as well as higher residence time and weathering process. The distribution of microplastic colors is shown in Fig. 4 for the water and sediment samples.

Polymer types were detected by analyzing 200 randomly selected particles with Raman Spectroscopy. 150 particles were successfully analyzed, whilst the remaining 50 particles could not be identified due to no match or below 60% similarity with the reference spectra. Amongst 150 particles, 134 of them were identified as microplastic (89%), whereas 16 particles were non-plastic (11%) including nine silicon dioxide (SiO_2) particles, six natural fibers (cotton and modal fibers), and one plastic additive (mortoperm blue). The majority of the particles that could not be identified by Raman were fiber. Although it was suspected that some of these may be non-plastic fibers, such as cotton, viscose and wool fibers, further analysis is needed before a definite conclusion. Previous research indicated that dyed fibers including cotton, viscose, acrylic and wool can have different peaks according to their colors and can be detected more clearly at different Raman laser wavelengths varying from 514 nm to 830 nm (Thomas et al., 2005; Was-Gubala and Machnowski, 2014; Buzzini and Massonnet, 2015; Prego Meleiro and García-Ruiz, 2016). Hence, future work should also focus on the relevant particles considering these factors to obtain a precise outcome about the type of these non-identified particles.

According to the results, many of the plastic particles in water were polyethylene terephthalate (PET, 28%) and polyamide (PA, 27%), which were followed by polyethylene (PE, 19%), polystyrene (PS, 19%), polypropylene (PP, 5%), and polyisoprene (PI, 2%). In sediment samples, PS (56%) was predominant, followed by PE (20%), PA (12%), PP (8%), and other (4%) including one polysulfone/polyamide composite polymer. The results of the Raman analysis are given in Fig. 5. Examples of plastic and non-plastic spectra with the reference spectra are given in Fig. S1-S11. PS, PA, PET and PE were the four most abundant polymers within the samples in total. PS is a hard and resistant thermoplastic resin, with a variety of applications from foam products used in disposable food containers to rigid materials in the electronics and automotive industries. The number of foam particles was relatively low, but many of the hard fragments found were identified as PS, which may have originated from the waste of machinery and automotive industries located in the neighboring area or degradation of plastic litter. PET is an aliphatic polyester that is used in the production of everyday items including clothes, carpets and other home textile products and water bottles, and it dominates synthetic fiber production for around 60% by volume (Radzi et al., 2021). PA (or nylon) is usually used in manufacturing items that require strength and flexibility, such as textile products, seatbelts, airbags, fishlines and parts of machines. After PET, PA is one of the most used polymers in the production of synthetic fibers (Dalla Fontana et al., 2020). Therefore, it can be concluded that the majority of PET and PA, particularly in shape of fiber, originated from the textile industries or discharges from WWTPs from the upstream river. It is likely that other PA particles came from the wastewaters of various industries manufacturing part of machines or automobile. PE is also a versatile polymer, mainly used for packaging and shopping bags, and can also be used in water pipes, housewares, insulating materials, agricultural mulches, etc. In our study area, the industries producing such materials can be sources of PE particles, yet, plastic mulches used in agricultural lands around the downstream of the Çorlu and Ergene tributaries can be another source of detected PE, as discussed previously. These sources derive secondary microplastics, which are assumed to constitute the majority of microplastics in the environment. PS and PE particles are also used in cosmetic and medical products in pellet form as primary microplastics (An et al., 2020). It is worthwhile to note that, some of the polymers mentioned above could originate from the degradation of anthropogenic litters, so it becomes necessary to implement a more comprehensive investigation to assess the exact contribution of each source to microplastic contamination in the river.

Amongst these polymers, PET has the highest density (1.38 g cm^{-3}), followed by PA (1.01–1.08 g cm^{-3}), PS (1.05 g cm^{-3}) and PE (0.88–0.96

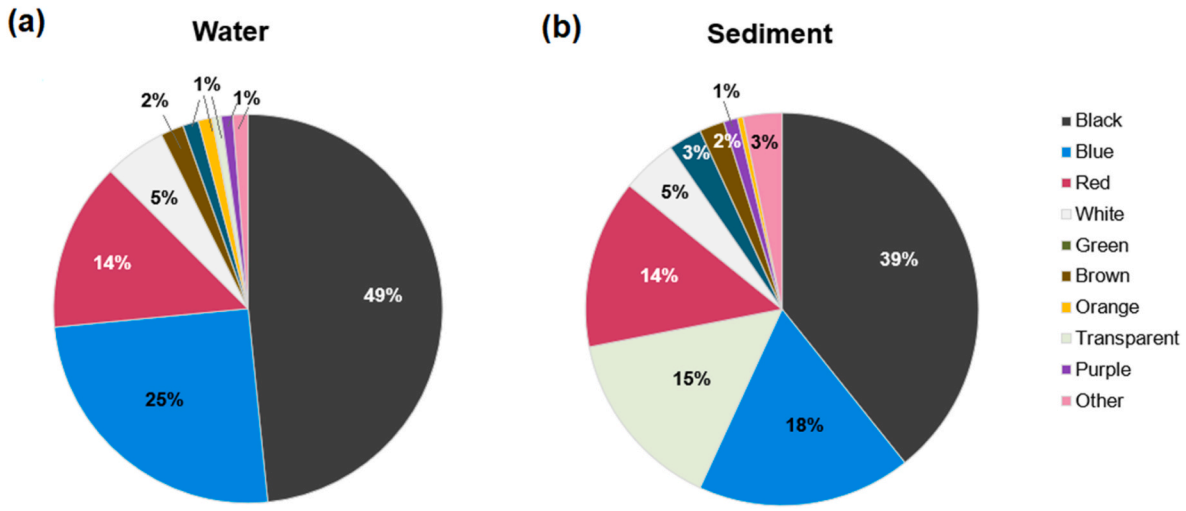

Fig. 4. Microplastic color distribution: (a) water samples and (b) sediment samples.

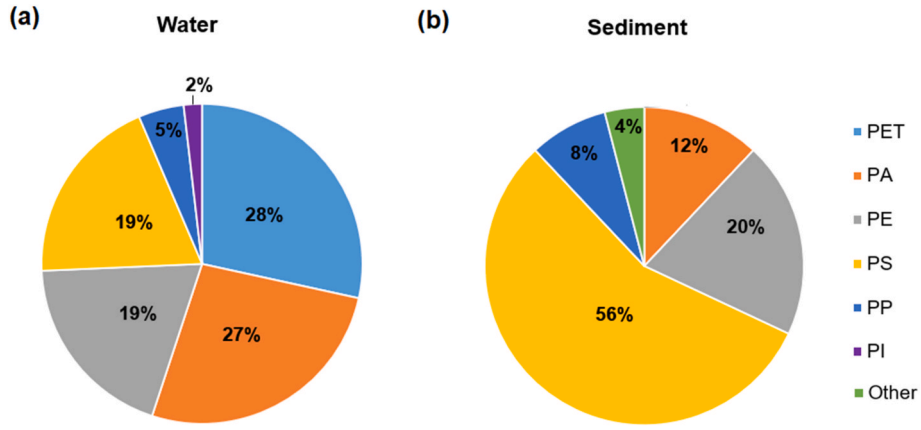

Fig. 5. Microplastic types in the river samples: (a) Polymer distribution in water, (b) Polymer distribution in sediment.

g cm^{-3}). According to our results, despite the higher densities of PET and PA than water, these polymers were identified in the water samples compared to other low-density plastics, such as PE. This can be attributed to river's hydrodynamical conditions, which may cause resuspension of denser polymers by turbulence. In addition, the occurrence of excessive levels of textile fibers in the water samples, which generally constitute PET and PA, results in these polymers being the most abundant types in water samples. PET is the most used material in manufacturing synthetic fibers, accounting for around 60% by volume (Radzi et al., 2021). Therefore, predominance of PET and PA in the production of plastic items in the study area and associated higher release of these particle types to the river, as well as river hydrodynamics can results in higher levels of these polymers in the surface water.

4. Conclusions

This study investigates the spatiotemporal distribution of microplastics in surface water and sediment of an industrially polluted river (i.e. the Ergene River, Turkey). According to the results, water samples had an overall average concentration of 4.65 ± 2.06 and 6.90 ± 5.16 items L^{-1} (mean ± standard deviation, n = 12) for the May 2019 and September 2020 periods, respectively, while those were 97.90 ± 71.72 and 277.76 ± 207.21 items kg^{-1} (n = 18) for sediment samples, respectively. Statistical analyses suggested a moderately positive correlation between microplastic levels in water and stream depth, whilst a moderately negative correlation was found between microplastic abundance and channel width. Fibers were found as the dominant shape both in water (88%) and sediment samples (70%). In total, medium-sized microplastics ranging between 1000 and 2000 μm (38%) were found as the most abundant particles in surface water, whilst the dominant size range was 45–1000 μm (55%) in sediment. Majority of the particles were black, which constitute 49% and 39% of all microplastics detected in water and sediment, respectively. According to the Raman analysis, most of the particles were PET (28%) and PA (27%) in water samples, while PS (56%) were dominating the sediment samples.

Rivers are both transport pathways and sinks of microplastics. This study focuses on the upstream of the Ergene River, an area of intensive industrialization, being close to the potential sources of microplastics (e.g. OIZs and WWTPs). Compared with the microplastic levels in other rivers (e.g. Yangtze, China and Mersin rivers, Turkey), Ergene River has excessive levels of microplastics, especially the upstream being a significant source of microplastic pollution. Domestic sewage and effluents of various industries, especially textile, are the main contributors of microplastics in the study area. Agricultural plastics, anthropogenic litters and other industrial effluents, such as plastic, machinery and automotive are also sources of microplastics, particularly in the form of fragments. Evidently, more comprehensive and holistic research should be adopted to determine the exact contribution of each source to the total amount of microplastics in the study site. In this context, accurate and effective management strategies, such as improved treatment technologies and waste management strategies can be developed for

mitigation of microplastics in the Ergene River. Further research should also investigate the source-to-sink variation of microplastics to precisely understand microplastic levels along the Ergene River and riverine loading to the sea.

Credit author statement

Zeynep Akdogan: Conceptualization, Methodology, Investigation, Writing - Original Draft. **Basak Guven:** Supervision, Funding acquisition, Writing - Review & Editing. **Ahmet E. Kideys:** Investigation, Writing - Review & Editing.

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envres.2023.116500.

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