microplastics

Article

Quantification and Characterization of Microplastics in Seven Urban Wastewater Treatment Plants

Erica Sparaventi [1], Rafael Nuez [2], María Pilar Yeste [2,*], Miguel Ángel Cauqui [2] and Marta Sendra [1]

[1] Department of Genetic Toxicology and Cancer Biology, National Institute of Biology, 1000 Ljubljana, Slovenia; erica.sparaventi@nib.si (E.S.); marta.sendra@nib.si (M.S.)
[2] Department of Materials Science and Metallurgic Engineering, and Inorganic Chemistry, University of Cádiz, MEYMAT: Institute of Research on Electron Microscopy and Materials of the University of Cádiz, Puerto Real, 11510 Cádiz, Spain
* Correspondence: pili.yeste@uca.es

Abstract

Microplastics (MPs) are routinely detected throughout wastewater treatment plants (WWTPs), yet current treatment trains were not designed specifically to remove them. This study quantified and characterized visually identified MPs in influent and effluent waters from seven urban WWTPs in Andalusia (southern Spain) during a six-month monitoring period (July–December 2020). The targeted analytical size range was 45–5000 µm, and a subset of particles was further characterized by FTIR. MPs were detected in all sampling campaigns. Concentrations ranged from 6 to 78 items/L in influent and from 12 to 65 items/L in effluent. Fibers were the dominant morphology, and the 100–500 µm size class was the most represented fraction. Among the subset analyzed by FTIR, PA, PP, PVC and LDPE were the most frequent polymer assignments, with PA predominating in the fiber-rich fraction. However, because influent and effluent 24 h time-composite samples were not hydraulic retention time (HRT)-paired and FTIR interpretation was based on a selected subset of particles, the dataset is best interpreted as describing spatiotemporal variability during the study period rather than robust process-specific removal efficiency. Overall, the results support WWTPs as an ongoing pathway for MP release to receiving environments.

Keywords: microplastics; wastewater treatment plants; six-month monitoring; FTIR; fibers; spatiotemporal variability

Academic Editor: Jose Manuel Cervantes-Uc

Received: 16 April 2026
Revised: 29 May 2026
Accepted: 5 June 2026
Published: 14 June 2026

1. Introduction

Plastic pollution is widely recognized as a major environmental problem affecting aquatic and terrestrial ecosystems. Global plastics production reached 413.8 Mt in 2023 and is projected to continue rising markedly in coming decades [1,2]. The most widely used polymers in Europe include polypropylene (PP), polyethylene (PE), polyvinyl chloride (PVC), polyurethane (PU), polyethylene terephthalate (PET), and polystyrene (PS), reflecting the breadth of applications for these durable and inexpensive materials [3,4].

Microplastics (MPs) are generally defined as plastic particles smaller than 5 mm, whereas nanoplastics occupy the submicrometer to nanometer range [5]. MPs may be primary (intentionally produced at small size) or secondary (generated by fragmentation and weathering of larger plastic items) [6]. They have been reported in multiple environmental compartments and are increasingly recognized as vectors of co-contaminants and as potential stressors to biota and human health [7–9]. In parallel with particulate MPs, recent

https://doi.org/10.3390/microplastics5020122

work shows that stressed plastics can also release amorphous polymer micropollutants into water, broadening the context of polymer-derived contamination beyond classical particle counts [10]. These concerns have driven regulatory efforts aimed at reducing plastic inputs and unnecessary uses [11].

Wastewater treatment plants (WWTPs) are important convergence points for MPs from domestic, industrial, and urban runoff sources. Current wastewater treatment processes were not originally designed to remove MPs, but rather to treat dissolved and suspended contaminants [12,13]. As a consequence, effluents from WWTPs can contain MPs, which can reach the freshwater system (rivers, lakes and aquifers) and may ultimately be transported to marine environments. There are several sources of MPs, including domestic wastewater, where personal care products (e.g., microbeads) contribute significantly to MPs pollution [14] and industrial waste (e.g., textile manufacturing, paint and resin) also introduce MPs into wastewater systems [15]. It has recently been reported that the particles identified were predominantly fibers and fragments, with sizes ranging from 10 µm to 5 mm [16]. WWTPs consist of successive treatment units that comprise a preliminary and primary treatment (physical and mechanical processes). These initial stages can remove 56.8% to 98.4% of MPs, primarily through sedimentation [12,17]. These are followed by biological units, or secondary treatment, where the activated sludge process shows variable removal efficiency, ranging from 42.1% to 99.2% [15]. Some WWTPs also include a tertiary treatment stage. Advanced technologies such as membrane bioreactors may achieve very high apparent removal rates [12]. However, despite the removal capabilities of WWTPs, significant quantities of MPs still enter natural water bodies due to the large volumes of treated effluent released daily [12,14]. During the treatment process, MPs are transferred to sludge, where they are widely present, becoming a sink of these pollutants. In a recent study from Spanish WWTP, it is estimated that between 8.05 × 104 and 1.77 × 109 MPs·day^{-1} were loaded to sludge; representing a major source of MPs in agriculture when sludge is used as additive [18]. This ongoing issue highlights the need for systematic monitoring, improved treatment technologies and better management practices to mitigate the environmental impact of MPs pollution.

The aim of this study was to quantify and characterize visually identified MPs in influent and effluent waters collected from seven municipal WWTPs in Andalusia, Spain, during a six-month monitoring period (July–December 2020). We focused on describing differences among plants and sampling campaigns, together with particle morphology, size class, and polymer composition of an FTIR-analyzed subset, while explicitly acknowledging the limitations of unpaired 24 h time-composite sampling for inferring true plant removal efficiencies.

2. Materials and Methods

2.1. Study Areas

The seven studied WWTPs are located in the south of Spain (Andalusia region), two coastal close to Cádiz and Malaga cities and five inlands, close to Seville city (Figure 1). All the WWTPs have secondary treatment, except one from Seville (Sevilla R) with a tertiary treatment (Table 1).

2.2. Sampling Strategy and Sample Preservation

Influent and effluent samples were collected at the WWTPs as 24 h time-composite samples using an automatic sampler that collected constant-volume aliquots every 30 min during July–December 2020. After 24 h, the aliquots were pooled, homogenized, and a final volume of 2 L was taken for laboratory analysis. Samples were transported immediately to the laboratory and stored at 4 °C until extraction. Accordingly, the samples analyzed in this study were not field-preconcentrated samples, and the reported concentrations refer to the analyzed 2 L composite wastewater sample volume.

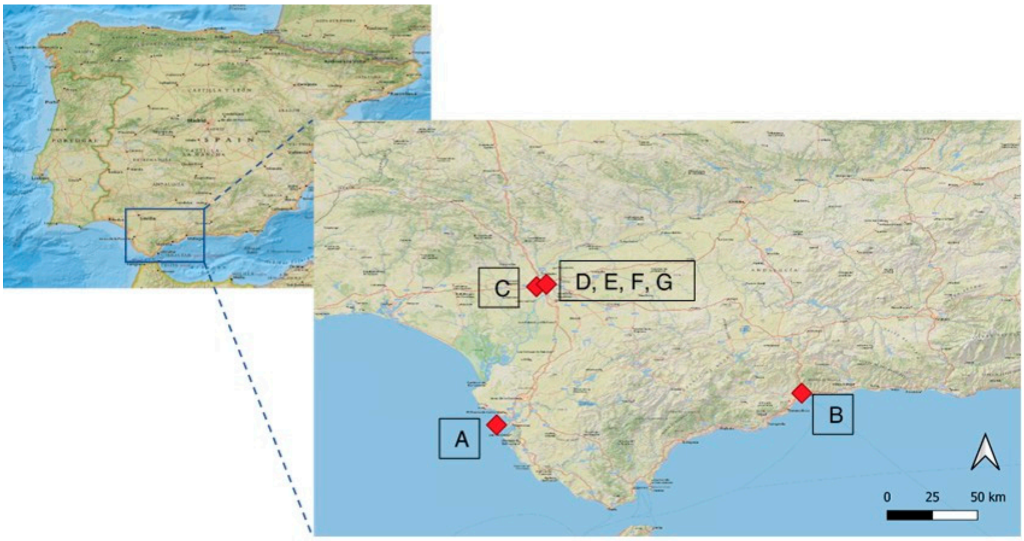

Figure 1. Location of the sample sites of the seven urban WWTPs in the south of Spain.

Table 1. Information about the seven WWTPs analyzed, in the Andalusian region.

WWTP	Location	Treatment Capacity (m^3/Day)	Population Equivalent	Influent Type	Type of Process	Water Treatment Processes
A **Cádiz**	Coast	75,000	375,000	Urban	Activated sludge, anaerobic digestion	Primary, secondary
B **Málaga**	Coast	177,000	1,300,000	Urban	Activated sludge, anaerobic digestion, ultrafiltration	Primary, secondary
C **Tomares**	Inland area	112,000	350,000	Urban	Activated sludge, anaerobic digestion	Primary, secondary
D **Sevilla N**	Inland area	90,000	350,000	Urban	Activated sludge, anaerobic digestion,	Primary, secondary
E **Sevilla R**	Inland area	90,000	350,000	Urban	Activated sludge, anaerobic digestion, nitrogen and phosphorus removal, tertiary treatment	Primary, secondary, tertiary
F **Sevilla T**	Inland area	50,000	200,000	Urban	Activated sludge, anaerobic digestion	Primary, secondary
G **Sevilla V**	Inland area	9220	41,000	Urban	Carrousel system, nitrogen and phosphorus removal, sludge dewatering	Primary, secondary

Given the hydraulic retention time (HRT) of the WWTPs, influent and effluent samples collected over the same 24 h period may not represent the same water parcel. Accordingly, influent–effluent comparisons in this study are treated as contemporaneous observations rather than true paired process tracking, and any apparent removal estimates are interpreted with caution.

2.3. Microplastics Extraction

Upon arrival at the laboratory, samples were equilibrated to room temperature and processed following a protocol adapted from Edo et al. [19]. For each sampling event, 2 L of homogenized composite sample was transferred to glassware, and the retained material was recovered with a stainless-steel spatula and rinsed with ultrapure water into a 250 mL glass beaker.

Organic matter was first digested with 10% (w/v) KOH at a 1:3 sample:KOH ratio (v/v) for 24 h at 40 °C. When visible organic material remained, a second oxidation step was applied by adding 15% H_2O_2 at a 1:1 sample:H_2O_2 ratio (v/v) and incubating at 40 °C for 72 h, extending the incubation when necessary until the digest appeared visually clear. In the original workflow, H_2O_2 was added directly to the post-KOH digest; we therefore acknowledge that oxidation efficiency may have been reduced by the alkaline matrix and discuss this as a methodological limitation.

After digestion, samples were sieved through a 5 mm mesh to remove large debris. The targeted size range was 45–5000 µm, consistent with the final filtration cut-off. Density separation was performed using 6.8 M NaCl, which preferentially recovers low-density polymers. After stirring and settling, the supernatant was vacuum-filtered through 45 µm cellulose microfiber filters (Whatman, Maidstone, UK). Glassware walls were rinsed thoroughly with ultrapure water and the rinsate was filtered to maximize recovery. Filters were dried at 40 °C for 48 h and stored in covered Petri dishes until analysis. Because cellulose filters and saturated NaCl can respectively affect fiber contamination control and recovery of high-density polymers, fiber counts and assignments of dense polymers (e.g., PVC) should be interpreted cautiously.

2.4. Microplastics Identification

All suspected MPs retained on the filters were first examined by visual inspection under a microscope. Items were categorized by morphology (fibers and fragments) and color. The fragment area and fiber length were measured. Table 2 reports visually identified items; polymer confirmation was performed only on a subset of particles.

Table 2. Concentrations of visually identified items during the six-month study period in influent and effluent waters from the seven WWTPs (items/L).

		Cádiz	Málaga	Tomares	Sevilla N	Sevilla R	Sevilla T	Sevilla V
July	Influent	34.5	72.0	77.5	28.0	38.5	21.5	21.5
	Effluent	14.5	34.0	38.0	30.0	13.0	27.5	61.5
August	Influent	49.0	22.0	45.5	25.5	45.0	34.0	25.5
	Effluent	30.0	27.5	46.5	55.0	16.5	30.5	27.0
September	Influent	50.5	48.0	60.0	29.0	25.0	68.5	40.0
	Effluent	25.0	17.0	45.5	18.5	41.5	40.5	46.5
October	Influent	36.0	55.5	66.0	69.5	76.0	65.0	70.5
	Effluent	11.5	41.5	47.0	22.5	12.0	28.0	34.0
November	Influent	35.0	47.5	24.0	30.5	54.0	47.0	41.5
	Effluent	13.5	29.5	52.5	24.5	48.5	17.0	24.0
December	Influent	62.0	61.0	29.5	52.0	53.5	44.5	5.5
	Effluent	18.0	25.5	18.5	29.0	11.5	55.5	25.5

Note: Values correspond to visually identified items. Only a subset of particles (45–5000 µm) was analyzed by FTIR for polymer assignment.

Specifically, for each WWTP and sampling month, filter membranes from both influent and effluent samples were divided into four equal quadrants. One quadrant was randomly selected for each filter, from which ten items were individually picked for further polymer identification. This subsampling approach was applied consistently across all WWTPs and sampling months to ensure comparability. A subset of 857 visually selected items was analyzed individually, and spectra were compared with the Bruker reference libraries. Tentative polymer assignment was carried out by Fourier-transform infrared spectroscopy (FTIR) using a Bruker ALPHA system and Bruker's dedicated software. Measurements were acquired in transmittance mode over a spectral range of 4000–500 cm^{-1}. Because the archived dataset does not contain a formal particle-selection log, this subset should be regarded as a practical, visually selected subset rather than a randomized sample. Spectral matches were manually reviewed before final assignment. However, because the original analytical workflow used a similarity threshold of $\geq$40%, polymer assignments are interpreted here as tentative and are discussed conservatively.

2.5. Quality Assurance/Quality Control

Procedural (process) blanks were included to assess potential contamination introduced during sample handling and processing. Blanks were run through the full procedure using ultrapure water and treated identically to samples. The mean blank mass (0.004 ± 0.008 g) was subtracted from sample filter masses prior to concentration calculations. Method performance was further evaluated using a positive control consisting of 0.1 g polystyrene particles (~100 µm) processed alongside samples, yielding a mean recovery of 99.267 ± 0.386%; however, this particulate control does not adequately represent the recovery behavior of fiber-dominated MPs. Likewise, blank mass subtraction cannot substitute for particle-level blank control by count, color, morphology, and polymer type. Contamination control data, including airborne contamination controls, procedural blanks, and working area contamination assessment, were obtained from a parallel study conducted simultaneously under identical laboratory conditions [20]. All controls indicated low contamination levels, with procedural blanks yielding a mean of 5.25 ± 0.96 fibers per blank. Blank correction was not applied to the present dataset for three reasons: first, sample microplastic counts were substantially higher than the contamination levels recorded in the parallel study, rendering blank correction negligible; second, as polymer identification was performed on items picked from a single randomly selected quadrant (one quarter of the total filter area), the proportional blank contribution to each subsample would represent approximately 1.3 fibers per sample; and third, as the primary aim of this study was comparative rather than absolute quantification, any uniform background contamination would not affect the relative differences observed among WWTPs or between influent and effluent samples. Additional fiber-specific QA/QC information (e.g., field blanks) was not available from the archived dataset and is therefore acknowledged as a limitation.

2.6. Statistical Analysis

Because concentration data were limited in number and not assumed to be normally distributed, non-parametric statistics were applied to the concentration values. Contemporaneous influent–effluent pairs were compared using a Wilcoxon signed-rank test ($n = 42$ paired observations). Differences among sampling months were explored separately for influent and effluent concentrations using a Friedman test across plants. Coastal versus inland WWTPs, and Sevilla R versus the remaining plants were explored using Mann–Whitney U tests. Statistical analyses were performed in RStudio (version 4.4.3), and $p < 0.05$ was considered statistically significant. Because samples were not paired according to hydraulic retention time, these comparisons were not interpreted as estimates of true plant removal efficiency.

3. Results and Discussion

A total of 6285 particles were found in the influent and effluent waters sampled from the seven urban WWTPs. Table 2 summarizes the concentrations of visually identified MPs (items/L) observed monthly during the six-month study period. Across all WWTPs, influent concentrations ranged from 6 to 78 items/L, whereas effluent concentrations ranged from 12 to 65 items/L. The highest concentration was observed in the influent of Tomares in July (77.5 items/L). Visually identified MPs were detected in all sampling campaigns and in both wastewater compartments.

A wide range of concentrations has been reported in WWTPs worldwide, although cross-study comparisons remain difficult because sampling design, target size range, analytical workflow, and reporting units differ substantially among studies [21–25]. In the present dataset, contemporaneous influent values were significantly higher than effluent values overall (Wilcoxon signed-rank test, n = 42, V = 724.5, $p = 0.00066$). However, this contrast should not be interpreted as a true process-specific removal efficiency because influent and effluent 24 h time-composite samples were not paired according to hydraulic retention time.

No robust temporal pattern was evident within the six-month study period, concentrations were broadly comparable across sampling months in the seven WWTPs, and no significant temporal variation was observed in influent (Friedman test, $\chi^2 = 7.73$, df = 5, $p = 0.17$) and effluent ($\chi^2 = 2.3469$, df = 5, $p = 0.7993$) particle counts (Table 3). Abundance of MPs was also very similar among the WWTPs, either coastal or inland (Mann–Whitney U test for influent samples, W = 214, $p = 0.351$; for effluent samples, W = 118, $p = 0.087$). Even in Sevilla R, where a tertiary treatment is present, no significant differences were found when compared to the other WWTPs for either the influent (Mann–Whitney U test, W = 92, $p = 0.577$) or the effluent samples (W = 151, $p = 0.126$). Overall, contemporaneous influent concentrations tended to be higher than effluent concentrations; however, this contrast should not be interpreted as a true process-specific removal efficiency because influent and effluent 24 h time-composite samples were not paired according to hydraulic retention time (HRT). No seasonal interpretation is attempted here because the monitoring period (July–December 2020) did not cover a full annual cycle. Instead, the dataset is interpreted as describing variability during the study period.

Sevilla R, the only plant described as having a tertiary treatment step, did not show a clear concentration advantage over the remaining WWTPs. This finding should be interpreted cautiously because tertiary treatment is not a single technology and its contribution depends strongly on the specific unit process, sampling point, and particle size range considered. In particular, disinfection-based tertiary steps are not designed to physically retain particles, whereas filtration-based tertiary treatments may improve retention [26].

Table 3. Summary of non-parametric tests comparing item concentrations across sampling months, geographical locations (Coastal vs. Inland), and specific facility performance (Sevilla_R, with tertiary treatment vs. Others).

Comparison Group	Target Sample	Statistical Test	Statistic (V, χ^2, W)	*p*-Value
All WWTPs	Influent-Effluent	Wilcoxon signed-rank	724.5	0.00066
Sampling months	Influent	Friedman	7.73	0.17

Table 3. *Cont.*

Comparison Group	Target Sample	Statistical Test	Statistic (V, χ^2, W)	*p*-Value
Sampling months	Effluent	Friedman	2.3469	0.7993
Coastal vs. Inland	Influent	Mann–Whitney U	214	0.350927
Coastal vs. Inland	Effluent	Mann–Whitney U	118	0.0867261
Sevilla R vs. Others	Influent	Mann–Whitney U	92	0.5774037
Sevilla R vs. Others	Effluent	Mann–Whitney U	151	0.1264658

Note: V = Wilcoxon signed-rank test statistic; χ^2 = Friedman Chi-square statistic; W = Mann–Whitney U statistic; $p < 0.05$ indicates statistical significance.

3.1. Polymer Composition of the FTIR-Analyzed Subset

Among the visually identified items retained on the filters, a subset of 857 particles (fibers and fragments) was visually selected for FTIR analysis. Of these, 849 yielded spectra assigned to synthetic polymers, representing 99% of the analyzed subset. The most frequent polymer assignments were polyamide (PA, 71%), polypropylene (PP, 9%), polyvinyl chloride (PVC, 6%), low-density polyethylene (LDPE, 5%), polystyrene (PS, 3%), and styrene acrylonitrile (SAN, 3%), with PA dominating across plants (Figure 2). These assignments are broadly consistent with the prevalence of textile-derived fibers and common consumer polymers in municipal wastewater [17,27–34].

The predominance of PA is plausible because the retained fraction was dominated by fibers, which are often associated with textile-derived inputs. However, polymer attribution should be interpreted cautiously for three reasons. First, FTIR was performed on a visually selected subset rather than on all retained particles. Second, 24% of the analyzed items did not yield polymer matches above the adopted threshold, suggesting that non-plastic particles, cellulosic fibers, semi-synthetic fibers (e.g., cotton, rayon/viscose), or residual organic material were also present. This is particularly relevant for fiber-rich samples, where visual classification alone can overestimate "plastic" fibers and where non-plastic fibers can be abundant in wastewater. Third, because the original analytical workflow used a similarity threshold of ≥40%, polymer assignments are interpreted here as tentative and are discussed conservatively.

In addition, the use of saturated NaCl favors recovery of low-density polymers, so the assignment of high-density polymers such as PVC should also be regarded cautiously in this dataset. No robust plant-specific or temporal shift in polymer composition was inferred from the available subset. Given the six-month monitoring window and the conservative interpretation adopted here, the polymer results are therefore best interpreted as indicative of the dominant types present in the analyzed fraction rather than as a fully resolved polymer budget.

Polyethylene is one of the cheapest polymers and is used in plastic bags, toys, packaging for detergent, shampoo and bleach [28,29]. Polystyrene is used in foam containers, plastic cutlery, and floats [35]. Finally, PVC is mainly used in pipes and electrical cables [36]. The polymers identified in this study corresponded to those previously reported in the inlet and outlet effluents of other European WWTPs [17]. However, the proportion of polymers was not consistent with studies in WWTPs elsewhere [30–32]. For example, in a WWTP in Turkey, the most prevalent polymers are PE and PP [32]. In the study by Hajji et al. [30], it was found that PE was the most abundant polymer in two WWTPs in Morocco. In a WWTP in China, one of the most frequent polymers has been PA, although PE and polyester have also been detected in great abundance [31]. The predominance of polyamide among the polymers identified here could be related to the fact that most of the MPs found were fibers.

Figure 2. MPs composition (%) detected in influent (INF) and effluent (EFF) of the seven WWTPs in southern Spain from July to December 2020.

This finding is in accordance with the study of Napper et al. [33], in which the effluent from WWTP outflows in the United Kingdom was analyzed. Furthermore, the presence of a high proportion of PA fibers in the WWTPs analyzed here could be attributed to the domestic origin of the wastewaters [34]. No differences were observed comparing the polymers found in each WWTP.

In addition, because FTIR characterization was performed on a subset of visually identified particles (selected as "representative"), the polymer profile may be affected by selection bias, for example, an over-representation of fibers or larger/easier-to-handle particles, potentially inflating the apparent dominance of PA. Future work would benefit from a fully randomized particle selection scheme (or complete filter mapping where feasible) and explicit reporting of the fraction of cellulosic/semi-synthetic fibers to better constrain polymer-specific trends.

The overall polymer composition was similar among plants, with variations only in their relative abundances [37]. However, in the case of two WWTPs analyzed in Thailand, the MP polymer content changes between the two plants were justified by the influence of the surrounding communities [14].

The composition of the inlet and outlet effluents remained the same during the six months, as urban wastewaters (same domestic sources) may present similar polymer composition over seasons, in agreement with the literature [30,38]. However, Ridall et al. [39] observed an increased presence of PA during winter, likely due to the increased amount of nylon clothing being washed during this season (Florida).

3.2. MP Characteristics (Size, Shape and Color)

A total of 6285 particles were measured. The most abundant size fraction in both influent and effluent samples was 100–500 µm, followed by 1000–5000 µm and 500–1000 µm. Because the size distribution was highly dispersed and the archived dataset available for manuscript revision did not contain the full particle-level measurements needed to recompute robust summary statistics, we focus here on dominant size classes and on the broad patterns shown in Figure 3 rather than on potentially inconsistent mean values. Particles smaller than 100 µm accounted for less than 5% of the visually identified fraction, while those larger than 5000 µm accounted for only 1%.

The predominant shape found in all samples from the WWTP waters was fiber (86%), followed by fragment (14%) (Figure 4). This is in agreement with other studies in the literature [40,41] where fibers are the most abundant type of MPs found in urban WWTPs released primarily from domestic laundry systems [24,42]. No flakes or spheres are found. The number of fibers released during a clothes wash can vary between 1.9×10^3 and 6×10^6 [43,44]. Fragments can be derived from several sources and enter the WWTP stream, and also they can be generated during the treatment stream from the degradation of plastic equipment, as hypothesized by Blair et al. [42]. The majority of MPs were predominantly black (Figure 5). Black particles were the most frequent color category, especially among fibers, whereas fragments displayed a broader range of colors.

Black fibers dominated the influent and effluent, 47.8% and 35.6% respectively. This is in agreement with a study of a French WWTP [21]. In contrast, fragments showed a more diverse color distribution, with a greater contribution from non-black particles. This proportion increased markedly in certain months.

3.3. Contemporaneous Influent–Effluent Differences and Study Limitations

The overall decrease from contemporaneous influent to effluent concentrations suggests that treated waters often contained fewer visually identified MPs than incoming waters at the time of sampling. Nevertheless, the design of the present study does not support a robust estimation of true removal efficiency. Influent and effluent 24 h time-composite samples were collected over the same sampling period and therefore did not necessarily correspond to the same water parcel after accounting for hydraulic retention time (HRT).

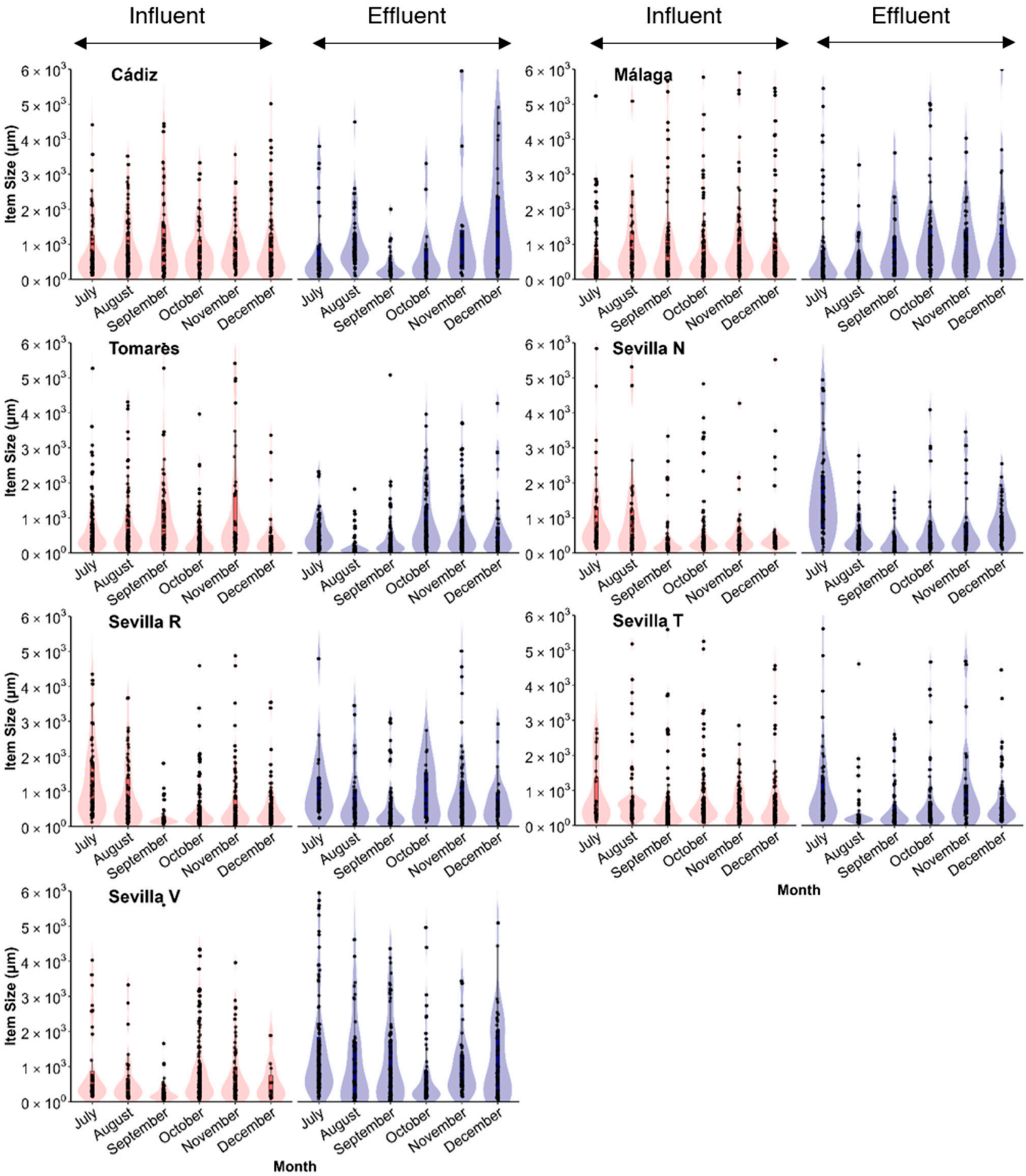

Figure 3. Size distribution of particles retained in influent (in red on the **left**) and effluent (in blue on the **right**) waters from the seven WWTPs in southern Spain during July–December 2020. Size is expressed in µm.

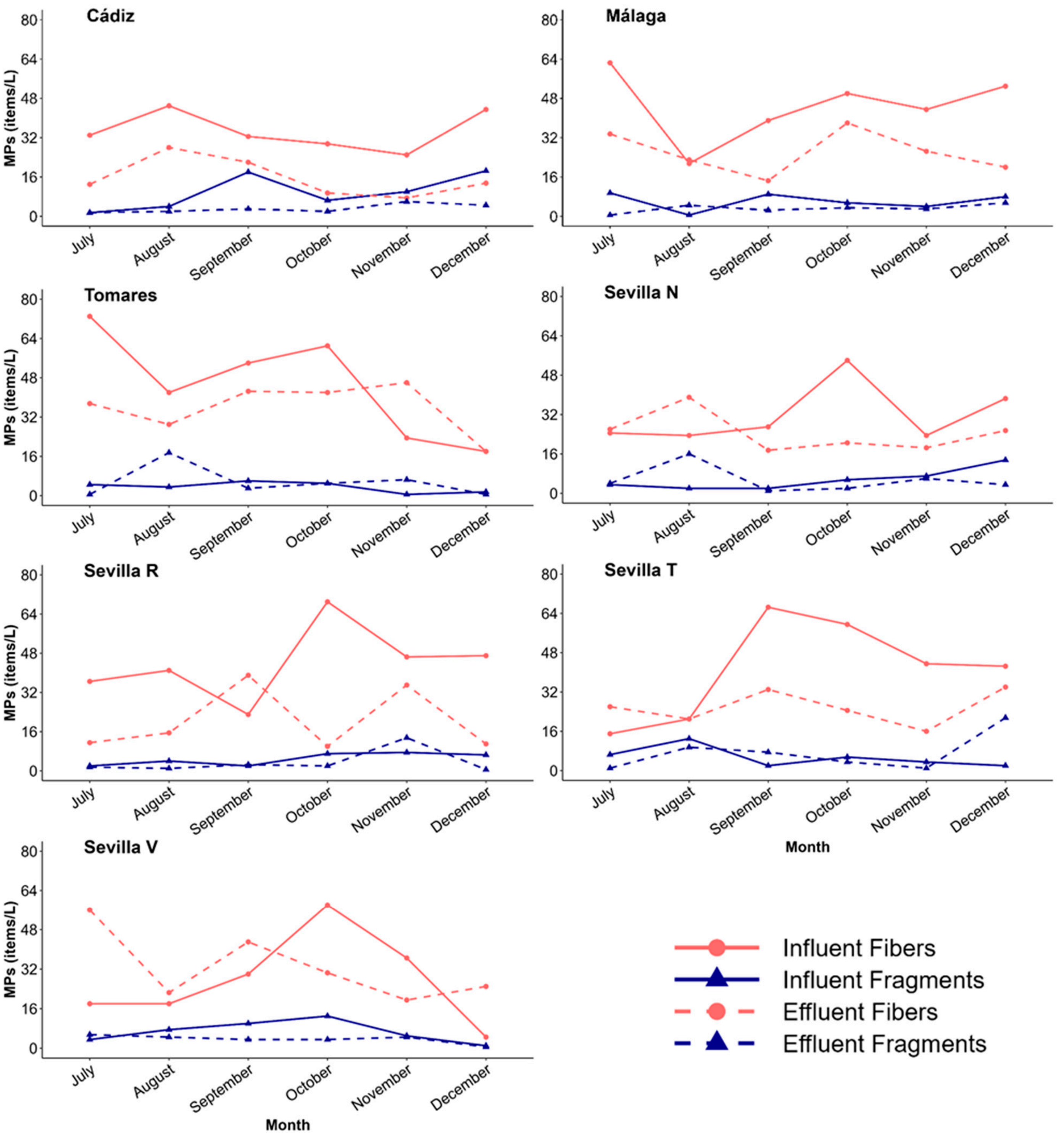

Figure 4. Relative abundance of fibers and fragments in the seven WWTPs in southern Spain during July–December 2020.

In addition, this study targeted only the 45–5000 µm fraction and therefore does not capture smaller MPs or nanoplastics, which may also be relevant in WWTPs. The dataset further relied on a subset for FTIR confirmation, used cellulose filters for final retention, and lacked field blanks and fiber-resolved blank correction in the archived QA/QC information. These constraints are sufficient to bias apparent influent–effluent contrasts and to complicate interpretation of polymer-specific patterns.

Accordingly, we refrain from interpreting the dataset in terms of definitive plant removal efficiencies. Instead, the main value of this study lies in its multi-site, repeated monitoring design, which documents the recurrent presence of MPs in both influent and effluent waters from urban WWTPs and highlights the need for future surveys based on HRT-informed pairing of composite samples, larger and explicitly recorded sampled volumes, stronger fiber-specific QA/QC, stricter polymer confirmation criteria, and improved spectral QA/QC workflows.

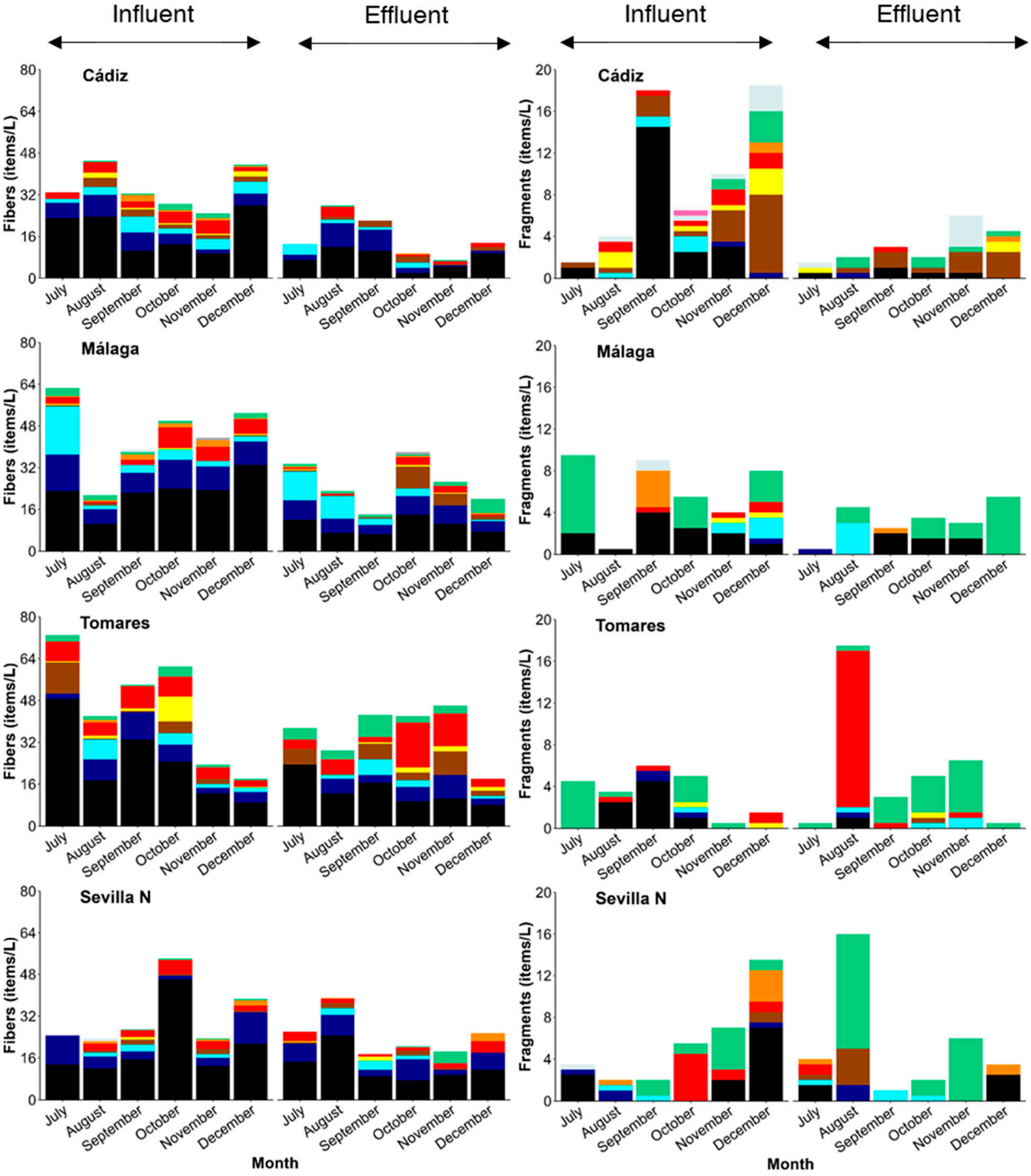

Figure 5. *Cont.*

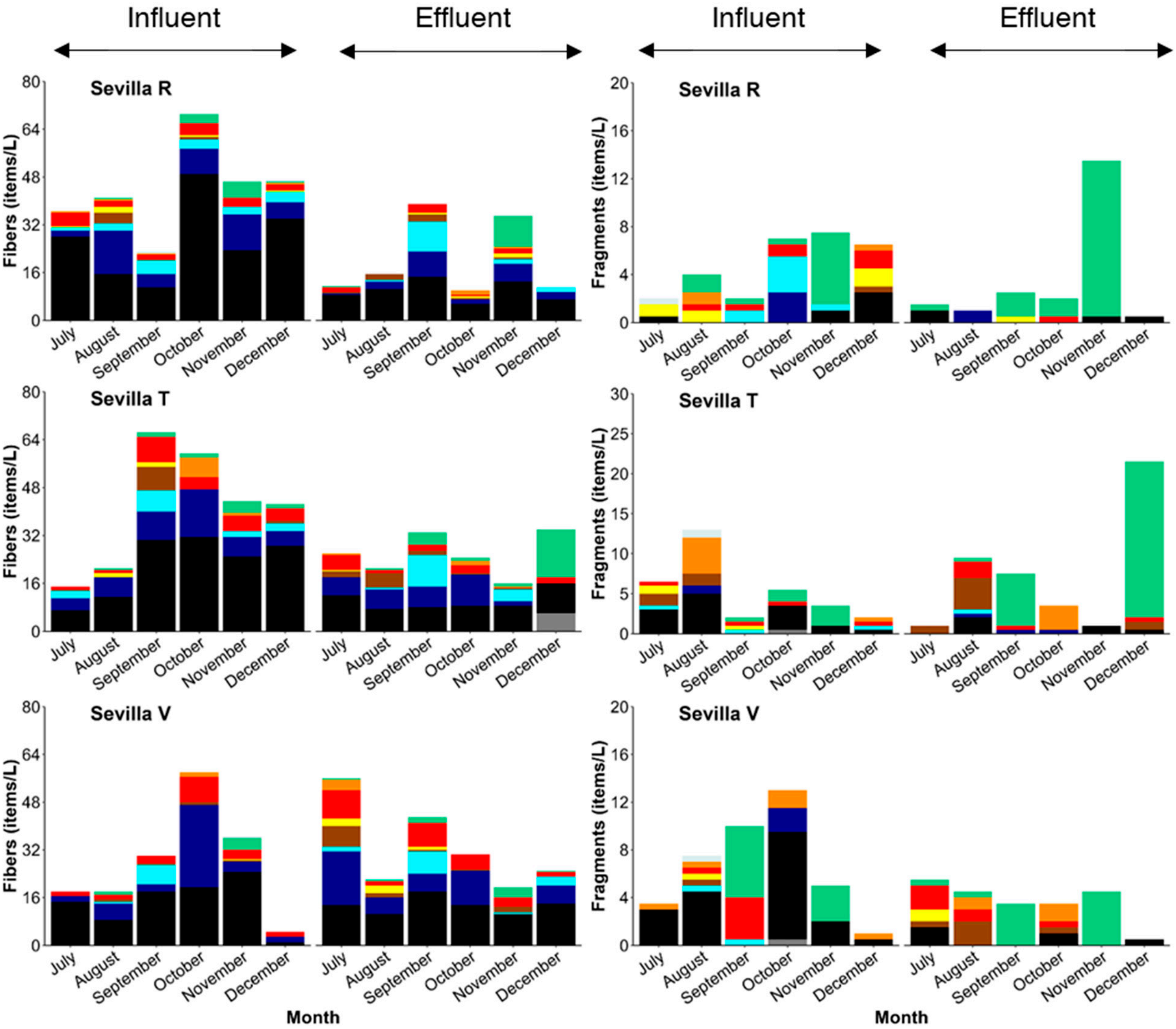

Figure 5. Color distribution of visually identified items in influent (**left**) and effluent (**right**) waters from the seven WWTPs in southern Spain during July–December 2020. The colors of the bars represent the colors of the plastics found.

4. Conclusions

Although no marked differences in concentration, predominant size class, morphology, or polymer type were observed, this study contributes to understanding the role of urban WWTPs as pathways for MP release into the environment. To our knowledge, this is the first study to monitor MPs in influent and effluent waters from seven urban WWTPs in Andalusia, southern Spain, over a six-month period. MPs were detected in all sampling campaigns, supporting the view that WWTPs remain an ongoing route of MP discharge to aquatic and terrestrial ecosystems. Within the dataset, effluent concentrations were generally lower than contemporaneous influent concentrations; however, because influent and effluent 24 h time-composite samples were not paired according to hydraulic retention time, these differences should not be interpreted as robust removal efficiencies. Fibers dominated the retained fraction, and PA was the most frequent polymer assignment within the FTIR-analyzed subset, consistent with textile-related inputs; however, polymer-level interpretation remains cautious because only a subset of particles was analyzed, the FTIR threshold was low, and not all analyzed items yielded confident synthetic polymer matches. No robust month effect, coastal–inland contrast, or clear tertiary treatment advantage was evident within the study period. Future monitoring should prioritize HRT-informed pairing of composite samples, longer time series, full reporting of sampled volumes, stronger fiber-specific QA/QC, more conservative polymer confirmation criteria, and explicit consideration of MPs smaller than 45 µm and nanoplastics.

Author Contributions: Conceptualization, M.P.Y. and M.S.; methodology, M.P.Y. and M.S.; formal analysis, E.S., R.N., M.P.Y. and M.S.; investigation, R.N., M.P.Y. and M.S.; data curation, E.S., R.N., M.P.Y. and M.S.; writing—original draft preparation, E.S.; writing—review and editing, M.P.Y., M.Á.C. and M.S.; funding acquisition, M.P.Y. and M.Á.C. All authors have read and agreed to the published version of the manuscript.

Funding: The authors gratefully acknowledge financial support from the Junta de Andalucía (Spain) to the FQM-110 and from the Junta de Andalucía Project (FEDER-UCA-2024-A2-37 DEGRABIOPLAS), by Marie Skłodowska-Curie Actions (MSCA) Postdoctoral Fellowships, part of the EU's research and innovation framework programme Horizon Europe under Grant Agreement N° 101244206 and by the Slovenian Research and Innovation Agency (ARIS) through the ARIS Strategic Projects/Gravitacija Call 2024, project Nº 25-ARIS-STRP-01.

Data Availability Statement: Data supporting this study's findings are available from the corresponding author upon reasonable request.

Conflicts of 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.

Abbreviations

The following abbreviations are used in this manuscript:

MPs	Microplastics
NPs	Nanoplastics
WWTPs	Wastewater Treatment Plants
PP	Polypropylene
PE	Polyethylene
LDPE	Low-Density Polyethylene
HDPE	High-Density Polyethylene
PVC	Polyvinyl Chloride
SAN	Styrene Acrylonitrile
PU	Polyurethane
PET	Polyethylene Terephthalate
PC	Polycarbonate
PS	Polystyrene
PA	Polyamide
HRT	Hydraulic Retention Time
RE	Removal Efficiency
QA	Quality Assurance
QC	Quality Control

References

1. PlasticEurope_TheFacts_2024_digital-1pager. Available online: https://plasticseurope.org/wp-content/uploads/2024/11/PE_TheFacts_24_digital-1pager.pdf (accessed on 1 October 2025).
2. OECD (Organisation for Economic Co-operation and Development). *OECD Global Plastics Outlook: Policy Scenarios to 2060*; OECD: Paris, France, 2022. [CrossRef]
3. Hildebrandt, J.; Thünemann, A.F. Aqueous Dispersions of Polypropylene: Toward Reference Materials for Characterizing Nanoplastics. *Macromol. Rapid Commun.* **2023**, *44*, 2200874. [CrossRef] [PubMed]
4. Magalhães, S.; Alves, L.; Medronho, B.; Romano, A.; Rasteiro, M.D.G. Microplastics in Ecosystems: From Current Trends to Bio-Based Removal Strategies. *Molecules* **2020**, *25*, 3954. [CrossRef]
5. Gigault, J.; Halle, A.T.; Baudrimont, M.; Pascal, P.-Y.; Gauffre, F.; Phi, T.-L.; El Hadri, H.; Grassl, B.; Reynaud, S. Current Opinion: What Is a Nanoplastic? *Environ. Pollut.* **2018**, *235*, 1030–1034. [CrossRef]

6. Duis, K.; Coors, A. Microplastics in the Aquatic and Terrestrial Environment: Sources (with a Specific Focus on Personal Care Products), Fate and Effects. *Environ. Sci. Eur.* **2016**, *28*, 2. [CrossRef]
7. Peng, J.; Wang, J.; Cai, L. Current Understanding of Microplastics in the Environment: Occurrence, Fate, Risks, and What We Should Do. *Integr. Environ. Assess. Manag.* **2017**, *13*, 476–482. [CrossRef]
8. Luo, G.; Liang, B.; Cui, H.; Kang, Y.; Zhou, X.; Tao, Y.; Lu, L.; Fan, L.; Guo, J.; Wang, A.; et al. Determining the Contribution of Micro/Nanoplastics to Antimicrobial Resistance: Challenges and Perspectives. *Environ. Sci. Technol.* **2023**, *57*, 12137–12152. [CrossRef] [PubMed]
9. Jeong, E.; Lee, J.-Y.; Redwan, M. Animal Exposure to Microplastics and Health Effects: A Review. *Emerg. Contam.* **2024**, *10*, 100369. [CrossRef]
10. Li, D.; Li, P.; Shi, Y.; Sheerin, E.D.; Zhang, Z.; Yang, L.; Xiao, L.; Hill, C.; Gordon, C.; Ruether, M.; et al. Stress-Induced Phase Separation in Plastics Drives the Release of Amorphous Polymer Micropollutants into Water. *Nat. Commun.* **2025**, *16*, 3814. [CrossRef]
11. European Parliament. *Microplastics: Sources, Effects and Solutions*; European Parliament: Strasbourg, France, 2018.
12. Bodzek, M.; Pohl, A.; Rosik-Dulewska, C. Microplastics in Wastewater Treatment Plants: Characteristics, Occurrence and Removal Technologies. *Water* **2024**, *16*, 3574. [CrossRef]
13. Freeman, S.; Booth, A.M.; Sabbah, I.; Tiller, R.; Dierking, J.; Klun, K.; Rotter, A.; Ben-David, E.; Javidpour, J.; Angel, D.L. Between Source and Sea: The Role of Wastewater Treatment in Reducing Marine Microplastics. *J. Environ. Manag.* **2020**, *266*, 110642. [CrossRef] [PubMed]
14. Tadsuwan, K.; Babel, S. Unraveling Microplastics Removal in Wastewater Treatment Plant: A Comparative Study of Two Wastewater Treatment Plants in Thailand. *Chemosphere* **2022**, *307*, 135733. [CrossRef] [PubMed]
15. Hu, Z.; Sun, Y.; Zhou, J.; Sun, W.; Shah, K.J. Microplastics in Wastewater Plants: A Review of Sources, Characteristics, Distribution and Removal Technologies. *J. Contam. Hydrol.* **2024**, *267*, 104448. [CrossRef]
16. Ormaniec, P.; Mikosz, J. Circulation of Microplastics in a Municipal Wastewater Treatment Plant with Multiphase Activated Sludge. *Desalin. Water Treat.* **2024**, *317*, 100265. [CrossRef]
17. Kurt, Z.; Özdemir, I.; James, R.A.M. Effectiveness of Microplastics Removal in Wastewater Treatment Plants: A Critical Analysis of Wastewater Treatment Processes. *J. Environ. Chem. Eng.* **2022**, *10*, 107831. [CrossRef]
18. Franco, A.A.; Martín-García, A.P.; Egea-Corbacho, A.; Arellano, J.M.; Albendín, G.; Rodríguez-Barroso, R.; Quiroga, J.M.; Coello, M.D. Assessment and Accumulation of Microplastics in Sewage Sludge at Wastewater Treatment Plants Located in Cádiz, Spain. *Environ. Pollut.* **2023**, *317*, 120689. [CrossRef]
19. Edo, C.; González-Pleiter, M.; Leganés, F.; Fernández-Piñas, F.; Rosal, R. Fate of Microplastics in Wastewater Treatment Plants and Their Environmental Dispersion with Effluent and Sludge. *Environ. Pollut.* **2020**, *259*, 113837. [CrossRef]
20. Sánchez-Guerrero-Hernández, M.J.; González-Fernández, D.; Sendra, M.; Ramos, F.; Yeste, M.P.; González-Ortegón, E. Contamination from Microplastics and Other Anthropogenic Particles in the Digestive Tracts of the Commercial Species *Engraulis encrasicolus* and *Sardina pilchardus*. *Sci. Total Environ.* **2023**, *860*, 160451. [CrossRef] [PubMed]
21. Kazour, M.; Terki, S.; Rabhi, K.; Jemaa, S.; Khalaf, G.; Amara, R. Sources of Microplastics Pollution in the Marine Environment: Importance of Wastewater Treatment Plant and Coastal Landfill. *Mar. Pollut. Bull.* **2019**, *146*, 608–618. [CrossRef]
22. Sun, J.; Dai, X.; Wang, Q.; Van Loosdrecht, M.C.M.; Ni, B.-J. Microplastics in Wastewater Treatment Plants: Detection, Occurrence and Removal. *Water Res.* **2019**, *152*, 21–37. [CrossRef]
23. Murphy, F.; Ewins, C.; Carbonnier, F.; Quinn, B. Wastewater Treatment Works (WwTW) as a Source of Microplastics in the Aquatic Environment. *Environ. Sci. Technol.* **2016**, *50*, 5800–5808. [CrossRef]
24. Franco, A.A.; Iglesias-Arroyo, D.; Egea-Corbacho, Á.; Martín-García, A.P.; Quiroga, J.M.; Coello, M.D. Influence of Tourism on Microplastic Contamination at Wastewater Treatment Plants in the Coastal Municipality of Chiclana de La Frontera. *Sci. Total Environ.* **2023**, *900*, 165573. [CrossRef]
25. Magni, S.; Binelli, A.; Pittura, L.; Avio, C.G.; Della Torre, C.; Parenti, C.C.; Gorbi, S.; Regoli, F. The Fate of Microplastics in an Italian Wastewater Treatment Plant. *Sci. Total Environ.* **2019**, *652*, 602–610. [CrossRef]
26. Prasetya, K.D.; Hassan, F.; Yen, Y.-T.; Chen, P.-Y.; Jiang, J.-J.; Wang, Y.-F.; You, S.-J. Assessing the Fate of Microplastics in Multi-Stage Treatment Units through Distribution Patterns and Settling Dynamics Models. *J. Contam. Hydrol.* **2026**, *276*, 104734. [CrossRef]
27. Nematollahi, M.J.; Mobasheri, M.; Esmaeili, Z.; Mahmoudi, M.; Yousefi, N.; Busquets, R. Distribution and Abundance of Microplastics in Urban and Industrial Wastewater Treatment Plants in Tabriz Metropolis. *Sci. Rep.* **2025**, *15*, 24577. [CrossRef]
28. Akarsu, C.; Kumbur, H.; Gökdağ, K.; Kıdeyş, A.E.; Sanchez-Vidal, A. Microplastics Composition and Load from Three Wastewater Treatment Plants Discharging into Mersin Bay, North Eastern Mediterranean Sea. *Mar. Pollut. Bull.* **2020**, *150*, 110776. [CrossRef] [PubMed]
29. Ziajahromi, S.; Neale, P.A.; Rintoul, L.; Leusch, F.D.L. Wastewater Treatment Plants as a Pathway for Microplastics: Development of a New Approach to Sample Wastewater-Based Microplastics. *Water Res.* **2017**, *112*, 93–99. [CrossRef] [PubMed]

30. Hajji, S.; Ben-Haddad, M.; Abelouah, M.R.; De-la-Torre, G.E.; Alla, A.A. Occurrence, Characteristics, and Removal of Microplastics in Wastewater Treatment Plants Located on the Moroccan Atlantic: The Case of Agadir Metropolis. *Sci. Total Environ.* **2023**, *862*, 160815. [CrossRef]
31. Luo, Y.; Xie, H.; Xu, H.; Zhou, C.; Wang, P.; Liu, Z.; Yang, Y.; Huang, J.; Wang, C.; Zhao, X. Wastewater Treatment Plant Serves as a Potentially Controllable Source of Microplastic: Association of Microplastic Removal and Operational Parameters and Water Quality Data. *J. Hazard. Mater.* **2023**, *441*, 129974. [CrossRef]
32. Üstün, G.E.; Bozdaş, K.; Can, T. Abundance and Characteristics of Microplastics in an Urban Wastewater Treatment Plant in Turkey. *Environ. Pollut.* **2022**, *310*, 119890. [CrossRef] [PubMed]
33. Napper, I.E.; Parker-Jurd, F.N.F.; Wright, S.L.; Thompson, R.C. Examining the Release of Synthetic Microfibres to the Environment via Two Major Pathways: Atmospheric Deposition and Treated Wastewater Effluent. *Sci. Total Environ.* **2023**, *857*, 159317. [CrossRef]
34. Gao, Z.; Cizdziel, J.V.; Wontor, K.; Vianello, A. Spatiotemporal Characteristics of Microplastics in a University Wastewater Treatment Plant: Influence of Sudden on-Campus Population Swings. *J. Environ. Chem. Eng.* **2022**, *10*, 108834. [CrossRef]
35. Andrady, A.L. Microplastics in the Marine Environment. *Mar. Pollut. Bull.* **2011**, *62*, 1596–1605. [CrossRef]
36. Kudzin, M.H.; Piwowarska, D.; Festinger, N.; Chruściel, J.J. Risks Associated with the Presence of Polyvinyl Chloride in the Environment and Methods for Its Disposal and Utilization. *Materials* **2023**, *17*, 173. [CrossRef] [PubMed]
37. Okoffo, E.D.; Rauert, C.; Thomas, K.V. Mass Quantification of Microplastic at Wastewater Treatment Plants by Pyrolysis-Gas Chromatography–Mass Spectrometry. *Sci. Total Environ.* **2023**, *856*, 159251. [CrossRef] [PubMed]
38. Zhang, Z.; Liu, W.; Gao, Q.; Huang, F.; Kang, Y.; Pan, Y.; Luo, G.; Zhou, X.; Fan, L.; Wang, A.; et al. Microplastics Extraction from Wastewater Treatment Plants: Two-Step Digestion Pre-Treatment and Application. *Water Res.* **2023**, *230*, 119569. [CrossRef]
39. Ridall, A.; Farrar, E.; Dansby, M.; Ingels, J. Influence of Wastewater Treatment Plants and Water Input Sources on Size, Shape, and Polymer Distributions of Microplastics in St. Andrew Bay, Florida, USA. *Mar. Pollut. Bull.* **2023**, *187*, 114552. [CrossRef] [PubMed]
40. Martín-García, A.P.; Egea-Corbacho, Á.; Franco, A.A.; Rodríguez-Barroso, R.; Coello, M.D.; Quiroga, J.M. Grab and Composite Samples: Variations in the Analysis of Microplastics in a Real Wastewater Treatment Plant in the South of Spain. *J. Environ. Chem. Eng.* **2023**, *11*, 109486. [CrossRef]
41. Bayo, J.; Olmos, S.; López-Castellanos, J. Microplastics in an Urban Wastewater Treatment Plant: The Influence of Physicochemical Parameters and Environmental Factors. *Chemosphere* **2020**, *238*, 124593. [CrossRef]
42. Blair, R.M.; Waldron, S.; Gauchotte-Lindsay, C. Average Daily Flow of Microplastics through a Tertiary Wastewater Treatment Plant over a Ten-Month Period. *Water Res.* **2019**, *163*, 114909. [CrossRef]
43. Browne, M.A.; Crump, P.; Niven, S.J.; Teuten, E.; Tonkin, A.; Galloway, T.; Thompson, R. Accumulation of Microplastic on Shorelines Woldwide: Sources and Sinks. *Environ. Sci. Technol.* **2011**, *45*, 9175–9179. [CrossRef]
44. De Falco, F.; Gullo, M.P.; Gentile, G.; Di Pace, E.; Cocca, M.; Gelabert, L.; Brouta-Agnésa, M.; Rovira, A.; Escudero, R.; Villalba, R.; et al. Evaluation of Microplastic Release Caused by Textile Washing Processes of Synthetic Fabrics. *Environ. Pollut.* **2018**, *236*, 916–925. [CrossRef] [PubMed]