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Microplastics removal from a primary settler tank in a wastewater treatment plant and estimations of contamination onto European agricultural land via sewage sludge recycling*

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ABSTRACT

Wastewater treatment plants (WwTPs) remove microplastics (MPs) from municipal sewage flow, with the resulting bulk of MPs being concentrated within generated sewage sludge which is frequently recycled back onto agricultural land as accepted practice in many European countries as a sustainable fertiliser resource. This circular process means that MPs successfully removed from WwTPs are deposited into the soil and able to return into the natural watercourse by means of run-off or infiltration to groundwater. This study quantifies the removal efficiency of MPs with size ranging between 1000 and 5000 µm in a primary settlement tank (PST) at a WwTP serving a population equivalent of 300,000 and provides MP concentrations in the generated sewage sludge. Our study revealed that the proportion of MPs partitioning in a PST to settled sludge, floating scum and effluent was 96%, 4% and 0% respectively, implying 100% removal of MPs of 1000–5000 μm in size. The generated sewage sludge was estimated to contain concentrations of approximately 0.01 g of MPs or 24.7 MP particles per g of dry sewage sludge solid, equivalent to ~1% of the sewage sludge weight. Using these figures and data from the European Commission and Eurostat, the potential yearly MP contamination onto soils throughout European nations is estimated to be equivalent to a mass of MPs ranging between 31,000 and 42,000 tonnes (considering MPs 1000–5000 μm in size) or 8.6×10^{13} – 7.1×10^{14} MP particles (considering MPs 25–5000 μm in size). An estimated maximum application rate of 4.8 g of MP/m²/yr or 11,489 MP particles/m²/yr, suggests that the practice of spreading sludge on agricultural land could potentially make them one of the largest global reservoirs of MP pollution. Hence, recycling raw sewage sludge onto agricultural soils should be reviewed to avoid introducing extreme MP pollution into the environment.

1. Introduction

Microplastic (MP) pollution, defined as plastic particles smaller than 5000 μ m in size, is a well-documented threat to aquatic and terrestrial ecosystems worldwide (Eerkes-Medrano et al., 2015; Hamid et al., 2018; de Souza Machado et al., 2018). MPs which have the capacity to absorb organic contaminants onto their surface, leach toxic chemical additives throughout the process of degradation, and can serve as attachment media for hazardous bacterial pathogens, are readily ingested by a range of organisms, owing to their small size (Galloway et al., 2017; Hermabessiere et al., 2017; de Souza Machado et al., 2018). The ingestion of MPs can cause negative health effects to organisms and the trophic

transfer of MPs from lower trophic organisms to top predators means that the impacts of MP exposure and ingestion not only effects organisms at an individual level but potentially impacts the whole food chain (Haegerbaeumer et al., 2019; D'Souza et al., 2020).

The pathways through which these emerging environmental contaminants enter the aquatic and terrestrial environments are currently not fully understood (Hardesty et al., 2017; Cera et al., 2020). One significant source of MPs entering the aquatic environment are the effluents from wastewater treatment plants (WwTPs) due to inefficient removal of MPs from incoming municipal sewage (Murphy et al., 2016; Talvitie et al., 2017). Whilst the quality and quantity of techniques used during the water treatment process determines the capacity of WwTPs to

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remove MPs, they can still release substantial amounts of MPs despite having high MP removal rates of 72–98% from inlet to effluent, exacerbated if large amounts of municipal sewages are treated every day (Iyare et al., 2020). For instance, a WwTP serving a 1.2 million population that treats 400 million litres of sewage per day, with a MP removal efficiency of 84%, could be responsible for a daily release of 1.6×10^8 MPs per day into the aquatic environment (Magni et al., 2019).

Although WwTPs are currently not purposefully designed to remove MPs, it is important to understand the prevalent MP removal processes in order to develop a suitable technology to curb the concentrations of MPs exiting these infrastructures and entering the environment. The primary stages of sewage treatment, that usually comprise of a primary settling tank (PST), are responsible for up to 68-98.4% of MP removal according to seven recent studies (Talvitie et al., 2015; Michielssen et al., 2016; Murphy et al., 2016; Gies et al., 2018; Blair et al., 2019; Sun et al., 2019; Yang et al., 2019). The purpose of the PST is to promote solid settling to minimise the size of suspended particles prior to the biological treatment (Riffat, 2013). The incoming sewage, with its MPs content, are separated by density in the PST; dense materials such as grit and organic solids settle to the bottom of the tank as 'sludge', where more than 90% of the MPs from the incoming sewage feed are deposited, while less dense fats, oils and grease (often known as FOGs) stay to the upper water column as 'scum' (Carr et al., 2016). Both the sludge and scum, along with the MP load from the incoming sewage, are combined to produce sewage sludge, which is then subjected to additional treatment such as thickening, aerobic digestion, and de-watering (Kelessidis and Stasinakis, 2012; Mintenig et al., 2017; Alavian Petroody et al., 2021). MPs have been found in sewage sludge and vary in abundance depending on the treatment processes used at the WwTP and the population equivalent it serves (Mahon et al., 2017; Edo et al., 2020; Rolsky et al., 2020). For example, at a Chinese WwTP serving a population equivalent of 100,000, 2.92 MP particles per g of dry sewage sludge was measured, corresponding to 1.14×10^{11} particles per year deposited in the sewage sludge generated, while from an Italian WwTP serving a population equivalent of 1.2 million, 113 \pm 57 MP particles per g of dry sewage sludge was observed, corresponding to over 1.24×10¹² MP particles per year deposited in the sewage sludge generated (Magni et al., 2019; Ren et al., 2020).

Sewage sludge is commonly recycled to agricultural land as a sustainable and renewable source of fertiliser throughout European countries, owing to the European Union's directives that promote diverting sewage sludge away from landfill and incineration, and towards energy production and agriculture, contributing to goals that lead to net-zero waste and sustainable economic growth (e.g. European Landfill (1999/31/EC(30)) and Renewable Energy (2009/28/EC(31)) (EU Commission, 2009a, 2009b; 1999; Mininni et al., 2015). The spreading of sewage sludge onto agricultural land has been acceptable practice until the ever-increasing MP presence appeared as a new environmental threat to terrestrial ecosystems through MP deposition onto agricultural lands. Concentrations as high as 541 MP particles per kg of soil were found in agricultural soils that had sewage sludge applied to them in Ontario (Canada), compared to 4 MP particles per kg in control non-sewage sludge applied soils, meaning that sewage sludge land application is contaminating agricultural soil with MPs (Crossman et al., 2020). Based on quantitative data from national MP inputs to WwTPs from Denmark, Sweden and Norway, it was projected that 63,000-430, 000 tonnes of MPs are applied onto European agricultural land each year, with average and maximum loadings per-capita of 2 and 80 g of MP/m²/yr, respectively (Nizzetto et al., 2016). In addition, Mohajerani and Karabatak (2020) estimated that between 26,000 and 151,000 tonnes of MPs are disposed onto European agricultural soils using figures from a review of three papers which report concentrations of MPs in generated biosolids, produced from WwTP sewage sludge. As a result, agricultural land may represent one of the largest potential reservoirs of MP pollution worldwide, mirroring MP concentrations in global ocean surface waters (Sebille et al., 2015).

These recent findings have highlighted a need for understanding the transport of MPs throughout the environment, processes involved in their removal at WwTPs, the MP concentration within generated sewage sludge, and to provide further insights into the MP budget and contamination on agricultural soils. This study provides a better understanding of the partitioning of removed MPs in the size range of 1000–5000 µm in a PST into settled sludge, surface scum, and effluent by mass and abundance, obtained at a WwTP in Newport (Wales, UK). These field sampling data were used to estimate the MP concentration within the generated sewage sludge and to better estimate the potential magnitude of MP pollution on European agricultural soils for most nations, adopting data from yearly sewage sludge production and application rates from individual European nations (EU Commission, 2015, 2018; Eurostat, 2019b). These data will aid to improve the management of the WwTP sewage sludge process in order to minimise or remove the number of MPs contained, as well as to aid in the development of policies that regulate the MPs input limits for sewage sludge spreading on agricultural soils.

2. Methodology

2.1. Nash WwTP background and production of sewage sludge

The Nash WwTP in Newport (South Wales, the United Kingdom) is operated by Dwr Cymru Welsh Water (DCWW) and treats the combined sewage from a total population equivalent of 300,000 from Newport and Chepstow, with a pass forward flow limit of 1,415 L per second. Fig. 1 shows a diagram flow depicting the Nash WwTP consisting of an initial 6 mm screening and grit removal, a primary settling tank (PST), biological activated sludge treatment in an aerated basin, and secondary settlement, before discharging the final effluent to Julian's Pill, a tributary of the River Severn. Waste sludge from the secondary settler is returned to the PST stage for co-settlement. The PST separates incoming municipal sewage into scum and sludge, combined to create sewage sludge, which is then thickened on-site with sludge imported from smaller rural WwTPs also managed by DCWW and transported for advanced anaerobic digestion (AAD) treatment at Cardiff WwTP before being recycled back to agricultural land. From all DCWW's sludge centres (35 WwTPs), around 97,854 tonnes of dry solid (T_{ds}) sewage sludge was produced in 2019–2020, of which 5,311 T_{ds} (5.43%) was contributed by the Nash WwTP (DCWW, personal communication).

2.2. Sampling locations and procedure

Four locations at the Nash WwTP (shown in Fig. 2) were sampled to quantify the abundance of MPs in a PST and assess the removal efficiency of the PST, and to calculate the concentration of MPs in generated sewage sludge. These include: (1) the screened incoming sewage before the PST (PST feed), (2) the effluent discharged from the PST (PST effluent), (3) settled sludge removed from the base of the PST tank (sludge), and (4) surface FOG's and debris collected from the surface scum traps (scum). A 'flow and load' survey was conducted, with 24-h monitoring throughout a seven-day dry weather period, which is consistent with a standardised sampling programme to understand typical treatment works loading for other environmental pollutants (Environmental Agency, 2017; Bertanza et al., 2022), providing a total of seven samples per location (28 samples in total).

PST feed and effluent samples were collected in 15-min intervals using an automatic sampling system over 24-h periods (Aquamatic Aquacell P2 Coolbox). PST feed sample pipework was placed within the central baffle boards where the tank inlet pipework was located, while the PST effluent sample pipework was installed at the tank's outer edge where clarified effluent weirs over at the surface. On collection, the bulk samples containers were mixed and decanted into a 1 L glass sampling bottles once per day (Fig. 3). Spot samples of the PST sludge and PST scum were also taken each day. Sludge is removed from the PST tank by

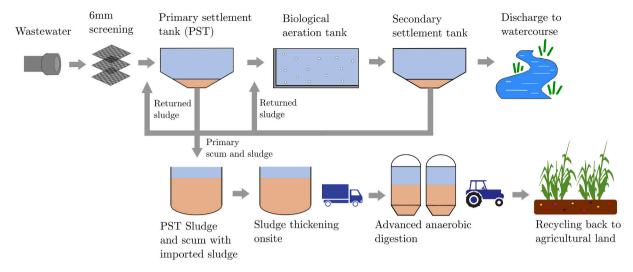


Fig. 1. Schematic diagram of the wastewater treatment flow process, sewage sludge production and environmental discharge at Nash WwTP (South Wales, UK).

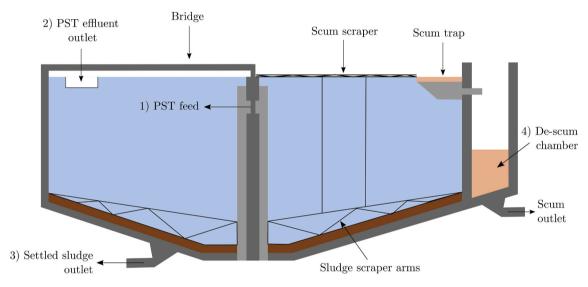


Fig. 2. A side-view cross-section of the PST and de-sludge chamber at the Nash WwTP showing the four sampling locations: 1) PST feed, 2) PST effluent, 3) settled sludge outlet, and 4) de-scum chamber.



Fig. 3. Photograph of collected samples stored in 1 L glass bottles. From left to right: PST feed, PST effluent, Sludge and Scum.

dedicated de-sludge pumps from the base of the PST tank once per hour for a maximum of 15 min or until the density of the sludge falls below 3.5% dry solids content. One litre of sludge sample was taken once per day using a tap located on the de-sludge pipework. For the scum samples, surface scrapers that revolve around the centre of the PST force the scum towards the outside of the tank into a scum trap (see Fig. 2) where the scum drains into a de-scum chamber once per revolution (approximately once per hour). A collection bucket was secured below the pipe connecting the scum trap to the de-scum chamber and decanted into 1 L glass sampling bottle (Koelmans et al., 2019). In addition, the total volume of incoming feed and outgoing effluent was recorded in litres, while the volume of scum and sludge generated by the PST was recorded in both litres and as grams of dry solid (gds) over a seven-day sampling period.

2.3. Extraction and identification of MPs from samples

MPs were recovered and identified from the samples collected at the four sampling locations (shown in Fig. 2) in four steps: (1) wet sieving, (2) wet peroxide oxidation, (3) density separation using zinc chloride (ZnCl₂), and (4) rose bengal staining and microscopic examination. Firstly, debris and organic material were removed from the samples using wet vacuum filtering with two custom graded wire meshes that divided the samples into 250–1000 μm and 1000–5000 μm size ranges; with only the latter fraction analysed in this study, as equipment to analyse the smaller size fraction was not available at the WwTP and would also require Water Companies to have specialised technicians and equipment. A volume of 200 mL from the 1 L glass bottles collected per day was used from the feed and effluent samples, while 10 mL of sludge and scum samples was used from the 1 L glass bottles collected per day, totalling 1.4 L of feed and effluent samples and 70 mL of sludge and scum samples, following recommendations by Koelmans et al. (2019) for feed samples. Due to the large amounts of FOGs that remained on the meshes during trial scum samples, an additional sample preparation step was added prior to wet sieving. This consisted of warming a mixture of 10 mL of scum sample, 100 mL of filtered water, and 10 mL of filtered washing-up liquid detergent in the oven at 50 °C for 1 h, which was then stirred for 10 min with a magnetic stirrer before sieving to remove the FOGs as they melted and disaggregated. The meshes were taken from the vacuum filtration equipment and dried in the oven overnight at 50 °C in accordance with recommendations made in Koelmans et al. (2019).

To digest the organic material in the samples, an iron catalyst and 30% hydrogen peroxide ($\rm H_{2}O_{2}$) (commonly known as Fenton's Reagent) was used (Masura et al., 2015; Liu et al., 2019). The meshes containing the samples were placed into an 800 mL glass beaker, 10 mL ferrous sulphate was added, followed by 23 mL 30% hydrogen peroxide, all stirred using a glass stirring rod and a foil cover placed over the beaker. This was left overnight to allow the reaction to complete. The meshes were then removed from the beaker and washed using filtered water into the beaker. The solids were recovered by passing the samples through the mesh using the vacuum filtration unit, from which the meshes were removed and the samples dried in the oven overnight at 50 °C.

The samples were then transferred from the meshes to a 400 mL glass beaker and a 250 mL solution of 1.7 g/mol $\rm ZnCl_2$ was added for density separation (Prata et al., 2019). The solution was stirred vigorously with a glass stirring rod and left for at least 20 h. To retrieve the MPs particles, the top quarter of the zinc chloride was sieved through the mesh. The process was repeated by adding up to 250 mL $\rm ZnCl_2$, stirring vigorously to agitate the samples and release any flocculated MPs, and then passing the top quarter liquid through the meshes to recover the MPs.

Finally, the meshes containing the MP samples were placed in a foil tray and 0.2 mg/mL rose bengal solution was added until the samples were submerged and left to stain for 5 min (Lares et al., 2019). The waste rose bengal solution was passed through the meshes to recover any MPs. The foil tray and meshes were rinsed with filtered water using the vacuum filtration unit to recover any MPs and remove the rose bengal residue. The meshes were removed from the foil tray and dried in the oven overnight at 50 °C. The remaining MP particles were then analysed and counted at $40\times$ magnification using a digital microscope. Particles that took up the stain were considered to be MPs and the remaining un-stained particles were discarded (Lares et al., 2019). Three rules for visual identification of MPs larger than 1000 µm proposed in

Hidalgo-Ruz et al. (2012) were also used where: (1) no cellular or organic structures are visible, (2) fibres should be uniform in thickness, and (3) length and colour should be clear or uniform. MP particles were then placed on a weighed foil tray and re-weighed. MP concentrations over the seven-day sampling period were calculated as g of MPs (g_{mp}/L) and as MP particle abundance per litre (MP $_p/L$).

It is acknowledged that analysing MPs 1000–5000 μ m in size over a seven-day sampling routine with a sample size of 200 mL per day may not fully represent the seasonally variability or total spectrum of MPs from a WwTP, while approaches such as using Fourier-transform infrared spectroscopy (FTIR) would have reduced uncertainty in the MP identification. However, the methods used in this present study are successful techniques for MP isolation and identification in WwTP sludge samples for MPs 1000–5000 μ m in size using readily available equipment and reagents accessible at an on-site WwTP laboratory (Ziajahromi et al., 2017; Campo et al., 2019; Lares et al., 2019). Furthermore, the methods are designed in such a way that the approach could be routinely carried out by a Water Company together with the regular monitoring of other environmental pollutants, thus providing a standardised monitoring and estimation framework of MP concentrations in PST feed, effluent and sewage sludge.

2.4. Control methods

As MPs are ubiquitous and can be found in tap water, on clothing, and in the air, a number of control measures were implemented to minimise contamination of the samples (Dris et al., 2017; Brander et al., 2020). A negative control of filtered water was used to check for any MP contamination from the automatic sampling systems and throughout laboratory procedures. Filtered water was used for all washing and rinsing of laboratory equipment, sample collection buckets and bottles, and glass and metal equipment was used wherever possible. Glassware was rinsed twice with filtered water between sample transfers from one procedure to another. Furthermore, sample containers were covered with aluminium foil during waiting periods to avoid airborne MP contamination.

2.5. Data interpretation and upscaling methods

The mean MP concentration (C) from the seven-day sample was used together with the mean daily volume (Vol) processed at each of the four sampling locations to calculate the daily incoming feed of MP to the PST, and MPs leaving the PST as effluent, scum, or sludge, reported as a daily MP mass flux (g_{mp}/day , Eq. (1)) and a daily MP particle flux (MP_p/day , Eq. (2)).

Daily MP mass flux
$$(g_{mp}/day)$$

= $Vol(L/day) \times C(g_{mp}/L)$ (1)

Daily MP particles flux
$$(MP_p/day)$$

= $Vol(L/day) \times C(MP_p/L)$ (2)

The total daily MP flux and the mean daily sewage sludge production (i.e. combined scum and sludge) at the WwTP were used to calculate the concentration of MPs per g of dry pre-thickened and pre-AAD treated sewage sludge (g_{ds}), as mass (g_{mp}/g_{ds} , Eq. (3)) and particle abundance (MP_p/g_{ds} , Eq. (4)).

MP mass per g of dry sewage sludge
$$(g_{mp}/g_{ds}) = \frac{Daily mass flux of MPs (g_{mp}/day)}{Daily production of sewage sludge (g_{ds}/day)}$$
 (3)

$$\textit{MP particles per g of dry sewage sludge} \left(MP_p \, \middle/ \, g_{ds} \right) = \frac{\textit{Daily MP particles flux} \left(MP_p \middle/ \text{day} \right)}{\textit{Daily production of sewage sludge} \left(g_{ds} \middle/ \text{day} \right)} \tag{4}$$

Eurostat (2019b) is the main source of data about sewage sludge management in Europe and publishes yearly datasets, collected biennially by means of questionnaires, on the production and disposal of sewage sludge (between 2009 and 2018) for each European nation. Figures from Eurostat (2019b) are collected by the European National Statistical Institutes, by which no specific data collection method is imposed by Eurostat, from a variety of data sources, including regional or local authorities, environmental administrations and industry. An additional source of data on yearly sewage sludge management in Europe is from reports published by the EU Commission (2015, 2018) that summarise and analyse the implementation of Sewage Sludge Directives (EU Commission, 1986, 1991, 1994) by each European nation, through means of a questionnaire. The EU Commission (2015, 2018) reports are a synopsis of the replies submitted by European nations for the period 2010–2015.

Based on both the EU Commission (2015, 2018) and Eurostat (2019b) datasets, between $8-10\times10^6$ tonnes of dry sewage sludge (T_{ds}) per year is produced from European WwTPs between 2009 and 2018. Using the amount of MPs per unit of sewage sludge from the Nash WwTP and the average sewage sludge production from WwTPs in individual European countries between the years 2009 and 2018, an estimate of the MP concentration in generated sewage sludge across Europe was calculated as yearly MP mass in tonnes (T_{mp}/yr , Eq. (5)) and particle abundance (MP_p/yr, Eq. (6)). As the concentration of MPs present in the generated sewage sludge may vary depending on sewage sludge production processes, WwTP capacity, and population served, the number of MPs per unit of sewage sludge from five other studies which sample European WwTP's are also included in calculations, as presented in Table 1 (Lusher et al., 2017; Mahon et al., 2017; Mintenig et al., 2017; Sujathan et al., 2017; Lares et al., 2018; Edo et al., 2020). The other European WwTPs were chosen to include different countries where the facilities are located, WwTP operations and range of population equivalents served by the WwTP. The five studies only report the abundance of MPs per unit of sewage sludge and isolated MPs down to 25 μm in size from sewage sludge, thus calculations consider MPs smaller than the current study ($<1000 \mu m$) for MP abundance.

Results for MP mass are displayed as the lower and upper limit is based on the standard error for the production of sewage sludge for each individual European country over the available years datasets (between 2009 and 2018) from EU Commission (2015, 2018) and Eurostat (2019b) datasets, while for MP abundance, lower and upper limits are based on the lowest and highest MP $_p$ per unit of sewage sludge from the

Nash WwTP and the five other European studies.

MP mass load from sewage sludge production
$$(T_{mp}/yr)$$

= MP mass per unit of sewage sludge (T_{mp}/T_{ds})
×EU production of sewage sludge (T_{ds}/yr) (5)

MP particles load from sewage sludge production
$$(MP_p/yr)$$

= MP particles per unit of sewage sludge (MP_p/T_{ds})
× EU production of sewage sludge (T_{ds}/yr)

The EU Commission (2015, 2018) and Eurostat (2019b) datasets also provide the amount of sewage sludge that is disposed to agricultural land by each individual European country between 2009 and 2018. Based on the average yearly disposal of sewage sludge onto agricultural land, 35–44% of total generated sewage sludge from European WwTPs was recycled for agricultural use between the years 2009 and 2018, equating to 3.5–3.8 million $T_{\rm ds}$ of sewage sludge applied to European agricultural lands. Therefore, the amount of MP application per year to European agricultural soils, as well as the upper and lower limits, were calculated using the amount of MPs per unit of sewage sludge from the Nash WwTP and the five other European studies, assuming that 100% of sewage sludge directed to agricultural use is recycled back onto European soils (Eqs. (7) and (8)). Furthermore, the fraction of sewage sludge that is recycled back to European agricultural soils was also calculated for each individual European country.

$$MP \ mass \ recycled \ to \ agricultural \ land \ (T_{mp}/yr) \\ = MPs \ mass \ per \ unit \ of \ sewage \ sludge \ (T_{mp}/T_{ds}) \\ \times sewage \ sludge \ recycled \ to \ agricultural \ land \ (T_{ds}/yr)$$
 (7)

MP particles recycled to agricultural land
$$(MP_p/yr)$$

= MP particles per unit of sewage sludge (MP_p/T_{ds})
× sewage sludge recycled to agricultural land (T_{ds}/yr)

Finally, the application rate of MPs to agricultural land as MP mass $(g_{mp}/m^2/yr, Eq.~(9))$ and MP particle abundance $(MP_p/m^2/yr, Eq.~(10))$ was calculated considering the maximum amount of total nitrogen/ha/yr permitted for European agricultural soils (250 kg of total nitrogen/ha/yr), which typically represents the limiting factor determining the rate of application of sewage sludge to agricultural land (EU Commission, 1991a,b; DEFRA, 2018; Collivignarelli et al., 2019). For a typical digested sewage sludge cake, such as that produced at DCWW's AAD site in Cardiff, which is where the sewage sludge from the Nash WwTP was sent for processing before direct application to agricultural land, 250 kg of total nitrogen/ha/yr equates to a maximum of 18.7 T of wet sludge at

Table 1
Comparison of the concentration of MP particles per g of dry sewage sludge, the population served by WwTP, the lower size limit used in isolating the MPs from the sample, and section of the sewage sludge generation sampled from five other European studies, in order of decreasing lower MP size limit.

Reference	Population served by WwTP	Country	Lower size limit	MP concentration (MP $_{\rm p}/g_{\rm ds}$ \pm SE)	Type of sewage sludge
Mintenig et al. (2017)	$7.0\times 10^{3} 2.1\times 10^{5}$	Germany	500 μm	1–24	Combined PST surface scum and settled sludge
Lares et al. (2018)	N/A	Finland	250 μm	23 ± 4.2	Activated sludge
				170.9 ± 28.7	Digested sludge
				27.3 ± 4.7	Membrane bioreactor sludge
Lusher et al. (2017)	$1.8 \times 10^4 – 6.1 \times 10^5$	Norway	50 μm	1.7–19.8	Dewatered sludge and dried sludge
Mahon et al. (2017)	$6.5 \times 10^2 – 2.4 \times 10^5$	Ireland	45 μm	4.0-15.4	Aerobically digested sludge, thermal dried sludge, and lime
					stabilized sludge
Edo et al. (2020)	3.0×10^5	Spain	25 μm	183 ± 84	Combined primary and secondary settled sludge