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Seasonal abundance and characteristics of microplastics in a peri-urban river determined by optoelectronic methods

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Microplastics are recognized as emerging contaminants in aquatic ecosystems, with potential impact on water quality. However, their seasonal dynamics in urban and peri-urban rivers remain insufficiently documented. In this context, the present study investigates the occurrence and seasonal variability of microplastics in surface waters, contributing to a better understanding of their distribution and temporal evolution in river systems. The results highlight the vulnerability of urban rivers to anthropogenic pressures and confirm the role of wastewater treatment plants as potential sources of microplastic contamination, emphasizing the need for effective monitoring strategies and control measures to reduce this type of pollution.

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1. Introduction

Plastic materials are widely used and, due to their persistence, have become pervasive pollutants in aquatic environments. Surface waters, particularly rivers, act as primary recipients of plastic debris originating from land-based human activities and play a key role in its transport through freshwater systems toward downstream environments [1-2]. Microplastics are commonly defined as plastic particles smaller than 5 mm and are classified, by origin, into primary and secondary microplastics [3-6]. Primary microplastics are intentionally manufactured at small sizes, such as microspheres used in personal care products, whereas secondary microplastics result from the fragmentation of larger plastic items under environmental stressors including ultraviolet radiation and mechanical abrasion [7-8]. In surface waters, microplastics occur predominantly as fibers, fragments, and microspheres, while synthetic textile fibers represent a major source associated with domestic washing and wastewater discharges [1, 3, 9]. Microplastics are now recognized as ubiquitous contaminants in freshwater systems and have been detected in rivers, sediments, aquatic organisms, and, indirectly, in humans. Their persistence and mobility allow interactions with toxic additives and persistent organic pollutants, raising concerns regarding ecological impacts and potential risks to human health [8, 10]. Rivers play a central role in the environmental fate of microplastics, functioning both as sinks for land-derived inputs and as transport pathways connecting terrestrial sources to lakes, reservoirs, and marine environments. Within river systems, microplastics may remain suspended, accumulate in depositional zones, or settle into sediments, where resuspension processes can lead to secondary contamination of the water column [4, 8]. Although research on microplastic pollution has traditionally focused on marine environments, studies addressing freshwater systems have increased in recent years. However, existing research has mainly targeted large rivers or highly urbanized areas, while small rivers and urban–periurban surface waters remain insufficiently studied, despite their importance as interfaces between land use, hydrology, and plastic transport [2, 10]. The abundance and distribution of microplastics in rivers are controlled by a combination of environmental and anthropogenic factors, including hydrological conditions, land use patterns, urbanization, wastewater inputs, and surface runoff. Nevertheless, strong spatial and temporal heterogeneity leads to substantial variability among studies, and the influence of seasonal processes in small and medium-sized urban and periurban rivers remains poorly understood [2, 3, 8]. Given the urgency of this environmental situation, scientists worldwide have tried to apply the best methods for characterizing water quality. Among the most commonly used optoelectronic methods for water quality characterization, fluorescence spectroscopy [11-12] and microscopy are used to evidence the organic matter content and particle contamination, respectively [13-15].

In this context, the present study aims to investigate the occurrence and seasonal variability of microplastics in urban and peri-urban surface waters, contributing to a better understanding of microplastic dynamics in river systems that are currently underrepresented in the literature. To this aim, state of the art optoelectronic techniques have been used for fast and reliable quantitative and qualitative results.

2. Materials and methods

2.1. Study area and sampling

Water samples were collected from two locations along an urban river (Ciorogarla River). Ciorogarla River has a total length of 57 km and a catchment of 149 km^2. The water quality is directly influenced by the surrounding residential and agricultural areas. Thus, it collects surface runoff, untreated sewage and treated wastewater effluents from Magurele city. The locations presented in Fig. 1 were chosen considering the relative position of the Magurele wastewater treatment plant (P.E. 14000). Therefore, Sample 1 (Ciorogarla 1) is situated upstream of the wastewater treatment plant, while Sample 2 (Ciorogarla 2) is situated downstream from the plant.

Four sample collection campaigns were undertaken in order to collect water samples in each season, starting from April 2023 and ending in February 2024.

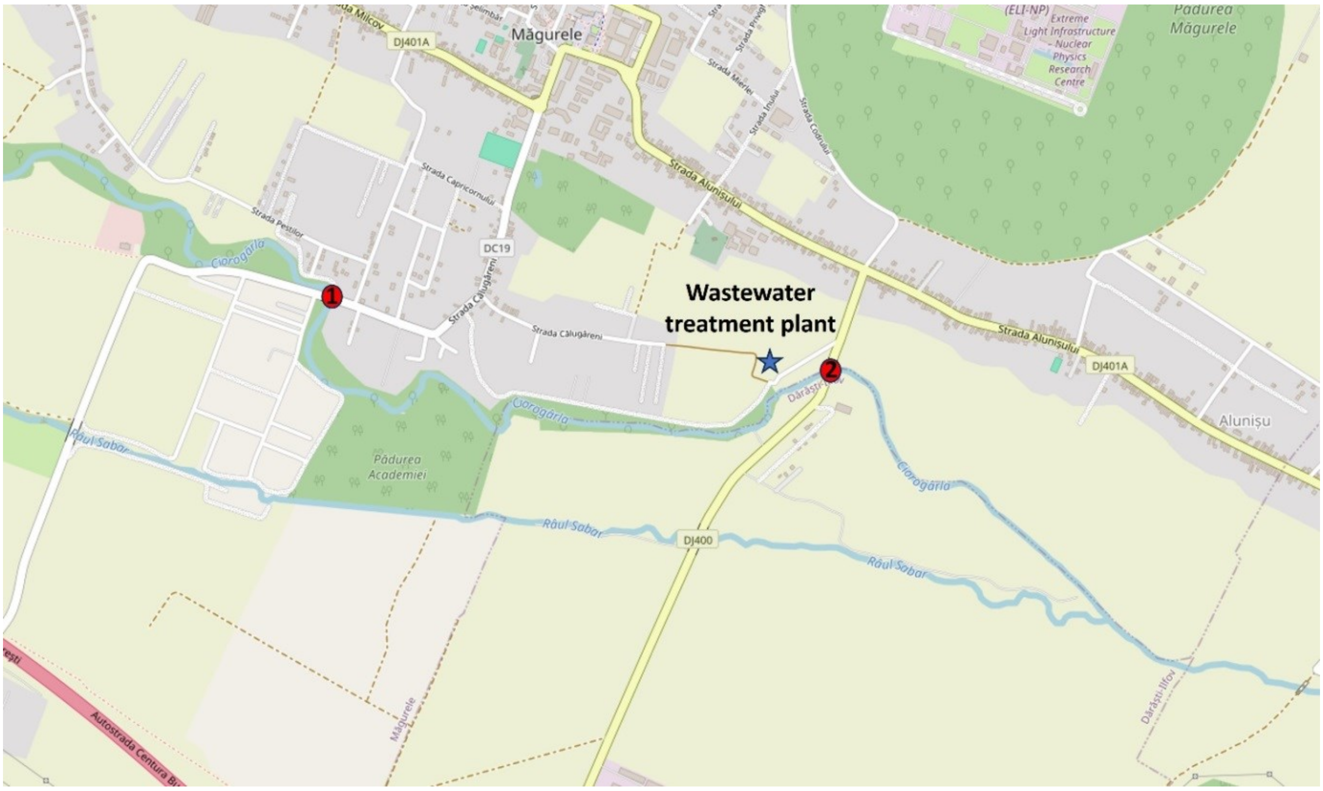

Fig. 1. Geographical distribution of the collection points along the Ciorogarla River: 1 (Ciorogarla 1) – on the Ciorogarla River before the wastewater treatment plant and 2 (Ciorogarla 2) – on the Ciorogarla River after the wastewater treatment plant (colour oonline)

The water samples were collected in glass bottles which were previously cleaned with a 20% RBS solution and rinsed with ultrapure water in order to prevent any potential contamination. On site, the bottles were rinsed with river water before sample collection and transport. All measurements were made within 24 hours from sample collection.

2.2. Ancillary measurements

In order to characterize the water quality of the collected samples, the following parameters were measured: pH, conductivity, turbidity, phosphate (PO_4), nitrate (NO_3), nitrite (NO_2), and total dissolved solids (TDS). For pH, conductivity, turbidity, and TDS, the Hanna Instruments HI 225 combined meter was used. For phosphate, nitrate, and nitrite, the PF-12Plus photometer and testing kits from Macherey-Nagel were utilized.

2.3. Optical microscopy analysis

To highlight the seasonal evolution of the abundance and type of microplastics present in the Ciorogarla River, the samples were subjected to a specific protocol. Before beginning the procedure, all beakers, graduated cylinders and micropipette tips were washed three times with ultrapure water. The 30% hydrogen peroxide solution was filtered with a 0.22 µm paper filter. After the water samples were filtered, the glass beakers were covered with aluminum foil prior to being weighed and placed in an electric oven.

The sample preparation process was comprised of several stages. In the first step, the water samples were filtered with a 1,000 µm sieve. The residues were washed with plenty of pure water, then collected into glass beakers, and subjected to evaporation at a temperature bellow 70 ^{o}C. The residues thus obtained were weighed and subjected to the digestion process, in order to destroy organic matter. For this purpose, approximately 20 mL of 30% hydrogen peroxide were added, in ambient atmosphere, until no more foam was formed. When the reaction with the hydrogen peroxide solution had completed (after approximately 5 days), the resulting mixture of residues and hydrogen peroxide solution was filtered through a 1 µm microfiber filter (Whatman, GF/B, 47 mm diameter, Global Life Sciences Solutions Operation UK, Ltd.). The particles loaded filter was placed on a Petri dish, dried at ambient temperature and stored in a desiccator before microplastics analysis with an optical microscope. The entire process was performed in triplicate. The particles isolated from the water samples were examined using an optical microscope (OLYMPUS BX 51 M, Olympus Corporation, Tokyo, Japan), operated with the Olympus Stream Essential 1.9.3 software, using X100 magnification. The microscope is equipped with a digital color camera with a resolution higher than 3 MP and a CCD sensor with a minimum of ½" for determining particle size, color and morphology. Optical microscopy is one of the most reliable techniques for quick particle identification due to its ability to differentiate plastics from other types of materials such as fats, mineral particles or cellulose fibers [16]. In order to prevent identification errors and underestimation of microplastics dimensions (> 1 mm), a series of criteria were applied in the process of interpreting the obtained results:

A. No organic or cellular structures are observed in the case of particles.
B. In the case of fibers, the diameter must remain constant throughout their entire length.
C. Particles must present clear and homogeneous colors.
D. In the case of transparent or white particles, higher magnifications must be used.

Ultrapure water was used as a control sample, and all measurements were performed in triplicate.

3. Results and discussions

3.1. Ancillary measurements

The pH values exhibited a gradual decrease from spring to autumn. For Ciorogarla 1 samples, the values decreased slightly from 8.04 in spring to 7.96 in summer and eventually to 7.86 in autumn, and for Ciorogarla 2 samples from 7.99 in spring to 7.88 in summer and 7.73 in autumn. The highest values were recorded in winter, 8.17, for both Ciorogarla 1 and Ciorogarla 2 samples.

In terms of turbidity, the results showed that the highest values were registered in spring for all samples, while the lowest values were recorded in autumn for Ciorogarla 2, which is located downstream from the wastewater treatment plant. The values at Ciorogarla 1 were slightly higher than those at Ciorogarla 2 in spring, summer and autumn, potentially due to the dilution effect of the wastewater effluent. In winter, the trend is reversed.

In order to further analyze the water quality of Ciorogarla River, PO_4, NO_3 and NO_2 concentrations were determined due to the fact that they are key indicators of nutrient pollution and of the nitrogen cycle health status and can indicate ecological imbalance. During spring and summer, nitrate concentrations were higher than 0.2 mg/L (Fig. 2), which are considered to be moderate values for water quality assessment according to national regulations [17]. This result could be influenced by the use of fertilizers in the fields or agricultural terrains adjacent of the river bed. The maxima nitrate concentrations did not exceed the national regulated limit for surface waters [17]. The value registered in autumn for the Ciorogarla 2 collection point exceeded 5.5 mg/L, which suggested a slight eutrophication process in the area [17]. Regarding the nitrite concentrations, the concentration values registered in spring and summer samples were relatively low (Fig. 2), which suggested that the river could be classified as being in good condition according to Romanian laws [17]. However, during winter and especially during autumn, the river water quality status differed, the nitrite concentrations being higher than those registered in the other seasons. In autumn the values were close to the maximum concentration admissible by law for effluents in wastewater treatment plants [18].

The conductivity, TDS, and salinity values registered seasonally showed a gradual increase from summer to winter, but the highest values did not exceed levels that could suggest a strong contamination (Fig. 2). These parameters are interconnected and their concentrations are related to sewage, agricultural runoff or road salts contamination.

The seasonal evolution of the studied parameters is presented in Fig. 2.

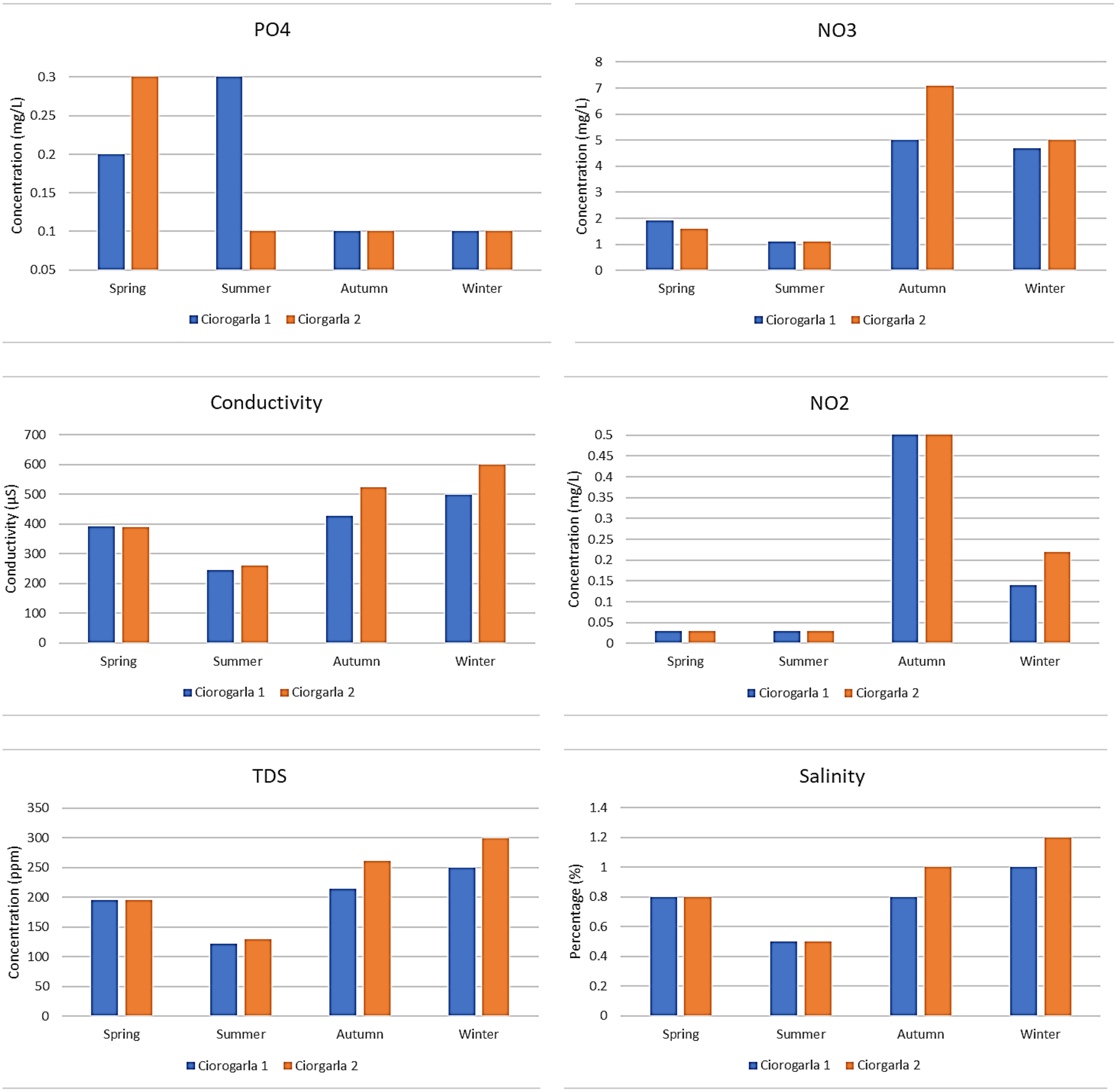

Fig. 2. Standard water quality parameters measured seasonally (colour online)

3.2. Optical microscopy analysis

The total number of microplastics investigated by optical microscopy varied between 15 and 45 particles/L in the case of the samples taken from Ciorogarla 1 and between 17 and 51 particles/L for the Ciorogarla 2 samples. In both cases, most particles were detected in samples collected in winter, and the lowest abundance of particles was recorded in spring. The seasonal distribution of microplastics abundance is highlighted in Fig. 3.

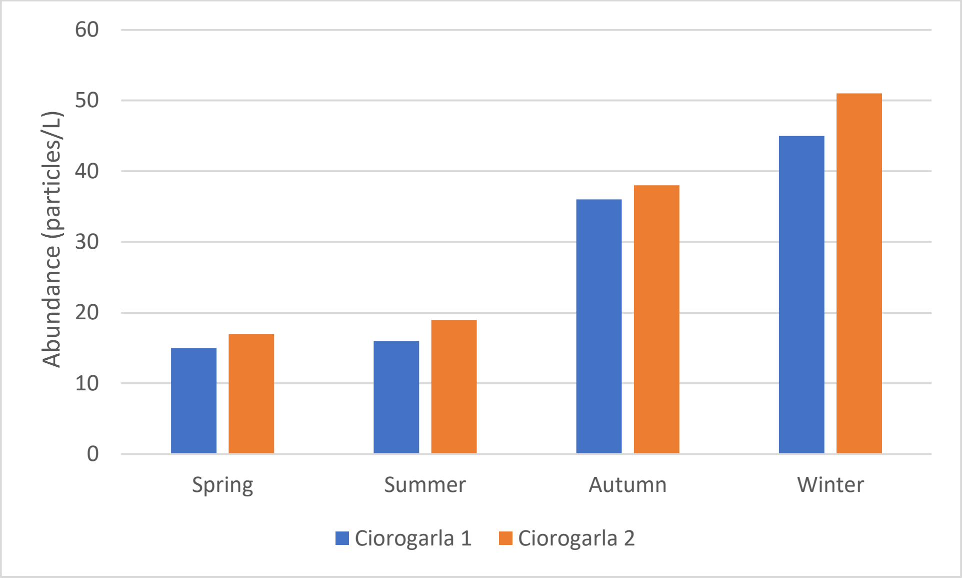

Fig. 3. Seasonal abundance distribution of microplastics (colour online)

With regard to the shape of the identified microplastics, they were divided into fibers, fragments and films. Relevant images of the observed particle types are presented in Fig. 4.

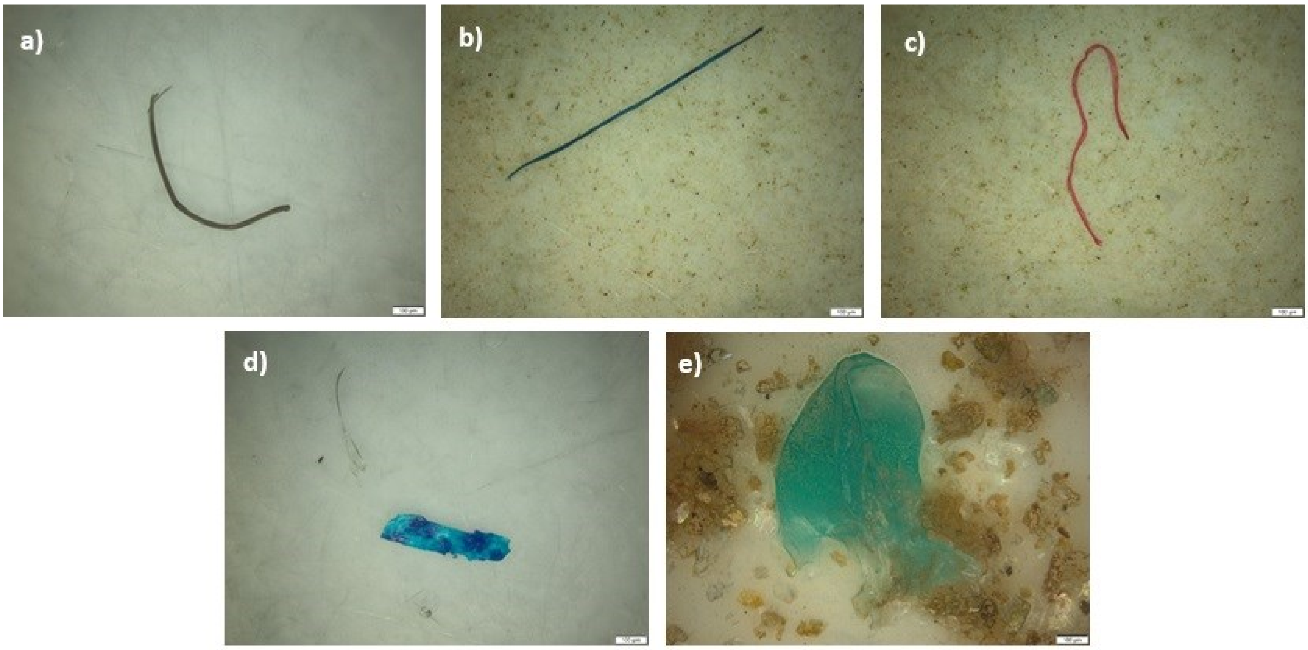

Fig. 4. Examples of types of microplastics found in Ciorogarla 1 and Ciorogarla 2 water samples in the form of a) black fiber, b) blue fiber, c) red fiber, d) blue fragment and e) blue foil (colour online)

A breakdown of microplastic abundance by particle type and seasonal occurrence is depicted in Fig. 5.

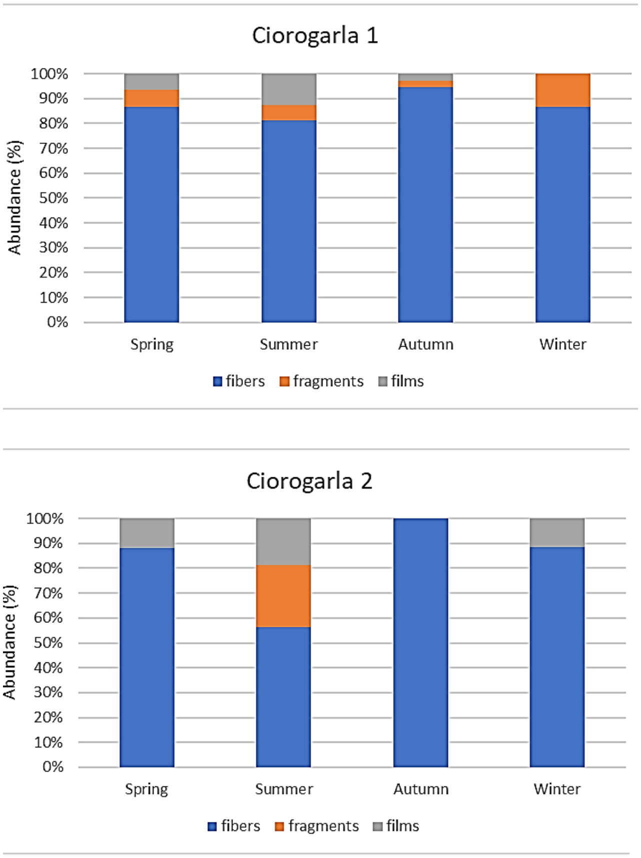

Fig. 5. Microplastics abundance depending on their shape and seasonal occurrence (colour online)

Microscopic analysis revealed a prevalence of fibers (88.39% for Ciorogarla 1 samples and 85.71% for Ciorogarla 2 samples). Other types of microplastic particles were substantially less abundant (8.04% fragments and 3.57% films for Ciorogarla 1 samples and 4.4% fragments and 9.89% films for Ciorogarla 2 samples). These findings are in accordance with the results obtained by Pojar et al. [4] in their study on microplastic occurrence. Moreover, an increased abundance of fibers was observed during spring, autumn and winter for both sampling points. Browne et al. [19] suggested a connection between this behavior and household activities that could induce this result. Their studies suggested that because people tended to wear more clothes during colder seasons than in warmer months, a large concentration of fibers might have been released through laundry.

When considering the color of the detected microplastics, it was observed that three dominant colors appeared more frequently: black, blue, and red. The color distribution of the detected microplastics is exhibited in Fig. 6. Out of the three colors, black was predominant, closely followed by blue. The prevalence of black, blue and colorless microplastics especially in the effluents of wastewater treatment plants was highlighted in previous studies [20]. On the other hand, other recent studies have avoided reporting the color of the detected microplastics [20-25] due to the possibility of color alteration produced by the chlorination and oxidation processes used in wastewater treatment plants. Our observation that black is the predominant color is in agreement with previous studies that attributed this phenomenon to the fact that black microplastics have multiple potential sources, such as car tires, industrial processes or are components in packings and various single-use plastic products [26-29].

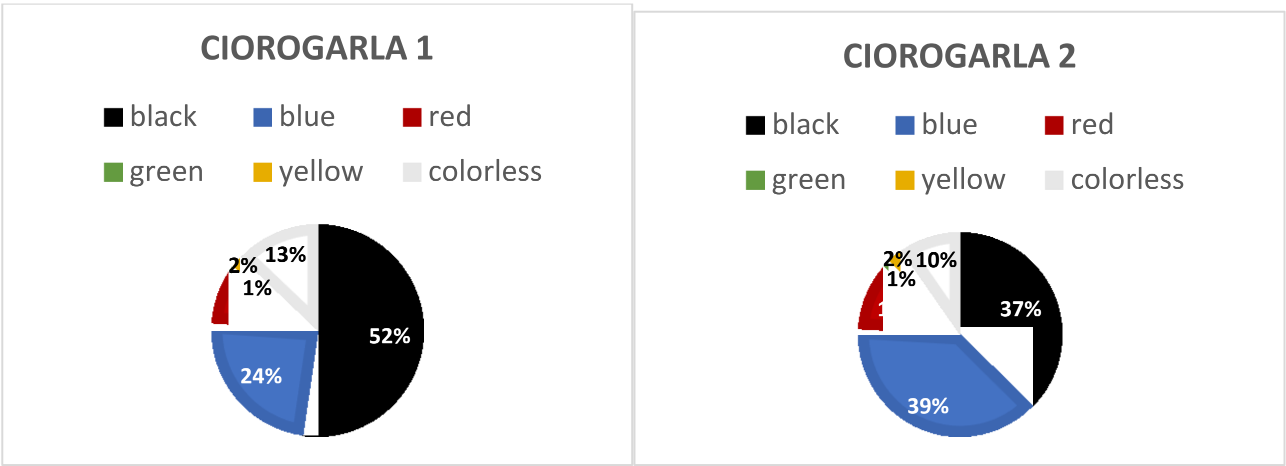

Fig. 6. Color distribution of microplastics (colour online)

Fig. 7 shows the color distribution depending on the season of occurrence. It can be observed that black microplastics are more numerous in Ciorogarla 1 point, before the wastewater treatment plant, while in Ciorogarla 2 point, blue microplastics are the most abundant. Also, colored microplastics are more abundant compared to transparent or colorless microplastics (Fig. 7).

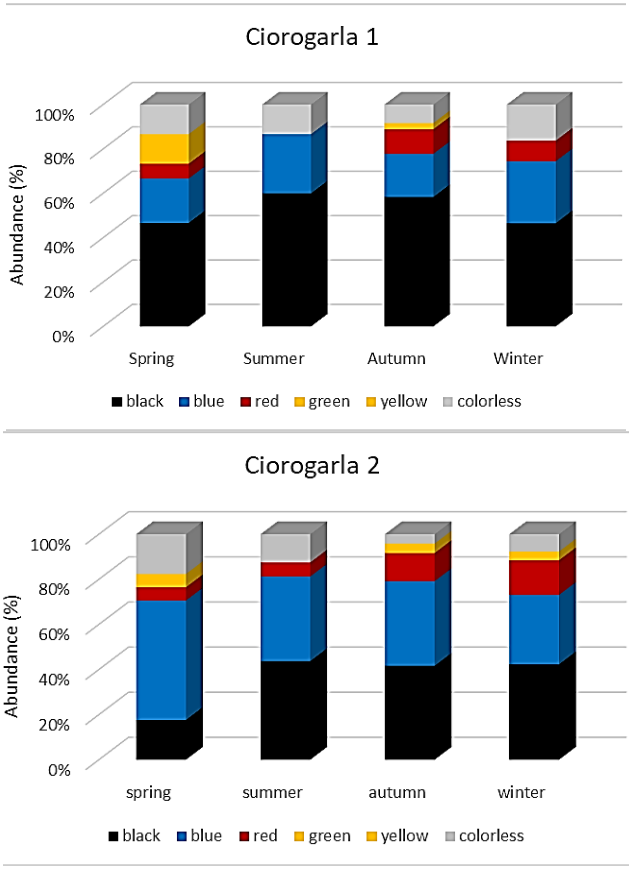

Fig. 7. Distribution of microplastic colors depending on the season of occurrence (colour online)

A significant increase of microplastics abundance during seasons has also been reported in previous studies. This behavior was observed in the case of several treatment plants in South Africa [29], and one explanation was that during winter in particular, the treatment plants could not process the very large quantities of microplastics that needed to be removed.

The seasonal variations in microplastic concentrations detected for both sampling points suggested that there was a direct influence from anthropogenic activity in the region. Moreover, the high abundance of fibers was represented by water discharged by washing machines [29].

4. Conclusion

The aim of this study was to monitor and determine the water quality parameters and microplastic abundance of Ciorogarla River given the fact that its water quality is directly influenced by the surrounding environment, this influence ranging from rainwater discharge to agricultural or wastewater treatment plant contamination. It emphasized the behavior of microplastics and the influence of wastewater treatment plant effluents on river water quality. Standard parameters measured seasonally evidenced the fact that the river water quality is highly susceptible to external influence and the human activities that take place adjacent to the river bed are a contamination factor. With regards to the presence of microplastics, it was concluded that the abundance and the shape distribution is influenced by the seasonal behaviors of human activities. Furthermore, it was observed that the wastewater treatment plant effluent, released into Ciorogarla River, is a potential source of microplastic contamination. The color distribution of the microplastics is also season dependent and is strongly influenced by human activities. The results presented in this paper highlight the need to adapt to current needs for removing microplastics from the environment.

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