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Araştırma Makalesi / Research Article

Microplastic pollution profile and potential ecological risk of in Aksu Stream (Giresun, Türkiye)

Arzu AYDIN UNCUMUSAOĞLU[1*]

Abstract

The microplastic (MP) profile in water and sediment samples collected from five stations along Aksu Stream, which serves as a drinking water source for Giresun province, was investigated. The presence and characterization of MPs in the samples were determined using both microscopic and ATR-FTIR spectroscopy techniques. The study revealed 910 items kg^{-1} of MPs in sediment samples, while 0.65 items L^{-1} were found in water samples. The dominant color of MPs observed in the study was transparent. The most prevalent size range of MPs was between 0-50 µm, and the dominant shape in the polymer analysis of MPs collected from Aksu Stream was fiber. According to FT-IR analysis results, polyethylene (PE) and polypropylene (PP) were the most abundant MP polimer types detected. When assessing the potential ecological risk of MPs, it was determined that the stream falls into damage and risk categories III (High) and IV (Hazard). This research adds to the expanding body of evidence indicating that microplastic contamination is pervasive in freshwater environments. It is urgent to reduce MP waste generation in order to protect aquatic ecosystems and human health.

Keywords: Microplastic, Fresh water, Sediment, Potential ecological risk.

Aksu Çayı'nın (Giresun, Türkiye) Mikroplastik Kirlilik Profili ve Potansiyel Ekolojik Riski

Öz

Giresun ili içme suyu teminini sağlayan, Aksu Deresi boyunca beş istasyondan toplanan, su ve sediment örneklerindeki mikroplastik (MP) profili araştırılmıştır. Örneklerde tespit edilen MP'lerin varlığı ve karakterizasyonu hem mikroskobik hem de ATR-FT-IR spektroskopisi teknikleri kullanılarak belirlenmiştir. Araştırmada, sediment örneklerinde 910 parça kg^{-1} MP tespit edilirken, su örneklerinde 0.65 parça L^{-1} MP bulunmuştur. Çalışmada gözlenen MP'lerin baskın rengi transparandır. MP'lerin en baskın boyut aralığı 0-50 µm arasında bulunurken ve Aksu Deresi'nden toplanan MP'lerin polimer şekli analizinde baskın grup olarak fiber bulunmuştur. FT-IR analiz sonuçlarına göre, PE ve PP tespit edilen en bol MP polimer türleri olmuştur. MP'lerin potansiyel ekolojik riski değerlendirildiğinde, derenin III (Yüksek) ve IV (Tehlike) hasar ve risk kategorilerine girdiği belirlenmiştir. Bu araştırma, mikroplastik kirliliğinin tatlı su ortamlarında yaygın olduğunu gösteren kanıtların artmasına katkıda bulunmaktadır. Sucul ekosistemlerin ve insan sağlığının korunması için MP atık üretiminin azaltılması acilen gerekmektedir.

Anahtar Kelimeler: Mikroplastik, Tatlı su, Sediment, Potansiyel ekolojik risk.

[1*]Giresun University, Environmental Engineering Department, Giresun, Türkiye, arzu.a.uncumusaoglu@gmail.com

[*]Sorumlu Yazar/Corresponding Author
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1. Introduction

Plastic, an indispensable element of modern life, is produced in quantities exceeding 335 million tons annually worldwide (Tiseo, 2021). Recently, the COVID-19 pandemic, which has had a global impact, has also unfortunately accelerated plastic usage due to the demand for hygienic products such as gloves and face masks. Despite efforts to reduce plastic consumption, their versatility, lightness, durability, and low production costs have led to an increase in their use in our daily lives (Kulkarni and Anantharama, 2020).

The buildup of plastic waste in both aquatic and terrestrial environments endangers ecosystems and poses risks to both wildlife and human health. Plastics are subject to numerous degradation processes over time, such as hydrolysis, photodegradation, thermal oxidation, mechanical abrasion, and biodegradation (Andrady, 2015). These processes result in the formation of smaller plastics known as microplastics (MPs) and even nanoplastics (NPs). MPs, defined as plastic particles smaller than 5 mm, encompass all types of plastics present in the environment regardless of their chemical composition (Masura et al., 2015).

MPs, due to their potential environmental risks, have been detected in numerous settings and can originate from human activities, terrestrial operations, atmospheric deposition, and activities within aquatic ecosystems. Sources of MPs include the textile industry, land and air transportation, tourism, shipping, aquaculture, and commercial fishing operations (Daana et al., 2018; Huang et al., 2023). MPs, which can be regular or irregular in shape, are insoluble in water and can arise from primary or secondary sources (Frias and Nash, 2019). They can disperse into the environment directly through wind and rainfall or indirectly via wastewater treatment plants (WWTPs), sewage sludge, irrigation, agricultural fields, landfills, and urban runoff (Xu and Ren, 2021; Xu et al., 2023). In addition to marine habitats, MPs have been identified in freshwater environments (rivers and lakes), agriculture, sewage sludge, wastewater systems, and the atmosphere (Gatidou et al., 2019; Chen et al., 2020; Wright et al., 2020; Mutlu et al., 2023; İpek et al., 2024).

The uncontrolled and unconscious discharge of wastewater into aquatic environments poses a substantial threat to these ecosystems (Dris et al., 2016; Liu et al., 2019; Vaid et al., 2021). Various materials accumulate in water bodies, leading to sediment formation. This sediment, characterized by different properties depending on the aquatic environment, results from geographical and natural erosion, the settling of organic and inorganic materials, and the accumulation of dead algae at the bottom (Aydın and Sunlu, 2004; Çil et al., 2023). Due to the water cycle, pollutants gradually settle into the sediment over time.

The most commonly used plastics include polypropylene (PP), polystyrene (PS), polyethylene terephthalate (PET), polyester, polyethylene (PE), polypropylene (PP), polyamide (nylon), and

polyvinyl chloride (PVC). Over the past five decades, plastic usage has surged due to industrial growth, resulting in an increased amount of plastic waste in our environment. Despite the recyclability of most plastic waste, it is estimated that 5-12 million tons of plastic waste enter aquatic environments annually (Baalkhuyur et al., 2020). As plastic production continues to rise, so do concerns about plastic accumulation in coastal and marine ecosystems. More than 80% of plastic waste reaching the oceans is terrestrial in origin, with a significant portion comprising MPs from rivers (Alprol et al., 2021; Tokatli et al., 2024).

Plastic debris in natural environments can restrict the movement of animals such as waterfowl, fish, and sea turtles, and can even cause drowning. Ingested plastics can lead to digestive system disorders in animals, resulting in weight loss and developmental issues. The accumulation of plastic marine debris on the seabed presents another level of danger, hindering gas exchange and threatening the survival of benthic organisms (Akkan et al., 2023; Bayhan and Aydin Uncumusaoglu, 2024).

This study aims to elucidate the profile of MPs found in water and sediment samples from the Aksu Stream within Giresun province and to evaluate their potential ecological risks. The findings are expected to enhance our understanding of MP pollution in the region and aid in the development of necessary mitigation measures.

2. Materials and Methods

2.1. Study area

The Aksu Stream, situated in the Eastern Black Sea Region, discharges into the Black Sea within the boundaries of Giresun province. The watershed area draining into the Aksu Stream covers 731 km², with a perimeter of 129.4 km, a main channel length of 58.8 km, and a gradient of 4.5%. Additionally, the median elevation of the basin is 2102.3 meters, the stream gradient is 4, the drainage density is 0.48 km-1, and the channel frequency is 0.16 channels/km². The total surface water potential of Giresun province is 4373 $hm^3.year^{-1}$, of which 562 $hm^3.year^{-1}$ is provided by the Aksu Stream. The alluvium derived from the Aksu Stream also forms the groundwater aquifers of Giresun. The Etbaşoğlu and Duroğlu wells, which supply drinking water to the central district of Giresun, are supported by the Aksu Stream.

2.2. Sampling of Water and Sediment

The sampling event was performed on May 27, 2021 in the Aksu Stream. Samples in S1-S5 were collected at the river bank due to ease of shoreline access (Figure 1-2).

Figure 1. Map of the study area and stations

Figure 2. Photographs from sampling stations

Samples of sediment and water were taken from five designated stations along the Aksu Stream to fully represent sampling from three points, consisting of two sides and a central point of the stream. Sediment samples were collected by scanning 1m^2 areas with a metal shovel to a depth of 5 cm from the surface (Zhou et al., 2021). Wet sediment samples were transported to the laboratory in glass jars. Sampling was conducted on days with no precipitation for at least three days. Water samples were also taken from each station at three points, using a 5-liter metal bucket, and were filtered through a plankton net with a mesh size of 35 µm, totaling 100 liters of stream water, and stored in glass jars for transportation to the laboratory (Masura et al., 2015).

2.3. Microplastic Analysis

MPs particles were isolated from samples using wet peroxidation (WPO) and density separation techniques (Masura et al., 2015). Water samples brought to the laboratory underwent wet peroxidation (WPO) using iron sulfate ($FeSO_4$-$7H_2O$) solution as a catalyst and 30% hydrogen peroxide (H_2O_2) solution. The oxidation process was conducted on a hotplate at 60-70°C with 80 rpm for 6 hours (Masura et al., 2015). The protocols for sample preparation and microplastic identification were modified from a National Oceanic and Atmospheric Administration (NOAA) protocol (Masura et al., 2015). After digestion, particles were extracted using density separation. To separate MPs from other components, a sufficient amount of NaCl (1.2 g cm-^{3}) was added to the solution and allowed to settle in an Imhoff funnel overnight. Subsequently, the MPs were filtered using glass filter paper (pore size 1.2 µm, Whatman, GE Healthcare, UK) and a vacuum pump. The glass filter papers were dried at room temperature in the laboratory (Masura et al., 2015).

Each wet sediment sample brought to the laboratory was initially dried in an oven at 70°C until a constant weight was reached, then sieved through a 5 mm porous steel sieve to remove stones and other residues. Dried sediment samples (50 g) were weighed and mixed with 100 mL of saturated sodium chloride (140 g L^{-1} NaCl) solution and stirred for 15 minutes. Each sample was allowed to settle for 24 hours. The upper phase containing MPs was transferred to a beaker with at least three repetitions. Iron sulfate ($FeSO_4$-$7H_2O$) solution and 30% hydrogen peroxide (H_2O_2) solution were used as the wet peroxidation (WPO) catalyst. The beakers were then subjected to oxidation on a hotplate at 60-70°C with 80 rpm for 6 hours. To separate MPs from other components, a sufficient amount of NaCl (1.2 g/cm^3) was added to the solution and allowed to settle in an Imhoff funnel overnight. The MPs in the obtained mixture were filtered using glass filter paper (pore size 1.2 µm, Whatman, GE Healthcare, UK) and a vacuum pump. The glass filter papers were dried at room temperature in the laboratory.

MPs were examined for size and shape using a stereo microscope (Nikon, Tokyo, Japan) (Hermsen et al., 2017). The largest dimensions of each particle were measured with a high-resolution digital camera attached to the microscope. Plastic-like materials were tested using a hot needle (Bellas et al., 2016). MPs were then sorted into six colors (red, green, blue, black, white, transparent) and visually identified as fragments, fibers, films, foam, or pellets. All particles were preserved for future polymer identification using Fourier transform infrared (FT-IR) spectroscopy.

To minimize airborne contamination from microplastics (MPs), samples were promptly dissected and transferred, while the workspace was sanitized with alcohol beforehand. Nitrile gloves, cotton lab coats, and glassware were consistently utilized throughout the study. Unused equipment was shielded with aluminum foil to prevent contamination. Additionally, blind sample tests were

conducted by placing three filter papers in distilled water in the laboratory for 24 hours, followed by observation under a stereomicroscope to detect any potential MPs contamination. No MPs were found on these filter papers (Zhao et al., 2023; Bayhan and Aydin Uncumusaoglu, 2024).

2.4. FT-IR Spectroscopy Analysis

In this study, Fourier Transform Infrared (FT-IR) spectra were acquired in attenuated total reflection (ATR) mode using a Particle Attenuated Total Reflection-Fourier Transform Infrared (FT-IR) spectrometer (VERTEX 70 Series, Bruker, Germany). The spectral range was set from 4000 to 400 cm-1, with a resolution of 2.0 cm^{-1} and 128 scans for polymer type identification. Spectra data were processed by linear baseline correction and normalization to the highest absorbance value using the "Speaktragryph© version 1.2.14" software. Absorbance spectra were then compared with recommended polymer types in the device library and analyzed based on their similarity (Gedik and Gözler, 2022; Bayhan and Aydin Uncumusaoglu, 2024).

2.5. Potential Health Impact of MPs

The polymer risk index (H) was utilized to assess the ecological risk of microplastics (MPs) in all samples. The potential ecological risk of MPs was evaluated using the polymer hazard index (PHI) method, as applied in previous studies (Lithner et al. 2011; Xu et al., 2018; Ranjani et al., 2021). The potential risks of plastic polymers to the environment and human health can be assessed based on the chemical toxicity of the monomers (Lithner et al. 2011). Table 3 provides the expression of hazard status, scores, and risk categories when evaluating polymer types. The polymer hazard index (PHI) for MPs can be calculated using the following formula:

$$\mathrm{PHI} = \sum PnxSn$$

Here, Pn represents the percentage of each type of MPs polymer detected in each sample, and Sn is the hazard score of the polymers in MPs provided in the study by Lithner et al. (2011). Additionally, the hazard categories and values were evaluated by following the study carried out by Ranjani et al. (2021).

2.6. Data analysis

One-way analysis of variance (ANOVA) was used to compare whether there was a significant difference between the stations according to the MPs abundance of the stations.

Multivariate Hierarchical Cluster Analysis (HCA) technique was used to classify the clusters that may form between the mean MPs abundances of the stations using Ward's method as a similarity measure. The significance level was set at $P < 0.05$. Statistical analyses were performed using SPSS Statistics for Windows, version 25.0 (IBM, USA). Results were expressed as graphs or tables.

3. Findings and Discussion

3.1. Descriptive Statistics on the MPs

In this study, microplastics (MPs) ranging from 1 to 5 mm in size were examined. These MPs were analyzed based on their size, color, and type in both water and sediment samples collected from each station. The analysis revealed a total of 910 pieces of MPs per kilogram in sediment samples across all stations, while 0.65 pieces per liter of MPs were detected in water samples (Figure 3). There was no significant difference in the size of MPs between the stations (one-way ANOVA; $P > 0.05$). However, a significant difference was observed between the stations for black and transparent colors in water (one-way ANOVA; $P < 0.05$), while no significant difference was found for other colors. Regarding the shape of MPs structures, a significant difference was observed for fiber and foam (one-way ANOVA; $P < 0.05$), while no significant difference was observed for other types. There was a significant difference between the stations for green and black colors in the sediment (one-way ANOVA; $P < 0.05$), while no significant difference was found for other colors. There was no significant difference in the shape of MPs structures (one-way ANOVA; $P > 0.05$). No statistically significant differences were observed for the types of polymers (one-way ANOVA; $P > 0.05$).

When the density of MPs according to all stations was analyzed, it was concluded that the highest abundance of MPs was found in S2, followed by stations 1, 3, 4 and 5, respectively. In water samples, the highest abundance of MPs was found in S2, followed by stations 3, 1, 5 and 4, respectively (Figure 3). The highest abundance of MPs in the sediment was found in S1, followed by stations 2, 4, 5 and 3, respectively. In water samples, the highest abundance of MPs was found in S2, followed by stations 3, 1, 5 and 4, respectively. The Aksu stream was exposed to MPs at lower concentrations than the Ergene river, which may be due to the fact that the Ergene river is exposed to the plastic wastes of the human population and industry (Akdogan et al., 2023).

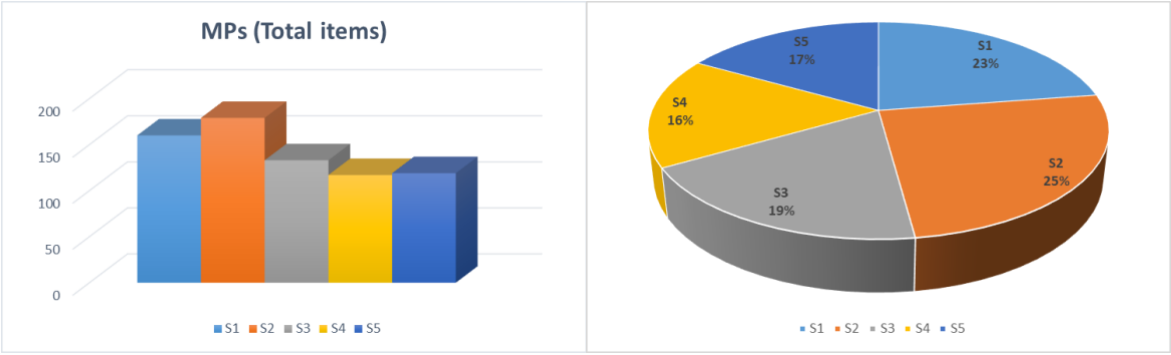
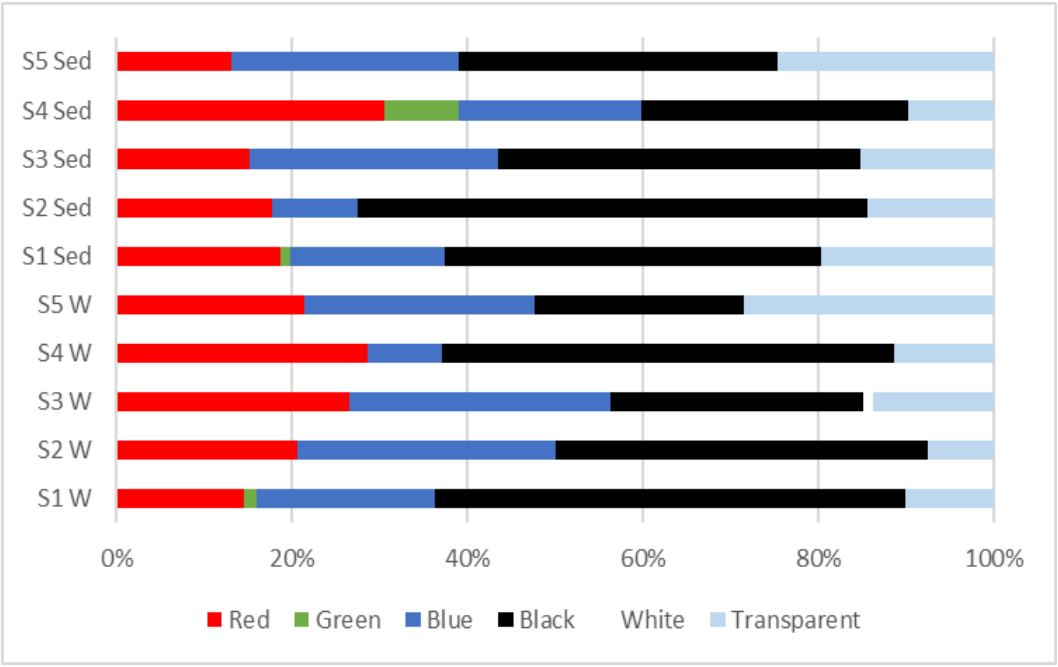


Figure 3. The abundance ratios (%) and items MPs values by the stations (S)

The most dominant color in the MPs of all samples was black, accounting for 41% of the total. This was followed by blue with 22%, red with 21%, transparent with 15% and green with 1% (Figure 4). The most dominant color in Aksu Creek sediment MPs samples was blue with 75%. This was followed by green with 22% and red with 3%. White and transparent colors were not detected in the sediment. Black was again the most dominant color in water as in sediment. This color was followed by blue with 25%, red with 22% and transparent with 13%. White MP was detected in water with one sample (Figure 4). The dominant color detected in the water and sediment in this study, Black, is similar to Ergene River and Batlama Stream (Akdogan et al., 2023; Çebi and Aydın Uncumusaoğlu, 2024).



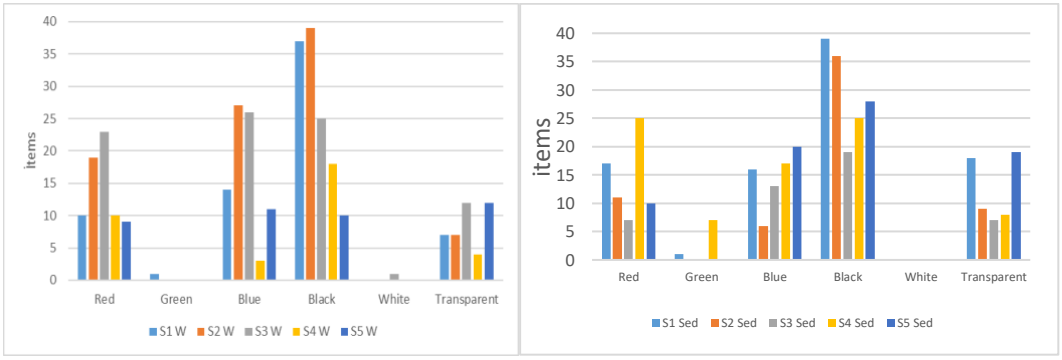
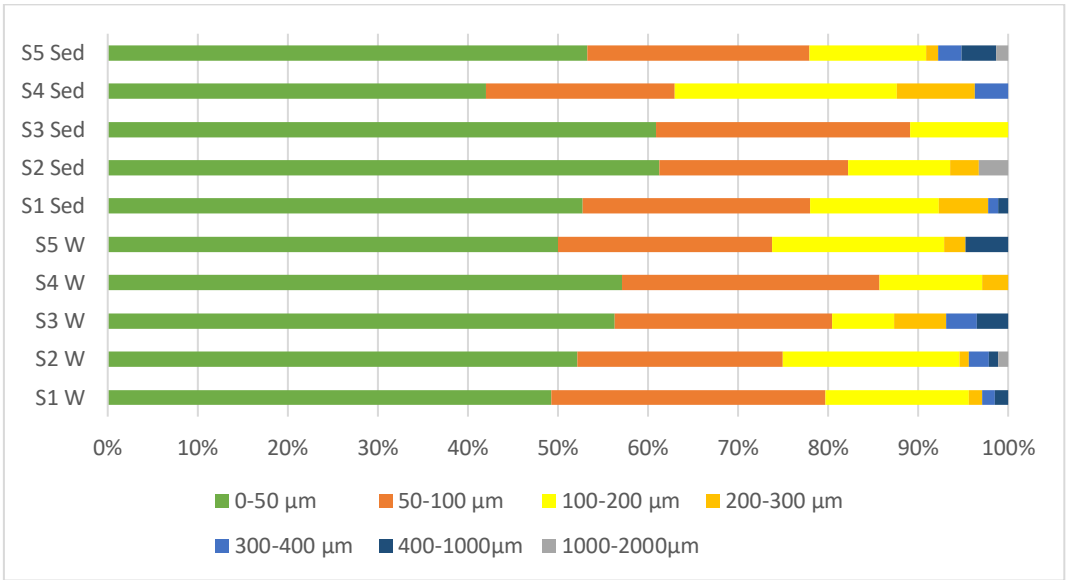


Figure 4. Distribution of MPs by color (%)

The size in the range of 0-50 µm was measured in 53% of all MPs samples. Then, MPs particles in the range of 50-100 µm, 100-200 µm, 200-300 µm and 300-400 µm were measured and identified at the rates of 18%, 10%, 5% and 1%, respectively. It was observed that 66.6% of the most common MPs in the sediment were in the 0-50 µm range, followed by the 50-100 µm range with 19.13%, 100-200 µm range with 9.8%, 200-300 µm range with 3.6% and 300-400 µm range with 0.83%.When the sizes of MPs in water were analyzed, it was found that 75.5% of the most common MPs size was in the 0-50 µm range, followed by 50-100 µm range with 12%, 100-200 µm range with 10%, 200-300 µm and 300-400 µm range with 1.5% (Figure 4). The predominant size range (50-100 µm) observed in this study aligns with findings from research conducted on the Maozhou River, Batlama Stream, and an urban river network in eastern China. In contrast, studies on the Hanjiang River and Yangtze River reported larger predominant sizes (Wang et al., 2020; Çebi and Aydın Uncumusaoğlu, 2024; Fan et al., 2022).



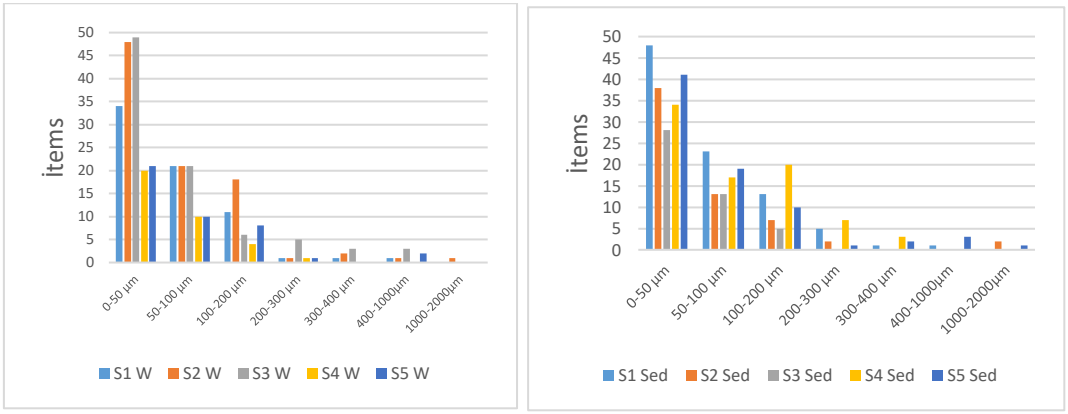
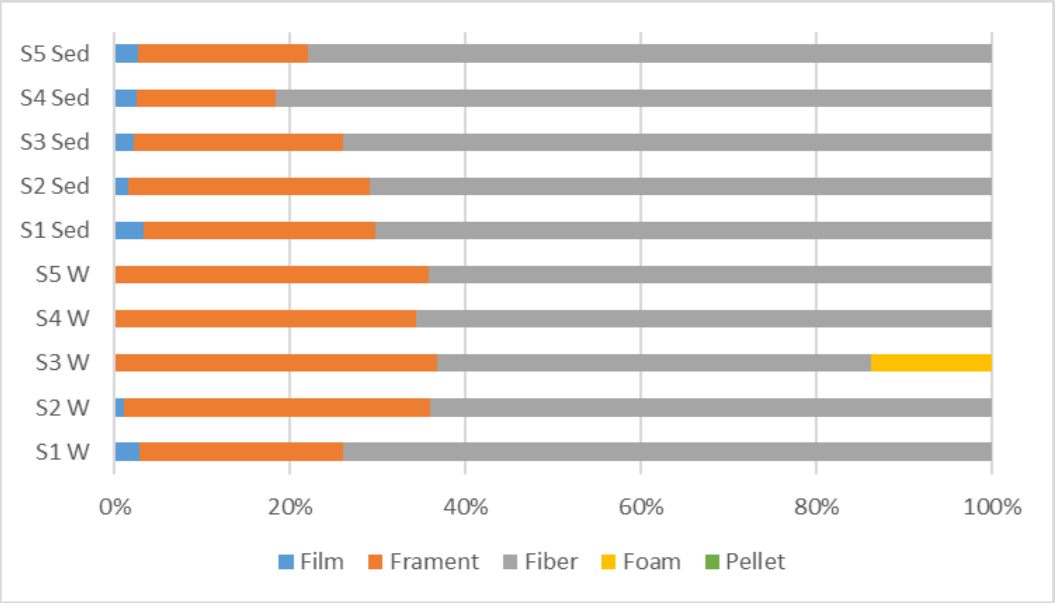


Figure 5. Sizes of MPs

Polymer shapes of MPs samples collected from Aksu Stream were fibers with 69%, followed by particles with 27%, both film and foam with 2% each, and pellets with 22%. In sediment samples, the most dominant MPs shape type was fibers with 75%, followed by plastic fragments with 22%. In water, 62% of the MPs were fibers, 33% were plastic fragments and 4% were foam (Figure 6). Visual representations of MPs shape types are given in Figure 7. Research on microplastic (MP) loads discharged from rivers such as the Hanjiang, Yangtze, Batlama and Manas Rivers, as well as Mersin Bay in the Mediterranean Sea, has shown that fibers are the most common form of MPs, consistent with the findings of this study (Wang et al., 2017; 2020; Çebi and Aydın Uncumusaoğlu, 2024; Özgüler, 2022). However, studies on MPs from the Küçükçekmece River basin, various lakes and river gorges along the Turkish coast of the Black Sea and Jajroud River indicated a dominant particle shape, differing from the results of this study (Çullu, 2021; Öztekin, 2021; Shekoohiyan and Akbarzadeh, 2022).



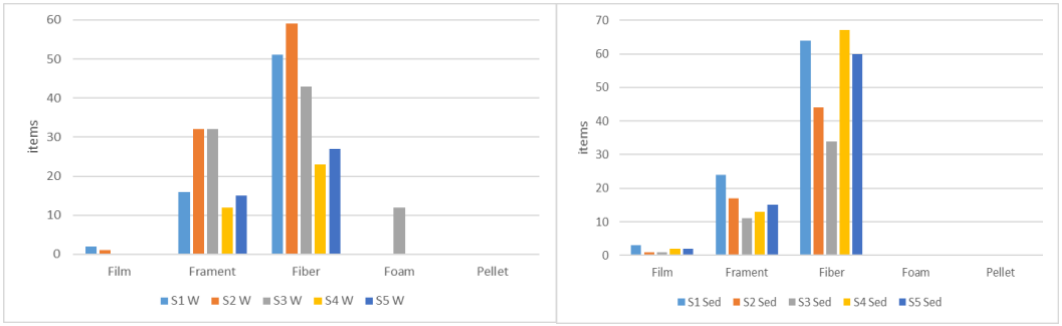


Figure 6. MPs shape

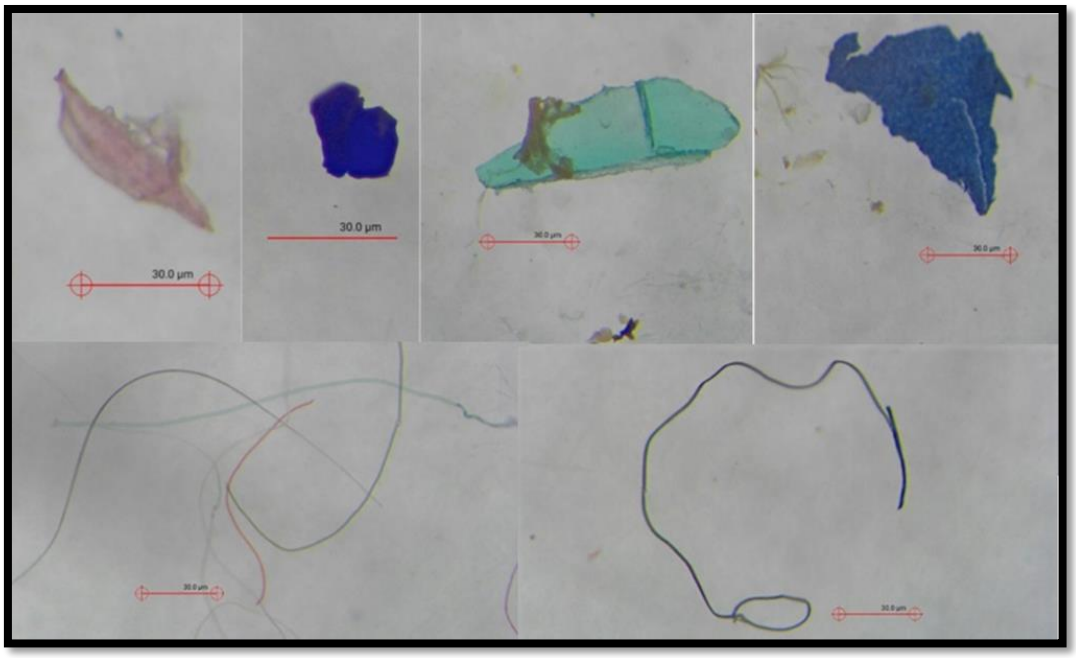


Figure 7. Shape types of MPs (1-5; fibres, 6-8; fragments, 9-12; film, scale bar= 0.3 mm).

FT-IR analysis was made for polymer types of MPs samples. The highest percentage was found to be Polypropylene (PP) 34%, followed by Polyethylene (PE) 26%, Polyethylene Terephthalate (PET) 24%, Polystyrene (PS) 7%, Polyamide (PA) 5% and Polyvinyl Acetate (PVAc) 4% . MPs types in water samples were 36% PP, 27% PE, 23% PET, 5% PS, 6% PA and PVAc with 3% (Figure 8). In terms of MPs in sediment samples, PE was the most abundant with 32% PP, followed by 26% PE, 23% PET, 11% PS, both PA and PVAc with 4% each. FT-IR spectra of Aksu stream microplastics in Figure 9. These conclusions are consistent with previous studies (Wang et al., 2017; 2020; Zhou et al., 2020; Enamul Kabir et al., 2022).

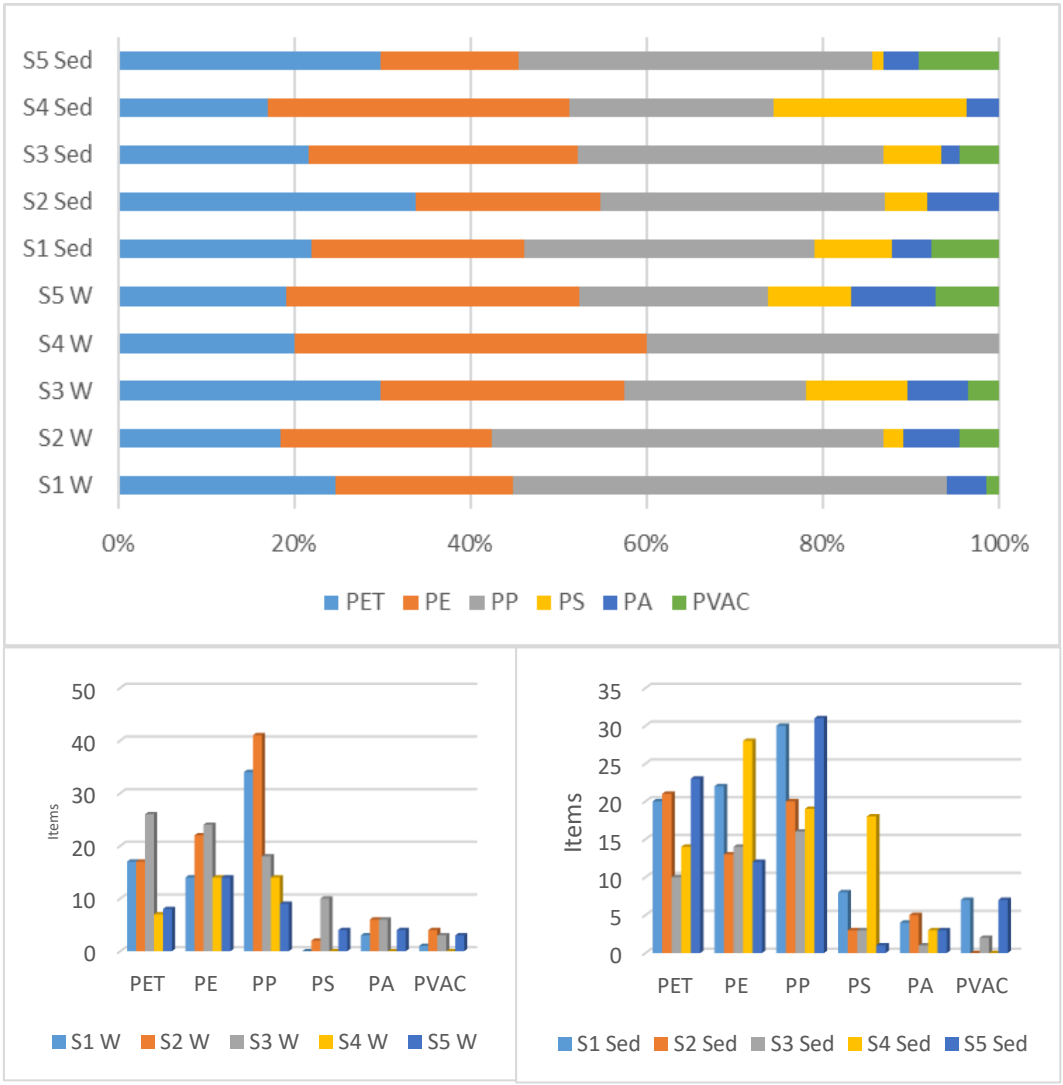


Figure 8. MPs polymer type

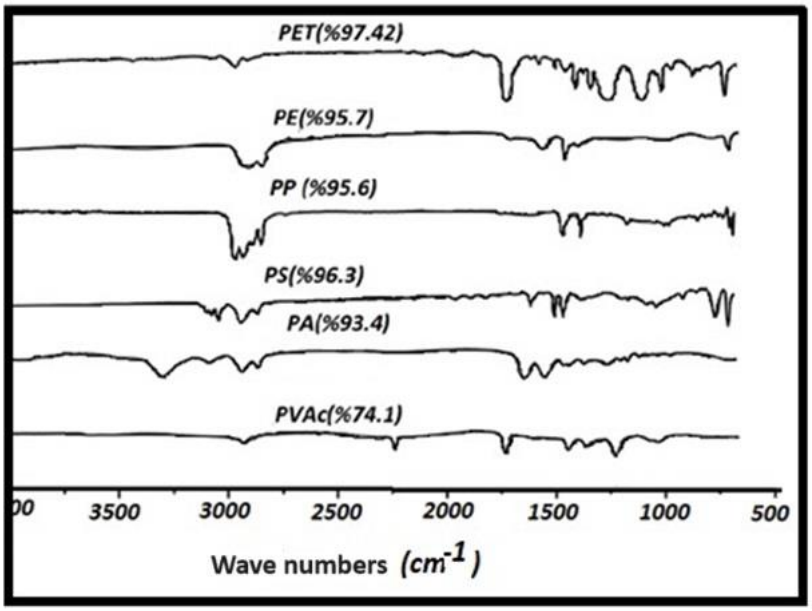


Figure 9. FT-IR spectra of Aksu stream microplastics. Values in parentheses indicate the average match rate with the standard spectrum for each polymer: PP (polypropylene), PS (polystyrene), PE (polyethylene), PVAc (polyvinyl acetate), and PA (polyamide), PET (polyethylene terephthalate).

3.2. Hierarchical Clustering Analysis

The HCA test, which clearly shows the similarity or difference of the stations according to their microplastic densities, was applied to the data of this study. Cluster A, which is the first of the two groups formed in the HCA test in the data obtained as a result of microplastic research in Aksu Stream sediments, consists of stations 1, 5, 4 and it was concluded that stations 4 and 5 are more similar to each other. It can be said that this is due to the fact that these stations are closer to the sea and the presence of settlements. Cluster B consists of stations 2 and 3. In general, Cluster B stations have higher similarity rates and it is thought that this similarity is due to the settlement pattern in the region. The related analysis graph is given in Figure 10. With the data obtained as a result of microplastic research in Aksu Stream water samples, Cluster A, the first of the two groups formed in the HCA test, is more similar than Cluster B. Given these results, Aksu Stream can be considered as upper basin (Stations 1–3) and lower basin (Stations 4–5).

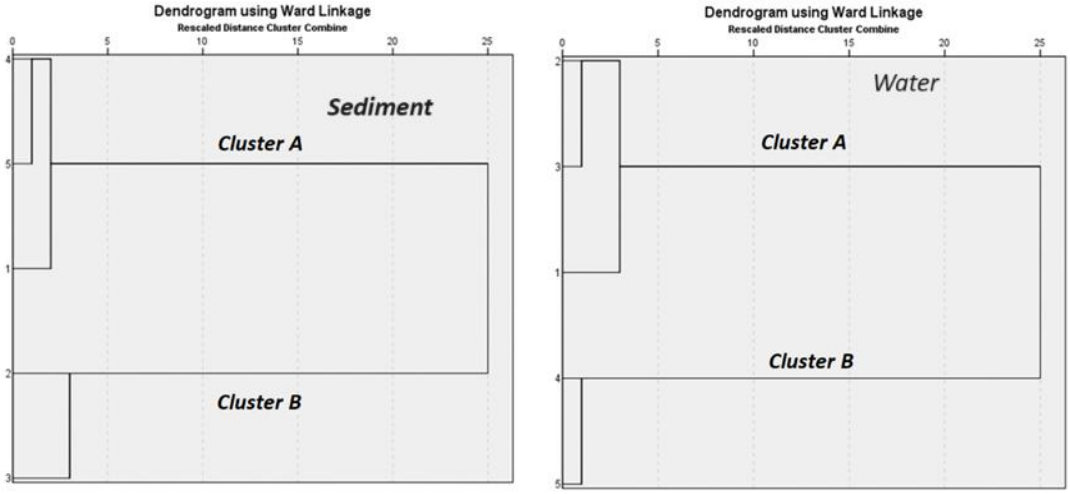


Figure 10. HCA Analysis of MPs of Aksu Stream

3.3. Potential Health Impact of MPs

PHI (Potential Health Impact) calculated in the present study in parallel with the studies carried out by Lithner et al. (2011) and Ranjani et al. (2021) was found to be in the damage and risk categories III (High) and IV (Danger). Detailed potential ecological risk of MPs results for water and sediment are given in Tables 1-3.

Table 1. Potential Health Impact of MPs (Total sampling)

Polymers	Proportion (%)	Hazard score *	PHI**	Hazard category**	Risk Category**
PET	23.66	4	94.63	III	High
PE	27.04	11	297.47	IV	High
PP	33.94	1	33.94	III	High
PS	6.66	30	199.78	IV	High
PA	4.95	47	232.55	IV	Danger

PVA_C	3.75	10.551	39,59	IV	Danger

* Lithner et al. (2011), **Ranjani et al. (2021).

Table 2. Potential Health Impact of MPs (Water)

Polymers (Water)	**Proportion (%)**	**Hazard score ***	**PHI****	**Hazard category****	**Risk Category****
PET	22.41	4	89.64	III	High
PE	29.02	11	319.27	IV	Danger
PP	35.19	1	35.19	III	High
PS	4.64	30	139.15	IV	Danger
PA	5.46	47	256.53	IV	Danger
PVA_C	3.28	10.551	34.58	III	High

* Lithner et al. (2011), **Ranjani et al. (2021).

Table 3. Potential Health Impact of MPs (Sediment)

Polymers (Sediment)	**Proportion (%)**	**Hazard score ***	**PHI****	**Hazard category****	**Risk Category****
PET	2491	4	99.63	III	High
PE	25.06	11	275.68	IV	Danger
PP	32.69	1	32.69	III	High
PS	8.68	30	260.41	IV	Danger
PA	4.44	47	208.57	IV	Danger
PVA_C	4.23	10.551	44.59	III	High

* Lithner et al. (2011), **Ranjani et al. (2021).

4. Conclusions and Recommendations

In accordance with the assessments made, it is predicted that the extent to which microplastic pollution affects the aquatic ecosystem and ultimately human health has reached increasingly serious dimensions. As a result, it is recommended to minimize the wastes, take necessary precautions and carry out monitoring studies in order to reduce the risks posed by the wastes flowing into the sea from the stream, which threaten both the aquatic ecosystem and human health. We are of the opinion that microplastics in the seas will be less risky by 2050 thanks to advanced treatment techniques applied or aimed to be applied in cities, reduction of point source pollution, recycling and reduction of consumption. It is predicted that the extent to which the aquatic ecosystem and ultimately human health are affected by microplastic pollution in line with the evaluations made and that it will be under a more serious danger day by day.

In today's world, plastics should be considered as a kind of pollutant at the point of production and microplastics should be managed by considering their effects on the environment and human health. This study contributes to the growing evidence that MPs contamination is very widespread, even in freshwater ecosystems. It also provides a basis for future risk assessment studies.

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Authors' Contributions

AAU: Conceptualization, methodology, software, validation, formal analysis, investigation, resources, data curation, writing—original draft preparation, writing—review and editing, visualization, supervision. Author has read and agreed to the published version of the manuscript

Statement of Conflicts of Interest

There is no conflict of interest between the author.

Statement of Research and Publication Ethics

The author declares that this study complies with Research and Publication Ethics.

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