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Investigation of Removal Efficiency of Microplastics at Different Process Stages of a Wastewater Treatment Plant in the Textile Industry in Southern China

Yanjing Zhu [1], Sijia Yang [1], Mathias Gustavsson [2], Wenli Huang [3], Si Gao [1] and Rui Wang [1,*]

[1] IVL Swedish Environmental Research Institute, Beijing Representative Office, Beijing 100005, China; yanjing.zhu@ivl.se (Y.Z.); sijia.yang@ivl.se (S.Y.); si.gao@ivl.se (S.G.)
[2] IVL Swedish Environmental Research Institute, 100 31 Stockholm, Sweden; mathias.gustavsson@ivl.se
[3] College of Environmental Science and Engineering, Nankai University, Tianjin 300350, China; huangwenli@nankai.edu.cn
* Correspondence: rui.wang@ivl.se

Abstract: Wastewater treatment plants (WWTPs) play a crucial role in mitigating microplastic (MP) release to the environment. In this paper, a WWTP of a textile manufacturing plant in Guangdong, China, was investigated to identify MP characteristics and the effectiveness of wastewater treatment within the plant. Laser Direct Infrared (LDIR) and Liquid Chromatography with Mass Spectrometry (LC-MS/MS) were applied to quantify both the number and the mass of the microplastics in the effluent of the textile manufacturing plant where most of the wastewater were from three printing and dyeing lines. The study further investigated the MP removal efficiency of each wastewater treatment process of the industry-owned WWTP and analysed the removal mechanism of each step, highlighting limitations in detecting and eliminating MPs. It is observed that (1) the results from LDIR and LC-MS/MS can be complementary to each other; (2) the MP concentration in the influent was 1730 n/L by number and 13.52 µg/L by mass; (3) the total removal efficiency of the WWTP were 99% by the number of MPs and 67.7% by the mass of MPs; (4) nine types of polymers have been identified in the influent, of which Polyamide (PA) was dominating; (5) hydrolysis acidification removed PA most; (6) aerobic tank, sand filter, and biological aerated filter (BAF) showed low removal efficiency; (7) coagulation and sedimentation tank had the highest removal efficiency to PET than any other processes.

Keywords: microplastics; textile production; printing and dyeing; removal efficiency; China; wastewater treatment

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1. Introduction

Microplastics (MPs) are small plastic particles, less than 5 mm in size, that have become a significant environmental concern due to their widespread distribution and persistence in marine and terrestrial environments. MPs are broadly categorised into primary MPs, which are intentionally manufactured at a small size (e.g., microbeads in cosmetics), and secondary MPs, which result from the breakdown of larger plastic items [1,2]. MPs can further be categorised into four morphological particle groups: plastic fibres (microfibres), plastic fragments, microbeads, and pellets [3]. Of these, microfibres, which are most commonly derived from synthetic textiles, are characterised by their elongated, thread-like structure, and are one of the most prevalent types of microplastics found in environmental samples [4].

MPs pose ecological and health risks by being ingested by marine organisms, accumulating in the food chain, and potentially transferring toxic substances [5–7] as well as additives and other substances used in the formulation of plastic materials [8]. Studies have documented microplastic ingestion in a variety of species, including zooplankton, fish, and shellfish, which can lead to physical harm, disrupted feeding patterns, and chemical toxicity [9–13]. MPs are reported to pose a risk to aquatic organisms by destroying their digestive systems, and bringing toxic substances and microorganisms into their bodies [14]. MPs can be more easily transferred and accumulated in the food chain [15,16] where human beings are the final receptors. Furthermore, studies in recent years have verified the existence of MPs in the human organs (e.g., lung and liver), gastrointestinal and respiratory tracts (faeces and sputum), and body fluids (e.g., blood and breast milk) [17–23]. Therefore, it is necessary to take action to reduce the emission of MPs into the environment.

Wastewater treatment plants (WWTPs) play a critical role in mitigating pollutants in water as they are designed to capture and remove contaminants from water before discharge into natural bodies. It has been claimed that the effluent from WWTPs is the most important contributor to MPs entering the aquatic ecosystem and the terrestrial environment, as most WWTPs are not designed for MP removal and cannot entirely remove MPs from wastewater [24–31]. Studies have summarised that the amount of MP concentration in the WWTPs' influent varied between 0.28 MP particles per litre (n/L) to 31,400 n/L [29]. Studies have also shown that most WWTPs can retain 70–95% of MPs, which indicates that a proportion of MPs continue to enter the water body from WWTPs [22,32]. Tang and Hadibarata [33] reviewed more than 80 scientific papers on MP removal efficiency by WWTPs which are equipped with primary treatment, secondary treatment, and tertiary treatment processes. They summarised the removal efficiency by different treatment processes as follows: the primary treatment can remove 16.5–98.4% MPs, the removal efficiency of secondary treatment ranges from 7.0% to 99.9%, and the tertiary treatment can remove 40.0% to 99.1% of MPs. Jiang's study [34] further proved that even the same treatment process can remove MPs at different levels. Additionally, research also shows that microplastic concentration tends to increase at different stages of the water treatment process, creating a potential hotspot of MPs spreading, especially in the events of sanitary sewer overflew occurring during flash flooding or snow melting [35]. In addition, as a part of the wastewater treatment process, many toxic substances can be absorbed by MPs, which makes MPs more harmful to the natural environment and ecosystems [6]. These findings indicate that the removal efficiency of WWTPs varies as the differences exist in the influent concentration, the treatment process of WWTP, and even the same process with different operation conditions.

Compared with the extensive study on WWTPs' removal efficiency on MPs, the research to explore MPs' abundance in the effluent of the textile industrial WWTP is less [36–41]. The reported MPs in the effluent of the textile manufacturing sites ranged from 361.6 ± 24.5 n/L to 54,100 n/L of wastewater [36,41] with 23.5–95% of removal efficiency [39,42]. Given the limited research available on the release of MPs from industrial sectors, further studies are needed in this area.

Meanwhile, the global production of synthetic textiles increased from 45 million tons in 2010 to 68 million tons in 2020 and the share of synthetic fibres as compared to total production went from 57% in 2010 to 62% in 2020 [42]. This increase in demand for synthetic textiles is driven by, among other things, fast fashion and durable synthetic fibres such as polyester and nylon [43]. This increase in synthetic textile production also increases potential risks for MP release from the textile production plants. Even though the recycling of fibres and textiles is expected to increase, this is from a low level [42,44]. Capturing MPs from the production sites to prevent their release to water recipients is one of the

many necessary steps to address the challenges of MPs in our environment. With a better understanding and more evidence of microfibres released by textile industries, it is crucial to understand how many MPs are contributed by the manufacturing plants and further identify its pathway within the industry-owned WWTPs.

This study aims to provide evidence of how MPs are reduced in wastewater through the different stages of the wastewater treatment process at a textile manufacturer in Southern China. The study adopted Laser Direct Infrared (LDIR) and Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS) to quantify both the number and the mass of the microfibres in the effluent of the textile manufacturing plant. The study further investigated the MP removal efficiency of each wastewater treatment process of the industry-owned WWTP, which may provide scientific support on the MP pollution elimination.

2. Material and Method

2.1. Sampling

The invested textile manufacturing plant is in Guangzhou City, Guangdong Province, China. The plant has production lines for spinning, knitting and weaving, printing and dyeing, finishing, as well as the production of final garments. The textile plant is equipped with a WWTP at the production site with a capacity of 25,000 ton of wastewater per day (Table 1). The treatment process, and where the sampling points were located, are shown in (Figure 1). Wastewater samples were collected in the influent of untreated wastewater from the textile production process (S1), and then of the effluent water from each treatment process (S2–S6) and the fully treated water (S7). During the sampling periods, the wastewater was mainly from three printing and dyeing lines but also some wastewater from other industrial workshops. Of the three printing and dyeing lines, three kinds of products were produced. Product 1 was formed by 90% Polyethylene terephthalate (PET) with 10% Polyurethane (PU, also known as Spandex). Product 2 was 80% PET with 20% PU, and product 3 contained 80% nylon with 20% PU.

Table 1. Key operational parameters of the WWTP stages.

Name of Process	Hydraulic Retention Time (min)	Capacity	Operational Parameters
pH regulation tank	45.0		pH: 9–11
Hydrolysis acidification tank	13.2	1.54 kg COD/m^3 filler per day	
Aerobic tank	672.0	Air-water ratio: 15:1, 0.48 kg BOD/m^3 filler per day	DO: 2–6 mg/L
Coagulation–sedimentation tank	31.5		PAC: 0.28 kg/m^3; PAM: 0.08 kg/m^3
Sand filter		0.66 $m^3/m^2/h$	$\leq$0.025 MPa
Ozone tank	96.0		Dosage of ozone: 25–35 ppm
Biological aerated filter (BAF)	86.0	0.34 kg BOD5/m^3/day	DO: 5–6 mg/L

Three parallel samples of 1 L each were taken from each sampling point. The samples were collected in stainless steel barrels and then transferred to glass bottles. All the equipment was carefully washed with ultrapure water before use. The samples were sealed and brought back to the laboratory and stored at 4 °C.

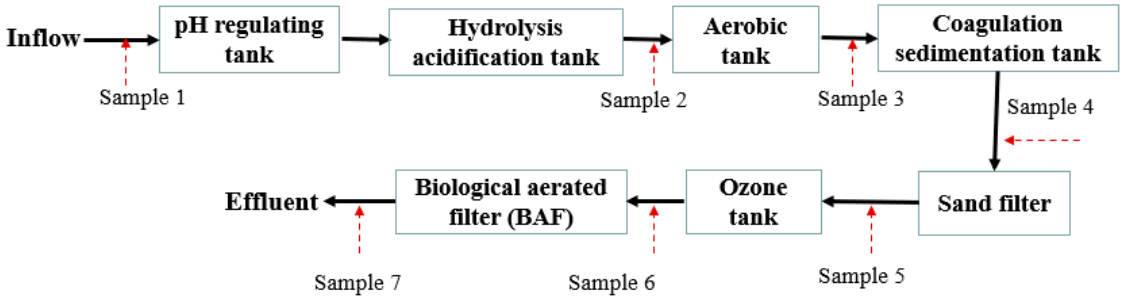

Figure 1. The schematic diagram of wastewater treatment processes and sampling points.

2.2. Isolation of MPs from Samples

For each sample, 500 mL of the sampled 1 L wastewater was filtered through a 5 mm stainless steel filter membrane (Jiuding High-tech Filter Equipment Co., Ltd., Nanjing, China). The membrane was rinsed three times with MilliQ water (Sigma Odrich, Biochemical Technology Co., Wuxi, China), and the sample was transferred to a new clean beaker. Then, the beaker was placed in an oven at a temperature of 60 °C until the water in the beaker evaporated completely.

Then, 20 mL of $FeSO_4$ (0.05 mol/L) (Aladdin Chemistry Co., Ltd., China, Shanghai, China) and 20 mL of 30% hydrogen peroxide (*v*/*v*) (Tianjin Solomon Biotechnology Co., Ltd., Tianjin, China) were added to the beaker successively [45]. Then, waiting for around 20 min to ensure the reaction was completed.

A saturated $ZnCl_2$ solution (Aladdin Chemistry Co., Ltd., China, Shanghai, China) with a density of 1.6–1.8 g/cm^3 was prepared following Coppock's method [46]. Then, it was applied to separate the mineral matters and polymers followed by the procedures of Schütze et al. [47]. As most of the density of most polymers ranges from 0.8 to 1.6 g/cm^3 [3], materials with a density higher than 1.6–1.8 g/cm^3 would fall to the bottom of the bottle and could be removed through a density separation process whereas the MPs floated on the surface.

2.3. Characterisation of the Polymer Composition of MPs

In the characterisation of MPs, two methods were applied, the Laser Direct Infrared Imaging (LDIR) method and Liquid Chromatography with Mass Spectrometry (LC-MS/MS).

2.3.1. LDIR Method

The detailed composition of MPs was determined by the LDIR chemical imaging system (8700 LDIR, Agilent Technologies Inc., Santa Clara, CA, USA), and MPs were detected in reflection mode by a Quantum Cascade Laser (QCL) equipped with the system. To determine detailed information on the MPs, the MPs on the stainless steel filter membrane were transferred to 1 mL of ethanol solution and then mixed. From this ethanol solution, 0.1 mL was removed for the detection and identification of MPs in the LDIR. The samples were initially located individually at 1442 cm^{-1} based on their X–Y coordinates and the composition of each particle was identified within the wavenumber range of 975–1800 cm^{-1}. The infrared spectrum analysis was carried out by the software in the LDIR system database (8700 LDIR, Agilent Technologies Inc.), and the material was identified when the matching degree with the standard spectrum was higher than 75%. MPs with a particle size greater than 20 µm were detected. Each sample was tested in triplicate to identify the types of polymers and the number of each kind of identified polymers. For the same kind of polymer, the average value of its number was taken.

2.3.2. LC-MS/MS Method

The LC-MS/MS (G6460C (ESI-MS/MS)) method was adopted to measure the mass of PET, polycarbonate (PC), and Polyamide 66 (PA66) [48,49]. Theoretically, PET, PC, and PA66 can all be depolymerized according to their reactions to obtain the corresponding monomers. Therefore, PET, PC, and PA66 can be quantitatively calculated based on the concentrations of their monomeric compounds Pure Terephthalic Acid (PTA), Bis Phenol-A (BPA), and Adipic Acid (AA) [48,49].

For PET and PC, the sample was added to 20 mL of 1-pentanol solution (Aladdin Chemistry Co., Ltd., China, Shanghai, China) along with KOH (Aladdin Chemistry Co., Ltd., China, Shanghai, China) and heated to reflux for 30 min. After the solution was cooled to room temperature, it was transferred to a 50 mL polypropylene (PP) tube, the beaker was washed two times with 20 mL of ultrapure water (Sichuan Youpu Ultrapure Technology Co., Ltd., Chengdu, China) and the washing liquid was transferred to the above tube. The PP tube was shaken at 180 rpm for 5 min and then centrifuged at 3000 rpm for 5 min by a centrifuge. The organic phase above the amyl alcohol solution (Aladdin Chemistry Co., Ltd., China, Shanghai, China) was then collected, and 20 mL of ultrapure water was added to the remaining solution to repeat the extraction process. The pH of the solution was adjusted to 2–3 before solid-phase extraction (SPE) and the volume was concentrated to 5 mL by a cartridge (Poly-SeryHLB 6 cc/200 mg; CNW, Shanghai, China). The solvent was evaporated under a gentle stream of nitrogen (Tianjin Baishida Gas Co., Ltd., Tianjin, China), reconstituted with 1 mL of water/acetonitrile (1:1, v/v) solution (Aladdin Chemistry Co., Ltd., China, Shanghai, China), and transferred to an autosampler vial for analysis. For PA66, we added the sample to 20 mL H_2SO_4 (40% v/v) at 135 °C ± 5 °C, mixed, and heated for 5 h. The solution was cooled and transferred to a 50 mL PP tube, and the flask was washed twice with 20 mL of ultrapure water and mixed with the depolymerization solution. When the solution was concentrated to 10 mL, an equal amount of ethyl acetate solution (Aladdin Chemistry Co., Ltd., China, Shanghai, China) was added to extract AA, and the sample was mixed by a shaker for 30 min and centrifuged at 3000 rpm for 5 min in a centrifuge. The experiment was repeated three times, ethyl acetate was collected in a new tube and evaporated to dryness, reconstituted with 1 mL of water/methanol (1:1, v/v), and transferred to an autosampler vial for the LC-MS/MS analysis.

3. Results

The result from the different sample points (S1–S7) in terms of the count of MP particles (n/L) at each sampling point is presented in Table 2, and Table 3 provides the results of the average size of MPs identified at each sampling point. Each sampling stage is presented in some detail in the following sections, including a presentation of the reduction over the full wastewater treatment system.

Table 2. Number of MPs in each sampling point (n/L).

Type of Polymers	Sampling Point						
	S1	**S2**	**S3**	**S4**	**S5**	**S6**	**S7**
ABS	10	150	0	0	0	0	0
PE-C	150	20	20	20	0	10	0
PA	1400	20	0	0	30	0	0

Table 2. *Cont.*

Type of Polymers	Sampling Point						
	S1	**S2**	**S3**	**S4**	**S5**	**S6**	**S7**
PE	60	0	0	10	10	10	0
PP	10	40	10	20	10	10	10
PTFE	10	0	70	10	0	10	0
POM	70	90	0	0	0	0	0
PC	10	0	0	0	0	0	0
PET	10	20	10	10	10	0	0
SUM	**1730**	**340**	**120**	**90**	**70**	**40**	**10**

Table 3. Average size polymers in each sampling point (μm). Grey cells did not have any MP identified and hence, no average size could be given.

Type of Polymer	Sampling Point, Average Size Polymers (μm)						
	S1	**S2**	**S3**	**S4**	**S5**	**S6**	**S7**
ABS	86	58	-	-	-	-	-
PE-C	143	73	58	58	-	57	-
PA	88	186	-	-	132	-	-
PE	105	-	-	73	133	63	-
PP	98	92	73	90	70	65	68
PTFE	70	-	90	186	-	98	-
POM	786	90	-	-	-	-	-
PC	324	-	-	-	-	-	-
PET	333	142	105	105	210	-	-
Average size of MP (all polymers)	124	84	77	73	116	71	68

3.1. MPs in Influent (S1)

The concentration of MPs in the influent (S1) was 1730 n/L covering nine types of polymers. These were Acrylonitrile Butadiene (ABS), Polyamide (PA), Chlorinated Polyethylene (PE-C), Polyethylene (PE), polypropylene (PP), Polytetrafluoroethylene (PTFE), Polyoxymethylene (POM), PC, and PET. PA was dominating, accounting for 81% of the total number of MPs. PE-C and POM were the second and third biggest contributors, responsible for 9% and 4% of the MPs in the influent, respectively. The total concentration of the rest 6 types of polymers was 6%. There was no PU MP detected in the influent.

The concentration of MPs (PA66, PE, and PC) by weight was 13.52 μg/L in the influent (S1). The separate concentrations of PET, PA66, and PC in the influent were 3.39 μg/L, 8.91 μg/L, and 1.21 μg/L, respectively. In line with the number concentration, PA was the dominating polymer by mass.

3.2. MPs in the Effluent After Each Treatment Process (S2–S7)

3.2.1. Hydrolysis Acidification Tank (S2)

The total MP concentration decreased from 1730 n/L in S1 to 340 n/L in S2 after the hydrolysis acidification tank (Table 1). The number of PAs decreased from 1400 n/L to 20 n/L. PE-C decreased from 150 n/L to 20 n/L. However, the numbers of ABS, POM, PP, and PET found increased from 10 n/L to 150 n/L, 70 to 90 n/L, 10 n/L to 40 n/L, and 10 n/L to 20 n/L, respectively. And, the top three types of polymers became ABS, POM, and PP in S2. PC, PE, and PTFE were not detected.

Through the hydrolysis acidification process, the total mass concentration of PA66, PET, and PC became 9.30 μg/L. The mass concentration of PA66, PET, and PC decreased from 8.91 μg/L to 6.46 μg/L, 3.39 μg/L to 3.21 μg/L, and 1.21 μg/L to 1.07 μg/L, respectively.

3.2.2. Aerobic Tank (S3)

The number concentration of MPs after the aerobic tank (S3) was 110 n/L. Of the identified polymers, the number concentration of PE-C, PP, PTFE, and PET were 20 n/L, 10 n/L, 70 n/L, and 10 n/L, respectively. No ABS, POM, and PC were detected in S3 and neither in the effluent of the subsequent processes (S4 to S7). No ABS and POM were identified after the aerobic tank (S3), while 150 n/L and 90 n/L, respectively, were found in S2.

The mass concentration of PA66, PET, and PC was 9.30 μg/L. Specifically, it was 2.71 μg/L, 1.02 μg/L, and 5.57 μg/L for PET, PC, and PA66.

3.2.3. Coagulation–Sedimentation Tank (S4)

After coagulation and sedimentation (S4), the total MP concentration was 70 n/L, and the number concentrations of PE-C, PE, PP, PTFE, and PET were 20 n/L, 10 n/L, 20 n/L, 10 n/L, and 10 n/L.

The total mass concentration of PA66, PC, and PET was 7.08 μg/L, of which, the PA66 was dominating with the concentration of 4.13 μg/L, followed by PET (1.98 μg/L). The concentration of PC was 0.96 μg/L.

3.2.4. Sand Filter (S5)

After sand filtration (S5), the total number concentration of MPs was 60 n/L. It was observed that the number concentration of PA increased from 0 n/L to 30 n/L. The number concentration of PE and PET was 10 n/L, which is the same as that in S4. No number data on PTFE was gained. PP's number concentration decreased from 20 n/L to 10 n/L.

The mass concentration of PET, PC, and PA66 was 1.81 μg/L, 0.89 μg/L, and 2.99 μg/L, respectively. The total mass concentration decreased from 7.08 μg/L to 5.69 μg/L.

3.2.5. Ozone Tank (S6)

The concentration of MPs was 40 n/L after the ozonation tank (S6). Only PTFE, PP, PE, and PE-C were detected with sizes between 50 μm and 100 μm. The total removal efficiency increased to 97.7%. While the mass concentration of PET, PC, and PA66 decreased from 1.81 μg/L to 1.18 μg/L, 0.89 μg/L to 0.83 μg/L, and 2.99 μg/L to 2.77 μg/L, respectively.

3.2.6. Biological Aerated Filter (S7)

After the biological aerated filter (BAF), which is also the effluent from the WWTP (S7), only PP was found with a concentration of 10 n/L. It can be noted that the reduction of PP throughout the process was found at a concentration of about 10 n/L also in the earlier stages (S1, S3, S5, and S6).

3.3. Removal of MP in the Different Treatment Stages

The total MP removal efficiency by concentration (n/L) of the wastewater treatment plant was 99.4% (Figure 2). The total removal efficiency of the wastewater treatment plant by mass was 68%. Specifically, the concentration of PET, PA66, and PC in the influent were 3.39 μg/L, 8.91 μg/L, and 1.21 μg/L, while they were 1.06 μg/L, 2.55 μg/L and 0.75 μg/L in the effluent of BAF, respectively. Correspondingly, the total MP removal efficiency by mass of the wastewater treatment plant for PET, PC, and PA66 was 69%, 38%, and 71%. The total removal efficiency for PET, PC, and PA 66 by mass was 67.7% (Figure 3).

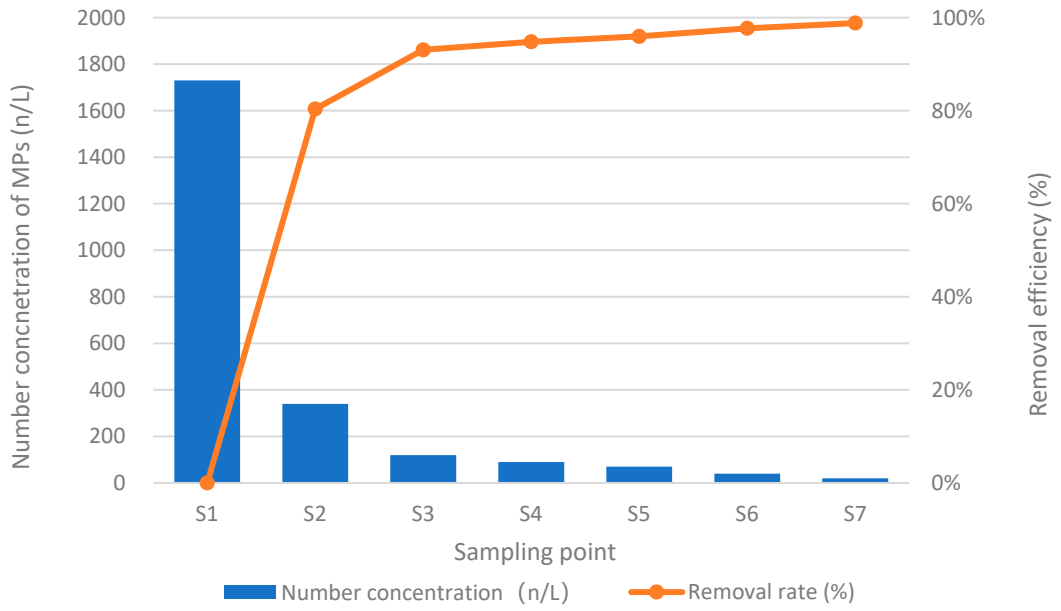

Figure 2. Removal efficiency of MPs by number.

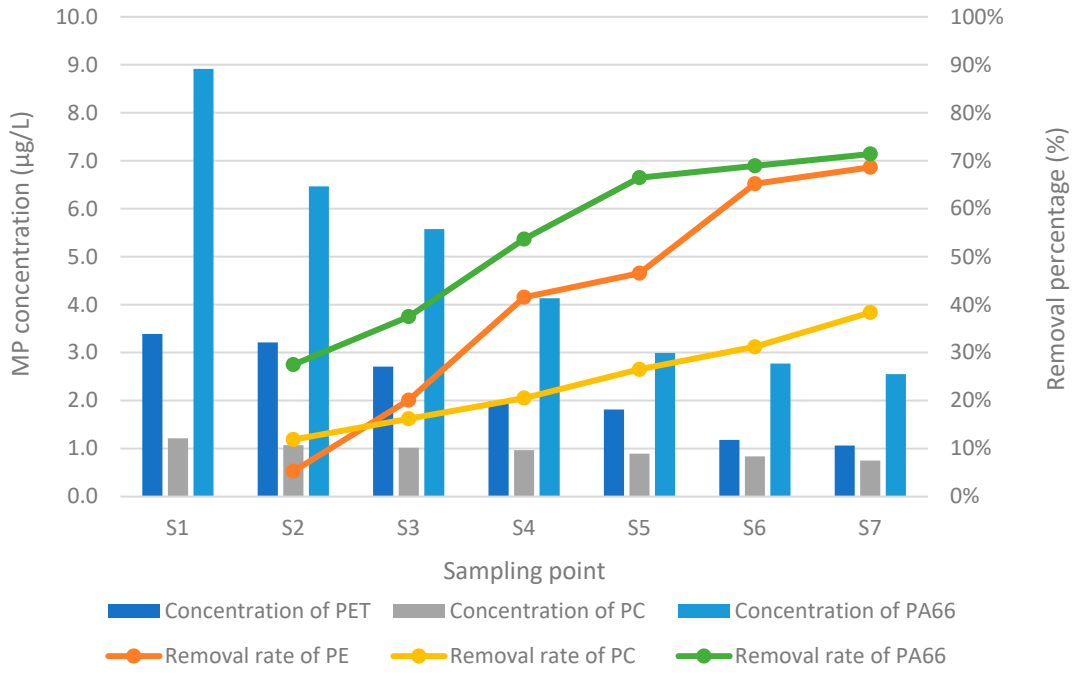

Figure 3. Removal efficiency of MPs by mass.

4. Discussion

4.1. MPs in Influent (S1)

The total number concentration of MPs in the influent in this study is higher than the number concentration of 57.6 n/L to 133 n/L and 361.6 ± 24.5 n/L reported in some previous studies [31,36]. At the same time, the sizes were smaller as compared to Zhou et al. [41] where 92% of the MPs were within the size range of 20–250 µm. In the present study, 77% of the MPs were less than 100 µm (Figure 4) which is in line with Haque [37] and Jian [50].

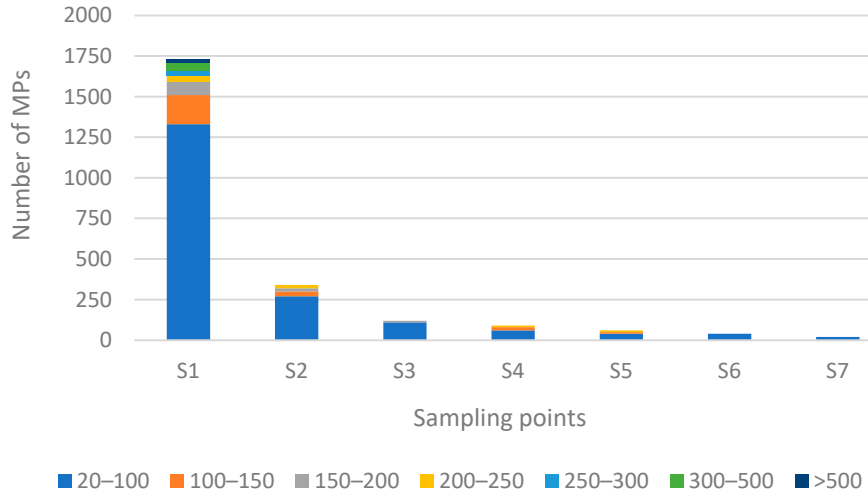

Figure 4. Size distribution (μm) of MPs at sampling points S1–S7.

The production information indicates that there should be PET, PU, and PA in wastewater. However, as there is no production volume information, it is impossible to connect the production volume with the number of MPs in the influent. In the detection of MP also PU was looked for, but none was found. One reason could be that the size of PU was lower than the detection limit (20 μm). Since some PU was part of the production process, it would be reasonable that some particles would have been detected.

4.2. Discussion of Removal Efficiency in the Treatment Process

4.2.1. Hydrolysis Acidification (S2)

The total removal efficiency of MPs by number in S2 was 80.3% by number (Figure 2). The total mass concentration of PA66, PET, and PC was 20.5%. Except for PA, all the other detected polymers' sizes decreased from S1 to S2. The deceased sizes, together with the removal efficiency both for number and mass prove the removal of MPs by hydrolysis acidification. Furthermore, it is observed that PA was removed most through the hydrolysis acidification supported by both the highest removal efficiency of PA (98.5%) by number and by mass (27%). A 5% PET removal efficiency on mass indicates that a small part of PET was transferred to the sludge. This result can be supported by the decreased size of PET (333 μm to 142 μm). The observation was in line with the study of Bayo, who found that a low pH value can facilitate a decrease in MP size [51]. The increased number concentration of PET, PP, POM, and ABS may be explained by the degradation caused by abrasion [50]. A 12% decreased mass concentration of PC means 12% of them was transferred into the sludge. No number of PCs was detected in S2, which can be explained by the fact that the size of PC here was lower than the detection limit. For PE-C, both the number and the sizes were decreased, meaning that part of PE-C was captured by the sludge and meanwhile, degradation happened. There was no PTFE found in the effluent of the hydrolysis acidification tank, but it was found again in the following processes. It may be because PTFE with a bigger size than the detection limit was removed from wastewater. However, PTFE was introduced again because of the PTFE application in wastewater transferring tubes or aerators.

4.2.2. Aerobic Tank (S3)

The aerobic tank removal rate was 12.7% of MPs by number in total. Of the removed MPs, the removal of ABS contributed to the highest share (9%) and POM to 5%. PET, PC, and PA66 were removed 11% in total in the process of aerobic by mass. PET, PC, and PA66 contributed to 3.7%, 0.4%, and 7%, respectively.

For PE-C, even though no change was recorded in the number concentration as compared to the previous stage, the average size recorded was reduced from 73.5 μm to 58 μm, indicating some PE-C mass was removed from wastewater. Both the number concentration result of PA and PET and the mass concentration results of PA66 and PET proved that the aerobic process could transfer PA and PET from wastewater to the sludge. Though no PA was found using the LDIR method, 5.57 μg/L of PA66 proved the existence of PA and indicated that the average size of PA was below the detection limits. The average size of PET (from 141.7 μm to 104.7 μm) decreased, which is reasonable as under anaerobic conditions, the generated OH^{-1} reacts with -C- and breaks down the PET chain supports [52,53]. There were no PC detected as part of the LDIR after the first sample point (S1). However, data on mass showed that around 4.3% of PC were removed. Combined with the removed PC in the above process, there was still 84% (by mass) of PC existing in wastewater with a size smaller than 20 μm. For PP, after the aerobic process, both the number and the size decreased (92 μm to 73 μm), which means a portion of PP was removed by this process.

4.2.3. Coagulation–Sedimentation Tank (S4)

The total number removal efficiency in S4 was 2.3%. The removal efficiency of PTFE was the highest (3.5%, from 70 μm to 10 n/L). The total mass removal efficiency of PA66, PC, and PET was 16% by mass.

The decreased mass concentration showed that the sedimentation tank is the most efficient process for removing PET and can remove 21.4% of the total PET. The reason could be that PET has a higher density than wastewater, and thus, was easily removed from the wastewater through physical sedimentation [5,24,53]. It is also applied to the PTFE, which has a bigger density than PET and has the highest number removal efficiency. There was no detection of the number of MP of PC and PA66 in S4. At the same time, the LC-MS/MS mass detection showed that there was a difference of 4.3% of PC and 16.2% of PA66 between S3 and S4. It indicates that the sizes of PC and PA were below the detecting limit (20 μm) of the LDIR method applied. The number and mass data of PET, PC, and PA show that the coagulation–sedimentation tank is more efficient in removing smaller size MPs. This is consistent with Na et al.'s [54] study but reasonable as the removal efficiency of coagulation–sedimentation is affected by both the operational factors (e.g., pH, coagulation dose, and interaction time) and characteristics of wastewater (e.g., size of MP, types of polymers, and the composition of the wastewater). The re-appearance of PE may be caused by the abrasion fragment of the tube, valves, and coagulants. It also may come from the sludge as Talvitie's study showed that 20% of MPs come back from sludge to wastewater [55].

4.2.4. Sand Filter (S5)

The total removal efficiency of MPs by sand filtration is 0.6% by number. The results show that it could remove 10.2% of PA66, PC, and PET by weight in total. Of them, PA66 has the highest removal efficiency (12.8%). Meanwhile, 5.0% of PET and 6.0% of PC were removed.

The MP removal mechanism of sand filtration is mainly mechanical straining, through which larger MPs are more likely to be retained on the filter [24,54,56]. Na et al.'s study showed that sand filtration can retain MPs bigger than 45 μm completely [54]. The low removal efficiency of the sand filter in this study is consistent with previous studies [57,58] and may be because most of the MPs with bigger sizes have been removed by previous processes and it could also be caused by the long-period operation limits the sand filter's capacity to retain much MPs [53]. Phillips and Prata reported that sand filtration can

contribute to degrading MPs into smaller pieces [57,58]. This may explain the increased number of PAs in our study. The concentration and average size of PP decreased to 50% and 22% of those in the effluent of sedimentation (S4), respectively. It means that the portion of PP was transferred to the sludge. The unchanged number concentration and decreased mass concentration of PET proved that smaller pieces were removed. No PC was detected by LDIR while LC-MS/MS caught the decreased mass concentration of PC, indicating the existence of PC in the effluent that was lower than the detecting limitation of LDIR, which is one main shortage of the LDIR method. For PP, the decreased size (90 µm to 70 µm) and number concentration indicated the removal of PP from wastewater.

4.2.5. Ozone Tank (S6)

The number concentration decreased by only 1.2%. However, the total removal efficiency of PET, PC, and PA66 was 6.7% on mass through the ozonation.

The decreasing trend of MPs in this process has proved that the existence of ozone can speed up the polymers' ageing by reacting with the polymers' main chains containing carbon–carbon double bonds, saturated hydro-carbon links, or aromatic rings [53,59,60]. It can explain why the number concentration did not change, but the sizes of PE and PP were observed to decrease in our study. For PET, PC, and PA66, the mass information confirmed their existence. Furthermore, 18.7% of PET, 4.7% of PC, and 2.5% PA66 were removed during this process, indicating that ozonation is more efficient in removing PET than PA and PC.

4.2.6. Biological Aerated Filter (S7)

The removal efficiency of MPs in the BAF was 1.7% by number. The mass removal efficiency of PET, PC, and PA66 was 3.2% in total. The biofilter removed the MPs through physical (filtration) and biological purification processes (adsorption and degradation) [38]. A previous study proved that after tertiary treatment, the remaining concentration of MPs was between 0.1 and 7.8% of the MP concentration in the influent. It is in line with our finding that the MPs in the effluent of BAF account for 0.6% of the MP number concentration in the influent (S1) [53]. The total removal efficiency of the BAF for PET, PC, and PA66 was only 3.1%. It is consistent with other studies conducted by [55,61]. However, it does not mean that BAF has a minor effect on MP removal as the previous processes (e.g., aerobic and sedimentation) have removed a significant share of the total MPs [24] where most of the MPs with bigger size and higher density have been removed [38]. The average size (68 µm) of MPs in the effluent of BAF was in line with other studies [62,63].

5. Conclusions

In this study, the direct emission of MPs from the outlet of the manufacturing workshop and the removal efficiency of the main processes of the industrial self-owned wastewater treatment plant were characterised by the LDIR and LC-MS/MS methods. The main findings are as follows: (i) Both methods have their limits but can be used together to complement each other. (ii) From the removal efficiency based on number, the total removal efficiency is quite high; however, when considering the mass removal efficiency, there is much room for improvement. (iii) For PA, hydrolysis acidification is most efficient; (iv) for PET, coagulation and sedimentation process is better than any other processes. (v) Aerobic tank, sand filtration, and the tertiary treatment of BAF showed less impact on the MPs' removal.

The total removal efficiency by number was 99%. However, the total removal efficiency of PA66, PET, and PC was 67.7%. The difference is mainly caused by that for the LDIR method, where most of the polymers can be identified; however, there was a detection limit

on size, which was 20 µm in our study, whereas the LC-MS/MS method has no detection limitation on size but is confined to limited type of polymers. It indicates the limits of each characterisation method.

It can be observed that in the WWTP's effluent, all of the PET, PC, and PA were removed totally. However, the adopted LC-MS/MS methods indicated that only 68.7% of PET, 71.4% of PA66 and 38.4% of PC were removed. Thus, the result analysis is much more convincing when combined with the results from the LC-MS/MS method and from the LDIR method as they are complementary to each other. Improved filtration technologies and innovations in MP capture are essential to enhance WWTP efficacy and reduce the environmental burden of textile-derived MPs from the production stage.

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