Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2021.27287

227 (2021) 26–33
July

Wastewater treatment plants' contribution in microplastics' contamination of surface water. Effluent concentration and detection techniques review

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Received 29 October 2020; Accepted 2 April 2021

ABSTRACT

Microplastics (MPs), recently gain notable attention since they are detected in water bodies. Increasing production and recurrent short-term usage of plastic products enhance their disposal rates. They are originated from land-based or sea-based sources related to anthropogenic activities. Wastewater treatment plants (WWTPs) and applied technologies are under investigation, as a potential source of microplastic contamination. Although typical WWTPs are found to be able to remove from 70% to more than 90% of microplastics from wastewater, these plastic particles, smaller than 5 mm, are detected in effluents. Even small concentrations of microplastics in WWTPs effluents can have a significant accumulative effect, since WWTPs release a large amount of treated wastewater continuously. It is worth mentioning that the daily discharge of microplastics from a WWTP, as estimated, can reach a few million particles. Microplastics' concentration that has been reported in effluents ranges between less than 0.5 particles/L to more than 50 particles/L. This study focuses on the contamination of surface waters from MPs, as a result of WWTPs' effluents. A thorough review of current research is targeted to summarize the reported MPs concentration measurements in the wastewater treatment processes, the WWTPs efficiency and MPs removal rates, as well as the prevailing MPs identification techniques.

Keywords: Microplastics; WWTPs efficiency; Microplastics' detection techniques; Wastewater treatment; Effluent concentration

1. Introduction

Plastic pollution is an increasingly major concern among scientific communities, as plastic production, consumption and waste disposal is intensifying. In the European Union alone 150,000–500,000 tons of plastic waste is identified in the ocean each year [1]. Not to mention that most plastic materials are non-biodegradable and their degradation can continue for 100 y, which is extremely disproportional with current plastic disposal rates. It is very important in the framework of waste management and taking into account the European Directive 2019/904 on the reduction of the impact of certain plastic products on the environment. Waste strategies [2,3] play a vital role in environmental protection.

Unfortunately, the COVID-19 pandemic pushes back against any effort that has recently been made in reducing plastic consumption and increasing public awareness. Plastic is the fundamental material for medical and protective equipment and single-use plastics are making people feel safer, during the pandemic, inducing an ongoing increase in single-use plastics and creating a new challenge in beating plastic pollution [4,5].

Microplastics (MPs) are extremely small plastic particles in the size of 1 nm–5 mm [6]. Microplastic pollution is caused

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Presented at the OpenEARTH Conference on Climate Change Adaptation and Mitigation, 12–14 February 2020, Thessaloniki, Greece

either by primarily produced particles on a micro-scale or by secondary produced particles as a result of degradation and fragmentation of larger particles [7].

Wastewater treatment plants (WWTPs) have recently been determined as a critical source of microplastics in the aquatic environment [8–10]. Urban wastewater, in the developed world, is collected and treated in WWTPs before released in the water aqueducts [11,12]. Apparently, WWTPs with existing treatment technology, cannot sufficiently remove microplastics, mainly due to their small size and the fact that they are not biodegradable. WWTPs can retain 70%–90% of microplastics during the first stages of treatment, that is, grit and grease removal chambers and primary sedimentation [13–16]. Although contemporary advanced treatment technologies such as membrane filtration can improve the microplastics removal competence, a small amount of MPs can still escape and reach surface water [17,18]. According to the literature, the most common MPs found in wastewater are fibers [14,18–20], which originate from personal care products and washed synthetic. It is estimated that 4,594–94,500 MPs can be released after a single usage of a personal care product containing micro-particles [21]; 6.0 kg washing machine cycle of synthetic textiles may release up to 700,000 plastic fibers to sewerage system [22]; 1.6 g of toothpaste could contain about 4,000 MP particles [13].

The contribution of this study is the collection and thorough review of the majority of recently published research (in the 7-y period 2013–2019) that focuses on MPs' detection in WWTPs. The identification of the reported detection techniques of MPs, and evaluation of the WWTPs' effluent concentration and removal rates of MPs after treatment, through this study, serve as the first step towards the recognition of the importance of emerging new technologies not only for the biological treatment of wastewater but also other constituents' removal.

2. Microplastics' detection in wastewater

According to recent bibliography, MPs' detection in wastewater comprises three main steps: a collection of samples, samples' preparation and pre-treatment, and particles' quantification and characterization [23]. In the following paragraphs, these three steps will be analyzed. MPs' concentration is expressed in a number of microplastics per mass or per volume of the sample matrix [18].

2.1. Sampling

Samples were usually taken from raw wastewater, from various stages of the treatment facility and from the WWTPs' effluents. In most cases, samples were taken from established sampling ports inside a WWTP, from outlet flumes or from treatment tanks, some centimeters below the surface of the water, or by skimming the surface of the water.

Sampling techniques included taking samples manually with a container or taking composite samples by automatic samplers. Samples can also be sieved in situ, in order to increase the volume of samples that can be analyzed [24–26]. Otherwise, wastewater can be sampled by pumping a large volume of water through one or a series of sieves with different mesh sizes. Researchers reported that when using containers in sampling, a lower volume of water can be collected, and samples are more susceptible to air-born contamination [27]. Precautionary measures must be taken during collection and analysis, to prevent sample contamination. Proper sampling requires rinsing of the equipment with water before use, avoiding synthetic textiles clothing, avoiding if possible plastic equipment, covering samples to protect them from contacting air, filtering of all used solutions [18,24,26,28,29]. It must be noted that, in all examined studies, blank samples were also analyzed to detect any sample contamination [15,16,25,26].

2.2. Samples' pre-treatment

The main target of pre-treatment is to remove all other particles that can be confounded with microplastics, such as organic and inorganic material, before quantification and characterization of microplastics. The most widely used pre-treatment procedures are organic matter removal and density separation.

A commonly applied method for organic matter removal is oxidation, by the use of hydrogen peroxide H_2O_2 [30]. This method requires a 3–7 d period to acquire sufficient results [20,31]. The required amount of time is reduced to some minutes (5–10 min) when applying H_2O_2 in the presence of an iron(II) catalyst, [14,29,32,33]. This process derives from Fenton's reaction and it is called wet peroxide oxidation (WPO) [34].

A more complex pre-treatment procedure includes samples' hydrolysis. It is applied by utilizing a cellulose digesting enzyme to digest cellulose fibers, followed by WPO [25,35], or by a multi-step plastic preservative enzymatic maceration, utilizing enzymes protease, lipase and cellulose, followed by hydrogen peroxide and chitinase solution application [15]. Furthermore, sodium dodecyl sulfate can be added to wastewater samples in order to achieve detachment of microplastic from other bigger organic and inorganic particles before filtration steps [15,25,35].

Density separation of microplastics from inorganic material, is another pre-treatment procedure which is suggested by using zinc chloride ($ZnCl_2$) solution [15,29], sodium chloride NaCl [18,26], or sodium iodide NaI [20].

Gies et al. [36] use a liquid–liquid extraction technique referred to as oil extraction protocol. This method takes advantage of the lipophilicity of plastic polymers. In this study, the canola oil was allowed to separate from the wastewater, and MPs were trapped in the canola oil layer.

Although a pre-treatment procedure is necessary for an efficient quantification and characterization of MPs in wastewater, it can have the opposite effect, if not applied properly. Pre-treatment may alter MPs' characteristics, such as their chemistry, structure and size [32]. However, the most frequently used WPO pre-treatment is found not to have any effect on the Fourier-transform infrared (FTIR) spectrum of microplastics [31].

2.3. Visual observation and characterization

The final stage in MPs' detection is visual quantification and characterization of microplastics. Visual analysis can be accomplished by the use of a microscope to identify the particles' characteristics, such as color and shape [23,37]. Under the microscope, organic material can be distinguished from other particles by observing characteristics such as apparent cellular structure or softness, friability and easy disintegration of material under mild pressure [13,27].

However, by using only visual observation, positive characterization of particles as MPs may be possibly false. Thus, FTIR and micro-FTIR analysis, which are based on characteristic spectra of MP particles, were applied in numerous studies sampling wastewater [18,20,24,26]. Up to 70%–90% of possible MPs detected by microscopy were not verified as microplastics by FTIR analysis [20,38,39]. Identification of MP particles, with a diameter of less than 1 mm, using solely microscopy could be arduous [40].

Ultimately, a combination of microscopy and spectroscopy can be applied. Microscopy serves as a preliminary step for the isolation of possible microplastic particles [26,39]. Then a subsample can be examined with spectroscopy. Alternatively, Song et al. [40] proposed the use of FTIR spectroscopy to define a set of criteria, which then will be used for identifying microplastics by examining samples in the microscope.

Other known techniques for particles characterization are Raman spectroscopy [41,42] and pyrolysis–gas chromatography–mass spectrometry (pyrolysis-GC–MS), even though it is not frequently used in analyzing wastewater samples.

FTIR microspectroscopy (micro-FTIR) is a tool that combines FTIR spectroscopy with microscopy [31] and enables the detection of small particles even down to 20 μm size. Application of modern focal plane array detectors, instead of single element detection, reduces required time for FTIR analysis, sustaining maximum resolution [15].

3. WWTPs' contribution to microplastic pollution

Recent studies reveal the presence of MPs in WWTPs effluents and report the efficiency of different treatment stages and technologies in MPs removal. Summarized reported effluent concentration and removal rates of MPs after wastewater treatment, are shown in Table 1, according to the recent bibliography (2013–2019).

Researchers examine WWTPs contribution to MPs pollution, by sampling raw wastewater, the outflow of each treatment stage of the plant and facilities' effluents. Both

Table 1
Reported effluent concentration and removal rates of MPs after wastewater treatment

Reference	Mean concentration (particles/L)	Removal rate (%)	Detection technique	Treatment level	Treatment type	Particles' size (μm)
[17]	52	–	Microscopy	Secondary	–	–
[17]	51	–	Microscopy	Tertiary	MBR	–
[19]	35	95	Microscopy	Secondary	Biofilters	100 – 5,000
[28]	8.6	98	Microscopy	Tertiary	Biological filtration	20 – > 200
[14]	0.06	–	Microscopy	Tertiary	Granular (gravel, sand, anthracite coal)/biological aerated filter	125 – > 355
[14]	0.05	–	Microscopy	Secondary	–	125 – > 355
[43]	5.9	93.8	Microscopy	Secondary	Activated sludge	20 – 4,750
[43]	2.6	97.2	Microscopy	Tertiary	Granular sand filter	20 – 4,750
[43]	0.5	99.4	Microscopy	Tertiary	Anaerobic MBR	20 – 4,750
[24]	0.25	98.4	Spectroscopy-(FTIR)	Secondary	Aeration basin	65 – > 5,000
[37]	0.089	–	Microscopy	Secondary	–	125 – > 355
[37]	0.083	–	Microscopy	Tertiary	–	125 – > 355
[20]	0.21	–	Spectroscopy-(FTIR)	Tertiary	Ultrafiltration – RO	25 – > 500
[20]	0.48	–	Spectroscopy-(FTIR)	Secondary	Secondary aeration	25 – > 500
[20]	1.54	–	Spectroscopy-(FTIR)	Primary	Sedimentation	25 – > 500
[18]	63	72	Spectroscopy-(FTIR)	Secondary	–	10 – 5,000
[18]	51		Spectroscopy-(FTIR)	Tertiary	MBR	10 – 5,000
[27]	0.03	98.5	Spectroscopy-(FTIR)	Tertiary	Discfilter	20 – > 300

Table 1 *Continued*

Reference	Mean concentration (particles/L)	Removal rate (%)	Detection technique	Treatment level	Treatment type	Particles' size (μm)
[27]	0.02	97.1	Spectroscopy-(FTIR)	Tertiary	Rapid sand filters	20 – > 300
[27]	0.1	95	Spectroscopy-(FTIR)	Tertiary	Dissolved air flotation	20 – > 300
[27]	0.005	99.9	Spectroscopy-(FTIR)	Tertiary	MBR	20 – > 300
[36]	0.5	98.3	Spectroscopy-(FTIR)	Secondary	Trickling filters	64 – > 64
[41]	0.0027	73	Spectroscopy-(Raman)	Secondary	Aeration tanks	55 – 5,000
[41]	0.0023	79	Spectroscopy-(Raman)	Secondary	Aeration tanks	55 – 5,000
[42]	1	98.3	Spectroscopy-(FTIR, Raman)	Secondary	Activated sludge	250 – > 5000
[42]	0.4	99.4	Spectroscopy-(FTIR, Raman)	Tertiary	MBR	250 – > 5000
[33]	0.44	98	Spectroscopy-(FTIR)	Secondary	Anaerobic-anoxic-aerobic (A2O)	106 – > 300
[33]	0.14		Spectroscopy-(FTIR)	Secondary	Sequence batch reactor (SBR)	106 – > 300
[33]	0.28		Spectroscopy-(FTIR)	Secondary	Media	106 – > 300
[25]	131.5	99.3	Spectroscopy-(FTIR)	Secondary	Activated sludge	10 – 500
[25]	19		Spectroscopy-(FTIR)	Tertiary	Rapid sand filtration	10–500
[44]	13.65	98.1	Microscopy	Secondary	Activated sludge	60 – > 418
[26]	0.4	84	Spectroscopy-(FTIR)	Tertiary	Sand filter	63 – 5,000

wastewaters originated from domestic, commercial and industrial uses or from combined sewer systems are tested. Sampled WWTPs show large heterogeneity in treatment types, however, in this research, the facilities are grouped by level of treatment, primary, secondary and tertiary. Primary treatment refers to screening, grit and grease removal including or not primary sedimentation; secondary treatment refers to biological treatment and sedimentation; tertiary refers to more advanced treatment, such as membrane bioreactor (MBR) or other advanced filtration techniques.

Detected effluent concentration varies between 0.0023–131.5 particles/L and removal rate between 72%–99.9%. The smallest particle that is considered by researchers, is 10 μm and the largest 5,000 μm, which is the upper size limit for a particle to be characterized as microplastic. These results are thoroughly discussed in the next section.

The statistical analysis was conducted with the implementation of the Python 3.7 programming language under the Spyder 3 integrated development environment, which are included in the Anaconda 3 package [45].

4. Results and discussion

After having identified the existence of MPs in wastewater and the prevailing detection techniques, the contribution of WWTPs to MP pollution remains to be addressed. That can be achieved either by estimating the concentration of MPs in the treated wastewater effluents, or by assessing the MP removal rates after the treatment.

The key findings of this research are presented in the following graphs and discussed in this section.

Fig. 1 summarizes the reported removal rates of MPs after wastewater treatment, included in the recent bibliography (2013–2019). Overall removal rates are between 72.0%–99.9%, and the majority of facilities are found to be able to remove more than 90% of inserted MPs. Whether this efficiency level

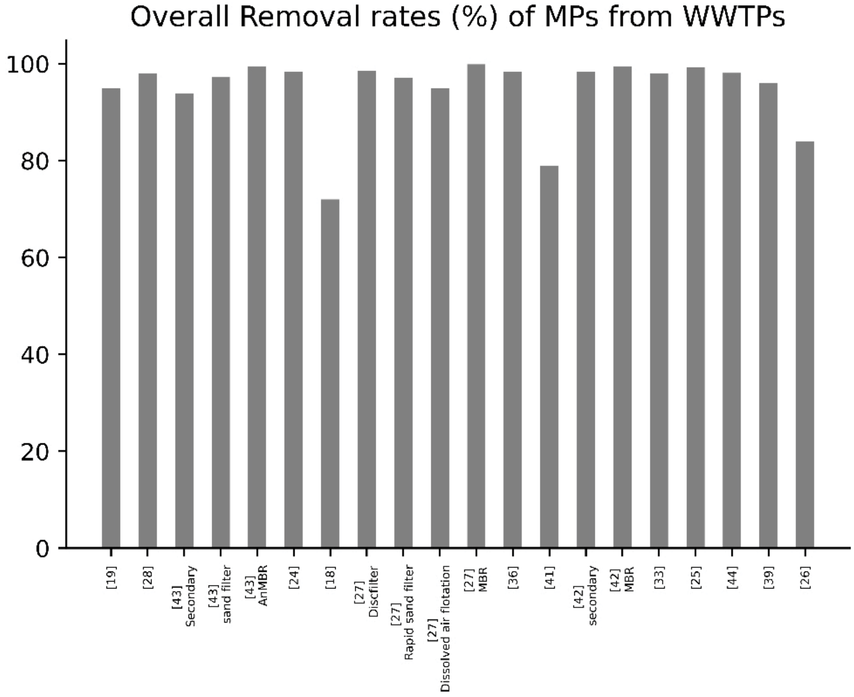

Fig. 1. Overall removal rates (%) of MPs from WWTPs.

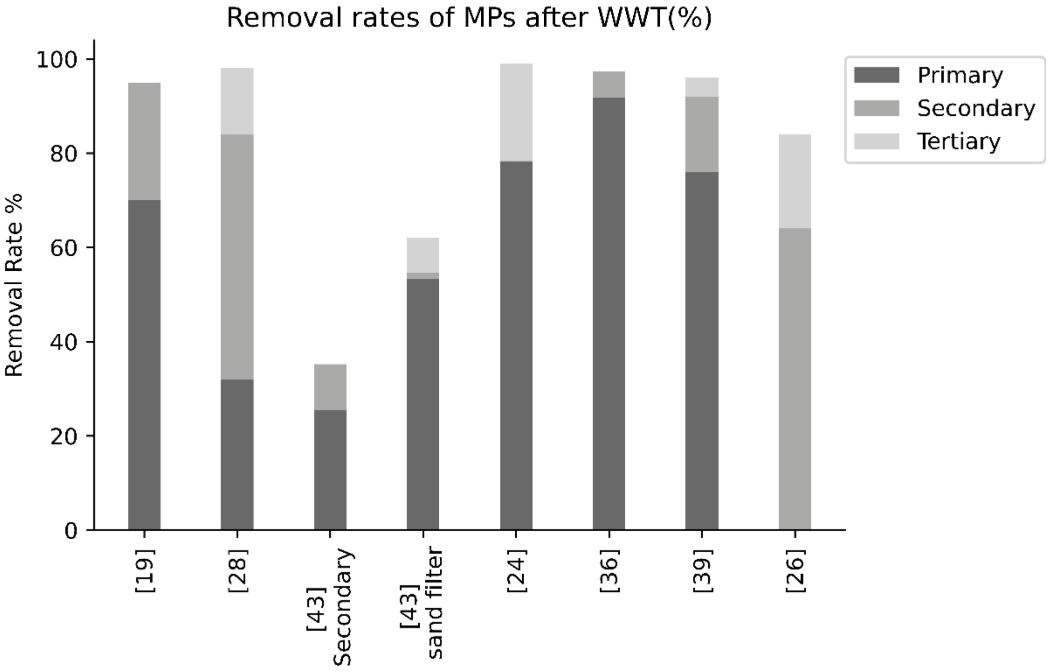

Fig. 2. MPs removal rates (%) after wastewater treatment.

of the WWTPs, with respect to the MPs removal, is satisfactory or not, in view of the environmental pollution and human health, remains to be investigated.

Fig. 2 presents the reported removal rates during different treatment stages of various WWTPs. It is worth mentioning that the MPs removal efficiency of WWTPs depends on the size of particles that are taken into consideration. Smaller particles are more abundant in the samples [25], and their removal rates are lower [15,16,19,44]. Among reviewed research papers, particles as small as 10 μm were measured only in two studies, while particles bigger than 20 μm were examined by the majority of the research teams. Nonetheless, MPs research has to develop methods to specifically target and detect nanoparticles (<<10 μm) in environmental matrices [18].

WWTPs are more effective in removing larger particles, according to studies that reported less or no large particles in effluent (>1 mm, in [19]), (>0.355 mm, in [14]), (>500 μm, in [15]), (>418 μm, in [44]). Higher retention percentages were reported in primary treatment stage (Fig. 2), whereas secondary and tertiary treatment exhibited significantly lower retention rates. It should be noted that particles which are removed from wastewater are re-directed to sludge treatment of the same facility [18,26,46]. As a result, wastewater sludge comprises another possible source of microplastics in the environment, which will be investigated separately.

Although reported MPs concentration in effluents is relatively low, in many cases lower than 0.5 particle/L, their accumulative effect cannot be neglected, since huge quantities of wastewater are treated and discharged every day [14,20,24,26,43]. Mason et al. [14] estimated a discharge of 4 million MPs/d from each facility and on average 13 billion MPs/d from all US WWTPs; Murphy et al. [24] assessed the discharged concentration to 65 million MPs/d from the facility under study; Magni et al. [26] measured 160 million MPs/d; Blair et al. [39] appraised 22 million MPs/d.

Reported MP concentration of effluents came from various WWTPs with either secondary or tertiary level of treatment. The results depict a very wide range of 0.005–131.0 particle/L. The three plants with recorded higher concentration are applying secondary treatment. However, there are also WWTPs that exhibit very low MP concentrations after biological treatment. There is no statistically significant difference between secondary and tertiary effluent concentration in reviewed data ($p = 0.12$, derived from Mann Whitney U-test with 95% confidence level). Given the small amount of available data, it is obvious that more data are needed in order to draw reliable results.

Simon et al. [25] exhibit the highest concentration of 131 particles/L, from secondary treatment, significantly higher than all other studies. Similarly, their results from tertiary treatment also show one of the highest effluent MP concentrations. They attributed this difference to smaller size particles that they were testing (10–500 μm). Other studies examined particles in the range of 20–500 μm, or bigger, up to 5 mm. Leslie et al. [18] also examined particles down to 10 μm and their results appear to have the second-largest concentration in the table. However, there are numerous other parameters concerning the WWTPs that can justify this wide range of values. Different treatment technologies, different treated volumes, different sampled volumes, land uses in the region, population served, sampling frequency and random variation in time can contribute to large heterogeneity of data. The lowest concentration values are lower than 0.5 particle/L. The lowest concentration (0.005 particle/L) was reported from Talvitie et al. [27] from the WWTP with tertiary treatment utilizing MBR technology. There are also secondary effluents with lower than 0.5 particle/L [14,20,24,41] from WWTPs that utilize activated sludge or aeration in secondary treatment and either microscopy or FTIR methods of characterization.

The data are still sparse and the influence of several factors, including seasonal and daily fluctuation and stormwater contribution in MPs concentration are not clear. Reported studies collect data with single sampling events and do not provide continuous data sets. Lares et al. [42], who sample every two weeks for a three months period, reported variability on MPs concentration during sampling campaign. On the other hand, Conley et al. [44], whose sampling campaign take place over the course of a year, do not notice any recognizable seasonal pattern across WWTPs.

Lee and Kim [33] found greater amounts of tire fragments and they attributed this to surface runoff, since they conducted their research during rainy weather. The concentration of other types of MPs appeared reduced, while precipitation increased, probably because of dilution.

5. Conclusions

MPs detection in wastewater treatment facilities comprises three main steps: wastewater sampling, pre-treatment for particles extraction and particles' quantification and characterization. Higher removal rates are achieved during primary treatment, grit and grease removal and primary sedimentation. It is documented that WWTPs retain more than 90% of inserted MPs; nonetheless, millions of tonnes of MPs can be discharged from an ordinary WWT facility every day.

There is not a clear correlation between effluent MP concentration values and applied final level of wastewater treatment. Therefore, removal efficiency of different tertiary treatment technologies should be more thoroughly examined.

The available data on WWTPs' effluent concentration is extremely heterogeneous and difficult to be assessed. Moreover, a universal laboratory analysis protocol for MPs detection in wastewater must be developed, in order to achieve standardization in results' assessment.

Finally, identification and assessment of fate, transport and removal of MPs from WWTPs is essential towards the containment and management of MPs in the environment. Towards this direction is also the detection and removal of MPs in the WWTPs sludge treatment procedures.

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