Chapter 8

Evaluating wastewater effluent as a source of microplastics in environmental samples

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8.1 INTRODUCTION

Microplastic's (GESAMP, 2015) association with wastewater discharge predates 21st Century concerns with microplastic pollution. Synthetic clothing fibers and plastic microbeads from household laundering and consumer products have long been routed to wastewater treatment plants where it is reported that most are captured in the sewage sludge and the remainder emitted in the effluent (Fendal & Swell, 2009; Gregory, 1996; Habib *et al.*, 1998; Ziajahromi *et al.*, 2016). In the late 1990s synthetic fibers from clothing were proposed as an indicator of wastewater environmental fluxes after it was found that concentrations of synthetic fibers decreased with increasing distance from sewage sludge application or from outfalls (Habib *et al.*, 1998). Today synthetic fibers are a recognized form of pollution that are increasingly monitored by environmental

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scientists (Browne *et al.*, 2011; Miller *et al.*, 2017), and microbeads have been banned from use in personal care products (CA State Legislature, 2015; US Congress, 2015). Over the ensuing decades we have learned that synthetic fibers also originate from atmospheric deposition (Baldwin *et al.*, 2016; Dris *et al.*, 2016). With this discovery, and further elucidation of other sources of plastic pollution, the ability to identify microplastic sources from environmental samples has come under increasing scrutiny (Leslie *et al.*, 2017).

Research papers assessing the provenance of microplastics in environmental samples have reported a wide range of confidence in their ability to attribute sources. Some reports have stated that locating the source of plastics from environmental samples is impossible, or that there is a high level of uncertainty in the assessment (Claessens *et al.*, 2011; Leslie *et al.*, 2017; Woodall *et al.*, 2014). Other reports state with confidence that the microplastics they observed in the environment originated from wastewater effluent (Estahbanati & Fahrenfeld, 2016; Vermaire *et al.*, 2017; Warrack *et al.*, 2018), or not (Campbell *et al.*, 2017). The differences in true levels of certainty arise primarily from differences in the technique used to identify sources.

To address the contribution of study design to this microplastic source uncertainty, 23 papers assessing wastewater effluent as a source of microplastics to the environment have been reviewed using evidence gathered from samples collected outside of the effluent discharge point. For details on the physical techniques for microplastics sampling strategies, see: Hidalgo-Ruz *et al.* (2012); Blair *et al.* (2017); Li *et al.* (2017); Hanvey *et al.* (2017); Shim *et al.* (2017); Mai *et al.* (2018); and Silva *et al.* (2018). This chapter focuses on the following question: *Is the way we are ascribing source to wastewater using environmental samples accurate, and how can it be improved?* In this context, source can be described as an absolute source (with an exact number of microplastics coming from wastewater) or as a relative source (with a proportion of microplastics from wastewater compared to from another source). The papers reviewed all assessed relative sources. As in other areas of interest in microplastics research, the number of papers that have discussed wastewater effluent source allocation has risen in the past few years (Gago *et al.*, 2018) (Figure 8.1). The 23 papers reviewed here represent globally distributed regions (Figure 8.1) and all continents are represented except Oceania and Antarctica. Eighteen of the papers suggested that wastewater treatment plant effluent is a source of microplastics in their study region (Table 8.1). Eleven of the papers explicitly stated that determining the source of plastic was a primary goal (Table 8.1). The studies leveraged samples from surface water, sediment, the water column and organisms to assess plastic pollution in streams, coastal oceans, lakes and estuaries. Marine environments were studied in eight of the reviewed papers, whilst the remainder studied freshwater. Six of the studies compared environmental samples to wastewater effluent samples (Table 8.1). In this critical review, the techniques used to assess sources of microplastics in the environment were reviewed and provide a framework for how

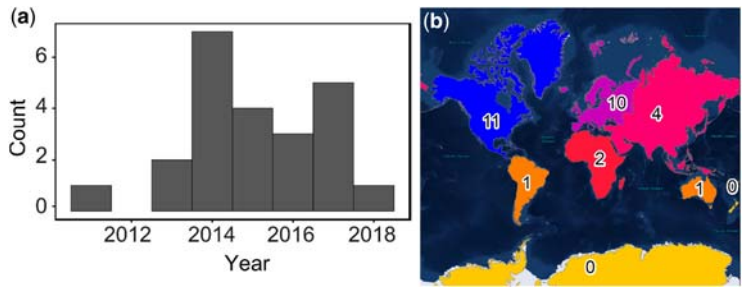

Figure 8.1 (a) Histogram of annual report numbers from the 23 studies in this review. (b) number of papers in each continent from this review.

these claims can be weighed by the scientific community, from lowest certainty (anecdotal evidence) to highest (full mass balance evaluation) (Figure 8.2).

8.2 ANECDOTAL EVIDENCE

Anecdotal evidence is obtained from experience, unverified third-party reports or *ad hoc* estimation of potential sources and is the point where most scientific inquiry begins. Although most scientists would agree that anecdotal evidence should not be a primary basis of scientific conclusions, sometimes this is the case. Free *et al.* (2014) and Zhang *et al.* (2015) collected plastics in populated regions that had no wastewater treatment plants and determined that, by default, wastewater could not be a source of the plastic pollution they found. Lechner *et al.* (2014) attributed the source of the plastic pollution they found to wastewater effluents because their sampling locations were downstream of wastewater treatment plants that were of low purification (primary and secondary treatment). These are contrasting examples of using anecdotal evidence. While Zhang *et al.* (2015) and Free *et al.* (2014) determined that a lack of municipal wastewater treatment and poor wastewater quality indicated a low likelihood for the source of the plastics to be from wastewater, Lechner *et al.* (2014) reached the opposite conclusion. This contrast seems to be centered around the question, "What is wastewater?"

We advance the argument that wastewater effluent should be defined as any sewage water discharged by humans and agree with Lechner *et al.* (2014) that a lack of treatment or lower quality treatment corresponds to a higher likelihood for wastewater discharge contaminated by plastics. In future studies, the degree of wastewater treatment could be classified as informal or formal (with an in-depth explanation of the degree of treatment) and evaluated in terms of connectivity to aquatic systems. For example, in developing countries with no formal treatment, waste effluent with high connectivity to receiving water bodies such as open sewers and clothes washing in streams could represent a substantial source of informal wastewater-based microplastics. Additionally, an

Table 8.1 Metadata from reviewed publications.

Source	Paper Type	Publisher Name	Quantification Technique	Taxonomy Techniques	Size Range	Density Separation	OM Destruction	Field Methods	Smallest Sample Volume (L)
Baldwin *et al.* (2016)	Article	Environmental Science and Technology	Count	Shape (Fibers, Fragments, Pellets/Beads, Foam, Film), Size (0.355–0.99, 1–4.75, >4.75)	>333 μm	NA	H_2O_2	Net	1000+
Browne *et al.* (2011)	Article	Environmental Science and Technology	Count	Polymer (Polyester, Acrylic, Polypropylene, Polyethylene, Polyamide)	NA	NaCl	NA	Grab Sample	0.25
Campbell *et al.* (2017)	Note	FACETS	Count	Shape (Fibers, Fragments, Beads, Foam, Film)	>5 μm and >80 μm water	NA	Yes	Traps	500
Castañeda *et al.* (2014)	Rapid Communication	NRC Press	Count	Shape (Microbeads)	>500 μm	NA	NA	Grab Sample	2.25
Claessens *et al.* (2011)	Article	Marine Pollution Bulletin	Count	Shape (Fibers, Granules, Films, Spherules)	>38 μm – 1 mm	NaCl	NA	Cores	0.5
Dris *et al.* (2015)	Research Paper	CSIRO Publishing	Count	Size (100–500, 500–1,000, 1,000–5,000)	>80 μm and >330 μm	NA	NA	Net	450
Dubaish and Liebzet (2013)	Research Article	Water Soil and Air Pollution	Count	Shape (Granular, Fibers)	>40 μm	NA	H_2O_2	Grab Sample	0.1
Eriksen *et al.* (2013)	Article	Marine Pollution Bulletin	Count	Shape (Fragment, Film, Foam, Pellet, Line), Size (0.355–0.999, 1–4.749, >4.75)	>333 μm	NA	NA	Net	1000+
Estahbanati and Fahrenfeld (2016)	Article	Chemosphere	Count	Shape (Primary, Secondary), Size (63–125, 125–250, 250–500, 500–2,000)	>125 μm	NaCl	H_2O_2	Net	1000+
Free *et al.* (2014)	Article	Marine Pollution Bulletin	Mass and Count	Shape (Fragment, Line/Fiber, Pellet, Film, Foam), Size (0.355–0.999, 1–4.749, >4.75)	>333 μm	NaCl	H_2O_2	Net	1000+
Gallagher *et al.* (2016)	Article	Marine Pollution Bulletin	Count	Shape (Fibre, Rounded, Irregular, Oval), Color (Black, White, Clear, Blue, White/Blue, Grey, Yellow, Green, Orange, Brown, Blue/Black)	>300 μm	NaCl	NA	Net	1000+
Lechner *et al.* (2014)	Short Communication	Environmental Pollution	Mass and Count	Shape (Pellets, Flakes, Spherules, Other)	>500 μm	Yes	NA	Net	1000+

Source	Access	Goal Includes Effluent or Source Allocation	Is Effluent a Contributor?	Technique	Media	Ecosystem	Country	Chemical Confirmation	Sampled Wastewater
Baldwin *et al.* (2016)	Yes	Yes	No	Correlation	Surface water	Stream	USA	No	No
Browne *et al.* (2011)	No	Yes	Yes	Taxonomic	Sediment	Shorelines	Multiple	FTIR	Yes
Campbell *et al.* (2017)	Yes	Yes	No	Correlation	Organism and Water	Stream	Canada	No	No
Castañeda *et al.* (2014)	No	No	Yes	Taxonomic	Sediment	Stream	Canada	No	No
Claessens *et al.* (2011)	Yes	No	Yes	Taxonomic	Sediment	Coastal Ocean	Belgium	FTIR	No
Dris *et al.* (2015)	No	Yes	No	Taxonomic	Surface water and Atmosphere	River	France	No	Yes
Dubaish and Liebzet (2013)	No	No	Yes	Taxonomic	Surface Water	Ocean	North Sea	No	No
Eriksen *et al.* (2013)	No	No	Yes	Taxonomic	Surface water	Lake	USA	No	No
Estahbanati *et al.* (2016)	No	Yes	Yes	Correlation	Surface water	Stream	USA	No	No
Free *et al.* (2014)	Yes	Yes	No	Anecdote and Taxonomic	Surface water	Lake	Mongolia	No	No
Gallagher *et al.* (2016)	No	No	Yes	Taxonomic	Water column	Estuary	UK	FTIR	No
Lechner *et al.* (2014)	No	No	Yes	Anecdote	Surface water	Stream	Europe	No	No

(*Continued*)

Table 8.1 Metadata from reviewed publications. (*Continued*)

Source	Paper Type	Publisher Name	Quantification Technique	Taxonomy Techniques	Size Range	Density Separation	OM Destruction	Field Methods	Smallest Sample Volume (L)
Leslie *et al.* (2017)	Research Article	Environment International	Count	Shape (Fibres, Spheres and Foils), Size (10–300, >300–5,000)	10–5,000 μm	NaCl	NA	Continuous Centrifuge	NA
Magnusson and Noren (2014)	Report	Swedish Environmental Research Institute	Count	Shape (Fibers, Particles, Flakes)	>300 um	NA	NA	Net	6600
McCormick *et al.* (2014)	Article	Environmental Science and Technology	Count	Bulk	>333 μm	NaCl	H_2O_2	Net	1000+
Miller *et al.* (2017)	Research Article	Marine Pollution Bulletin	Count	Color (Blue, Red, Black, Transparent, Other), Shape (Fiber, Round, Other), Length (100–1.5 mm, 1.6–3.2 mm, 3.3–9.6 mm)	> 100 um	NA	NA	Grab Sample	1
Smith *et al.* (2017)	Report	Mohawk Watershed Symposium	Count	Shape (Fibers, Films, Pellets, Foams, Fragments)	>333 μm	NaCl	H_2O_2	Net and Grab Sample	1000+
Talvitie *et al.* (2015)	Article	Water Science and Technology	Count	Shape (Fibers, Particles)	>20 um and >200 um	NaCl	NA	Filtration	20
Vermaire *et al.* (2017)	Article	FACETS	Count	Shape (Fibers, Microbeads, Other Plastics)	>100 μm	NaCl	H_2O_2	Net, Grab Sample, Pump	3.5
Warrack *et al.* (2018)	Research Article	Frontiers of Undergraduate Research	Count	Shape (Fragments, Foams, Fibers, Pellets, Films)	>333 μm	NA	H_2O_2	Net	1000+
Woodall *et al.* (2014)	Research Article	Royal Society Open Science	Count	Polymer (Polyester, Acrylic, Other Synthetics), Shape (Fiber), Color (All)	>32 μm	NaCl	NA	Cores	0.01
Zhang *et al.* (2015)	Research Article	Environmental Pollution	Count	Shape (Fragment, Sheet, Line, Foam, Microbeads), Polymer (PE, PP, PS), Size (112–300, 400–500, 500–1,600, 1,600–5,000)	>112 μm	Yes	NA	Net	2000
Zhao *et al.* (2014)	Baseline	Marine Pollution Bulletin	Count	Shape (Fibres, Films, Granules, Spherules), Size (>0.5–1 mm, 1–2.5, 2.5–5, >5)	>333 μm	ZnCl	H_2O_2	Net and Filtration	20

Source	Access	Goal Includes Effluent or Source Allocation	Is Effluent a Contributor?	Technique	Media	Ecosystem	Country	Chemical Confirmation	Sampled Wastewater
Leslie *et al.* (2017)	Yes	Yes	Yes	Taxonomic	Many	Ocean and Stream	Netherlands	FTIR	Yes
Magnusson and Noren (2014)	No	Yes	Yes	Correlation	Surface water	Stream	Sweden	FTIR	Yes
McCormick *et al.* (2014)	No	Yes	Yes	Correlation and Other Sources	Surface water	Stream	USA	No	No
Miller *et al.* (2017)	No	No	Yes	Taxonomic and Correlation	Surface water	Stream	USA	FTIR	No
Smith *et al.* (2017)	No	No	Yes	Taxonomic and Correlation	Surface water and Sediment	Stream	USA	Raman	No
Talvitie *et al.* (2015)	No	Yes	Yes	Other Sources	Water column and Sediment	Coastal Ocean	Finland	No	Yes
Vermaire *et al.* (2017)	No	Yes	Yes	Correlation	Sediment and Water	Stream	Canada	No	Yes
Warrack *et al.* (2018)	No	No	Yes	Correlation	Surface water	Stream	Canada	No	No
Woodall *et al.* (2014)	Yes	No	Yes	Taxonomic	Sediment	Ocean	Multiple	FTIR	No
Zhang *et al.* (2015)	No	No	No	Anecdote and Taxonomic	Surface water	Stream	China	FTIR	No
Zhao *et al.* (2014)	No	No	Yes	Taxonomic	Surface water	Estuary and Coastal Ocean	China	No	No

Figure 8.2 From left to right: this chapter's section headings, which correspond to a spectrum of increasing certainty from source evaluation techniques.

increasing degree of formal wastewater treatment has been found to decrease the concentration of microplastics discharged by effluent (Carr *et al.*, 2016). However, there is a large range of variability in wastewater purification techniques, particularly tertiary treatment, which can be very effective when microfiltration technologies are employed. Thus, further information on whether and by what processes wastewater is treated is of great importance for understanding wastewater derived microplastic discharges. The contribution of wastewater-borne microplastics also depends upon the connectivity between discharge and receiving body. If plastic transport time/distance from the site of waste generation to the sampled receiving body is long relative to the characteristic transport length of microplastic particles (Pizzuto *et al.*, 2017), small communities with a lower level of development (such as those studied by Free *et al.* (2014) and Zhang *et al.* (2015)) may indeed contribute a lower flux of wastewater-borne microplastics to a given waterbody relative to a plumbed system conveying primary waste.

These issues highlight the fact that anecdotal evidence is not the most effective or accurate means of determining the source of microplastics and should be used with caution. More accurate and quantitative assessments of microplastic sources and their connectivity to the environmental system of interest can often be employed.

8.3 TAXONOMIC EVIDENCE

Taxonomic approaches utilize the characteristics of microplastics in the environment, such as the shapes of microbeads and fibers (Figure 8.3), to assess microplastic provenance. Depending on the specific approach, taxonomic evidence can provide qualitative to semi-quantitative evidence of source. Of the 23 papers reviewed, 14 used some type of taxonomic evidence to assign source. Taxonomic groups from microplastics include shape, size, color, polymer type and item type. Unfortunately, the nomenclature used for these taxonomies are not standardized, and in cases where taxonomies are standard their ability to be used to determine source has come into question (Leslie *et al.*, 2017). However, by utilizing multiple forms of taxonomic evidence from microplastics, macroplastics (plastics >5 mm) and nonplastics, confidence provided by taxonomic evidence may be increased.

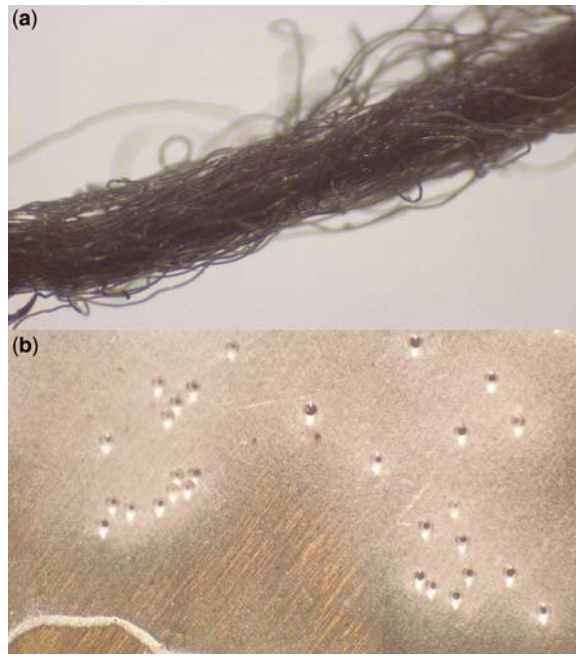

Figure 8.3 Examples of taxonomic classifications. (a) microfibers from clothing; (b) 90 μm clear plastic microbeads surrounded by 2 μm microbeads creating a halo glow (Photo credit: Win Cowger).

8.3.1 Microplastic indicators

A lack of standardized taxonomy has resulted in 19 unique categorization terms used in the literature (Figure 8.4). Though many studies do not explicitly define their taxonomic classifications, we used our best judgement and context or image examples to define the relationships between them in Figure 8.4. Ambiguity introduced from the choices of taxa and their definitions can impede cross-study comparisons and larger scale meta-analysis of existing microplastic datasets. The utility and comparability of taxonomic features would benefit from the optimization of an effective, standardized taxonomic scheme (Helm, 2017).

Some plastic taxonomies overlap in their definition. "Microbeads" and "pellets" seem to overlap in their spherical shape definition but differ based on size. Castañeda *et al.* (2014) only quantified microbeads and included particles up to 2 mm in diameter. Eriksen *et al.* (2013) analyzed consumer microbeads and classified any spherical particles in their environmental samples smaller than 1 mm as microbeads. Fendall and Sewell (2009) found very few cosmetic microbeads larger than 1 mm. We suggest that the spherical plastic particles size threshold between microbeads and pellets should be 1 mm which is a common

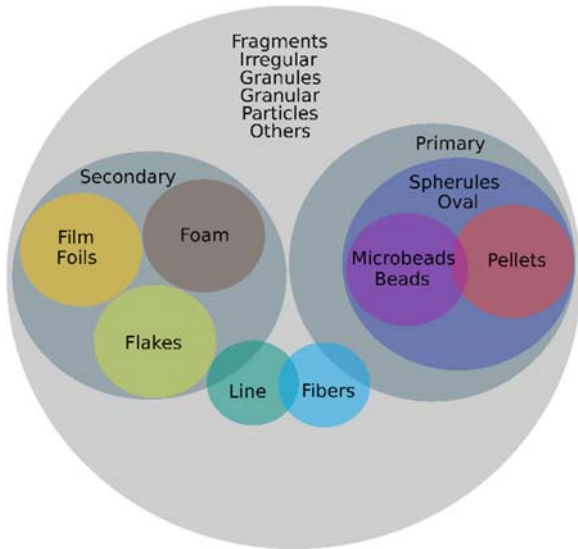

Figure 8.4 Relationships between taxonomic nomenclature. Overlapping circles represent shared definitions within studied publications. Multiple words in a circle means that those words have been used to represent the same classification.

boundary used to distinguish microplastics from mesoplastics (1–25 mm), and that all studies should report the size thresholds used to make this taxonomic distinction. Similarly, there is little that distinguishes "fibers" from "line" and some papers use the two interchangeably (Free *et al.*, 2014). Fibers and line classifications could be separated by defining fibers as from a cloth origin and line as a linear fragment, and by creating a new category for monofilament fishing line. A rigorous study on the uncertainties involved in microplastic taxonomies would greatly benefit the field (Helm, 2017).

The use of microplastic taxa alone to identify the source of microplastics has recently been challenged (Leslie *et al.*, 2017). The two most commonly encountered taxa ascribed to a wastewater origin in our review were microbeads and fibers (Figure 8.3). Microbeads and fibers are shapes that have been found to be abundant in the environment and common in wastewater effluent (Mason *et al.*, 2016). Fibers are elongated linear objects known to come from textiles (Helm, 2017) and are also abundant in atmospheric fallout (Dris *et al.*, 2016), potentially rendering fibers an unreliable source allocation tool for wastewater. Similarly, microbeads are spherical to irregular shaped plastic objects which originate from consumer products (facial washes, cosmetics and toothpastes) – reaching the environment through wastewater effluent – and from sandblasting media (Castañeda *et al.*, 2014; Eriksen *et al.*, 2013; Free *et al.*, 2014; Gallagher *et al.*, 2016; Smith *et al.*, 2017). Size and density have aided in identifying the

sources for each of these taxonomies. Microbeads are said to float if they are from a wastewater origin (cosmetic products) and sink if they are from sandblasting (Eriksen *et al.*, 2013). However, Castañeda *et al.* (2014) found non-floating microbeads that they attributed to wastewater, based on the mean diameter of the beads being similar to those found in cosmetic products. Similarly, fiber size was used by Dris *et al.* (2015) to attribute the origin of the fibers they found in stream samples to an atmospheric origin because the lengths were more similar to their atmospheric samples than to their wastewater effluent samples. By comparing fibers in wastewater effluent to environmental samples, Browne *et al.* (2011) suggested that the fingerprint from the proportions of the polymer types they found in sediments was similar to the fingerprint of fibers from laundry effluent. In this way, the strength of the evidence is amplified using multiple taxonomic characteristics beyond merely attributing all fibers or microbeads from environmental samples to wastewater. It is apparent that a thorough study of wastewater-derived microbead and fiber characteristics would be a significant contribution to the field.

8.3.2 Macroplastic indicators

Parallel to the discussion on using small microplastic taxonomies to locate sources, macroplastics (GESAMP, 2015) are distinguishable to the naked eye and can aid the identification of a microplastic source. Macroplastics can become microplastics and are often used as indicators of plastic source. Macroplastics from wastewater outfalls often reflect trash items accumulated in storm drains or items flushed into municipal sewer systems which may bypass waste treatment facilities and travel directly into waterways. When Morritt *et al.* (2014) sampled submerged litter in the river Thames (UK), they observed sanitary items and abundant litter near wastewater treatment plants, suggesting a higher proportion of plastic taxa and count can be geographically associated with wastewater outfalls. On beaches in the Bristol Channel (UK), Williams and Simmons (1997) reported macroplastics that could be assigned to wastewater outflows (sanitary items) and they attributed this to combined sewage overflow (CSO) systems whereby untreated sewage and stormwater is released to the environment during high runoff events. Similar results were reported from other beaches in the UK and elsewhere (Ross *et al.*, 1991; Storrier *et al.*, 2007; Velander & Mocogni, 1998). Since wastewater treatment technology and coverage has improved during the last decades, the occurrence of these items on beaches has decreased (Williams *et al.*, 2014), although the problem of improperly treated wastewater seems to persist even in industrialized countries (Axelsson & van Sebille, 2017), contributing significant amounts of micro- and macroplastics to rivers and the marine environment (e.g. Lahens *et al.*, 2018).

The following examples highlight the potential utility of assessing the spatial distribution of macroplastic types to identify provenance. In 2016 on an expedition to the North Atlantic Subtropical Gyre, the 5 Gyres Institute collected

38 samples with a neuston net from the sea surface between the Bahamas and New York City (5 Gyres Institute, Personal Communication 2018). The last sample was collected from the Hudson River, where a 60-minute tow in the shadow of New York City netted more plastic by weight than all the other 37 samples combined. The items were clearly associated with CSO, including plastic sticks from earbuds, tampon applicators, condoms, cigarette filters and plastic toothpicks (Figure 8.5a). Also collected were over 400 pre-production

Figure 8.5 (a) results of a 60-minute surface tow in the Hudson River, showing items discharged from CSO (Photo credit: Marcus Eriksen); (b) another example of macro debris associated with wastewater discharge (Photo credit: Martin Thiel).

plastic pellets. All of these items were caked with bentonite, a clay mineral commonly used to enhance flocculation for the removal of fine particles through sedimentation. In another case, along the coast of Chile near Coquimbo, similar types of sanitary products were collected from the shore adjacent to a submarine wastewater outflow (Figure 8.5b). Though an abundance of hygiene products certainly suggests a likely wastewater source, such an approach does not produce an absolute quantitative estimation of wastewater contributions to the total population of sampled macroplastics.

8.4 WASTEWATER INDICATORS

Non-plastic indicators can help to strengthen the evidence for a microplastic source. McCormick *et al.* (2014) found microplastics in the streams they studied and used two additional forms of evidence to determine the source of the microplastics. Elevated levels of nutrients signaled an input of wastewater that corresponded to elevated levels of microplastics (McCormick *et al.*, 2014). Additionally, microbial assemblages on the microplastics were similar to those associated with wastewater (McCormick *et al.*, 2014). Talvitie *et al.* (2015) found snail shells in environmental samples that were also common in their wastewater effluent samples and concluded that the source of the microplastics was wastewater effluent. Additional indicators of wastewater that could be used in accordance with microplastic sampling are chemicals commonly used or produced as biproducts of wastewater treatment processes, such as ethylenediamintetraacetic acid, nitrilotriacetic acid, alkylphenolethoxy carboxylates, and haloacetic acids (Ding *et al.*, 1999). While multiple taxa increase certainty in source apportionment, measuring the correlation between taxa abundances and effluent may serve as further evidence.

8.5 CORRELATION

Investigating the relationship between microplastic abundance and wastewater discharge can provide a quantitative test of the hypothesis that microplastics are being introduced by wastewater effluent (Baldwin *et al.*, 2016). There are two strategies for correlating wastewater effluent to microplastic concentrations: one, based on proximity to wastewater discharge (Campbell *et al.*, 2017; Estahbanati & Fahrenfeld, 2016; Magnusson & Noren, 2014; McCormick *et al.*, 2014; Miller *et al.*, 2017; Smith *et al.*, 2017; Vermaire *et al.*, 2017) and the other on the quantity of wastewater discharged (Baldwin *et al.*, 2016; Warrack *et al.*, 2018). To date, studies applying these techniques have not addressed potential confounding factors present in their correlations.

Sampling in proximity to wastewater effluent is typically stratified by the directional fluxes of the matrix studied, as with discharge to streams (Estahbanati *et al.*, 2016) (Figure 8.6). Monitoring sites may be located above and below the

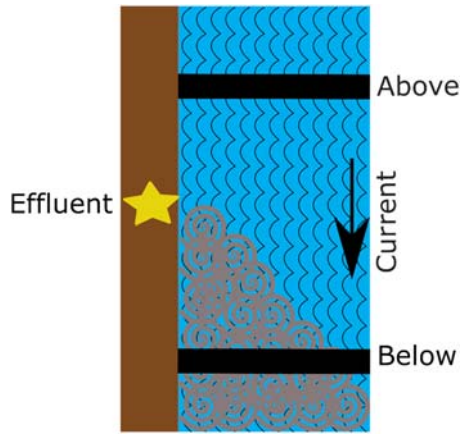

Figure 8.6 Generic sampling plan for assessing wastewater effluent impacts on microplastic concentrations in a linear flow system like a stream. Lines at "Above" and "Below" indicate generalized transect lines and represent sampling locations for a system where effluent plume structure is not known.

effluent outfall (McCormick *et al.*, 2014) or over increasing distance downstream from the outfall (Smith *et al.*, 2017). If concentrations are higher downstream or with greater proximity to the outfall, wastewater is inferred as the source. The benefit of this approach is that discharge from the effluent does not have to be known (which can be challenging data to acquire and assess) but sample sites must be stratified by effluent location.

To correlate wastewater discharge to microplastic concentration, Baldwin *et al.* (2016) sampled watershed outlets to the United States Great Lakes and, using Spearman rank procedures, did not find a significant correlation between plastic concentration and the percentage of the total streamflow from wastewater effluent. Conversely, Warrack *et al.* (2018) found that the highest season of wastewater discharge contribution corresponded with the highest concentration of microplastics found. This approach has advantages because the sites do not have to be stratified above and below the effluent pipe, but it also requires sampling a range of effluent contributions to adequately assess correlation. However, such approaches require a number of complicating assumptions, detailed below.

Complications of the proximity approach include potential issues with effluent plume mixing characteristics, confounding interactions between variables that can negatively impact the utility of correlation approaches, and the absence of flux-based considerations. While streamflow is predominantly unidirectional, the distance required for a wastewater effluent plume to fully mix across the flow field depends on the geomorphic and hydraulic conditions of the stream, the

location of the effluent outfall relative to the stream (e.g. at the bank or thalweg), and differences in the physical and chemical characteristics of the effluent and river water (Roberts & Webster, 2002). Next, confounding factors from other population driven variables are likely to be present in the correlation's signal. Wastewater quantity may positively scale with population size, development intensity (Baldwin *et al.*, 2016), or tourist seasons confounding the signal from the wastewater effluent with other potential sources of microplastics in the region. These potential confounding factors have yet to be explored explicitly. The correlation between wastewater proportional quantities such as percentage flow contribution (Baldwin *et al.*, 2016) may avoid these confounding factors when the connectivity between the wastewater outfall and the sampling location can be assumed equal among studied sites. Additionally, investigating correlations between microplastic characteristic taxa like microbead, fiber and fragment concentrations could provide more information than derived from correlative analyses of bulk concentrations alone (Baldwin *et al.*, 2016). However, potential exchange of microplastics with channel banks and beds may complicate even simple cases of stratified outfall sampling in a channelized system (Klein *et al.*, 2015). Furthermore, investigation of concentration without corresponding water discharge data omits the possibility of estimating absolute microplastic mass flux from effluent to receiving bodies, which may be present despite relative dilution from the effluent.

8.6 MASS BALANCE

The most rigorous approach to quantifying the impact of wastewater effluent on the abundance and character of microplastics in an aquatic system is a complete microplastic mass balance. To date, no studies have used this method. The components of a generic mass balance are: (i) identifying the boundaries of the aquatic system of interest; (ii) determining which boundaries are relevant to sample; and (iii) measuring or estimating the flux of microplastics across each boundary (Edwards & Glysson, 1999). Here we discuss the application of the mass balance approach to a river or stream setting (which are the most common systems studied in this review), but the approach can be adapted to other systems.

The microplastic boundary conditions of a given stream include at least the flux of waterborne microplastics from upstream, the flux of wastewater effluent microplastics and the efflux of microplastics out of the stream reach. However, additional boundaries that may serve as sources or sinks include the channel bed and banks, other surface water compartments and the atmosphere. Microplastics in atmospheric fallout are common and a likely source of contamination in samples (Dris *et al.*, 2016). Erosion or aggradation of stream bed and bank material can release or sequester microplastics to or from the flow field (Besseling *et al.*, 2017) but, even in cases of stable bed elevation, exchange of microplastic material likely occurs (Walling *et al.*, 1998). The first step toward better understanding the communication of microplastics between stream flow

and channel bed and bank materials must include further study of changes of in-channel microplastic storage over time, including spatio-temporal details of aggradational/degradational processes.

Choosing which boundaries are important requires prior knowledge of the various sources. In most cases potential sources and sinks could be reasonably assumed to be insignificant, thereby simplifying the mass balance scheme. For example, concrete drainage canals retaining little to no sediment could be assumed to have no bed and bank exchange of microplastics, an assumption that may also be applicable to "natural" channels found to be in dynamic equilibrium over the course of a study. Aeolian fluxes over the areas and time scales of sampling may also be found to be inconsequential. If the upstream channel length is much longer than the study reach, one would expect that a given parcel of water would be exposed to much more atmospheric fallout of microplastics during the travel time to, rather than through, the study reach. In most stream mass balance scenarios, one would expect that the flux of microplastics from upstream and from the wastewater effluent would be the most important components for assessing the importance of the wastewater contribution.

Spatio-temporal dynamics of particle transport and study constraints can impact decisions about how to measure microplastics. The geomorphology and hydrology of the stream channel can greatly affect the concentrations of suspended particles over short distances or times (Walling, 1983). Eddy currents in a stream can concentrate particles, and turbulent fluctuations can carry bursts of sediment and potentially denser microplastics to the surface (where most microplastic sampling has taken place) (Gray & Gartner, 2010). A comprehensive water sampling scheme should seek to dampen these short-term/range variations through samples that integrate over time periods and distances long enough to remove potential bias and outliers. The large sample requirement for microplastic analysis (often on the order of cubic meters) necessitates longer sampling times, and such considerations provide additional support for cross-channel sampling transects (Figure 8.6). Quantifying microplastic storage adjacent to the system and the flux across system boundaries imposes additional logistical constraints. Some fluxes, like aeolian microplastic deposition rates, may be relatively easily monitored with deposition pans (Dris *et al.*, 2016). However, measuring microplastic flux to/from the channel bed and banks is challenging and requires prior knowledge about the depositional morphology of the location (Hurley *et al.*, 2018). Obtaining effluent water flux and microplastic abundance and character from the wastewater source itself would be ideal and could make a mass balance unnecessary when answering the question "How much microplastic is coming from the effluent?"

8.7 STANDARDIZATION

How should source allocation of microplastics to wastewater be standardized? The reviewed studies nearly all monitored aquatic systems by sampling only the top of

the water column, and in some cases channel bed sediments, leaving most of the stream water column and channel banks unmonitored. Sample sizes ranged from 1 L grab samples (Miller *et al.*, 2017) to long trawls through many cubic meters of water (Eriksen *et al.*, 2013), with smaller samples generally resulting in much higher concentrations (Barrows *et al.*, 2017). Minimum particle size thresholds ranged from 5–500 μm and there were 20 unique microplastic size ranges introduced in the reviewed literature. To merge these data requires a number of assumptions about the total size distribution of microplastics sampled in each study. Only two of the studies quantified microplastic mass (Free *et al.*, 2014; Lechner *et al.*, 2014); the rest measured count alone. However, the range of error in converting between count and mass could be as high as five orders of magnitude (Schmidt *et al.*, 2017) (Figure 8.7). If researchers measured the size of each particle directly, scientists could be able to more readily and accurately compare results (Mintenig *et al.*, 2018). Lechner *et al.* (2014) compared mass to count and found that the proportional abundance of shape taxonomies changed, consequently redistributing the rank of the taxonomies – which begs the question: should researchers be using count (which is not a conserved unit) to measure flux? Furthermore, access to data is a requirement for repeating results and comparing literature; however, our investigation indicates that only six of the reviewed papers had published data through an open access portal by the date of this review. To standardize future research, sampling and analysis protocols should be developed for the full water column and stream bank; additionally, efforts toward quantifying mass, count, and particle size characteristics should be emphasized, and adopting a community open access policy for data archiving and dissemination should be prioritized.

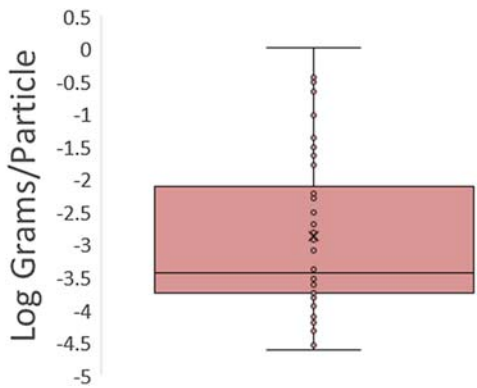

Figure 8.7 The log normalized grams per particle extracted from stream microplastics studies in Schmidt *et al.* (2017), showing that there is a range of five orders of magnitude involved in converting between particle count and mass.

After reviewing these recent papers on microplastic pollution from wastewater effluent, we must address a fundamental question: are these studies reliable? The majority of them (14) employed no chemical confirmation to guarantee plastic as the material and relied on visual observation alone. The smaller the particle, the less accurate the plastic identification by visual techniques, with particles below 1.5 mm having a significant increase in error of identification (Löder *et al.*, 2017; Kroon *et al.*, 2018). Raman and FTIR spectroscopy are complimentary techniques for determining microplastic polymer types, as they are able to characterize extremely small microplastics (with a minimum size of 1 μm and 20 μm, respectively) (Käppler *et al.*, 2016). The cost and time of analysis is substantial (in our experience ~$500–1000 and ~10–50 h per sample), and it is evident that automated techniques will be required in the future (Primpke *et al.*, 2018). However, the field of microplastic pollution is rapidly moving in the direction of spectral verification and we expect that wastewater studies employing analytical chemistry techniques will become the norm.

8.8 CONCLUSIONS

This chapter has organized existing approaches into a framework that can be used in a multiple lines of evidence approach to assess the source of microplastics in the environment. Some forms of evidence should be given more weight than others. While anecdotal evidence provides a reason for further investigation, it should be followed by investigation founded on more quantitative techniques. Taxonomic evidence can define a fingerprint that may be matched to a source but, as yet, there is no accepted or standardized taxonomic system or strategy for microplastics. For now, multiple forms of taxonomic evidence are essential to any source identification. Other forms of nonplastic evidence related to water sources should be combined with information on the various environmental forces acting on the sample area. Correlation can provide a more quantitative source attribution technique as long as potential confounding factors are explored in depth. By explicitly considering – if not fully elucidating – the mass balance of microplastics in the aquatic system of interest, improved source location from environmental samples can be achieved. However, we emphasize that sampling the effluent itself, if possible, remains the most accurate and valuable component of a wastewater source investigation. Looking into the future, we propose that standardization and validation efforts are immediately needed to increase the utility and reliability of environmental microplastic source allocation, including wider adoption of molecular characterization techniques such as FTIR and Raman Spectroscopy.

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