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REVIEW PAPER

Current research trends on microplastic pollution from wastewater systems: a critical review

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Abstract Microplastics have been widely considered as contaminants for the environment and biota. Till now, most previous studies have focused on the identification and characterization of microplastics in freshwater, sea water, and the terrestrial environment. Although microplastics have been extensively detected in the wastewater, research in this area is still lacking and not thoroughly understood. To fill this knowledge gap, the current review article covers the analytical methods of microplastics originating from wastewater streams and describes their sources and occurrences in wastewater treatment plants (WWTPs). Studies indicated that microplastic pollution caused by domestic washing of synthetic fibers could be detected in the effluent; however, most microplastics from personal care and cosmetic products (PCCPs) can be efficiently removed during wastewater treatment. Moreover, various techniques for sampling and analyzing microplastics from wastewater systems are reviewed; while, the implementation of standardized protocols for microplastics is required. Finally, the fate of microplastics during wastewater treatments and the environmental contamination of effluent to environment are presented. Previous studies reported that the advanced wastewater treatment (e.g., membrane bioreactor) is needed for improving the removal efficiency of small-sized microplastics (< 100 μm). Although the role of microplastics as transport vectors for persistent organic pollutants (POPs) is still under debate, they have demonstrated abilities to absorb harmful agents like pharmaceuticals.

Keywords Microplastics · Wastewater systems · Analytical techniques · Wastewater treatment plant · Environmental contamination

Abbreviations

ATR	Attenuated total reflectance
$CaCl_2$	Calcium chloride
FTIR	Fourier-transform infrared spectroscopy
Fe	Iron
$FeSO_4$	Iron (II) sulfate
FPA	Focal plane array
H_2O_2	Hydrogen peroxide
HNO_3	Nitric acid
HDPE	High density polyethylene
KOH	Potassium hydroxide
KI	Potassium iodide
NaCl	Sodium chloride
NaI	Sodium iodide
POP	Persistent organic pollutant

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PCCP	Personal care and cosmetic product
PE	Polyethylene
PEST	Polyester
PVC	Polyvinyl chloride
PS	Polystyrene
POM	Polyoxymethylene
PP	Polypropylene
PET	Polyethylene terephthalate
PC	Polycarbonate
PU	Polyurethane
PEVA	Poly(ethylene–vinyl acetate)
PA	Polyamide
Py–GC/MS	Pyrolysis–gas chromatography/mass spectrometry
PO	Polyolefin
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
SPT	Sodium polytungstate
TGA–DSC	Thermogravimetric analysis–differential scanning calorimetry
UV	Ultraviolet
WWTP	Wastewater treatment plant
WPO	Wet peroxide oxidation
$ZnCl_2$	Zinc chloride

1 Introduction

Microplastics, commonly defined as plastics smaller than 5 mm in diameter, are ubiquitous in various aquatic and terrestrial environments and biota (Hanvey et al. 2017; Alimi et al. 2018). This microplastics pollution could affect both the food chain and human health (Barboza et al. 2018; Carbery et al. 2018; Waring et al. 2018). In general, according to the routes in which they are generated, the microplastics can be broadly classified into: (1) primary microplastics originating from everyday use products (e.g., facial cleansers, scrubbers, and tooth paste) and (2) secondary microplastics are caused by the fragmentation of large plastics into small debris through photo-oxidation, mechanical, chemical, or biological interactions (Li et al. 2016; Auta et al. 2017).

In recent decades, the pollution by microplastics in both aquatic and terrestrial environments is of growing global concern, owing to: (1) poor degradability and thus leads to accumulation; (2) the possibility to sorb persistent organic pollutants (POPs) due to their large specific surface areas; and (3) they may be potentially ingested by fish and other living organisms (Jiang 2017; Wang et al. 2018a). Previous literature has stated that the microplastics can be detected in all environmental systems and biota, such as rivers, lakes, oceans, sediments, marine animals, and soils (Ng and Obbard 2006; Vianello et al. 2013; Cheung et al. 2018; Pellini et al. 2018; Rodrigues et al. 2018; Scheurer and Bigalke 2018; Xiong et al. 2018).

Until now, most recent reviews have focused on the microplastics in the freshwater, ocean, and terrestrial environments (Andrady 2011; Cole et al. 2011; Wright et al. 2013; do Sul and Costa 2014; Barboza and Gimenez 2015; Anderson et al. 2016; Duis and Coors 2016; Auta et al. 2017; Alimi et al. 2018; He et al. 2018; Li et al. 2018a; Mai et al. 2018; Rezania et al. 2018; Wang and Wang 2018; Wang et al. 2018b; Xiong et al. 2018; Castro et al. 2018; Chae and An 2018; Fahrenfeld et al. 2019; Koelmans et al. 2019; Stock et al. 2019; Strungaru et al. 2019; Triebskorn et al. 2019; Barletta et al. 2019). Whereas, based on the previous studies, a high concentration of microplastics is often found in the effluent and sludge from wastewater treatment plants (WWTPs) and the incomplete removal of these microplastics might cause pollution in the receiving water (Estahbanati and Fahrenfeld 2016). So far, only a few review articles have discussed the microplastics in wastewater systems (Morris et al. 2017; Burns and Boxall 2018; Kang et al. 2018; Prata 2018; Raju et al. 2018; Sun et al. 2019; Ziajahromi et al. 2016). In addition, no review has summarized the current analytical approaches including sampling, extraction, and characterization used for microplastics present in the wastewater systems. Therefore, in this article, a comprehensive review of microplastics originating from wastewater systems is presented.

2 Sources of microplastics in WWTPs

2.1 Personal care and cosmetic products (PCCPs)

Currently, the natural exfoliating materials such as walnut husk, pumice, and apricot in the facial cleanser have been substituted by microplastics. In the industry, the term ‘microbeads’ is usually used to describe the existence of microplastics as ingredients in the

PCCPs (Napper et al. 2015). Based on the American Academy of Dermatology, polyethylene (PE) microbeads are often applied in the facial cleansers, owing to their smoothness in nature and thus leads to less damage and redness to the skin (Chang 2015). It is worthwhile to mention that the majority of microplastics resulting from PCCPs can be removed by conventional wastewater treatments and the most frequently detected microplastics in the effluent are fibers and fragments rather than microbeads. The images of microplastics extracted from commercially available PCCPs are shown in Fig. 1 (Carr et al. 2016).

2.1.1 Characterizations

Fendall and Sewell (2009) identified and characterized the microplastics from four commercially available facial cleansers, and the size of the most extracted microplastics was observed to be smaller than 0.5 mm. In addition, all microplastics in the facial cleansers showed various irregular shapes. Chang (2015) characterized the PE microbeads in the facial exfoliating cleansers, such as size, color, and concentration. The results showed that the average size of microbeads was between 60 and 800 μm in diameter. Besides, most microbeads were found to be white and opaque. Cheung and Fok (2017) extracted the microbeads from nine different commercially available facial cleansers from mainland China, and the authors reported that the particle size of microbeads was in the range of 24–800 μm. In addition, it was observed that the average amount of microbeads detected was between 5219 microbeads/$g_{product}$ and 50,391 microbeads/$g_{product}$. Additionally, the predominant shape of the microbeads from facial cleansers was irregular. In another study, Kalčíková et al. (2017) characterized the microbeads in the body washes and facial cleansers, and the size of most microbeads was less than 100 μm. Besides, a higher abundance of the larger sized microplastics was found in the body washes, while, in contrast, facial cleansers contained mostly smaller sized microplastics. The average concentration of PE in the body washes (4.82 $g_{particles}$/100 mL) was considerably higher than that observed in the facial cleansers (0.74 $g_{particles}$/100 mL). Table 1 summarizes the recent studies investigating the microplastic pollution caused by PCCPs.

2.1.2 Estimated releasing amount

Kalčíková et al. (2017) estimated the daily emission of microbeads originating from cosmetics, and around 15.2 mg/person can be released every day. Besides, based on a 52% removal efficiency of microbeads by a biological WWTP, about 1,125,500,000 particles/day of PE microbeads could be discharged to the receiving river, which leads to the concentration of microbeads

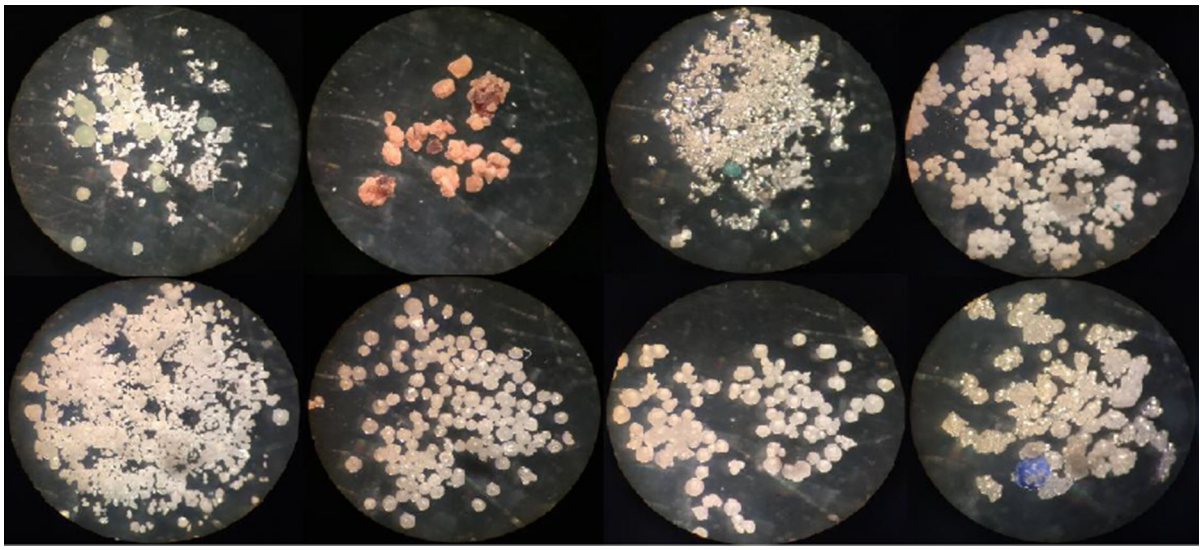

Fig. 1 Images of microplastic samples extracting from commercially available cosmetics and personal care products (Carr et al. 2016)

Table 1 Recent studies on microplastic pollution originating from personal care and cosmetic products

Sources	Size (μm)	Shape	Color	Polymer type	References
Facial cleansers	< 500	Ellipse; rod; thread; granular	Blue; orange	–	Fendall and Sewell (2009)
Facial cleansers	60–800	Uniform; granular	White; opaque	–	Chang (2015)
Facial cleanser	80–186	Irregular (mainly granular)	White; orange; dark blue; light blue; opaque	PE; LDPE; Wax; Luwax; PVC	Cheung and Fok (2017)
Facial cleanser; body wash	100	Irregular	White; red; blue; brown	PE	Kalčíková et al. (2017)
Facial cleanser	313 ± 130	Round; spherical; ellipse; filament; long stripe; grape-shaped; fragment	White; opaque	PE	Lei et al. (2017)
Shower gel	422 ± 185				

to be 21 particles/m^3. Cheung and Fok (2017) reported that approximately 209.7 trillion microbeads (equivalent to 306.9 tonnes) originating from facial cleanser might be released into the aquatic environment in mainland China on an annual basis. van Wezel et al. (2016) employed a mathematic model [Eq. (1)] to estimate the amount of microplastic pollution caused by PCCPs. According to three different emission scenarios including minimum, average, and maximum, the estimated concentration of microplastics was 0.2 μg/L, 2.7 μg/L, and 66 μg/L in the final effluent, respectively.

$$PEC_{eff} = \frac{C_{mp} \times U_{prod} \times (1 - F_{wt}) \times F_{pen}}{W_{inf}} \quad (1)$$

where PEC_{eff} and C_{mp} represents the predicted concentration of microplastics in the final effluent ($g_{microparticle}/L_{effluent}$) and the concentration of microplastics in a commercial product ($g_{microparticle}/g_{product}$), respectively. U_{prod} is the usage of the product on a daily basis (g per capita per day) and F_{wt} is the fraction of microplastics removed by wastewater treatments. In addition, F_{pen} and W_{inf} represents the market penetration of products containing plastic microbeads and the amount of wastewater generated on a daily basis (L per capita per day), respectively.

Although some regulations have been introduced by the government to monitor the amount of microplastics applied as ingredients in the PCCPs, the alternatives to microbeads must be developed when considering the large quantities of microplastic pollution originating from PCCPs.

2.2 Synthetic fibers

Microfibers as secondary microplastics that results from the synthetic polymers in garments are considered to be the most prevalent type of microplastic pollution in the environment (Gago et al. 2018). Similar to microbeads, microfibers can be detected in a variety of environmental systems. Additionally, microfibers could accidentally be ingested by living organism and hence enter the food chain and harm human health. Except for entering the aquatic environment, microfibers can also be discharged into the terrestrial soils through the application of sludge as a fertilizer (Hartline et al. 2016). During conventional domestic washing of synthetic fibers, thousands of microfibers can be released and the resulting washing effluent is either released to the soils or to WWTPs through sewers (Browne et al. 2011). According to previous literature, approximately 35% of the microplastics identified in the aquatic environment might be released from synthetic fibers during washing (Prata 2018). Table 2 summarizes the recent studies on microplastic pollution resulting from the domestic washing of synthetic fibers.

Table 2 Recent studies on microplastics pollution resulting from domestic washing of synthetic fibers

Synthetic fabric types	Main conclusions	References
Polyester-cotton blend Polyester Acrylic	Loss of fiber decreased with increasing times of wash Polyester-cotton blend led to the lowest fibers More fibers were released when using conditioner or detergent	Napper and Thompson (2016)
Plain weave polyester Double knit jersey polyester Plain weave polypropylene	Woven polyester released the highest amount of microfibers The use of softner led to the lowest amount of microfibers release Powder detergent, high temperature, higher water hardness, and mechanical action increased microfibers release	De Falco et al. (2018)
100% polyester interlock Plain singlt kit jersey polyester and 2% Spandex plating	The use of detergent was the most important factor affecting microfibers release The length of fiber (100–800 μm) was not affected by the washing conditions and fabric type	Hernandez et al. (2017)
Polyester Polyester-elastane blend Cotton	The highest fibers release was from polyester-elastane blend, followed by cotton The thickness and length for most microfibers was 20 μm and 100–1000 μm, respectively	Sillanpää and Sainio (2017)
Polyester Polyacrylic Polyamide	Polyester fleece fabrics released the highest number of microfibers (7360 fibers/m^{-2}/L^{-1} in one wash) High twist yarns are preferable for reducing microfibers releasing	Carney Almroth et al. (2018)

2.2.1 *Effects of washing conditions on the estimated releasing amount*

Napper and Thompson (2016) investigated the effects of washing temperature (30–40 °C), detergent, and conditioner on the quantity of microfibers releasing from polyester (PEST), acrylic, and PEST-cotton during laundering. In the case of PEST, the amount of microfiber loss gradually reduced from 2.79 mg at 1st wash to 1.63 mg at 5th wash, which was similar to the trend observed for acrylic and PEST-cotton. The result could indicate that old garments release fewer microfibers than new clothing. In addition, the usage of conditioner and detergent significantly affected the amount of fibers released. Clearly, more fibers would be released during washing in the presence of detergent and conditioner. Based on the assumption of 6 kg of washing load, the authors estimated that the amount of microfibers caused by the washing of PEST, acrylic, and PEST-cotton fabrics was 496,030, 728,789, and 137,951 fibers, respectively.

In another study, De Falco et al. (2018) quantified the released microfibers from three different types of synthetic fabrics during domestic washing, and the highest releasing extent of microfibers was found in the woven PEST. Moreover, the effects of detergents, washing parameters (temperature, time, water hardness, and mechanical action), and industrial washes on the release of microfibers were examined. The use of detergent led to an increase in the microfibers release. Specifically, the release of microfiber considerably increased from 162 ± 52 microfibers/g of fabric to 1273 ± 177 or 3538 ± 664 microfibers/g of fabric when using liquid or powder detergent, respectively. This increase might be due to the inorganics (i.e., zeolite) in the detergent, which could cause friction with synthetic fabrics in the process of domestic washing. Another reason could be related to the high pH value of detergent, especially when using powder detergent, since the alkaline detergent is able to damage the surface of PEST fabrics through slow surface hydrolysis. A similar observance was reported by Hernandez et al. (2017), in which the use of detergent was observed to be the most important factor affecting the total mass of microfibers released. Besides, a greater level of microfibers releasing was achieved by applying higher temperatures, washing times, and water hardness in the washing process. The higher washing temperature may accelerate the slow surface hydrolysis of PEST fabrics in the presence of alkaline detergent, and this extent of chemical damage of fabrics can be increased by using a longer washing

time. The utilization of hard water triggers the abrasion to PEST during domestic laundering. Additionally, an estimation of 6,000,000 microfibers can be generated after domestic washing when based on an assumption of 5 kg wash load of PEST fabrics.

Carney Almroth et al. (2018) evaluated the amount of microfibers released from three common synthetic materials (acrylic, nylon, and PEST) under varying washing conditions, and it was observed that PEST fleece fabrics produced the highest amount of microfibers during washing. The authors also reported that the amount of microfibers shredded in laundering was dependent on the yarn and needle gauge. Specifically, a higher release degree of microfibers was obtained when washing tightly knitted PEST fabrics. The aging of clothing was found to be another significant factor affecting the release of microfibers in domestic washing, and the use of aged clothing resulted in higher masses of microfibers than that obtained from new clothing.

In addition, the effect of number of wash cycle on microplastics releasing was investigated by Sillanpää and Sainio (2017), in which four different types of PEST textiles and two garments of cotton were evaluated. The authors found that the amount of released microfibers reduced from 2.1×10^5–1.3×10^7 microfibers/kg fabrics into one tenth by fifth washes, which was in agreement with the results reported by Napper and Thompson (2016).

In conclusion, it is very difficult to control and monitor the releasing amount of microfibers due to their prevalence in our daily life. Additionally, the interaction between microfibers and sediment/plankton has been reported, causing accidental ingestion of microfibers by animals and thus enters the food chain (Gago et al. 2018). Thus, appropriate regulations for controlling the amount of microfiber released by domestic washing must be established.

3 Sampling approaches

Until now, different sampling methods have been employed for collecting microplastics-containing wastewater, including container collection, pumping coupled with filtration, surface filtration, and autosampler collection (Sun et al. 2019). Table 3 summarizes the sampling approaches from recent studies on microplastics present in the wastewater streams.

3.1 Container collection and pumping coupled with filtration

Even though sampling collecting by container is easy to operate, only a limited amount of wastewater can be sampled. As a result, container collection is desirable for the influent of WWTPs due to the high concentration of organic materials and solids (Talvitie et al. 2017b). Magnusson et al. (2014) adopted a Ruttner sampler consisting of a cylinder which is immersed into the wastewater and closed with a plummet. The wastewater sample was then transferred into a stainless steel filter holder with a mesh size of 300 μm, followed by vacuum filtration.

On the contrary, sampling by separate pumping coupled with filtration can be used for the collection of hundreds liters of wastewater, thereby making it favorable for sampling the effluent of WWTPs. A newly designed sampling method using a mobile pumping device was developed by Mintenig et al. (2017), as shown in Fig. 2. This sampling device is composed of a flexible PVC hose connected with a weighted end-piece, membrane pump, flow meter, and a filter housing containing stainless steel cartridge filter (mesh size: 10 μm). Prior to sampling, the pumping system of the device was flushed with wastewater for 5 min. Afterwards, the weighed end-piece was placed below the surface of wastewater ($\sim$ 10 cm) to collect the sample.

Ziajahromi et al. (2017) developed a sampling device (Fig. 3) that contains four stainless steel mesh screens with a size of 25, 100, 190, and 500 μm, respectively, for the identification and characterization of microplastics in the effluents. As depicted in Fig. 3, all mesh screens were stacked on top of each other, and the one with the largest size was placed on the top. The stacked mesh screens were sheltered by a cover made of polyvinyl chloride (PVC), and a baffle was placed inside the inlet to ensure an even distribution of sampled wastewater. This sampling device has two main advantages: (1) provides an in-situ division of microplastics, which is performed based on their size distribution; and (2) a large volume of sample can be treated continuously. The recovery efficiency of polystyrene (PS) microplastics obtained by this sampling technique was observed to be 92% when using a 25 μm mesh screen, and the adoption of 500 μm mesh screen led to a 99% recovery efficiency. This observance might suggest that it is effective for capturing

Table 3 A summary of sampling approaches adopted for collecting microplastic samples originating from wastewater systems

Methods	Location	Device	Lowest mesh size	References
Container collection				
	Glasgow	10 L of steel buckets	65 µm	Murphy et al. (2016)
	Derby	Telescopic sampling tool	0.2 µm	Tagg et al. (2015)
	Lysekil	Ruttner sampler	300 µm	Magnusson et al. (2014)
	Vancouver	Glass jar	1 µm	Gies et al. (2018)
	Kenkäveronniemi	10 L stainless steel bucket	0.25 mm	Lares et al. (2018)
	South Korea	100 L UV sterilization tank	106 µm	Lee and Kim (2018)
	Northern Italy	Steel bucket	63 µm	Magni et al. (2019)
Pumping coupled with filtration				
	Lysekil	Filter holder made from plankton net and a suction pump	300 µm	Magnusson et al. (2014)
	Northern California	A set of Tyler sieves and an extraction pump	0.125 mm	Mason et al. (2016)
	Western New York			
	Eastern New York			
	Central New York			
	Eastern Wisconsin			
	Oldenburg	Custom made mobile membrane pump	10 µm	Mintenig et al. (2017)
	Helsinki	A specific filtering assembly with an electric pump	20 µm	Talvitie et al. (2017b)
	Sydney	A stacked units consisting of several stainless steel mesh screens	25 µm	Ziajahromi et al. (2017)
	Los Angeles County	A stacked units consisting of several stainless steel mesh screens	20 µm	Carr et al. (2016)
	East Bay	A stacked units consisting of several stainless steel mesh screens	0.125 mm	Dyachenko et al. (2017)
Surface filtration				
	Los Angeles County	A surface filtering assembly designed for skimming the water surface at effluent discharge outfall	–	Carr et al. (2016)
Autosampler collection				
	Helsinki	Automated sampler (ISCO 3700)	20 µm	Talvitie et al. (2017b)
	Adana	Endress + Hauser ASP-Station 2000, vacuum system RPS20 model sampler	55 µm	Gündoğdu et al. (2018)

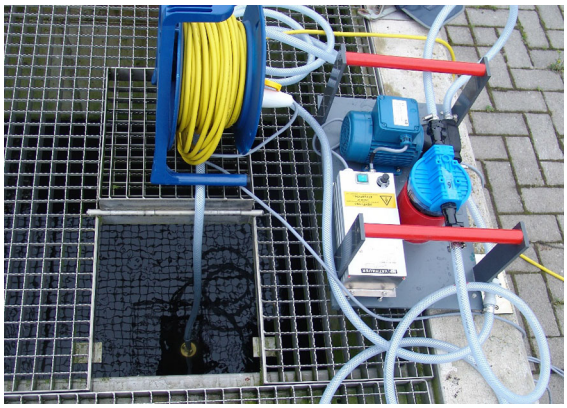

Fig. 2 A newly designed mobile pumping system for sampling (Mintenig et al. 2017)

microplastics with a wide range of particle sizes. More importantly, no contamination caused by sampling process can be found.

3.2 Surface filtration

Surface filtration is an highly effective sampling technique for collecting thousands of cubic meters of wastewater. As depicted in Fig. 4, a surface filtration unit for sampling was developed by Carr et al. (2016), which can be used for skimming the water surface at the effluent discharge outfall. Based on the flow rate and water quality, different deployment times were applied during sampling period. Nevertheless, sampling by surface filtration has several limitations that are required to be addressed: (1) is only applicable at water falls; and (2) the airborne contamination can not be eliminated as the surface filtration unit should be placed in an open channel. Additionally, the amount of microplastics can be underestimated by surface filtration since skimming the water surface might only collect the low-density microplastics (Sun et al. 2019).

In general, based on the current studies on wastewater sampling, we found that the most used are container collection and pumping coupled with filtration. In the case of collecting effluent samples, pumping coupled with filtration is a favorable sampling technique as a large amount of samples can be taken. On the other hand, for the microplastics present in the sludge, the most commonly adopted method is described as follows: the sludge samples can be directly collected in a beaker/glass jar and then stored in a refrigerator/cooler (i.e., − 4 °C) prior to analysis (Gies et al. 2018).

3.3 Contamination

It is significant to avoid contamination throughout the whole experiment. Mintenig et al. (2017) adopted several steps to reduce contamination, such as: (1) all lab materials were rinsed using ultrapure water and

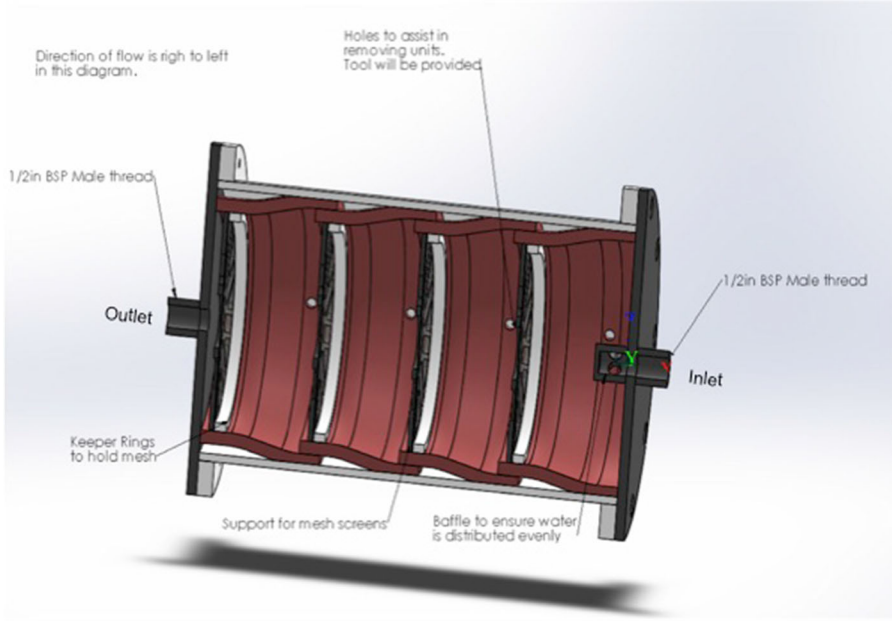

Fig. 3 A stacked sampling device with four different mesh sizes (Ziajahromi et al. 2017)

Fig. 4 A surface filtering assembly used for collecting microplastics-containing wastewater system (Carr et al. 2016)

30% ethanol; (2) all samples and lab materials were covered by aluminium foil to minimize airborne contamination; and (3) the limited use of plastic materials during the experiment. Additionally, the blank experiments must be conducted in order to control the airborne contamination (Gies et al. 2018). Briefly, a membrane filter is placed in a petri dish without a cover in the area where the experimental work is carried out, followed by microscope analysis.

4 Pre-treatment

4.1 Density separation

The extraction of microplastics from liquid is commonly achieved through density separation which involves the mixing of a liquid of defined density with the sample. The saturated sodium chloride (NaCl) solution with a density of 1.2 kg/L is the most commonly used liquid in the density separation, owing to its cheap price and non-toxic characteristics (Li et al. 2018a). During separation, a suspension is created and then vigorously shaken, followed by incubation until two clear phases are formed. The upper layer of water contains low-density particles like microplastics and high-density particles (e.g., clay) usually separates into the bottom layer. Consequently, the microplastic particles can be recovered from the supernatant (Li et al. 2018a). Magni et al. (2019) applied an saturated NaCl solution to separate the microplastics in the influent, effluent, and sludge samples from primary, secondary, and tertiary treatments, and it was observed that the high-density particles [PVC: 1.16–1.58 kg/L; polyoxymethylene (POM): 1.41–1.61 kg/L] cannot be separated.

Except that, other solutions like sodium polytungstate (SPT) solution, zinc chloride ($ZnCl_2$), calcium chloride ($CaCl_2$), and sodium iodide (NaI) solution have exhibited their effectiveness for the extraction of microplastics (Corcoran 2015; Stolte et al. 2015; Coppock et al. 2017; Crichton et al. 2017). For example, Mahon et al. (2017) adopted 1 M $ZnCl_2$ solution to separate microplastics from three effluents samples after treatment by lime stabilization, anaerobic digestion, or thermal drying. Whereas, the oily appearance was observed in the samples obtained after lime stabilization, making this density separation method undesirable for extracting microplastics. In addition, the high cost and toxicity of the above solutions represent barrier for their practical applications. Table 4 summarizes the different solutions adopted in the density separation of microplastics originating from wastewater systems.

4.2 Digestion

Due to the complexity in the composition of wastewater, digestion is required to remove biogenic materials from wastewater prior to further characterizations via FTIR and Raman spectroscopy. Table 4 summarizes the digestion methods adopted by analyzing the microplastics from wastewater streams.

To date, the most effective digestion approach involves the incubation of microplastics-containing samples in 30% (v/v) hydrogen peroxide (H_2O_2). The digestion by H_2O_2 can destroy most organics present in the sample (Stock et al. 2019). Li et al. (2018b) extracted microplastics from dewatered sewage sludge using saturated NaCl solution followed by digestion with 30% H_2O_2 (as shown in Fig. 5) and the results showed that an average 78.27% of microplastics can

Table 4 Separation and digestion methods adopted for analyzing microplastics originating from wastewater streams

Source	Separation	Digestion	Recovery (%)	References
Sludge, Spain	NaCl	–	–	Bayo et al. (2016)
Effluent, USA	–	30% H_2O_2 with0.05 M $FeSO_4$ as the catalyst	87	Dyachenko et al. (2017)
Upstream and downstream, USA	NaCl	30% H_2O_2 with0.05 M $FeSO_4$ as the catalyst	–	Estahbanati and Fahrenfeld (2016)
Effluent and sludge, Canada	–	30% H_2O_2	–	Gies et al. (2018)
Effluent, Turkey	NaI	30% H_2O_2 with 0.05 M $FeSO_4$ as the catalyst	–	Gündoğdu et al. (2018)
Effluent and sludge, Finland	–	30% H_2O_2 with 0.05 M $FeSO_4$ as the catalyst	–	Lares et al. (2018)
Effluent, South Korea	$ZnCl_2$	30% H_2O_2 with0.05 M $FeSO_4$ as the catalyst		Lee and Kim (2018)
Sludge, China	NaCl	30% H_2O_2	67–98	Li et al. (2018b)
Effluent ad sludge, Italy	NaCl	15% H_2O_2	78–98	Magni et al. (2019)
Effluent, Denmark	–	30% H_2O_2 with0.05 M $FeSO_4$ as the catalyst	57–78	Simon et al. (2018)
Effluent, UK	–	30% H_2O_2	–	Tagg et al. (2015)
Effluent, Australia	NaI	30% H_2O_2	–	Ziajahromi et al. (2017)

be recovered, as evidenced by Fourier-transform infrared spectroscopy (FTIR) analysis.

The main drawback for the digestion by H_2O_2 is the requirement of a large amount of H_2O_2; thus, it is less efficient for treating a larger sample volume in the economic perspective. To solve this, Tagg et al. (2017) studied the effectiveness of Fenton's reagent [a mixture of H_2O_2 and ferrous ion (Fe^{2+})] on the isolation of microplastics from wastewater, and no significant alternation in the microplastic properties was observed. To compare, Fenton's reagent has advantage over H_2O_2 pretreatment: (1) time-efficient; and (2) the ability to deal with large sample volumes. Gündoğdu et al. (2018) digested the secondary effluent by a mixture of 30% H_2O_2 and 0.05 M Iron (Fe) (II) at 75 °C, and then NaI solution was added for density separation. Following this, the suspension underwent centrifugation for 5 min at 3500 rpm and then filter over a 55 μm filter, and the collected matters were characterized by microscopy and μ-Raman spectroscopy.

In another study, the digestion by wet peroxide oxidation (WPO) for characterizing the microplastics in secondary effluent was evaluated by Dyachenko et al. (2017). Initially, 80 mL of 30% H_2O_2 and 40 mL of 0.05 M Iron (II) sulfate ($FeSO_4$) were added into the effluent samples and then heated at 70 °C for 30 min with constant stirring. An extra addition of 30% H_2O_2 was added when the digestion of organics was not completed. Subsequently, the extract was recovered by vacuum filtration over a 0.8 μm membrane filter. As confirmed by a dissecting stereomicroscope, WPO method led to a 87% recovery of the microplastics. While, due to the existence of cellulose fibers and fatty acids as two major interferents, a sequential WPO procedure was employed after a single cycle of digestion. Briefly, after a single WPO digestion, the extract was filtered through a 0.125 mm sieve and then washed with hexane and distilled water. The solid materials in the sieve was then digested again using a 30% H_2O_2–0.05 M $FeSO_4$ mixture for 3–6 digestion cycles in order to completely remove cellulose and other organic materials.

4.3 New approaches developed for extracting microplastics

Recently, Phuong et al. (2018) developed a new extraction method by centrifugation with ultrapure water for microplastics present in sediments without the use of H_2O_2 as the digestion agent. It was reported that

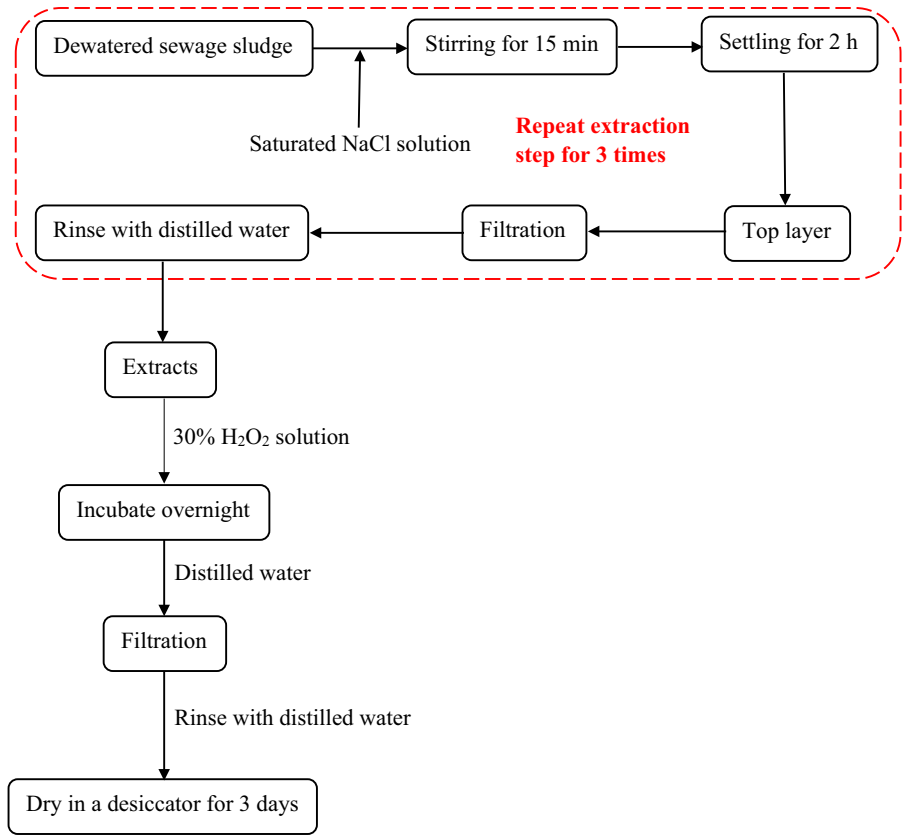

Fig. 5 A schematic diagram of the extraction procedure adopted for microplastics in the sewage sludge (Li et al. 2018b)

potassium hydroxide (KOH) was ineffective in the digestion treatment since the precipitation still occurred even after centrifugation (Phuong et al. 2018). Besides, the use of 65% nitric acid (HNO_3) as the digestion agent resulted in a 66–100% recovery efficiency for PE and polypropylene (PP). However, it has been reported that HNO_3 shows negative impact on the microplastics, such as: (1) destroy microplastics when the digestion temperature above 60 °C; (2) damage the polymeric structure of nylon; (3) discolor PE; and (4) react with the surface of PVC and thus affects its surface morphology (Claessens et al. 2013; Li et al. 2018a).Subsequently, two different separation methods including ultrapure water and 50% potassium iodide (KI) were investigated before centrifugation. In the case of KI solution, large amounts of materials were observed in the suspension and at the surface of supernatant after centrifugation, resulting in large-sized microplastics being analyzed by μ-FTIR and thus a longer processing time is required. On the other hand, for the separation by ultrapure water, an amount of 20 mL of ultrapure water was added to 25 g of 1 mm-sieved sediment samples, followed by centrifugation and filtration over a 12 μm pore-size filter. This procedure was validated by spiking the sediment samples with four commercially available polymers including PE, PP, PVC, and polyethylene terephthalate (PET). However, to the best of our knowledge, no study so far has adopted this method to extract microplastics present in the wastewater; therefore, future studies should be conducted to evaluate the recovery efficiency of extraction by ultrapure water and centrifugation for microplastics from various wastewater systems.

5 Identification and characterization of microplastics

5.1 FTIR and Raman spectroscopy

FTIR and Raman spectroscopy are two commonly used methods for characterizing microplastics present in various environmental systems (Carr et al. 2016;

Dyachenko et al. 2017; Gies et al. 2018; Lares et al. 2018). Two different working principles are applied: (1) the change in the dipole moment of chemical bonds produces an infrared spectrum in the FTIR analysis; and (2) Raman analysis offers a molecular fingerprint spectrum according to the polarizabilities of chemical bonds (Strungaru et al. 2019). A range of FTIR technologies have been developed, among which attenuated total reflectance (ATR)-FTIR is suitable for characterizing irregular microplastics and μ-FTIR is applicable for small-sized microplastic samples (around 20 μm) (Prata et al. 2019). Despite FTIR analysis is a non-destructive and reproducible technology, this approach is very time-consuming and a trained operator is needed (Rocha-Santos and Duarte 2015). Moreover, Raman spectroscopy is an analytical approach that can only be applied for the detection of compounds with aromatic bonds, C–H, and C=C bonds since the alteration of the polarizability of a chemical bond is necessary for the analysis. This approach allows for characterizing the size of microplastics down to 1–2 μm and it is not constrained by the samples properties like shape and thickness (Li et al. 2018a; Strungaru et al. 2019). Raman technique can analyze the sample containing organic or inorganic fillers, pigments, and those cannot be detected by FTIR spectroscopy (Imhof et al. 2016). Notwithstanding, the long processing time, polymer heating and degradation, as well as fluorescence inference are the main drawbacks of Raman spectroscopy (Strungaru et al. 2019).

5.2 Thermal degradation approaches

To overcome the time-consuming problem of FTIR and Raman spectroscopy, pyrolysis–gas chromatography coupled with mass spectrometry (Py–GC/MS) has been utilized for microplastics. During this analysis, the large-molecular weight compounds can be thermally degraded into small-molecular weight compounds under inert conditions (Fischer and Scholz-bottcher 2017). Py–GC/MS technique is capable of determining some polymeric compositions and organic plastic additives which cannot be easily separated, dissolved, and extracted (Qiu et al. 2016). Hermabessiere et al. (2018) performed an optimization study on the identification of microplastics in the bivalve, beach, and sea water surface using Py–GC/MS and Raman spectroscopy. The optimized operating conditions were as follows: injector temperature of 300 °C, split ratio of 5, and final temperature of 700 °C. Besides, it was found that Py–GC/MS offered several advantages over Raman spectroscopy like the feasibility for identifying co-polymer without the use of chemometric method. Nevertheless, some types of polymers with polar subunits, like PEST and polyether, can not be detected by Py–GC/MS because of the formation of polar pyrolyzate(Challinor 1989). Additionally, this technique is only applicable for certain selected types of polymers such as PE and PP due to the limitation on the pyrolysis database (Li et al. 2018a). Whereas, Py–GC/MS might be favorable for the analysis of microplastics due to the fact that PE and PP are two common polymeric type in the microplastics.

Other thermal degradation approaches have also been employed for analyzing the characterization. For instance, Dümichen et al. (2017) studied the polymeric composition of microplastics originating from a biogas plant by TED-GC/MS technology. This technology is a combination of thermal solid-phase extraction and thermal desorption.

In addition, the thermogravimetric analysis coupled with differential scanning calorimetry (TGA–DSC) has been used for analyzing the microplastics present in the wastewater streams. Majewsky et al. (2016) adopted TGA–DSC technique for determining the amount of microplastics in two effluent samples from a WWTP in Karlsruhe. The authors reported that the PE in the effluents represented 17–34% (81–257 $mg_{PE}/m^3_{effluent}$) of the total extracted materials, while, no PP was not detected in the effluents. Even though TGA–DSC offers an alternative or complementary approach to the time-consuming FTIR analysis in the qualitative and quantitative measurements of microplastics, only PE and PP can be identified owing to the overlapping transition temperature of the different polymers. Besides, the size, number, color, and shape of microplastics can not be provided by TGA–DSC technique, as the breakage of microplastic samples can be observed during the analysis (Strungaru et al. 2019).

5.3 Nile red staining

The use of staining dye such as Nile red is a low-cost method for the analysis of microplastics. Previous studies reported that the characterization of

microplastics using Nile red staining has showed a similar accuracy when compared with FTIR and Raman spectroscopy (Shim et al. 2016; Maes et al. 2017; Catarino et al. 2018). Moreover, Nile red staining technique has the advantages of: (1) short incubation time (10–30 min); (2) high recovery efficiency (up to 96.6%); and (3) with or without a cleaning step by bleach when using vibrational spectroscopy (Prata et al. 2019). Catarino et al. (2018) identified the presence of microplastics in *Mytilus edulis* by staining with Nile red dye in methanol at 1 μg/mL. This method led to a 48% of recovery efficiency of microplastics, which was comparable to that obtained from FTIR analysis (50%). Shim et al. (2016) optimized the microplastics identification by Nile red staining, and it was observed that 5 mg/L Nile red solution in hexane was effective for staining a variety of types of microplastics including PE, PP, PS, polycarbonate (PC), polyurethane (PU), poly(ethylene–vinyl acetate) (PEVA), polyamide (PA), and PEST, with the exception of PVC. However, to the best of our knowledge, no study so far has utilized Nile red dye to analyze the microplastics from wastewater systems. Therefore, future studies are necessary to explore the feasibility of Nile red staining for the characterization of microplastics present in the organic-rich samples like wastewater. However, its major drawback is correlated to: (1) the formation of weak signals for certain types of polymers like PET; (2) the microfibers are reported to be very difficult to stain by Nile red dye; and (3) a fluoresce microscopy is needed (Tamminga et al. 2017).

5.4 FPA-based FTIR imaging

Even though a wide range of analytical methods have been applied for microplastics in marine organisms and sediments, no specific technique has been established for the analysis of microplastics originating from wastewater systems. To fill this gap, Tagg et al. (2015) identified and characterized the microplastics present in the wastewater via focal plane array (FPA)-assisted reflectance micro-Fourier-transform imaging analysis combined with a pre-treatment using 30% H_2O_2. FPA is a detector that is very useful for identifying the small-sized microplastic beads on their surface areas. The authors reported FPA-based μ-FTIR analysis was observed to be effective for the characterization of PE, PP, PVC, and PS microplastics in the organics-rich wastewater. Mintenig et al. (2017) studied the microplastics in effluents via FPA-assisted transmission μ-FTIR, and this technique is allowable for the detection of small-sized microplastics ($\sim$ 20 μm). For all effluent samples, the abundance of microplastics was found to be 10–9000 microplastics/m^3 and 0–50 microplastics/m^3 for the microplastics with a size $<$ 500 μm and $>$ 500 μm, respectively. In addition, PE was identified as the most dominant polymeric type in both size groups of microplastic samples. A similar method by FPA-assisted FTIR imaging was adopted by Simon et al. (2018) where the abundance of microplastics present in the influent and effluent was determined. The authors stated that the FPA-based FTIR imaging is an effective method for investigating the polymeric type in microplastics; however, it can only provide an estimation of the amount of microplastic instead of providing an absolute value.

In summary, it is very difficult to analyze the microplastics in organic-rich wastewater. Current methods used for characterizing microplastics originating from wastewater systems are incomparable since the unit of the concentration of microplastics adopted is not consistent. Moreover, the differences in the sampling sites and methods and the contamination control render the comparison among analytical approaches more difficult. As a result, a standardized analytical protocol for microplastics present in the wastewater streams should be established (He et al. 2018).

6 What happens to microplastics in WWTPs

6.1 Treatments and removal efficiency of microplastic in WWTPs

Typically, a wastewater treatment plant is capable of operating primary, secondary, and tertiary treatment to remove contaminants prior to discharging the effluents into the surrounding environment. As suggested by Carr et al. (2016), most microplastics can be removed by primary treatment (e.g., skimming and settling) and fewer amounts of microplastics can be observed in the tertiary effluent. A flow diagram describing the fate of microplastics in a WWTP is depicted in Fig. 6. Most recent studies investigating the microplastics present in the sludge and effluent are summarized in Table 5.

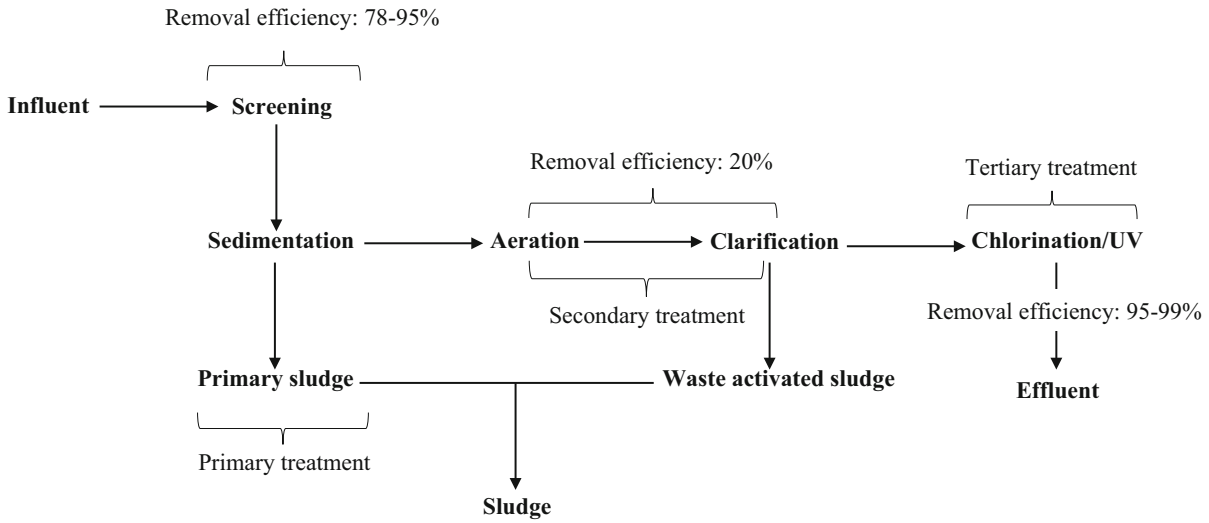

Fig. 6 A flow diagram describing the transport of microplastics in a tertiary wastewater treatment plant (*Note* The removal efficiency of each treatment is determined based on the amount of microplastics present in the influent) (Raju et al. 2018; Sun et al. 2019)

6.1.1 Effect of treatment process

Previous studies have explored the effect of treatment process on the removal efficiency of microplastics. Lee and Kim (2018) compared three biological treatments namely, anaerobic-anoxic-aerobic, sequence batch reactor, and the Media processes on the removal rate of microplastics. The authors reported that the concentration of microplastics in the effluents of anaerobic-anoxic-aerobic, sequence batch reactor, and the Media processes was 0.44 particles/L, 0.14 particles/L, and 0.28 particles/L, respectively, and the removal efficiency for microplastics with a size > 106 µm was estimated to be in the range of 98–99% for all three treatments. No difference of removal efficiencies was found among the tested treatments. This might suggest that most microplastics were removed by grease and grit removal treatment and primary settling. Besides, a higher removal efficiency was obtained from smaller sized microplastics (106–300 µm) when compared with that obtained from larger sized microplastics (> 300 µm). This result might be attributed to the affinity of microplastics with a small particle size to the sticky media (e.g., biofilm and floc).

In another study, Mahon et al. (2017) examined the effects of three different wastewater treatment processes including anaerobic digestion, thermal drying, and lime stabilization on the amount of microplastics present in the sludge. The treatment by anaerobic digestion led to a reduction in the abundance of microplastics, which might be attributed to the polymer degradation by microorganism within the anaerobic digestion system; however, this role needs to be elucidated.

6.1.2 Removal efficiency

Gündoğdu et al. (2018), for example, studied the microplastics concentration in the influent and secondary effluent from two municipal WWTPs in Turkey. It was found that the influent and secondary effluent contained 4,665,778 and 3333.3 microplastics/day, respectively, thus leading to a 91% of removal rate.

Gies et al. (2018) reported that an amount of 1.76 ± 0.31 trillion microplastics/year is discharged into the WWTP in Vancouver, Canada, among them 1.28 ± 0.54 trillion microplastics/year is retained in the primary sludge and 0.36 ± 0.22 trillion microplastics/year is detected in the secondary sludge. Based on this, a high removal efficiency (99%) of microplastics can be achieved by the WWTP.

Kalčíková et al. (2017) studied the transport and fate of PE microbeads in the WWTP was explored. The microbeads removal efficiency by secondary treatment was about 52%. Besides, it was observed that the PE microbeads at small size classes were

Table 5 Recent studies on microplastic pollution detected in the effluent and sludge

Sources	Analytical approaches	Main conclusions	References
Effluent			
	FPA-based μ-FTIR	FPA-based FT-IR coupled with H_2O_2 pre-treatment was an effective approach for analyzing microplastics in wastewater	Tagg et al. (2015)
	Microscopy	Most microplastics removed by primary treatment (skimming and settling)	Carr et al. (2016)
	FTIR	Tertiary effluent contributed less to the microplastic pollution	
		Some types of toothpaste formulation were significant sources of microplastics	
	FTIR	Microplastic removal efficiency of primary and secondary treatment was 78.34% and 20.1%, respectively	Murphy et al. (2016)
		High amounts of microplastics can be observed in the sludge	
	FTIR	Around 0.28, 0.48, and 1.54 microplastics/L can be found in the effluent after tertiary, secondary, and primary treatment, respectively	Ziajahromi et al. (2017)
		Fibers were the dominant type of microplastics detected in the effluent	
	Microscopy	All treatment provided a removal efficiency of 98%	Lee and Kim (2018)
	FTIR	A high fraction of blue and black fibers can be observed in all effluent samples	
	FTIR	PEST and PE were the most dominant polymer in the microplastics	Talvitie et al. (2017a)
		Membrane bioreator resulted in the highest removal efficiency of microplastics (99%)	
		The size of 20–100 μm and fibers were the most common size fraction and types of microplastics, respectively	
	Microscopy	PEST fibers and PE fragments were the most dominant type of microplastics	Lares et al. (2018)
	FTIR	98.3% of microplastics can be removed by conventional wastewater treatment	
	Raman microscopy	Size: 0.5–1 mm	
	Microscopy	Fibers and fragments were the most common type of microplastics	Gies et al. (2018)
	FTIR	No difference can be observed in the composition of microplastics between primary and secondary effluent	
		A microplastics removal of 99% can be achieved	
	Microscopy	PEST was the most predominant chemical composition of microplastics	Gündoğdu et al. (2018)
	μ-Raman microscopy	The removal efficiency of microplastics was 73–79%	
		Type: fibers (60%), films (20%), and fragments (20%)	
	Microscopy	Chemical composition: PEST and PA	Magni et al. (2019)
	FTIR	Type: lines (41%), films (38%), and fragments (21%)	
		Size fraction: 0.1–0.5 mm (52%), 0.01–0.1 mm (27%), 1–5 mm (14%), and 0.5–1 mm (7%)	
Sludge			
	Microscopy	Type: films (51%), fragments (34%), and lines (15%)	Magni et al. (2019)
	FTIR	Size fraction: 0.1–0.5 mm (54%), 0.01–0.1 mm (24%), 0.5–1 mm (12%), and 1–5 mm (10%)	
		Chemical composition: acrylonitrile-butadiene (27%), PE (18%), and PEST (15%)	
	Microscopy	Chemical composition: PP, nylon, PA, ethyl acrylate, and norbornene	Bayo et al. (2016)
	FTIR		
	DSC		
	Microscopy	Abundance: 4196–15,385 microplastics/$kg_{dry\ sludge}$	Mahon et al. (2017)
	FTIR	Chemical composition: HDPE, PE, PEST, acrylic, PET, PP, and PA	
	SEM	Type: fibers (78.5%), fragments (18.4%), films (1.9%), other unidentified particles (0.6%), and spheres (0.3%)	

Table 5 continued

Sources	Analytical approaches	Main conclusions	References
	Microscopy SEM M-FTIR	Abundance: 22.7 ± 12.1 × 10^3 microplastics/$kg_{dry\ sludge}$ Color: white (59.6%), black (17.6%), red (9.0%), orange (3.3%), green (2.3%), blue (1.7%), and others (6.5%) Type: fibers (63%), shafts (15%), films (14%), flakes (7.3%), and spheres (1.3%) Chemical composition: PO (fibers and shafts), PC (fibers), PE (films), PA (films), alkyd resin (flakes), PS (spheres)	Li et al. (2018b)
	Microscopy FTIR	Blue and black fibers and black fragments were the most dominant type microplastics	Lee and Kim (2018)
	Microscopy FTIR Raman microscopy	Chemical composition: PEST, PA, PE, and various polymers (PE, PP, and PEST fragments with similar kind of appearance) Type: fibers and fragments	Lares et al. (2018)
	Microscopy FTIR	Abundance: 14.9 ± 6.3 microplastics/kg dry primary sludge; 4.4 ± 2.9 microplastics/kg dry secondary sludge Fibers was the most common type in both primary and secondary sludge	Gies et al. (2018)

likely retained in the activated sludge, whereas larger sized microbeads tended to partition into the effluent. Most importantly, a high affinity of PE to the negatively charged activated sludge flocks was found, which suggests the potential of microplastics being accumulated in the sediments and affects marine organisms (Teuten et al. 2007). This could be due to the electrostatic attraction between the positively charged surface of PE microbeads and negatively charged activated sludge flocks.

Murphy et al. (2016) studied the fate and removal rate of microplastics during a secondary WWTP, and a reduction of ~ 15.70 microplastics/L of influent to ~ 0.25 microplastics/L of effluent was achieved, corresponding to a removal efficiency of 98.41% for microplastics. While, in the consideration of flow rate of WWTP, an estimation of 23 billion microplastics might be released from the final effluent annually. Specifically, the number of microplastics detected in the grit and grease removal stage (second stage) was ~ 8.7 microplastics/L, leading to the highest reduction efficiency (44.59%) for microplastics. Following this, in the third stage by primary settling, around 3.4 microplastics/L can be found and this results in a removal efficiency of 33.75% (based on the amount of microplastics in the influent). Finally, the amount of microplastics was further reduced to 0.25 microplastics/L by final treatment stage (aeration and clarification), which corresponds to a removal efficiency of 20.07% (based on the amount of microplastics in the influent) before releasing into the receiving water.

6.1.3 The presence of microplastics in the effluent

Gündoğdu et al. (2018) found that the most commonly detected microplastic type in the effluent was fiber (60%) followed by film (20%) and fragment (20%). μ-Raman spectroscopy analysis showed that the PE, PEST, PVC, acrylonitrile butadiene styrene, POM, nylon-6, and PP were identified as the main polymeric compositions of microplastics in the effluent, among which PEST was the most frequently observed polymer. PEST is a preferable material used in the textile industry owing to its strength and durability, and thus substantial amounts of PEST microfibers are expected in the wastewater (Napper and Thompson 2016). In a work by Carr et al. (2016), the most frequently identified microplastics in the WWTPs were blue and irregular PE particles, which are commonly utilized in the whitening toothpaste formulation. This suggests that some types of toothpaste formulations make contribution to the amount of microplastics detecting in the WWTPs.

In general, due to the ineffective for conventional treatments in removing small-sized microplastics

(< 100 μm), further research must be carried out to establish new treatment technologies with a focus on the removal of microplastics with a size smaller than 100 μm.

6.2 Advanced treatment and removal efficiency of microplastics

Despite the conventional wastewater treatments exhibit high efficiencies (~ 99%) for removing microplastics, substantial amounts of microplastic pollutants can still be released into the aquatic environment in the consideration of a large amount of effluent being discharged every day. To solve this, Lares et al. (2018) evaluated the removal efficiency of microplastics by an advanced treatment (i.e., membrane bioreactor), and this treatment led to a higher removal efficiency (99.4%) than that obtained from conventional activated sludge process (98.3%).

Talvitie et al. (2017a) investigated the removal efficiency of microplastics from effluent using four different advanced final-stage treatment technologies including membrane bioreactor, discfilter, rapid sand filtration, and dissolved air flotation. The authors reported that the number of microplastics in the primary effluent significantly reduced from 6.9 microplastics/L to 0.005 microplastics/L with the use of membrane bioreactor, which represents a 99.9% of microplastics removal efficiency. Similar to membrane bioreactor, the usage of rapid sand filter and dissolved air flotation led to 97% and 95% efficiency, respectively, for removing microplastics from secondary effluent. To compare, the discfilter removed the microplastics in the secondary effluent from 0.5 to 2.0 microplastics/L to 0.003–0.3 microplastics/L, resulting in a 40–98.5% removal efficiency. Moreover, the small sized microplastics (20–100 μm) were identified to be dominant in three out of four investigated wastewater treatment plants, with the exception for treatment by membrane bioreactor. Therefore, it is necessary to employ an advanced final treatment to remove the microplastics with a size smaller than 100 μm.

6.3 Characterizations of microplastics in sludge

Throughout the settling process of wastewater treatment, the majority of microplastics could be entrained and ended up in the sludge. Sludge as a semi-solid slurry is normally generated from primary and secondary wastewater treatment. Previous literature claimed that around 99% of microplastics remain in the sludge after multiple stages of wastewater treatment processes, making agriculturally-applied sludge (also known as biosolid) an important source of microplastic pollution to the environment (Mahon et al. 2017). Many studies have reported the abundance of microplastics in the sludge (Gies et al. 2018; Lares et al. 2018; Li et al. 2018b).

Li et al. (2018b) investigated the abundance and characterization of microplastics in 79 different sludge samples collected from 28 WWTPs across China, and the results showed that the average concentration of microplastics in the dried sludge was 22.7×10^3 microplastics/$kg_{dried\ sludge}$. Microscopic analysis indicated that fibers (63%) and white (59.6%) were found to be the predominant type and color, respectively, of the microplastics in the dewatered sludge. As indicated by FTIR analysis, the main polymeric compositions of sludge-based microplastics were low-density plastics such as polyolefin (PO), acrylic fibers, PE, PA, alkyd resin, and PS. Nevertheless, the high-density plastics like PVC (ρ = 1.16–1.58 g/mL) and PET (ρ = 1.37–1.45 g/mL) were not detected since their higher densities could complicate the density separation by saturated NaCl solution (ρ = 1.2 g/mL) (Hidalgo-Ruz et al. 2012).

In the work by Bayo et al. (2016), different sludge samples were collected after primary treatment and anaerobic digestion. The authors found that the ethyl acrylate (an important monomer for producing resins, plastics, rubbers, or dental materials) was identified as one of the main compounds in the sludge. It should be noted that ethyl acrylate as a toxic compound has demonstrated the feasibility for transferring the adsorbed metals (e.g., Pb, Cd, and Zn) into the food chain (Browne et al. 2013).

Mahon et al. (2017) examined the abundance and properties of microplastics in the sludge from seven WWTPs in Ireland, and the amount of microplastics extracted from sludge was in the range of 4196–15,385 microplastics/kg of sludge (on a dry basis). Besides, the majority of extracted microplastics were composed of 75.8% of fibers and small amounts of fragments (18.4%), films (1.9%), spheres (0.3%), and other (0.9%). In addition, the main polymeric components detected in the microplastic from sludge included high-density polyethylene (HDPE), PE, and

PA. As shown in Fig. 7a–c, the surface morphology of microplastics originating from the treated sludge after thermal drying exhibited fracturing and blistering; however, the microplastics, which were treated by lime stabilization, showed a more shredded and weathered appearance (Fig. 7d–f). Additionally, Fig. 7g–i indicated that the surface texture of microplastics isolated from the treated sludge after anaerobic digestion had a deep cleavage, which was distinct from microplastics extracted from sludge after thermal drying or lime stabilization.

Furthermore, Lares et al. (2018) stated that the effect of treatment approach on the polymeric composition of microplastics was insignificant. In general, PEST (79.1%) accounted for the most abundant in all microplastic samples, followed by PE (11.4%) and PA (3.7%). The type and size for the majority of microplastics was observed to be fibers and < 1 mm, respectively.

7 Potential environmental contamination by microplastics

As indicated by literature, considerable amounts of microplastics can still be released into the aquatic environment after wastewater treatments (Raju et al. 2018). The feasibility of microplastics for absorbing harmful agents like pharmaceuticals has been evaluated. Prata et al. (2018) evaluated the influence of the mixture of microplastics and pharmaceutical (procainamide and doxycycline) on their toxicity for microalgae (*Tetraselmis chuii*), and the results showed the toxicity of individual pharmaceutical was increased in the presence of microplastics. This phenomenon might be attributed to the interaction between microplastics and the cell wall of microalgae, thus facilitating the uptake and/or the toxic action of doxycycline/procainamide. In another study, the toxic effects of florfenicol, microplastics, and their mixture on the freshwater exotic invasive bivalve *Corbicula fluminea* were studied by Guilhermino et al. (2018). It

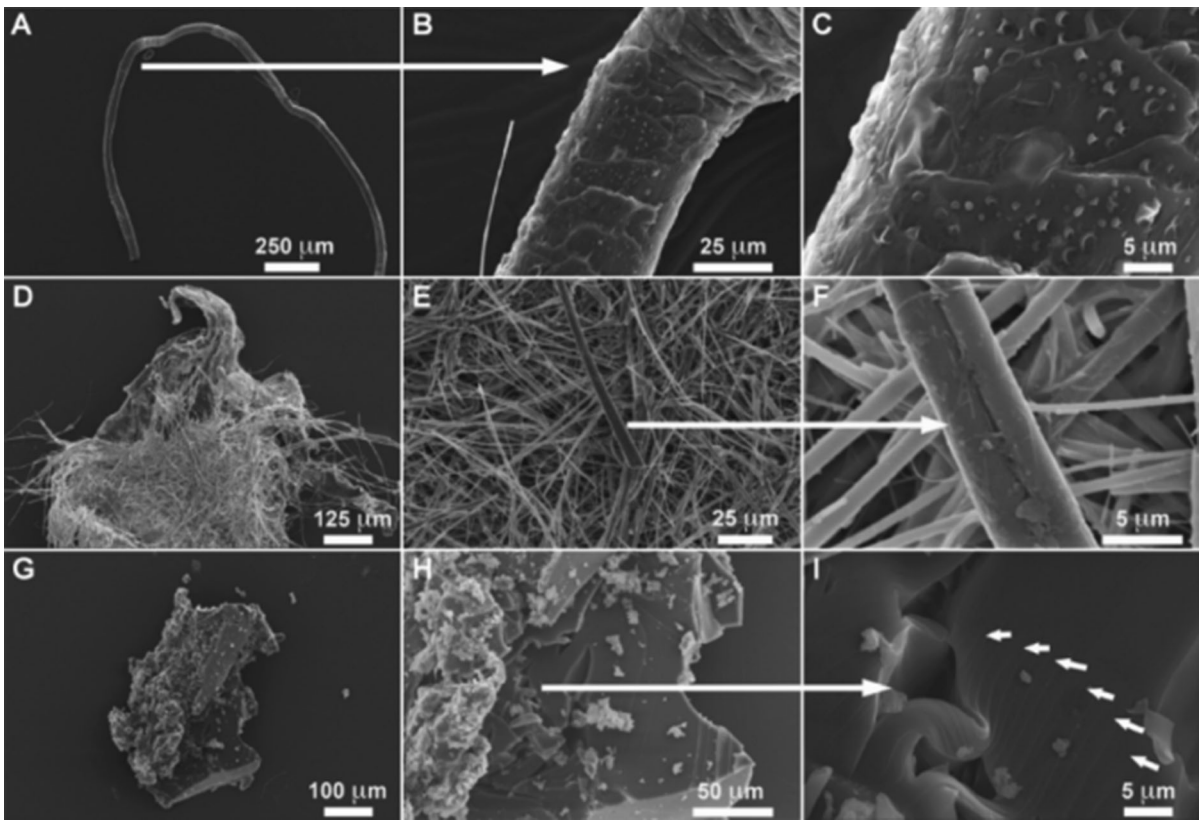

Fig. 7 Surface morphology of microplastics isolated from various treated sewage sludge samples after thermal drying (**a–c**), lime stabilization (**d–f**), and anaerobic digestion (**g–i**) (Mahon et al. 2017)

was found that both microplastics and florfenicol exhibited toxicity to *C. fluminea*; while a stronger toxic effect was observed in the mixture of florfenicol and microplastics. Fonte et al. (2016) also observed the toxicological interaction between microplastics and cefalexin to *Pomatoschistus microps*. Besides, it was found that an increase in the temperature led to a higher toxicity of cefalexin alone and in the mixture with microplastics.

On the other hand, microplastics may act as carriers or transport vectors for POPs like polycyclic aromatic hydrocarbons and (PAHs) polychlorinated biphenyls (PCBs) owing to their large specific surface areas (Wang et al. 2018a). Microplastics could float in the microlayer of sea surface due to a lower density than water, in which hydrophobic contaminants such as PCBs and 2,2-bis(p-chlorophenyl)-1,1-dichloroethene can be concentrated by 10^5–10^6 times (Mato et al. 2001). Chen et al. (2019) investigated the accumulation of PHAs and PCBs on the plastics, and the results showed that PS exhibited the highest concentrations of organic pollutants (PAHs = 23,696.7 μg/kg; PCBs = 234.6 μg/kg). This is possibly due to the fact that: (1) PS can act as a sink for some types of PAHs; and (2) PS is an amorphous polymer, whereas other plastics like PP contain crystalline regions and therefore more energy is required to disrupt their polymeric structures (Rochman et al. 2013). However, the role of microplastics as vectors of absorbed pollutants is still under debate. Beckingham and Ghosh (2017) carried out a comparative study on the bioavailability of PCBs in microplastics, wood, biochar, and coal by measuring the solid-water distribution coefficient, gut fluid solubilization, and bioaccumulation with *Lumbriculus variegatus*. It was found that the bioavailability of PCBs in microplastics was significantly lower than that from natural sediments. The authors concluded that the contribution of organic contaminants to the aquatic environment from microplastics was smaller than that from natural pathways. Bakir et al. (2016) also reported that the ingestion of microplastics by animals did not offer a route for transferring adsorbed organic pollutants from ocean to biota.

When considering the widespread of microplastic pollution and their interactions with harmful agents, future research is needed to thoroughly understand: (1) the synergistic toxic effects of widely used pharmaceuticals and microplastics; and (2) the role of microplastics as vectors for POPs.

8 Challenges and future perspectives

As indicated by literature, microplastics could undergo a series of degradation processes, such as thermal degradation, mechanical action, and biodegradation, thereby causing changes in their surface morphology and polymeric composition (Cooper and Corcoran 2010; Hidalgo-Ruz et al. 2012). Thus, a database containing the properties of microplastics in terms of chemical composition and surface morphology when subjected to various degradation processes must be established in the future. Moreover, microorganism might readily colonize the surface of microplastics, which could lead to the formation of biofilm and complicate the subsequent spectroscopic analysis (Li et al. 2018a). Consequently, it could be very difficult to differ the microplastics from natural polymers (e.g., chitin, cellulose, and chitosan) when considering the small size of microplastics (< 5 mm) (Li et al. 2018a). Besides, microorganism might be transported along with microplastics and thus could introduce pathogens into the surrounding environment (Jiang 2017).

To reduce the microfiber released from synthetic clothing during domestic laundering, there are several significant directions for future research such as: (1) conduct comparative studies on the varying types of washing machines; (2) evaluate the effects of wash duration and spinning speed on the microfiber released; (3) examine the influences of fabric design and textile choice on the microfiber loss; and (4) investigate the temporal dynamics of release throughout the life time of a garment product (Napper and Thompson 2016).

In addition, the standardized and effective analytical techniques used for the identification and characterization of microplastics in the wastewater systems must be developed and verified by extensive amounts of research (Bayo et al. 2016). For example, the use of different units (e.g., number/volume and mass/volume) makes the direct comparison among the current studies impossible (Silva et al. 2018). Besides, there are several challenges in the existing sampling techniques for microplastics, including: (1) the highly-efficient and detailed sampling methods are still lacking; (2) the seasonal or inter-annual variants of environmental parameters have not been considered in the previous literature; and (3) most studies don't

consider the short spatial and/or temporal (hours and meters) variations (Costa et al. 2018; Silva et al. 2018).

Furthermore, the underlying mechanism of accelerated proliferation of microplastics throughout the multiple stages of wastewater treatment processes requires to be examined. For instance, the role of microorganism in the anaerobic digestion process must be thoroughly understood as it might serve as a promising remediation approach (Mahon et al. 2017).

9 Conclusions

Microplastics as one of the serious environmental problems have received a great deal of attention. However, there is still a lack of thorough understanding about microplastics originating from the wastewater systems. Thereby, this review article summarizes the following topics: (1) sources of microplastics in WWTPs; (2) sampling, extraction, and characterization methods; (3) fate and transport of microplastics during wastewater treatments; and (4) environmental contamination caused by microplastics-containing effluent. The key conclusions are:

1. Microfiber caused by domestic washing of clothing significantly contributes to the microplastic pollution to the environment since they can pass through the wastewater treatments and be detected in the effluent; however, most microbeads originating from PCCPs can be removed by conventional wastewater treatments.
2. Pumping coupled with filtration is a favourable sampling technique for collecting effluent of WWTP and the sampling technique by container collection is more useful for the collection of influent.
3. Although FTIR and Raman spectroscopy are two effective techniques for analyzing the chemical composition of microplastics, there is still a lack of methods specially designed for the characterizations of microplastics present in the organic-rich wastewater.
4. Despite the traditional wastewater treatments can efficiently remove microplastics, an advanced treatment is still required in order to improve the removal efficiency of small-sized microplastics (< 100 μm).
5. Even though the role of microplastics as vectors for POPs is still undetermined (e.g., PAHs and PCBs), microplastics have demonstrated the possibilities for absorbing harmful agents like pharmaceuticals which can be identified in the wastewater.

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