

Norwegian University of Life Sciences

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Microplastics in river sediments, Norway: Evaluation of a recent technique for the detection of microplastic particles



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# Preface

This thesis marks the end of my Master's degree in Ecology at the Norwegian University of Life Sciences, autumn 2016. A special thank you to the Norwegian Institute of Water Research (NIVA) and everyone there for financial support and access to facilities. Working at NIVA was a great experience in all regards, and everyone made me feel right at home. A warm thank you to Ingar Becsan and Kirk Meyer for the much needed space and use of all equipment, hope you got everything back.

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To my parents, for opening my eyes to the beauty of books and of the natural world.

Thank you Ida Mosli, light of my life. It would take another thesis just to put into words what your support have meant for me during these last few years. So instead I will only say this;

"No more trains."

Norwegian University of Life Sciences

Ås, 13.12.16

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# Abstract

Plastic debris is one of the more visible pollutants in the aquatic environment. While much attention has been given to marine environments, fewer studies have explored the fate of plastics in freshwater environments. This is surprising since most of the plastic that is found in the oceans has a terrestrial source and is transported to the sea by rivers and estuaries. Due to their durability and long degradation time many polymers may last for years in the environment and continuously become more fragmented until only microscopic particles remain.

Presently there are no standardized techniques for quantifying microplastic particles from sediments. However, a recently proposed technique intended for marine beach sediments is an elutriation device (ED). Elutriation is a separation technique that entails an upward stream of gas or liquid to isolate lighter particles from heavier ones. In this thesis, an ED was constructed and evaluated for use in river sediments by testing the effects of water flow and aeration intensity on capture of supplemented microplastic pellets. In addition, microplastic particles from six sites in an urban stream in Oslo (Alna river) were quantified after sediment volume reduction with the ED. This thesis documents for the first time the occurrence of microplastics (< 5 mm) in Norwegian river sediments.

To separate microplastics from sediments a water flow of 300 L/h<sup>-1</sup> and an aeration intensity of 20 L/min<sup>-1</sup> was found to be optimal for the ED. Polymers encountered that have a 1.2 g/cm<sup>-3</sup> or higher density may not be detectable using this elutriation technique. 35 particles from the Alna river were identified by Fourier transform infrared spectroscopy (FT-IR) as plastic, synthetic polymers. 43% of the particles were microplastic, 51% were mesoplastic (5-25 mm) and 6% were macroplastic (> 25 mm). Polyethylene was the most common polymer found (34%), then in descending order polyethylene terephthalate (PET), polyurethane (PUR), polypropylene (PP) and polystyrene (PS). 17% of the polymers were so-called "road dust", i.e. they represent remains of auto paint surfaces and car tire rubbers. All polymers found in this study are consistent with similar studies in freshwater environments around the world. These findings can be groundwork for further studies on microplastic pollution in Norwegian freshwater environments.

# **Table of contents**

Preface	i
Abstract	iii
Glossary	v
Introduction	1
Methodologies and density separation	5
Materials and methods	8
Study area	8
Elutriation device	
Water flow intensity testing (artificial sediment)	11
Aeration intensity testing (river sediment)	
Field-test sediments and laboratory handling procedure	
FT-IR analysis	14
Data analysis	14
Results	
Water flow	15
Aeration	16
Field test	17
Discussion	19
Elutriation device calibration and assessment	19
Plastic items in a small urban stream in Norway	23
Conclusion	
References	
Appendix	
A1 Microplastic in freshwater studies	
A2 Water flow intensity data	
A3 Aeration intensity data	
A4 ED recovered items	

# Glossary

ED	Elutriation device
EEA	Ethylene/ethyl acrylate copolymer
FT-IR	Fourier transform infrared spectroscopy
GESAMP	Joint Group of Experts on the Scientific
	Aspects of Marine Environmental Protection
GLP	Good laboratory practice
HDPE	High-density polyethylene
LDPE	Low- density polyethylene
PAN	Polyacrylonitrile
PE	Polyethylene
PET	Polyethylene terephthalate
PMMA	Poly(methyl methacrylate)
POP	Persistent organic pollutions
РР	Polypropylene
PUR	Polyurethane
PVC	Polyvinyl chloride
SBR	Poly(butadiene:styrene)

## Introduction

Since the middle of the last century the mass production of plastics has had a steady increase in yearly global production that reached 311 million tons in 2014 (PlasticsEurope 2015). While the plastic industry has taken steps toward recycling and energy recovery processes, only 29.7% of produced plastics was recycled in Europe in 2014 (PlasticsEurope 2015) which suggest large quantities of post-consumer plastics still end up in the environment in a variety of ways. The last decade has seen an increasing public and scientific interest in the magnitude of plastic pollution and its effects on the environment. It has even been suggested that plastic waste should be classified as "hazardous" so that environmental agencies around the world could be empowered to stop the accumulation of plastic debris and to restore impacted ecosystems (Rochman et al. 2013).

Extensive literature exists that explore the contamination and littering of marine environments (e.g. Coe & Rogers 1997, Barnes 2002, Derraik 2002, UNEP 2005, Allsopp et al. 2006, Macfayden et al. 2009, Eriksen et al. 2014). Between 70 and 80% of all marine litter is estimated to derive from land-based sources (GESAMP 2010) that are being transported from inland through drainage systems, estuaries and rivers that eventually enters the oceans (Liffmann & Boogaerts 1997). Marine litter can affect human livelihoods by damaging fishing gear, fouling propellers, clot up water intakes, not to mention the aesthetic concerns at beaches or in coastal communities (UNEP 2005). There is consensus that the rate of marine debris input is increasing (e.g. Ryan & Maloney 1993, Thompson et al. 2004, Barnes 2005, UNEP 2005, Claessens et al. 2011, Bergmann & Klages 2012, Jambeck et al. 2015) making it increasingly important to identify knowledge gaps and to explore alternatives for detection and waste management.

A large part of marine litter is plastics and estimates vary between 60 and 90% of the total waste (Gregory & Ryan 1997, Galgani et al. 2015). This can be attributed to the durability and long degradation time of many plastic polymers (Zheng et al. 2005, Webb et al. 2013). Unfortunately, there is not enough comprehensive information as of yet about the rate of degradation or fate of plastic particles in natural elements. Depending on water conditions, oxidation, photodegradation and the physical abrasion it encounters some plastics may last up to 600 years in aquatic environments (Macfayden et al. 2009, *reviews see* Zheng et al. 2005, Cole et al. 2011) continuously becoming more fragmented until only microscopic particles remain.

Plastic polymer	Abbr.	%	Density	Buoyancy	Some examples of
		(demand)	(g/cm <sup>-3</sup> )	(when clean)	applications
Polyethylene	PE	29.3	0.91 - 0.96	+	Toys, bottles, pipes. Lower density PE for films, packaging, reusable bags
Polypropylene	РР	19.2	0.90 - 0.91	+	Folders/hinges, food packaging, diapers, ropes, furniture, wastebaskets
Polyvinyl chloride	PVC	10.3	1.14 - 1.58	÷	Window frames, flooring, pipes, construction, atm-cards
Polyurethanes	PUR	7.5	1.20	÷	Insulation, foams, skateboard wheels, spandex, condoms, adhesives, car tires
Polystyrene	PS	7	1.04 - 1.1	÷	Plastic cups, electronic packaging, cd cases, disposable razors
Polyethylene terephthalate	PET	7	1.32 - 1.45	÷	Soda bottles, frozen dinner packages, polyester clothing
Other polymers		19.7		±	Teflon, tires, rubbers, paints, epoxy

**Table 1.** Most common plastic polymers listed after global production and demand in 2014. Density, buoyancy and some select examples of applications (Hidalgo-Ruz et al. 2012, Nerland et al. 2014, PlasticsEurope 2015).

\* Seawater density 1.02-1.03 g/cm<sup>-3</sup>, freshwater density  $\approx$  1.00 g/cm<sup>-3</sup>, sand/sediment density  $\approx$  2.65 g/cm<sup>-3</sup>

Marine plastic debris are found everywhere from deep-sea sediments, throughout the watercolumn, floating on the surface or deposited on shorelines worldwide (Barnes et al. 2009). Eriksen et al. (2014) estimated the amount of plastic afloat on the world's oceans to be more than five trillion pieces with a combined weight reaching 250,000 tons. And Charles J. Moore and colleagues even estimated the mass of plastics in the North Pacific Ocean to be six times the mass of plankton (Moore et al. 2001). Considering that these estimates mainly take floating plastics into account, the actual mass of plastics present in oceans may be even higher than these estimates.

Due to the physical and chemical properties of individual types of plastic polymers the density of a clean polymer as compared to surrounding seawater will be a determinant if the polymer sinks or remain afloat (Filella 2015). The most common plastic polymers are polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), polyurethanes (PUR), polystyrene (PS) and polyethylene terephthalate (PET) based on global demand (PlasticsEurope 2015). Some of the more common plastics have a density similar or less than seawater (1.02-1.03 g/mg<sup>-3</sup>), for instance PE and PP, while other polymers have a higher density and will subsequently sink (Table 1). However, the addition of additives such as inorganic fillers or biofouling may increase the density of a polymer, or polymeric foaming may decrease it, changing their fates in the environment (Filella

2015). Plastic litter can have severe deleterious effects on the biota. Entanglement in litter may lead to suffocation, strangulation or eventual drowning, while ingestion may reduce foraging efficiency or give a false sense of satiety due to the stomach being filled or blocked. This may in turn cause malnutrition or even starvation (Allsopp et al. 2006, Gregory 2009). There is also a lot of concern regarding plastics' capability to transport and accumulate persistent organic pollutions (POPs) (Mato et al. 2001, Teuten et al. 2009, Wright et al. 2013) that might negatively impact aquatic wildlife.

The term of microplastics was first defined by Thompson et al. (2004) after finding small plastic particles in their plankton samples. Now most scientific literature is defining microplastic as all plastic particles < 5 mm based on the definition from GESAMP (2015). Primary microplastics are manufactured to be of small size and include particles used by the cosmetic industry. Virgin plastic production pellets can also be included as primaries (Cole et al. 2011). Secondary microplastics is the result of environmentally deteriorated larger pieces of plastics while some particles, such as synthetic fabric fibers, may be considered belonging to both groups (Dris et al. 2015a). Microplastics find their way into the environment through several pathways (Fig. 1), for instance via effluent from wastewater directly or from wastewater treatment (Magnusson 2014). Road dust and runoff together with storm water drainage, sewer overflows and agricultural runoff are also major sources of microplastic contamination to the aquatic environment (Sundt et al. 2015).



**Fig. 1.** Conceptual representation of microplastic pathways from land to aquatic environments which includes the production and runoff from domestic and industrial sectors, roads, sewage overflows, wastewater treatment effluents, atmospheric fallout and landfill leakages (*Modified after* Dris et al. 2015b).

While rivers and freshwater ecosystems can function as transporters or conduits of plastics to oceans, they are also themselves impacted by plastic pollution (e.g. Wagner et al. 2014, Dris et al. 2015a, Eerkes-Medrano et al. 2015). With a lot of the research being focused on marine environments, fewer studies have explored the fate of plastics that is retained in freshwater environments. Recent studies on the occurrence of microplastics in freshwater (Table A-1) undertaken on lakes and rivers show a less or to a certain extent similar concentration of plastics as described in marine environments (Dris et al. 2015a).

Since freshwater has a lower density than seawater it is possible that several plastic polymers can experience increased sedimentation in lakes and rivers (Filella 2015). Weathering from UV radiation, mechanical abrasion, shredding and biofouling of plastic particles can also increase the density of a polymer making it less likely to stay afloat over time (Morét-Ferguson et al. 2010, Fazey & Ryan 2016, Kowalski et al. 2016). However, Wang et al. (2016) in their recent study of surface waters of Wuhan, China, found microplastics in their samples with a higher density than the surrounding surface water. PET (density 1.37 g/mg<sup>-3</sup>), nylon (density 1.15 g/mg<sup>-3</sup>) and PS (density 1.05 g/mg<sup>-3</sup>) was collected in their samples and the authors acknowledge that not only density should be considered when addressing the distribution of microplastics in water (Wang et al. 2016). This is consistent with data from the Rhine river in Europe where Mani et al. (2016) reported PS as the highest component of the estimated 191 million plastic particles that the river transports daily. The river Danube, which is the second largest river in Europe, is estimated to empty 4.2 tons of plastic into the Black Sea every single day (Lechner et al. 2014). This equals 1533 tons every year and gives an indication of the amount of plastic possibly being transported by the large European rivers. In comparison the plastic input into the ocean from Norway is estimated to be 4000 tons annually (Sundt et al. 2015).

#### Methodologies and density separation

A standardization of methodologies is needed within the field of microplastic research. This is true for all aspects of sampling, separation, extraction and identification of microplastics. There are large discrepancies within published data making comparisons between studies or areas difficult to achieve (*extensive reviews see;* Wagner et al. 2014, Dris et al. 2015a, Van Cauwenberghe et al. 2015). One particular issue is contamination of samples. A study conducted in Paris, France, measured total atmospheric microplastic fallout from 29 to 280 particles m<sup>-2</sup> day<sup>-1</sup> with an average of 118 particles m<sup>-2</sup> day<sup>-1</sup> depending on rainfall (Dris et al. 2015b). This demonstrates the importance of employing standardized controls which detects any unintentional air contamination, especially concerning synthetic fibers. Another challenge to standardization is the diversity of the studied systems, if only considering river sampling there are many variables that may have a great impact on the results, examples include:

- Sampling station placements along the river continuum and the distance to cities/coasts/industrial areas.
- The river's structure and fluvial processes, water level, density, discharge, organic matter content and how this change in space and time.
- A passive or active sampling technique, the mesh size of nets used to capture microplastics, the placements of the nets, how long the experiment lasts.
- The depth of sediment sampling and distance to riverbank and/or bed, transect measurements, sediment size, sediment sample size, sediment quantity.

Using density separation may be the most practical way of extracting microplastics from sediments by adding salt solutions to increase the density of the added water within a sample (Hidalgo-Ruz et al. 2012). A salt solution induced sample is homogenized for a certain amount of time before left to sediment and the supernatant containing the lighter plastic particles are subsequently removed and analyzed. Thompson et al. (2004) introduced the technique using a sodium chloride solution of 1.2 kg/L<sup>-1</sup>, but this may lead to a serious underestimate since no polymers with a higher density than 1.2 g/cm<sup>-3</sup> will be extracted. Other attempts include a sodium polytungstate solution (density of 1.4 g/ml<sup>-3</sup>) (Corcoran et al. 2009), a zinc chloride solution (density of 1.6-1.7 g/ml<sup>-3</sup>) (Imhof et al. 2012) and a sodium iodine solution (density of 1.8 g/ml<sup>-3</sup>) (Claessens et al. 2013, Nuelle et al.



Fig. 2. Graphical representation of the elutriation device used for separating microplastics from sediment (*From Claessens et al. 2013*).

2014) to improve microplastic capture. The use of these solutions may not be very costeffective when considering the amount of sediment that are being analyzed (i.e. 1 kg sodium chloride about €1, 1 kg of sodium iodine approx. €70). Claessens et al. (2013) proposed the use of an elutriation device (ED) to reduce the amount of sediment in a sample before adding the salt solution. The process of elutriation is a separation technique that entails an upward stream of gas or liquid to isolate lighter particles from heavier ones. Consequently, less sodium iodine is needed to extract the finer particles. The ED is part of a two-step process of separation. The first part entails the density separation by elutriation to make the sample volume smaller, while the second consists of using part the aforementioned salt solutions to manipulate density. The following part will focus on this

first step only. Claessens et al. (2013) produced a ED (Fig. 2) based on elutriation that consisted of a PVC column that was 147 cm long and 15 cm in diameter, connected to a tap water outlet with a regular garden hose at the bottom of the column. The column was fitted with a sturdy 1 mm mesh screen on top with a sieve cover to prevent contamination of airborne particles or fibers. A second 1 mm screen with a 35  $\mu$ m mesh screen directly below it was placed at the bottom of the column to hold the weight of the sediment. They provided aeration using three air stones (50 x 25 x 25) that were placed on the bottom of the column to prevent dead zones. Aeration together with the water flow from beneath could effectively separate sediments and microplastics and let the rising water take lighter particles up where they could be captured on a 38  $\mu$ m sieve. The authors experimentally determined that a water flow of 300 L/h<sup>-1</sup> for 15 minutes would be the most efficient and the least likely to cause contamination. However, they did not report how they came to this conclusion of optimal flow rate, nor did they make any mention of the level of air pressure used in their experiments (Claessens et al. 2013).

In the scientific literature only five studies have investigated microplastic pollution in Norwegian environments (i.e. Magnusson 2014, Herzke et al. 2015, Lusher et al. 2015, Sundt et al. 2015, Bråte et al. 2016) with all focusing on marine habitats. To our knowledge no studies exist on the occurrence of microplastics in Norwegian sediments. Since microplastic pollution is a rather new field of research, especially concerning freshwater systems, many questions are still unanswered relating to both sources, fates and effects of microplastic pollution. The aim of this thesis is to establish if the recent technique for detection of microplastics from river sediments. Firstly, to optimize the use and handling of the ED the amount of air pressure and flow of water have to be evaluated and calibrated. Secondly, a field test was conducted as a first step towards quantifying the extent of microplastic pollution in an urban stream in Norway.

#### **Materials and methods**

#### Study area

With its 17 km the Alna river is the longest river in Oslo, Norway. The headwaters are Aln lake (59° 58' 0.12" N, 10° 51' 32.4" E) and the river connects with several smaller tributaries from other lakes in the 69 km<sup>2</sup> large catchment area before emptying into the Oslo fjord (NVE 2016). The river was important for the founding of Oslo about 1000 years ago since ships of that time could be pulled far upstream making the river and the surrounding coastal area good for anchoring. Over the centuries the town grew and the river continued to be important for agricultural purposes and especially saw-mills and brickworks were abundant along the course of the river from the thirteenth century (Nilsen 2010). With the onset of large-scale industrial development during the eighteenth century the river was heavily utilized and more of the original landscape was lost due to urbanization and building projects. Large parts of the river were placed in culverts, but over the last 20 years a great deal of effort has been undertaken to restore the river to its former self and now more than two-thirds of the river is easily accessible (Nilsen 2010). Steps to further restoration of this river is a priority in municipal planning towards the year 2020 and includes improving water quality, safeguarding biological diversity, improving recreational possibilities, opening the remaining culverts and preserving the remaining cultural and historical areas (Museth et al. 2008, Kommunedelplan for Alna miljøpark 2013). Several places the river is in close proximity to heavily used motorways and railroads. 20 storm water pipes and around 200 regular water pipes also encircle the river and leakages from these pipe lines and sewers combined with the remains of industry and old landfills still contribute to making Alna river one of the more polluted waterways in Oslo (Ranneklev 2009).

Sediment samples were gathered along the Alna river from six locations during summer of 2016 (Fig. 3). Each sediment sample (n = 3 at each location, each sample collected  $\leq 2$  m apart along the river bank) was collected using a standard river sediment-core sampler (diameter 5.5 cm, length 21 cm = 498 ml) and the contents was transferred into 500 ml glass sampling jars. A layer of tin foil was placed over the full glass container before attaching the plastic-coated lid while in the field. The first three sample locations were in the upper reaches of the river in Grorud valley before large railroad yards and the current industrial area. Sample location 1 was below a bridge of one of the more heavily used highways leading north from Oslo (European highway E6) and just before a

newly restored park area (Grorudparken). This upper part of the river underwent large restorations starting with the Hølaløkka park (sample location 3) that was finished in 2004 (Museth et al. 2008) and ended with Grorudparken in 2014 (pers. obs.). Sample location 2 was located in between location 1 and 3 at a small dam before reaching a small culvert area. The remaining three sample locations were in the lower reaches of the river in Svartdalsparken, at the beginning, middle and end. The last sample location (sample location 6) was roughly 40 m from where the river reaches its final culvert taking it into the Oslo fjord.



**Fig. 3.** Map of the main rivers of Oslo, Norway. Alna river to the right. Black arrows mark the six field-sample locations. Blue lines represent open parts of the river. Red lines represent areas where the river is underground in culverts (*Map modified from Oslo River Forum*).

#### **Elutriation device**

Our ED (Fig. 4a) was made to the same specifications as Claessens et al. (2013) with some modifications (*see introduction*). The PVC column was 132 cm long, measuring 105 cm from the internal 1 mm mesh screen up to where sediments and microplastics exited the column. Base of the device measured 35 cm so total length of base and column was 167 cm. The internal 1 mm mesh screen that supported the weight of sediments within the column was treated with several layers of silicone (CASCO Sanitary Transp. 300 ml) on a 1 cm<sup>2</sup> area around the entire outer part of the screen to be able to seal off water from exiting between the screen and the flanges. The mesh screen itself was cut to a diameter of 18.5 cm to prevent the silicone layer to intrude into the column. The 35  $\mu$ m mesh screen below was also cut to a diameter of 18.5 cm as to fit better with the 1 mm screen. Handling in this area was made possible by unfastening the bolts circling the flanges and four bolts placed diagonally from each other was enough to maintain column stability and to prevent leakage during all experiments.



**Fig. 4. a**) The elutriation device used in microplastic separation. **b**) 1 cm steel grate placed atop a steel bucket to remove all > 1cm pieces of stone and organic material from the collected sediments prior to entering the elutriation device.

Three standard aquarium air stones measuring 50 x 20 x 20 mm was placed in a "Y" pattern to minimize the formation of dead zones inside the column. The "Y" pattern was held in place by a small square galvanized steel ring (5 cm diameter) and fastened with copper wire which also improved handling and cleaning of the air stones between each experiment. The air stones were placed inside resting on the internal 1 mm mesh screen and activated before the commencement of each experiment. Aeration was provided by a Hiblow HP-80 air pump with a capacity of 80 L/min<sup>-1</sup>. A 9 mm one-way valve that controlled the amount of air flow connected the pump to a Manifold 6 Outlet (4 mm diameter) of which three outlets was closed off and the remaining three connected by three PVC hoses (each 2.10 m in length) to each of the air stones. By manipulating the one-way valve an airflow of approximately 40 and 20 L/min<sup>-1</sup> was achieved for aeration testing.

Adding sediments containing larger debris (> 1 cm) to a column empty of water could possibly damage both the inside of the column and the air stones, therefore no sediments were added until the water level inside the column reached ~65% height. Due to the height of the device and the use of freshwater sediments the 1 mm sieve on top and the sieve cover was replaced with a 1 cm steel grate placed atop a steel bucket to accommodate the size and handling of all collected sediments (Fig. 4b).

#### Water flow intensity testing (artificial sediment)

A standardized artificial sediment (OECD 2004) generally used in good laboratory practice (GLP) toxicity experiments on sediment dwelling biota, was produced to make sure that water flow testing could be easily replicated. The artificial sediment consisted of 75% sand, 20% kaolinite and 5% dry-weight *Sphagnum* peat. The wet- to dry-weight determination of peat was done by measuring the average weight difference after drying three individual small samples. The artificial sediment was mixed with deionized water to obtain homogeneity and a water content of 50% and a pH of 5.5. The produced sediment was poured into small plastic zip-lock bags to contain 50 ml (83.4 g  $\pm$  0.5 g) each and stored in a freezer while awaiting further use.

To analyze water flow effect on microplastic retrieval, each 50 ml artificial sediment sample was poured into a steel bucket and was spiked with ten different polymer types of factory microplastic virgin pellets ranging from 2.5 - 4 mm in size. Each individual 50 ml sample was spiked with 10

pellets of low-density PE (LDPE), 10 pellets high-density PE (HDPE), 10 pellets PET, 10 pellets polycarbonate (PC), 10 pellets PP and 10 pellets PS making it a total of 60 pellets for each sample. The sample was then stirred vigorously with a steel spoon to make sure all microplastic surfaces had come into contact with the sediment. A total of 18 samples was produced following the steps outlined above. Six of these samples were subjected to a 200 L/h<sup>-1</sup> water flow, six were subjected to a 300 L/h<sup>-1</sup> water flow and the remaining six were subjected to a 400 L/h<sup>-1</sup> water flow. Aeration was set to 20 L/min<sup>-1</sup> for all 18 samples and elutriation time for each sample was set to 25 min. The floated material was retained on a 500  $\mu$ m mesh screen and all microplastic virgin pellets were identified and counted. All the floated pellets and the ones retained within the column was accounted for between each test.

## Aeration intensity testing (river sediment)

The aeration experiment was conducted using river sediment from location 6. All sediment samples (n = 20) were collected using the standard river sediment-core sampler. After transport the sediments were taken directly from their sealed glass sampling jars and placed onto the steel grate atop the steel bucket and subsequently pushed through the openings with the help of a steel spoon. All larger pieces of stone or wood (> 1 cm) and the sieve was rinsed with < 150 ml of tap water before the larger pieces were removed by hand.

To analyze the aeration effect on microplastic retrieval each 500 ml sample was spiked with six different polymer types of factory microplastic virgin pellets ranging from 2.5 - 4 mm in size. Each individual 500 ml sample was added 3 pellets of LDPE, HDPE, PET, PC, PP and PS making it a total of 18 pellets for each sample. The sample was then stirred vigorously with the same steel spoon and poured into the top of the ED. A total of 20 samples were produced following the steps described above. Ten of the samples were subjected to a high aeration setting (40 L/min<sup>-1</sup>) and the remaining ten samples were subjected to a low aeration setting (20 L/min<sup>-1</sup>). Elutriation time for each sample was set to 40 min. The floated material was retained on a 500  $\mu$ m mesh screen and all microplastic virgin pellets were identified and counted. All the floated pellets and the ones retained within the column was accounted for between each test.

#### Field-test sediments and laboratory handling procedure

To prevent contamination of the samples, all field collection and later handling of sediments was performed wearing all cotton clothing, and cotton lab coats was used during all analysis. To test the feasibility of the ED on river sediment 500 ml of sediment was collected in each of three subsamples from all six locations along the Alna river and transferred into glass containers (n = 18). A layer of tin foil was placed over the glass container before attaching the plastic-coated lid while in the field as described above. After transport the glass sampling jars was emptied directly onto the steel grate atop the steel bucket and subsequently pushed through the openings with the help of a steel spoon. Three recognizable LDPE virgin pellets were added to each 500 ml sediment sample as a control. All larger pieces of stone or wood (> 1 cm) were removed by hand and the sieve rinsed with < 150 ml of tap water to make sure all smaller particles retained on the grate got included before being emptied into the ED. Elutriation time for each sample was set to 25 min and aeration was set to 20 L/min<sup>-1</sup>.

All the floated material collected on the 500 µm mesh screen was transferred to petri dishes and sealed until further analysis. Once ready for analysis, the samples were taken to a clean laboratory used to process biological samples. To prevent contamination during analysis, all work tools and surfaces were cleaned with ethanol and all tools used in the handling was visually inspected for contamination under a stereomicroscope prior to further work. The laboratory itself was closed off during all analysis. A Whatman<sup>®</sup> glass microfiber filter (GF-F) was placed as a control next to the stereomicroscope and examined for airborne contamination before and after each analyzed sample. Using various small tweezers and needle tools the floated samples were visually inspected on glass petri dishes under the stereomicroscope and all fragments that were deemed anthropogenic in origin was removed and placed in a smaller petri dish on top a new GF-F filter. Subsequently all fragments were catalogued, measured and photographed through the stereomicroscope using Lumen*era* Infinity Analyze Software v6.5.2.

#### **FT-IR** analysis

All collected fragments suspected to be plastic was analyzed by infrared spectroscopy. The sample surface of each individual fragment was exposed to a beam of infrared light (4000-400 cm<sup>-1</sup>) passing through contact with an attenuated total reflectance (ATR) diamond crystal using 32 iterations and with a resolution of 8 on a Thermo Scientific iS550 FT-IR. The infrared absorption spectrum recorded was then corrected for background and automatically compared to several spectral libraries to ascertain exactly what substance the sample represented.

## Data analysis

All handling of data and production of bar graphs was executed using Microsoft Office Excel 2016. An analysis of variance (ANOVA) was performed to test if differences between aeration, water flow and polymer type were significant by the use of the open source statistical program R Studio version 1.0.44.

# Results

#### Water flow

Water flow intensity significantly affected microplastic retrieval (Fig. 5, Table 2a). All LDPE, HDPE and PP was recovered in all 18 experiments on all water flow settings (Table A-2). At 200  $L/h^{-1}$  water flow only 1 PC was retrieved in total from the six replicated experiments at this setting. PS yielded an average of 6 (SD = 1.7). No PET was recovered in any of the test runs. At 300  $L/h^{-1}$  water flow PC had its highest retrieval rate (average 1.7, SD = 0.8) from all three water flow settings. PS obtained an average of 9.3 (SD = 0.5) from the six replicated experiments at this setting. At 400  $L/h^{-1}$  water flow PS had its highest retrieval rate with an average of 9.7 (SD = 0.5) from the six experiments at this setting. One single PET pellet was retrieved in one test run at this water flow setting.



**Fig. 5.** Retrieval of microplastics under a 200  $L/h^{-1}$  (blue bars), 300  $L/h^{-1}$  (orange bars) and 400  $L/h^{-1}$  (grey bars) water flow intensity.

**Table 2. a)** Summary of ANOVA on water flow, polymer type and interactions. **b)** Summary of ANOVA on aeration, polymer type and interactions.

a)	Df	Sum Sq	Mean Sq	F value	<b>Pr</b> (> <b>F</b> )
Water flow	2	15.6	7.8	22.98	< 0.001
Polymer type	5	2013.6	402.7	1188.36	< 0.001
Water flow : Polymer type	10	40.9	4.1	12.06	< 0.001
Residuals	90	30.5	0.3		

b)	Df	Sum Sq	Mean Sq	F value	Pr (>F)	
Aeration	1	0.41	00.41	3.316	0.071	
Polymer type	5	196.44	39.29	319.033	< 0.001	
Aeration : Polymer type	5	2.84	0.57	4.615	< 0.001	
Residuals	108	13.30	0.12			

Table 2 cont. b) Summary of ANOVA on aeration, polymer type and interactions.

#### Aeration

Manipulating aeration intensity had no overall significant effect on microplastic retrieval (Table A-3), but there was a significant interaction between aeration and polymer type, i.e. the effect of aeration varied between different polymer types (Fig. 6, Table 2b). No PC particles were recovered at low aeration intensity, while at high an average of 0.8 (SD = 0.8) was recovered. A high aeration setting (40 L/min<sup>-1</sup>) for each sample yielded in average 2.9 LDPE (SD = 0.3) and 2.6 PS (SD = 0.6) pellets from ten replicated experiments at this setting. All HDPE and PP pellets was retained in every test. No PET pellets were recovered in any of the test runs. A low aeration setting (20 L/min<sup>-1</sup>) for each sample yielded an average of 2.6 PS (SD = 0.5) pellets from ten replicated experiments at this setting. All LDPE, HDPE and PP pellets was retained in every test. No PET or PC pellets were recovered in any of the test runs.



**Fig. 6.** Retrieval of different types of microplastics under a high 40 L/min<sup>-1</sup> (blue bars) and low 20 L/min<sup>-1</sup> (orange bars) aeration intensity from natural river sediment.

#### Field test

The 18 collected sediment samples from along the Alna river yielded 54 particles and fiber bundles suspected of being plastic. The particles ranged from 0.49 mm to 39 mm in size. During analysis with FT-IR (Table A-4) six of the particles were lost due to handling error. Three particles were identified by FT-IR as natural materials while six had other anthropogenic origins. Four particles had too low match to be determined by FT-IR. The remaining 35 particles were plastic. 15 of the plastic particles were less than 5 mm in size and can be considered microplastic, 18 were mesoplastic and two macroplastic (Fig. 7).



Fig. 7. Size distribution of plastic items from Alna river

Of the six sampling locations along Alna river the first location yielded 54% of all plastic material found (Fig. 8). Not a single item suspected of being plastic was encountered in the samples from location number three. In every sample investigated possible textile fibers and other anthropogenic particles ( $< 500 \ \mu m$ ) were observed, however the concentration of these particles could neither be qualitatively distinguished nor mechanically collected. No attempt was made to assess or quantify plastic items retained within the ED, however five larger pieces that was discovered during cleanup between experiments where confirmed as a PE plastic bag piece, a PS bag closer, a PUR paint flake, a polyamide coated glass particle and a epoxy polymer particle (Table A-4).



Fig. 8. No. of plastic items retrieved from the six sample locations along Alna river.

The 35 particles were identified as 7 polymer types and 2 gathered groups (road dust and other polymers) (Fig. 9) with PE being the most common polymer (34%). FT-IR also matched one HDPE, one LDPE and a PE olefin fiber, these were grouped together with PE. Six PET, three PP, two PS, one poly(methyl methacrylate) (PMMA), one ethylene/ethyl acrylate copolymer (EEA) and one polyacrylonitrile (PAN) was also confirmed. Two thermoset epoxy polymers and a polyolefin fiber which can be a range of individual or mixed polymers were grouped together as other polymers. The remaining six polymers found was attributed to road dust that included three PUR particles from rust sprays, oil enamels or similar, and three rubber particles (two Michelin tire rubbers and one poly(butadiene:styrene (SBR) tire rubber).



**Fig. 9.** Plastic polymer types (n = 35) identified by FT-IR from Alna river. The Road dust category includes tire rubbers and PUR particles, Other polymers category include mixed polymers.

#### Discussion

#### Elutriation device calibration and assessment

An ED was built and tested in order to separate microplastics from freshwater sediments, based on Claessens et al. (2013). With the help of an upwards water flow and aeration the device was successful in separating the plastic particles one could expect based on their density (Table 1) from river sediments. Before using the ED on environmental samples, several test regimes were initiated by adding different microplastic polymers as described in the method section. We found a 100% recovery rate for HDPE and PP under any test regime. For other polymers it was not consistent (Fig. 5, Fig. 6), for example most PC pellets retained in the water flow intensity experiments was found at the medium setting of water flow (300 L/h<sup>-1</sup>). However, we cannot conclude on the reason for the inconsistent recovery rate of PC, and since so few PC particles were being retained in total, this is probably negligible in the bigger picture.

With PS there was a large discrepancy between the low water flow setting (200 L/h<sup>-1</sup>) and the two higher settings, which suggest that an increased water flow may increase the retrieval of PS. However, since the averages of all test runs only differed from 9.3 at medium to 9.6 at the highest setting (Fig. 5) we saw no clear benefit of using the highest water flow simply to accommodate the possibility of retaining a fraction more PS. Operating the elutriation device at the highest water flow however, increased the potential of spillage since the flow of water sometimes would re-float the pellets that had already been retained on the external 500  $\mu$ m mesh screen. Based on these findings it was concluded that a 300 L/h<sup>-1</sup> water flow setting on the ED was optimal.

The second variable we tested, aeration intensity within the column, had little effect on microplastic retrieval. Less particles of LDPE were retrieved under a high aeration intensity, something that was unanticipated considering the very light density of this polymer. It was an interaction between polymer type and aeration (Fig. 6, Table 2b), which is most likely PC, but the overall effects appear too random to be of any consequence for this study. The high aeration intensity had a tendency to form large air-bubbles on the surface within the column that spilled out through the column opening at great speed and sometimes bringing microplastic pellets with it and over the side of the external mesh screen. All the pellets were accounted for when this overflow transpired during the aeration experiments, but it is a possible issue that can seriously compromise trials with field collected

samples. Accordingly, it was determined that a 20 L/min<sup>-1</sup> aeration would be the optimal alternative for this study.

The column size in our study conveniently accommodate up to 500 ml of sediment, and this is a sample size that is effortless to gather and handle. The column size itself is operable by one person although a two-person team greatly improves handling efficiency and time. Strapping the elutriation onto a folding trolley also improves handling and transportation, and at the same time safeguards the device from falling while operating.

The opening where water containing microplastics exit the column is large and the water have a tendency to adhere to the underside of the opening. This may result in some microplastics only hitting the side of the mesh screen and the water (especially at flows >  $300 \text{ L/h}^{-1}$ ) may overflow and decrease the amount of microplastics being retained. The solution for our experiments was to place the mesh screen under and in contact with the column opening, but small amounts of water were still able to flow over the side of the sieve. If the amount of organic material in a sample was high, it had a tendency to be retained in that same area after elutriation so the mesh screen has to be physically turned every few minutes to keep it from clogging.

In my opinion the opening where water containing microplastic exits the column to the sieve and the microplastics are retained has the highest need of design improvements. To further enhance the device a 90° elbow joint attached to the opening is proposed (Fig. 10). This will increase the chance of all microplastic particles that exits the column will be retained on the mesh screens and not accidentally disappear over the side of the sieve due to excessive discharge. Since there is a problem with possible contamination from airborne plastic particles, the elbow joint will also help to reduce or prevent any contamination of this kind during the flotation procedure.

A system of several sieves in a descending order of mesh sizes will definitely better accommodate a high organic matter content and avoid clogging. Each sieve should be individually examined under stereomicroscope for plastic particles after flotation and subsequently be removed by hand or by hand-held instruments. Implementing these additions to the column will most likely enhance microplastic quantification and benefit the next step of density separation with salt solutions (*see introduction*, Thompson et al. 2004). The density separation with salt solutions is instrumental to qualitatively collect and examine very small sized particles and should complete most studies that aim to quantify microplastic abundance in freshwater environments.



**Fig. 10.** Elutriation device with new suggested components to decrease possible air contamination of samples and to increase microplastic retention. A 90° elbow joint attached to a system of several sieves in a descending order of mesh size (*Modified from Claessens et al. 2013*).

The 300  $L/h^{-1}$  water flow we used is in accordance with the recommendation of Claessens et al. (2013), but Zhu (2015) found that a water flow of 385  $L/h^{-1}$  was optimal for microplastic retrieval. Another aspect to consider is the diameter of the column. The columns used by Zhu (2015) measured from 5.06 cm to 10.16 cm in diameter, while the diameter of our column was 15 cm (*see methods*), and she tested only using one kind of polymer (unknown polymer from water bottles). Her efforts to optimize the ED demonstrate that microplastic retrieval increases as diameter decrease, because the aeration intensity will be stronger in a smaller space if kept constant (Zhu

2015). While this may be mathematically sound, when reducing the diameter of the device, it will give restrictions on the amount of sediment that can be successfully processed with this technique. The aeration (and water pressure) is also contradictory to our experience since an increased intensity will conceivably also increase overflows and accidental spillage. Another aspect is that using a smaller ED will most likely involve designing a new sieve to retain the microplastics, perhaps something similar to what is described above only on a smaller scale (Fig. 10). A minisized ED would be more practical to transport and store, and cheaper to obtain, but in my opinion further studies would have to be done into optimization to insure efficiency for all types of polymers.

Our experiments were conducted with water flow provided by tap water. It would be beneficial to use water with a higher density to increase the extraction of denser polymers (Table 1), but this was not possible for our experiments. A potential venture of the ED would be to connect it to seawater. Marine research stations that conduct experimental indoor mesocosm studies with seawater could be possible locations for operating an ED with freshwater sediments. A continuous supply of water with the density that seawater possess may potentially extract more microplastics from freshwater sediments than is currently achieved using tap water.

From the aeration and water flow testing of the elutriation device it is clear that some polymers are more readily found than others. With confidence we can extract HDPE, LDPE and PP pellets under the parameters described in this study. Most, but not all PS (density  $1.04 - 1.1 \text{ g/cm}^{-3}$ ) will be found and it seems to be affected mostly by water flow and that a higher intensity increases PS capture. The polymers with a higher density, i.e. PC (density  $1.2-1.22 \text{ g/cm}^{-3}$ ) and PET (density  $1.32 - 1.45 \text{ g/cm}^{-3}$ ) may be found, but their capture appear very random and we cannot with certainty say that they are detectable with this technique. From the polymers' density we may speculate that the upper limit of the elutriation device is somewhere between  $1.1 \text{ and } 1.2 \text{ g/cm}^{-3}$  and that all polymers with a lower density than this should be captured. Other polymers encountered in natural environments that have a  $1.2 \text{ g/cm}^{-3}$  or higher density may not be detectable.

#### Plastic items in a small urban stream in Norway

From the six sample locations along Alna river, a total number of 35 plastic items were found using the ED. Almost half (43%) of the particles collected can be classified as microplastic particles. Since the mesh screen used to retain plastic items in this study was 500  $\mu$ m many smaller particles will naturally pass through the sieve. Several fibers and black "spots" (< 0.5 mm) were observed in several of the samples, but no attempt was made to quantify these since they were too small to be mechanically handled. These are only mentioned as an observation and no data on this is presented in this study. Since our assessment of microplastics only examined 9 kg of sediments, and excluded all smaller particles that could have been collected by the use of salt solutions (*see above*), our findings is most likely a substantial underestimate of what is currently interred in Norwegian river sediments.

Of the six sample locations the first and uppermost location yielded 54% of all plastic material found. The reason for this may be that it is above the areas that were recently restored (2004 - 2014) by Oslo municipality and that the sample location is directly below the E6 European highway upstream a small dam. On each side of the E6 bridge vacant areas flank the river and much garbage is deposited nearby or thrown off the bridge. On location 3 not a single item suspected of having anthropogenic origins was encountered in the samples collected. The samples at this location was taken inside the restored Hølaløkka park, a popular recreational area for locals. Post-sampling it was discovered that only 15% of the river runs through the park area itself, the remaining part of the river is still in a culvert below-ground (Museth et al. 2008). It is possible that the water directed towards the park has less litter and waste compared to the part that runs below it, but this could not be confirmed. It is however a valid interpretation of why no items was recovered from this site in any of the three sub-samples.

The remaining sample locations at the lower part of the river (location 4, 5 and 6) were initially suspected to have a higher plastic item content than further upstream. The reason for believing so was because large and plentiful pieces of plastic waste was clearly visible around the riverbanks and riverbed. The lower part of the river is also the area that receive the largest part of municipal and industrial effluent compared with up-river. The last three locations however did not show any increased levels of plastic items as compared with the three first sites. This may be due to sampling errors since the three uppermost sample locations were all directly upstream in front of small dams

that could easily retain plastic debris. The three lower sampling locations were all situated in a more natural and wider meandering parts of the river. Care was taken in selection of the sample site, however environmental factors as riverbank topography and river currents that affect sediment deposition will not necessarily guarantee that plastics should be found in the same section of river.

The 35 particles confirmed as plastic in our samples belonged to several groups (Fig. 11). PE, followed by PET, PUR, PP and PS was the most common plastics found in Alna river. These findings are similar to other studies, e.g. Imhof et al. (2013) who found predominantly PS, PE and PP in lakeshore sediments in Italy and Castañeda et al. (2014) who reported that PE accounted for most of the plastic particles in St. Lawrence river in Canada. Klein et al. (2015) reported that PE, PP and PS did cover more than 75% of all polymer types found in the Rhine-Main rivers in Germany. Interestingly, our results indicate more PET and PUR than is mentioned in other studies of freshwater environments (*see references* Table A-1) with the exception of Wang et al. (2016) who also found PET then PP in in rivers and lakes of Wuhan, China.



Fig. 11. A selection of plastic items retrieved from Alna river.

The PUR from auto paint surfaces together with the tire rubbers amounted to 17% of all the plastics recovered in this study, and we grouped this as "road dust". This supports the findings of Sundt et al. (2015) who lists road dust as the largest contributor of microplastic pollution from Norway.

The amount of PET collected in the field samples are in contrast to the initial tests of the elutriation device where only a single PET pellet was recovered from all test runs. This may be due to the thick and square shape of the PET pellets that was used in the experiments, in comparison to the fibrous PET that were found in Alna river (*see polyesters in Fig. 11*). A fibrous appearance can possibly make them more susceptible to be incorporated together with organic material thereby keeping the polymer afloat. The fibrous PET particles may also have a higher surface/volume ratio than the added pellets and this may have facilitated the re-suspension during the elutriation process and subsequent capture on the external mesh screen. Suspension of PET particles with higher surface/volume ratio have been found in freshwater in other studies (Zhao et al. 2015, Wang et al. 2016) and is currently the most probable explanation as to the difference in obtaining PET in their experiments. The addition of additives such as inorganic fillers may also affect the density of a polymer (Filella 2015) and we cannot disregard that this may be the case for the PET particles found in Alna river.

Another explanation could be that the pellets used in all experiments here were clean when they were added to the test sediments. The PET particles found in Alna river would probably have been in contact with sediments for a longer time, and several natural weathering processes might have altered physical properties on the polymers. UV radiation, mechanical abrasion, shredding and biofouling of plastic particles for instance, have been shown to affect particle density and to facilitate increased sinking of particles in marine environments and in laboratory trials (Morét-Ferguson et al. 2010, Fazey & Ryan 2016, Kowalski et al. 2016). To my knowledge no studies have as of yet explored biofouling or weathering effects of microplastics in freshwater environments.

# Conclusion

This study provides both new insights into microplastic retrieval by using an ED, and also regarding microplastic distribution in river sediments in Norway which has not been previously studied. Although initially developed for marine beach sediments, the ED can also separate microplastics from river sediments. A sample of sediments will most likely have to be floated several times to retrieve most of the microplastics according to Claessens et al. (2013). This means that even though the device works it does so at a reduced efficiency and several runs with the same sediment may have to be performed. Some polymers however can have a density too high to be retrieved with this technique.

To improve this device a few ideas are suggested. Firstly, the addition of components that will reduce possible contamination from airborne particles and increase the potential of the device to handle sediments with heavier loads of organic material. Secondly, by increasing the density of the water that enters the column more microplastics will theoretically be able to detach itself from sediments and become retained after elutriation.

Several of the same polymers that were successfully retained during our experiments was also found in the remaining sediments contained within the column after each experiment. More work is needed in quantifying what is left within the elutriation column so this can be compared to what is actually retained on the sieves after elutriation.

The field test of river sediments confirmed that microplastic particles are being retained in river sediments in Norway. The polymers found in Alna river in descending order of magnitude is PE, PET, PUR, PP and PS and resembles the global production of polymers based on demand. These findings are consistent with similar studies of microplastic pollution in freshwater environments undertaken across the world. More data is needed on the magnitude of microplastic pollution in Norwegian sediments, and larger quantities of sediment should be analyzed to obtain a better understanding of the abundance and load of different polymers.

Since the topic of microplastic pollution is still in its infancy several research areas should be explored. There is the need for standardization of methods used in sampling, measuring and determining microplastic. This groundwork is fundamental when considering how data can be evaluated and compared. Further, measuring the magnitude and effects of microplastics in

freshwater ecosystems is essential, also in remote locations. Improving legislation and waste management infrastructure is imperative to decrease plastic pollution. A greater focus on freshwater cleanup and waste management will help curb the amount of plastics that discharge into oceans.

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# A1 Microplastic in freshwater studies

Table A-1. List of studies related to microplastic contamination of freshwater environments. Shown are what part of the aquatic environment that was the study focus, general method used for sampling and what type of identification of microplastics utilized.

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Setting	Year	Location	Focus	Method	D	Reference
Lakes	2011	Lake Huron (Canada/USA)	Shoreline sediments	Manual gathering	FT-IR	Zbyszewski & Corcoran 2011
	2012	Lake Geneva (France)	Sediments/biota/water	Manual gathering, manta trawl	Visual	Faure et al. 2012
	2013	Lake Garda (Italy)	Sediments	Random grid sediment sampling	RM	Imhof et al. 2013
	2014	Lakes Erie, St. Clair (Canada/USA)	Shorelines	Manual gathering	FT-IR	Zbyszewski et al. 2014
	2014	Lake Hovsgol (Mongolia)	Water	Manta trawl	Visual	Free et al. 2014
	2016	Lake Victoria (Tanzania/Uganda/Kenya	Biota	Manual gathering	FT-IR	Biginagwa et al. 2016
	2016	Lakes Bolsena, Chiusi (Italy)	Water/sediments	Manta trawl, manual gathering, density separation	UV microscope / SEM	Fischer et al. 2016
Rivers	2011	Rivers Los Angeles, San Gabriel (USA)	Water	Manta net, streambed samples, hand net	Visual	Moore et al. 2011
	2014	Rivers Elqui, Maipo, Maule, BioBio (Chile)	Riverbank sediments	Visual sampling $> 1,5 \text{ cm}$	Visual	Rech et al. 2014
	2014	River Thames (England)	Subsurface water	Eel nets	Visual	Morrit et al. 2014
	2014	River Danube (Germany/Austria)	Water	Stationary driftnets (0.5 mm mesh size), density separation	Visual	Lechner et al. 2014
	2014	Rivers Elbe, Mosel, Neckar, Rhine (Germany)	Sediments	Manual gathering, density separation	Visual	Wagner et al. 2014
	2014	River St. Lawrence (Canada)	Riverbank sediments	Grab samplers	DSC	Castañeda et al. 2014
	2015	Rivers Seine, Marne (France)	Water	Manta trawl, plankton net	Visual	Dris et al. 2015b
	2015	River Rhine (Switzerland, Netherlands)	Water	Manta trawl	FT-IR	Mani et al. 2015
	2015	River Rhine-Main (Germany)	Sediments	Steel spoon/ sieving/ density senaration	FT-IR	Klein et al. 2015
	2016	Rivers, various in Wuhan province, China	Water	Surface water sampler	Visual / FT-IR	Wang et al. 2016
* FT-IR (Fc	vurier-tran:	sform infrared spectroscopy), RM (Raman microsp	pectroscopy), SEM (Scanni	ing Electronic Microscope), DSC (	Differential Scannin	g Caliometry).

200 L/h <sup>-1</sup>	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
LDPE	10	10	10	10	10	10
HDPE	10	10	10	10	10	10
PET	0	0	0	0	0	0
PC	1	0	0	0	0	0
РР	10	10	10	10	10	10
PS	9	8	8	5	5	4
300 L/h <sup>-1</sup>	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
LDPE	10	10	10	10	10	10
HDPE	10	10	10	10	10	10
PET	0	0	0	0	0	0
PC	1	ŝ	2	2	1	1
PP	10	10	10	10	10	10
PS	6	6	6	6	10	10
400 L/h <sup>-1</sup>	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
LDPE	10	10	10	10	10	10
HDPE	10	10	10	10	10	10
PET	0	0	1	0	0	0
PC	0	2	3	0	0	2
РР	10	10	10	10	10	10
DC	10	U	10	10	10	C

Table A-2. Water flow intensity data. Six replicate tests each containing 60 virgin plastic pellets from six different polymers under three different water flow

A2 Water flow intensity data

High aeration 40 L/min <sup>-1</sup>	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9	Test 10
LDPE	3	3	3	3	2	3	3	3	3	3
HDPE	3	3	3	3	3	3	3	3	3	3
PET	0	0	0	0	0	0	0	0	0	0
PC	2	1	1	0	0	1	2	0	1	0
PP	3	3	3	3	3	3	3	3	3	3
PS	С	3	б	1	7	3	3	б	7	С
Low aeration 20 L/min <sup>-1</sup>	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9	Test 10
LDPE	3	3	3	3	3	3	3	3	3	3
HDPE	3	3	3	3	3	3	3	3	3	3
PET	0	0	0	0	0	0	0	0	0	0
PC	0	0	0	0	0	0	0	0	0	0

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A3 Aeration intensity data

Sample ID	Sub-sample	Plastic Sample no	Color	Size(mm)	Comment for FT-IR	Polymer
la	A	1	None	8,00	Unidentified	
	а	2	Blue	1,73	Unidentified	
	а	3	None	5,00		PET
	а	4	Green	2,93		PMMA
	а	5	None	7,74		PET
	а	6	White	0,51	Unidentified	
	а	7	Red	7,97		PET
	а	8	White	23,00		LDPE
	а	6	White	5,32	Anthropogenic item, not plastic	
	а	10	White	20,00		PE
	а	11	White	16,00		PE
	а	12	White	18,00		HDPE
	а	13	White	12,00		PE
	а	14	White	39,00		PE
	а	15	White	36,00		PE
1b	Ą	1	None	4,69	Unidentified	
	þ	2	Green	6,25		PET
	р	3	Green	9,31		Other polymer (Polyolefin)
	þ	4	None	4,50		Road dust (Polyurethane)
	þ	5	White	1,35	Anthropogenic item, not plastic	
	þ	9	Blue	1,75	Unidentified	
	þ	7	White	3,56		Other polymer (epoxy)
	þ	8	Green/white	2,21		PP
1c	c	1	Blue/white	2,24	Anthropogenic item, not plastic	

Table A-4 c	ont. Items from	the elutriation device.	Sample ID re	sfers to sample l	ocation 1 through 6. A, b, c, represent s	ub-samples taken at each location.
Sample ID	Sub-sample	Plastic Sample no	Colour	Size(mm)	Comment for FT-IR	Polymer
	c	2	Black	5,57		Road dust
	c	.0	Silver	2,35	Anthropogenic item, not plastic	
	С	4	Red	4,35		PAN
	c	5	Green	25,00		PE
2a	а	1	Red	0,98		Road dust (polyurethane)
	а	2	White	2,01		PS
	а	3	Brown	8,01	Natural	
	а	4	None	6,71		PP
	а	5	White	4,15		PS
	а	9	None	4,01		PE
2b	þ	1	Red	2,25	Unidentified	
2c			ı	ı	1	
3a, b, c	ľ	I		ı	ı	
4a	ı	I	ı	ı	ı	
4b	þ	1	Metallic	5,37	Unidentified	
	þ	2	None	14,00		PE
	þ	3	None	5,86		PE
	þ	4	Red	2,33		Road dust (polyurethane)
	q	5	None	3,74		PE
4c	ı		I	I		

Table A-4 c Sample ID	<i>cont.</i> Items from Sub-sample	n the elutriation device. Plastic Sample no	Sample ID ref. Color	ers to sample le Size(mm)	ocation 1 through 6. A, b, c, represent su Comment for FT-IR	b-samples taken at each location. Polymer
5a						
5b	q	1	Black	3,13		Road dust (SBR)
	þ	2	None	19,00	Anthropogenic item, not plastic	
	þ	3	Black	6,91		Road dust
5c	C	1	Green	9,70		PP
6a	а	1	Red	0,49	Unidentified	
	а	7	Blue	0,72	Unidentified	
6b	q	1	Green/white		Natural	
	þ	2	Red	1,59	Anthropogenic item, not plastic	
	þ	3	None	1,89	Unidentified	
	þ	4	Blue	2,18		PET (polyester)
	þ	5	Red	0,62	Natural	
	þ	6	None	2,70		PET (polyester)
	þ	7	White	1,02		Other polymers (Epoxy)
6c	c	1	None	5,70		EEA
Items		1	White			Sd
recovered		2	None			PE
inside the elutriation		3	Off white			Other polymers (polyurethane)
device		4	Shiny white			Pumice glass with polyamide coating
		5	Green gray			Other polymers (Epoxy)



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