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# **Microplastic pollution in an Urban Norwegian River Sediment – An Investigation of Freshwater Sediment Extraction by Elutriation**

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Master of Science in Natural Resource Management

## Glossary

### Polymers

DAP	Diallyl phtalate
HDPE	High-density polyethylene
LDPE	Low-density polyethylene
PF	Phenolic resin
PET	Poly(ethylene terephtalate)
PEVA	Poly(ethylene-vinyl acetate)
PMMA	Poly(methyl methacrylate)
PC	Polycarbonate
PP	Polypropylene
PS	Polystyrene
PUR	Polyurethane
PVA	Polyvinyl alcohol
PVC	Polyvinyl chloride

### Other

ANOVA	Analysis of Variance
ATR	Attenuated Total Reflection
ED	Elutriation device
FT-IR	Fourier transform infrared spectroscopy
GPS	Global Positioning System
GLP	Good laboratory practice
MSDF	Marine Strategy Framework Directive
NIVA	Norwegian Institute for Water Research
KHiO	Oslo National Academy of the Arts
POPs	Persisten organic pollutants
GF-F	Whatman <sup>®</sup> Glass Microfiber Filter

### Chemicals

HCl	Hydrochloric acid
H <sub>2</sub> O <sub>2</sub>	Hydrogen peroxide
HNO <sub>3</sub>	Nitric acid
NaCl	Sodium chloride
NaI	Sodium iodine
SPT	Sodium polystungstate
H <sub>2</sub> SO <sub>4</sub>	Sulfuric acid
ZnCl	Zinc chloride

## Preface

his thesis marks the end of my Master's degree in Natural Resource Management at the Norwegian University of Life Science (NMBU), spring 2017. A special thanks to the Norwegian Institute of Water Research (NIVA) for funding materials and lab room access, and to all the staff whom have been of great help. Being a guest at this institute was enlightening, educational and fun. A special shout-out and thank you to the two most patient and well sought after engineers Kirk Meyer and Ingar Becsan, sorry we made such a mess in the garage but it was fun, wasn't it?!

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To my mom, thank you for being gutsy and strong-willed. And my dad, your love for science and all things strange is something we will always share. You both are my inspiration.

Last but not least, to "Food", you are my boulder, I am drawn to you like a compass to the Earth's magnetic field. Your love will never be taken for granite.

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Nina Tuscano Buenaventura

## Abstract

Plastic materials in the environment are the most conspicuous and easily recognisable litter that are of great concern all over the world, including Norway. As these plastic debris continuously break up to even smaller pieces such as microplastics, they become more easily distributed to terrestrial, marine and freshwater environments. These microplastics (< 5 mm) are of great concern due to their long degradation time and that they can accumulate in habitats and be ingested by organisms. The effects of microplastics in the different environments and its effects on organisms are still not well known. Thus, the presence or absence of microplastic pollution is of pressing need specifically where data is lacking, in rivers and terrestrial environments.

Six sediment samples were taken along an urban Norwegian river in Oslo, Akers River to quantify microplastics in freshwater sediment. The first sample was taken at the mouth of a preserved lake, Maridal and the rest of the samples were taken further downstream where the river eventually enters the city of Oslo. There are however currently no standardised techniques for sampling or quantifying microplastics from sediments. Nonetheless, based on Claessens et al (2013) elutriation device (ED) to extract microplastics from marine sediments, freshwater sediments were used for the same purpose. Elutriation is a technique where the upward water flow and gas in a column is used to separate lighter from heavier particles, causing the lighter particles to float and exit the ED. Heavier particles will thus sink and be retained. The polymers used to spike the standardised artificial and natural sediments were virgin plastic pellets of high-density polyethylene (HDPE), low-density polyethylene (LDPE), polypropylene (PP), polycarbonate (PC), polyethylene terephthalate (PET) and polystyrene (PS). Both types of sediments were elutriated by the ED and the experiments showed that the aeration intensity was ideal at low ( $20 \text{ L min}^{-1}$ ), and the run time set to 25 minutes. Water flow was at  $300 \text{ L h}^{-1}$  and pre-determined by Claessens et al (2013). HDPE, LDPE and PP had a 100 % recovery rate, PET and PC were not extracted while PS had a maximum recovery rate of 85 %. From these results, it was expected to extract light plastics such as HDPE, LDPE, PP and some PS from Akers River sediments.

Microplastics of both light and heavy plastics in Akers River sediment were found. Polymers with densities  $\geq 1.0 \text{ g cm}^{-3}$  may not however all be extracted, or not extracted at all from samples as elutriation was shown to be strongly density-dependent. Due to time limitations only Site 1 (all three subsamples) and Site 6 (one subsample) underwent FT-IR analysis. At Site 1, six plastic particles were found, and at Site 6 a total of 101 plastic particles. Of the total 107 plastics identified, polyurethane (PUR) was the most common plastic found followed by polyethylene (PE), polyvinyl-alcohol (PVA), acrylics and poly(methyl methacrylate) (PMMA). The two polymers PUR and acrylics were mostly associated with paint fragments which coincides with literature reporting that microplastics from paint is the second most common microplastic pollution found in Norway. Furthermore, many of the plastic types found in Akers River are also consistent with what other studies have found in other freshwater environments.

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# 1. Introduction

## 1.1 Plastics and Plastic Production

Plastics are ubiquitous and indispensable in the modern world. The commercial development of plastic started in the 1930s and 1940s and in 2016, 322 million tons of plastic were produced worldwide (Plastic Europe 2016). Unfortunately, not all plastic waste is handled in an appropriate manner, and it is estimated that 12.7 million metric tons of plastic litter entered the ocean in 2010 (Jambeck et al., 2015). Plastics are inexpensive, durable and versatile but due to the high accumulation of plastic debris, plastic waste has become a global issue (Sadri and Thompson, 2014).

The word plastic itself originates from the Greek word “plastikos” meaning “able to be moulded into different shapes”, and the label “plastic” is therefore given to any synthetic or semi-synthetic, organic polymers with high molecular mass (Mukherjee and Chatterjee, 2014). This wide family of materials is derived from cellulose, coal, natural gas, salt and crude oil (Plasticseurope, 2016) and hydrocarbons and other naturally occurring compounds usually make up the plastic polymers molecular backbone (Nerland et al., 2014). To provide the suitable properties for specific usage, other chemicals and additives are added, such as plasticizers to improve the structural flexibility of certain polymers (Nerland et al., 2014, Rahman and Brazel, 2004). Examples of common plastic types are polyethylene; high and low density (HDPE and LDPE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC) and polyethylene terephthalate (PET) (Plasticseurope, 2016) (Table 1).

**Table 1.** Density, demand, buoyancy and examples of applications for common plastic polymers (Duis and Coors, 2016, Ghosh et al., 2013, Hidalgo-Ruz et al., 2012, Nerland et al., 2014, Plasticseurope, 2016)

Plastic polymer	Abbr.	% (demand)	Density (g cm <sup>-3</sup> )	Buoyancy (virgin)	Some examples of application
Polypropylene	PP	19.1	0.90-0.91	+	Food packaging and wrappers, hinged caps, microwave proof containers, pipes, automotive Toys, bottles, houseware,
Polyethylene	PE	29.4	0.917-0.965	+	reusable bags, agricultural films, food packaging film
Polystyrene	PS	6.9	1.04-1.1	-	Eyeglass frames, plastic cups, egg trays (packaging), building insulation
Polymethyl methacrylate (acrylic)	PMMA		1.09-1.20	-	Touch screens, contact lenses, Plexiglas
Polyvinyl chloride	PVC	10.1	1.14-1.58	-	Window frames, floor and wall covering, pipes, cable insulation, harden hoses, etc.
Polyvinyl acetate/alcohol	PVA		1.19-1.31	-	
Polycarbonate	PC		1.20	-	Eyeglass lenses, roofing sheets, traffic lights, riot shields

Polyurethanes	PUR	7.5	1.20	-	Building insulation, pillows, mattresses, insulating foams for fridges, surface coatings, commonly used in cars
Polyethylene terephthalate	PET	7.1	1.32-1.45	-	Bottles for water, soft drinks, juices, cleaners
Other polymers		19.9		NA	Hub caps, cable coating, medical implants, surgical devices

\*Seawater density  $\approx 1.02\text{-}1.03 \text{ g cm}^{-3}$ ; freshwater density  $\approx 1 \text{ g cm}^{-3}$ ; sand and sediment  $\approx 2.65 \text{ g cm}^{-3}$  (Imhof et al., 2012, Nerland et al., 2014, Rocha-Santos and Duarte, 2015).

Plastic Europe (2016) estimated that in 2014 alone, 25.8 million tonnes of post-consumer plastics waste ended up in the official waste streams. In 2015, Europe produced 58 million tonnes of plastic (322 million produced worldwide), of which 29.7 % was recycled, 39.5 % was recovered through energy, and 30.8 % ended up in landfills. These plastics were used for packaging (39.5 %); consumer and household appliances, furniture, sport, health and safety (22.4 %), for buildings and construction (19.7 %); and the remaining used for/in automotive, electrical and electronics, and agriculture (18 %) (Plasticseurope, 2016). The specific traits of plastics have not only led to multipurpose applications but also to a high number of single-use disposable items (Sadri and Thompson, 2014). According to Barnes et. al. (2009) plastic is the largest component of litter on land and marine environments, and may be as high as 80 % (Barnes et al., 2009). The accumulation of plastics includes all sizes of the polymers (Dris et al., 2015a). Macroplastic debris has been a focal point for environmental reasons due to its conspicuous manner, whilst smaller plastic particles, fibres and granules have only recently received attention (Cole et al., 2011). Nonetheless, it is now apparent that these small plastic particles can cause severe and lasting environmental problems, as will be discussed further below. The size classes of plastic fragments in the literature are not uniformly set, and there is an urgent need for a standardised definition (Dekiff et al., 2014, Hidalgo-Ruz et al., 2012, Nerland et al., 2014). In this report, the following size classes are adopted: macroplastics ( $> 25000 \mu\text{m}$ ) mesoplastics ( $5000 \mu\text{m} - 25000 \mu\text{m}$ ) and microplastics ( $< 5000 \mu\text{m}$ ) (Bråte et al., 2016, Kershaw, 2015).

Newer types of plastics that biodegrade have been developed for environmental purposes (Plasticseurope, 2016). Biodegradable plastics may seem to be a good replacement for non-degradable plastics. They consist of starch, vegetable oils or chemicals specialised to speed up degradation time. However, some of the biodegradable plastics also consist of synthetic polymers. This type of mixed plastic only biodegrades the non-synthetic materials if disposed of appropriately, such as industrial composting plants under hot, humid and well-aerated conditions. In the environment, however, such as in the cold marine environment, the degradation will be slow due to the lack of microbial degradation. Once it has broken down, there is still the issue of the remaining synthetic microplastic polymers (Cole et al., 2011). Since plastics in general do not fully degrade, there are growing concerns of smaller plastic particles termed microplastics accumulating in the environment and in organisms (Wagner et al., 2014).

Plastic waste in the environment is causing problems for the wildlife. More than 260 species have been documented to ingest or become entangled in plastics, this include fish, seabirds, turtles and marine mammals (Teuten et al., 2007). Plastics can also contain additives and plasticizers, hydrophobic persistent organic pollutants (POPs) and metals that are absorbed in plastics and can be harmful to the environment and organisms (Koelmans et al., 2013). Contaminants can transfer to organisms by inhalation and dermal sorption, and through ingestion. Once smaller particles are inside the organisms, there is the potential for chemicals to translocate to the host itself (Hidalgo-Ruz et al., 2012, Mathalon and Hill, 2014). This was made evident by Avio et al. (2015), showing that microplastics absorbed polycyclic aromatic hydrocarbons (PAHs) and that mussels exposed to microplastics containing PAHs had accumulated pyrene in their tissues (Avio et al., 2015). Von Moos et al. (2012) documented that immunological responses could have also been caused by the fragments sharp shapes that may have worsen the effects (Von Moos et al., 2012). Also, Pierce et al. (2004) found a lethargic and weak seabird that had ingested a plastic cap, most likely causing starvation (Pierce et al., 2004). Furthermore, Wright et al. (2013) observed reduced energy reserves in lugworms due to a combination of reduced feeding activity, prolonged gut residence time of ingested material and inflammation when microplastics were ingested. The authors also suggest that microplastics can have an impact on growth, reproduction and survival (Wright et al., 2013). The ingested plastics by organism may not only have deleterious effects on that specific organism but can also affect higher trophic levels as well. A study conducted by Setälä et al. (2014) subjected shrimps, copepods, cladocerans, rotifers, polychaete larvae and ciliates to 10 µm polystyrene (PS) where all taxa ingested the plastic particles. They also found ingested zooplankton containing PS and PS in shrimp intestines showing that microplastics may potentially be transferred from prey to predator and possible accumulation in food webs (Setälä et al., 2014).

## 1.2 Microplastics

### 1.2.2 Defining Microplastics

Microplastic particles (< 5mm) can be classified into two main categories based on their origin; primary and secondary microplastics (Kershaw, 2015). The term “Primary microplastics” is usually used to describe pre-production pellets or microplastics in personal care products such as microbeads found in exfoliators and toothpaste. These primary particles can also be used in air blasting technologies for clearing boat surfaces and machinery from rust, and even in medicine where the microbeads serve as vectors for drug delivery (Nerland et al., 2014, Browne et al., 2007). Secondary microplastic particles on the other hand result from degradation of macro- and mesoplastics caused by UV radiation, mechanical abrasion, biological degradation and further breakup (Dris et al., 2015a). Different plastic types may degrade differently despite being in the same environment, and degradation may differ between marine and freshwater environments (Biesinger et al., 2011). One type of secondary microplastics of growing concern are synthetic fibres which stem from clothing when washed. The material is broken down to fibres that are released into the environment through sewage sludge reaching land, freshwater and marine environments (Browne et al., 2011, Browne, 2015, Dris et al., 2016).



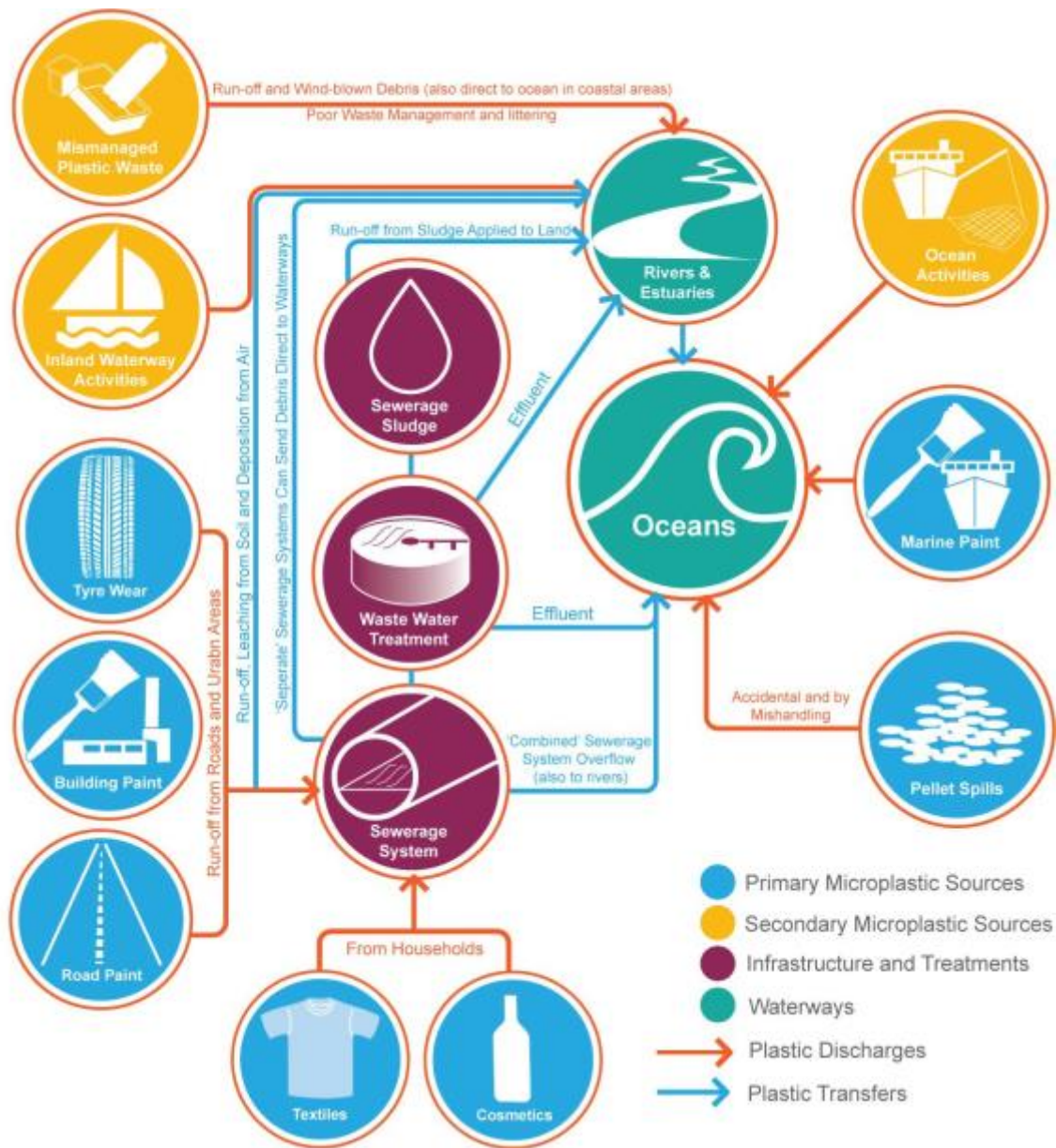
As Hidalgo-Ruz et al. (2012) are highlighting, microplastics are derived from different starting materials subjected to different degradation processes, and that their physical properties are highly variable. The form may be oblate, prolate, cylindrical, fibrous, or otherwise irregular, but most often spherical to prolate with rounded ends. The fragmentation process defines the shape of the plastic fragment including the residence time in a specific environment. Sharp edges may be an indicator that the fragment was recently broken off a larger item or newly introduced to the environment. Smooth edges usually characterise older pieces that have undergone abrasions from other items (Hidalgo-Ruz et al., 2012). These characteristics, in addition to the different densities of the different polymers, are key factors contributing to their distribution in the environment throughout the deep-sea sediments, water column, floating on the surface, in rivers and even deposited on shores worldwide (Barnes et al., 2009, Lagarde et al., 2016, Morét-Ferguson et al., 2010). For example, with regard to form/shape, it has been shown that fibres, many of which are synthetic, can travel great distances through air and water (Browne et al., 2011, Dris et al., 2016). Dris et al. (2017) concluded that larger fibres settle more rapidly and gather on soil surfaces, whilst small fibres may easily become airborne, and therefore be inhaled by organisms outside as well as indoors (Dris et al., 2017). The further breakdown of the plastic particle is probable to be a continuous process, likely leading to nanoparticles (Dris et al., 2015a).

### 1.2.3 Microplastic in Aquatic Environments

The majority of microplastic research has been conducted in the marine environment, and they have since been recorded along the coasts of every continent. This includes remote locations such as the sub Antarctic islands (Eriksson and Burton, 2003, Eriksson et al., 2013), the Arctic (Lusher et al., 2015) and deep-sea habitats (Van Cauwenberghe et al., 2013). In contrast to studies conducted on marine microplastic, only few studies have been done on freshwater microplastics (Dris et al., 2015b, Eerkes-Medrano et al., 2015) and microplastics in marine sediment (Thompson et al., 2004).

As much as 70-80% of the marine plastic pollution is thought to come from land-based sources (Bowmer T, 2010) such as from run-off from storm waters and it is also seen that rivers are a big source of microplastic particles to the marine environment (Sundt et al., 2016). Since freshwater environments are studied to a less extent and due to their role as contributors to the overall plastic load to the marine environment, it is important to collect more data on microplastics from freshwater environments.

In general, there are several pathways for microplastics to enter the terrestrial and aquatic environment, and understanding these pathways is of great importance (Sherrington et al., 2016). Some pathways can be fibres transported by air/wind (Dris et al., 2016), via effluent directly from wastewater or by wastewater treatment (Magnusson et al., 2016), road dust (Sundt et al., 2014) and direct runoff after rain events (Dris et al., 2015b) (Fig. 1).



**Figure 1.** Possible microplastic pathways to aquatic environments (Sherrington et al., 2016).

Since there is a clear link between other types of pollutants from municipal discharges, sewage, urban runoff and storm water from rivers to oceans, it is important to establish how significant rivers are a major source of microplastics to the marine environment (Eerkes-Medrano et al., 2015). Lechner et al. (2014) documented a high amount of microplastics with 79 % being of raw industrial materials in the Danube River (Lechner et al., 2014) and Moore et al. (2011) documented that most of the plastics found in the Los Angeles River were microplastics (Moore et al., 2011).

Similar to vegetation and wood debris, plastics can undergo sedimentation and become trapped in organic debris brought in by waves and currents along lake shorelines or riverbanks (Zbyszewski et al., 2014). Plastics such as PE may accumulate directly among the channel bedload (Williams and Simmons, 1996), where aggressive mechanical erosion processes may further break up particles into yet more microplastics (Andrady, 2011). As



#### 1.2.4 Microplastics in Rivers and in Norwegian Environments

Some emerging studies showed high concentrations of microplastic particles in rivers and lakes, and have given an insight into the role of urbanisation and plastic litter (Dris et al., 2015b, Sherrington et al., 2016, Wagner et al., 2014). An example of this is in the Great Lakes where Eriksen et al. (2013) documented an average abundance of microplastic litter of 43,000 items km<sup>-2</sup>, with a hotspot near metropolitan areas (Eriksen et al., 2013). Research on plastic pollution has shown a positive relationship between microplastic abundance and human population density, and the increase in both human population and plastic production may probably lead to a higher occurrence of microplastics in the environment ((Rocha-Santos and Duarte, 2015, Plasticseurope, 2016).

Little is known on microplastic pollution in Norwegian freshwater environments. A literature search on Google Scholar (excluding thesis's) gave three published articles, all on marine environments (Bråte et al., 2016, Herzke et al., 2016, Lusher et al., 2015) ; and three technical reports, one on microplastics and wastewater treatment plants (Magnusson, 2014), one on microplastic contribution to marine environments in Norway (Sundt et al., 2014) and one on overall microplastic contribution in Norway including rivers as a source to microplastics in marine environments (Sundt et al., 2016). A table from Sundt et al. (2016), Table 2, shows the pressing need for further research in Norway as the contribution of microplastic sources are only at the stage of "best guess" (Sundt et al., 2016). Even less is known about the impact of microplastics, especially in freshwater environments.

**Table 2.** A comparison of estimated gross microplastic pollution (at source) in grams per capita, comparing Norway, Germany, Denmark and EU estimates. The estimates range from 171 g/capita to nearly 4 kg/capita with Norway only providing "best guess" numbers of contribution (Sundt et al., 2016).

Population	5165k	80688k		5699k		508000k	
Country Estimates	Norway 2014	Germany 2015		Denmark 2015		EU	
	Buest guess	Low	High	Low	High	Low	High
Personal care & cosmetics	8		6	2	5	8	
Pellets (raw material)	87	260	2603	1	10	112	1122
Paints	238			28	228	55	
Tyres	871	744	1376	737	1158	63	
Textiles	136			35	175	53	
Road markings	62			19	121		
Others	210		2	143	742		
Totals	1612		3992	965	2439	171	1302

The aims of this thesis are 1) to build and test an elutriation device (ED) for the extraction of microplastic particles from river sediments and 2) to quantify microplastic particles in sediments of an urban stream, Akers River in Norway. The surrounding area and along Akers River in Oslo has become urbanised due to anthropogenic activities and settlements, exposing the river habitat to enhanced risk of plastic litter and microplastic sources. The

water flowing from Akers Rivers is in high/good condition at the top (Frysja), and in good/moderate condition closer to its outlet to the sea (Nybrua) (Statistikkbanken, Oslo kommune). Akers river flows through the city of Oslo, along settlements, old/closed industry buildings, offices and high trafficked roads and parks before culminating at Grønland (Ranneklev et al., 2009). These anthropogenic activities along the river may contribute to polluting the relatively unpolluted upstream water, increasing the pollution in the water as it culminates downstream. It is therefore expected to see a gradient in microplastic pollution. To investigate the microplastic content, the elutriation device (ED) based on Claessens et al. (2013) design was utilised and tested on artificial sediment for standardisation, and in situ sediment before commencing the case study of quantifying microplastics in Akers River. During the trial experiments on artificial and on in situ sediments the optimal aeration intensity and running time (in minutes) of the ED was determined. The ED was also chosen because it could easily be replicated and affordable for other institutes.

### 1.3 Issues Concerning Microplastic Sampling

Numbers of microplastic studies in the environment have been increasing rapidly in recent years, and so has our understanding of microplastics fate in the environment. Many challenges and key questions nonetheless remains. Significantly, the detection, collection and identification of microplastics can be challenging for several reasons:

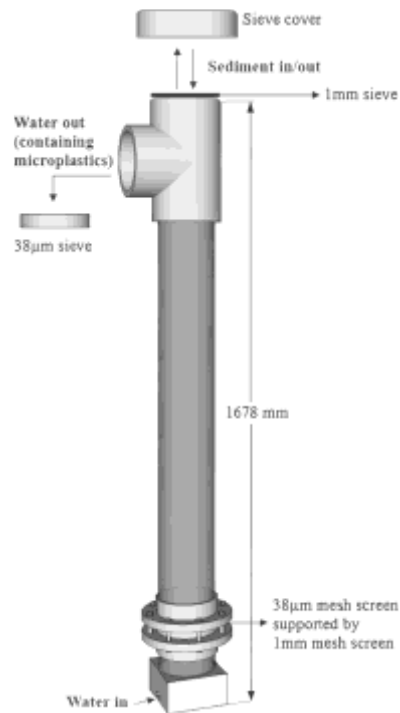
- The ability to capture plastic particles from a sample of water or sediment
- Separating plastic fragments from other particles in a sample
- Identifying correct particles as plastics
- Identifying the type of plastic polymer

In addition to the detection of microplastics, other problems include the inconsistency of techniques used for sampling, separating, extracting and identifying (Dris et al., 2015b, Hidalgo-Ruz et al., 2012, Ivleva et al., 2016, Wagner et al., 2014). Due to the variety of techniques and different units used, it can be a challenge to compare the results from one study to another (Hidalgo-Ruz et al., 2012). Another issue to consider is the diversity of the studied environments. Terrestrial, estuarine, marine and freshwater environments, each have intricate and unique characteristics that affect microplastic distribution differently (Eerkes-Medrano et al., 2015). Physical factors that can influence particle transport and the sedimentation of suspended particles include water flow velocity, water depth, substrate type, bottom topography, and the seasonal variability of flow (Simpson et al., 2005). Some events such as tidal cycles (only estuaries), storms, floods or anthropogenic activities such as dam releases may be infrequent, but can have profound effects on the distribution of microplastics. The combination of physical forces, temporal aspects and particle density may determine if it is in the benthic or pelagic transport route. Particles of high densities will most likely be in the benthic transport route as bedload and be deposited in the lower reaches of the river. Lower density and finer particles may occupy the pelagic transport route, suspended in the water and end up in marine environments. When particles reach estuarine conditions, turbulence and salinity can alter their density, size and charge, resulting in increased flocculation and particle deposition. Plastic particles may show similar

behaviour in aquatic environments leading to increased deposition where fresh and saline waters meet (Eerkes-Medrano et al., 2015).

Several methods have been used to separate microplastics from sediment samples. The most commonly used method is separation by density, or flotation. This is a process where lighter plastic particles are separated from the heavier sediment grains (sand/sediments density  $2.65 \text{ g cm}^{-3}$ ) by mixing a sediment sample with a saturated salt solution such as; sodium chloride (NaCl) (Thompson et al., 2004); sodium polystyrene sulfonate (SPT) (Corcoran et al., 2009); zinc chloride ( $\text{ZnCl}_2$ ) (Imhof et al., 2012); and sodium iodide (NaI) (Claessens et al., 2013, Nuelle et al., 2014) and shaking the mixture for a certain amount of time. It is expected that the heavier particles will settle to the bottom, while the lighter, lower density particles remain suspended or float to the surface of the solution. The lighter particles are then removed for further analysis (Hidalgo-Ruz et al., 2012). The use of NaCl solution was introduced by Thompson et al. (2004) for density separation, but with a density of  $1.2 \text{ g cm}^{-3}$ , it could lead to an underestimate if used, since plastic polymers with densities higher than  $1.2 \text{ g cm}^{-3}$  may not float and are thus excluded from the results (Claessens et al., 2013, Thompson et al., 2004). Other solutions applied have been SPT with a density of  $1.4 \text{ g cm}^{-3}$  (Corcoran et al., 2009),  $\text{ZnCl}_2$  with density between  $1.6\text{-}1.7 \text{ g cm}^{-3}$  (Imhof et al., 2012), and NaI with a density of  $1.8 \text{ g cm}^{-3}$  (Claessens et al., 2013, Nuelle et al., 2014). A review by Nuelle et al. (2014) points out issues concerning the extraction of microplastics from sediments is the sediment mass or volume applied for extraction. When NaCl was used, 1 kg of sediment sample was used. With the heavier and more costly salt solutions however, only 500 ml and 68 ml of sample volume was used with SPT, and a sample mass between 150 g and 190 g was used for  $\text{ZnCl}_2$ . The high-density salts are costly, and may be the reason why the volumes and masses applied to the solutions were low. Since the distribution of microplastics in sediments may be heterogeneous, however, and it is thus advantageous to have a large sample quantity. By increasing the sample volume, the chance of detecting microplastics is increased (Nuelle et al., 2014).

To aid this separation method, Claessens et al. (2013) developed an elutriation device (ED) (Fig. 3) allowing high recovery of even dense microplastics by firstly undergoing elutriation and then adding the recovered particles to a salt solution. The principle of elutriation is to separate lighter from heavier particles by an upward stream of gas or liquid (Southwood and Henderson, 2000). Claessens et al (2013) firstly separated lighter particles from heavier by forcing tap water up a column and out, particles that floated were retained by a sieve. To minimise dead zones in the column, aeration was added at the bottom (fig Claessens et al 2013). The step of flotation would thus require less volume of the chosen salt solution to extract microplastics.



**Figure 3.** A schematic representation of Claessens et al. (2013) elutriation device used for separating microplastics from marine sediments (Claessens et al., 2013).

## 2. Materials and Methods

### 2.1 Sample Area

Lake Maridal is the main source of drinking water (85 - 90 %) for the inhabitants of Oslo, Norway. It's located in the forest to the north of Oslo, Nordmarka with restricted access for anthropogenic activities. There is however considerable wildlife in the catchment that may potentially contaminate the lake. Lake Maridal has an area of approximately 4 km<sup>2</sup>, lies almost 150 meters above sea level and is the largest lake in Oslo (Robertson et al., 2009, Paruch et al., 2016) with a salinity between 23-52 mg L (Økland and Økland, 1998).

Akers River flows out of Lake Maridal, drains a 250 km<sup>2</sup> catchment in Nordmarka and is the largest river system in the municipality of Oslo. The river is regulated and the water flow at the outlet should be at least 1.5 m<sup>3</sup>/s between April 1<sup>st</sup> to November 31<sup>st</sup>, and the rest of the year, at least 1.0 m<sup>3</sup>/s. The 9 km long river runs through downtown Oslo before culminating out of the Oslo fjord in Bjørvika. Akers River is also broken up by several waterfalls, where there are in most cases dams (Bækken et al., 2011a).

In the 19<sup>th</sup> century Oslo's drinking water supply came from Akers River. However, as the industry grew from 1850's the river became increasingly polluted. The municipalities main objectives to counteract pollution from factories and sewage became a priority and together with Akerselva Miljøpark the water quality has improved (Bækken et al., 2011b) There is nevertheless a need for further improvements as the water quality decreased further downstream the river ((Statistikkbanken, Oslo kommune) (Van De Bund and Solimini, 2007)).

## 2.2. Extracting Microplastic Particles from Sediment by Elutriation

### 2.2.1 Construction of the Elutriation Device

Based on Claessens et al. 2013 design, GPA Flowsystem AS in Oslo constructed the PVC parts for the elutriation device (ED) that was assembled at NIVA (Oslo) before the experiments were conducted (Fig. 4).



**Figure 4a).** The elutriation device used for extracting microplastics and **b)** a sediment sample container with a metal spatula and a 1 cm steel grate on top of a metal bowl. Photo a) personal; and photo b) by Thomas Botolfsen.

The total length of the ED was 167 cm but the actual poly(vinyl chloride) (PVC) column from the inner meshes to where the water pours out, measured 105 cm with an internal diameter of 15 cm. The top of the device was 27 cm long and the base (where water pours in from) was 35 cm long.

Between the column and the base, two flanges were used to avoid water escaping through the device. To prevent any particle contamination from tap-water to the samples, and to support the sediment and the aeration system, a bottom 35  $\mu\text{m}$  and a top 1000  $\mu\text{m}$  metal support sieve mesh was also placed between the two flanges. The 1000  $\mu\text{m}$  sieve mesh had to be sealed with several layers of about 1.5  $\text{cm}^2$  silicone (CASCO Sanitary Wet Room Silicone) around the edge to prevent leakage. At the base, a hose attached to a tap was fitted to create an upward flow of tap-water forcing the water up the column and out of the second opening on top.

To prevent damage to the aeration system the sediment samples were poured in the ED after the water level in the ED was above 60 %. To maximise separation of particles and to avoid dead zones (zones free of circulation from air and water), three aquarium air stones (50 X 20 X 20 mm) were lowered down the column in a Y-form to create an aeration system.



To hold the Y-form, a small galvanized steel ring (5 cm diameter) was placed between the air hoses and a square metal construction surrounded the air stones for stabilisation and easy handling. The air stones were turned on before each sediment samples went into the elutriation device. Air was pumped into the water by an air pump (HiBlow HP-80) with a maximum air flow at 80 L min<sup>-1</sup>. To accurately pump out a defined volume of air into the water, a one-way valve was attached to the air pump to increase or decrease the amount of air supply. It was determined to have two settings, High (half of full capacity, 40 L min<sup>-1</sup>) and Low (a quarter of full capacity, 20 L min<sup>-1</sup>).

When operating the ED the lighter materials arose or floated to the top and was retained by a 500 µm metal sieve resting on top of the funnel. Overflowing water was thus directed to a drain by the funnel that was connected to a hose.

### 2.2.2 Artificial Sediment Preparation

A standardised artificial sediment (OECD 2004) was used for the recovery rate experiments. This sediment is used in “good laboratory practice (GLP)” toxicity experiments on sediment dwelling biota. Since the sediment is standardised, it is a favourable type of sediment to use due to its known particle size and content, and ensures that the experiments we performed can easily be replicated. Artificial sediment was made following the OECD/OCDE 218, ANNEX 3. Preparation of Artificial Sediment.

In short, the artificial sediment was prepared by mixing 75 % sand (SIGMA-ALDRICH; 50-70 mesh particle size), 20 % kaolinite (EMD Millipore Corporation, powder) and 5 % *Sphagnum* peat (d/w). The wet- to dry-weight was determined by measuring the average weight of three dried peat samples. Once the peat was dried, it was finely ground to ≤ 1mm. Sand, kaolinite and peat were thoroughly mixed together adding 50 % of deionized water until a homogenous mixture was achieved. The artificial sediment had a pH of 4.16 but adding a further 50 ml deionized water increased the pH to 5.5. The homogeneous artificial sediment was divided into 50 ml (83.4 g ± 0.5 g) bags and stored in a freezer and thawed prior to use.

## 2.3 Test of Microplastic Extraction Efficiency using the Elutriation Device

### 2.3.1 In Natural Sediment

Natural sediment was firstly used to determine the run time of an experiment as the technical information from Claessens et al. (2013) did not specify how they determined the length of their experiment. The trial experiment with natural/in situ sediment had a volume of 500 ml from the Akers River. Each sample was taken using a sediment corer and to ensure that a vacuum would occur to lift the corer containing the sample without any loss the sediment sample was taken under water. The inner diameter of the corer was 5.5 cm, and to extract approximately 500 ml of sediment, the corer had to be inserted 21 cm into the sediment. The volume of the sediment taken with the core was calculated from following equation:

$$498 \text{ ml} = \pi 2.75^2 21$$

The sediment inside the corer was then pushed out and into a glass jar with tin foil between the lid and the container to prevent contamination from the plastic coating inside the lid. Each sub-sample was transferred into a metal bowl with a 1 cm mesh screen to prevent particles >1 cm to enter the ED. The glass jar was also rinsed with < 150 ml tap water and poured over the screen mesh to obtain as many particles as possible. Prior to pouring the samples into the ED, the collected sediment was spiked with six types of virgin microplastic pellets, three of each type; low-density polyethylene (LDPE); high-density polyethylene (HDPE); polyethylene terephthalate (PET); polycarbonate (PC); polystyrene (PS); and polypropylene (PP) (total n = 18). The pellets were approximately 2-4 mm in size and obtained from Örebro University where they are used to performed microplastic exposure studies in a standardised way.

### 2.3.2 In Artificial Sediment

The technical specifications in Claessens et al. (2013) was not detailed concerning aeration intensities and it was therefore decided to further investigate this. Standardisation of experiments was also needed, it was thus important to examine if the previous trial experiments with natural sediments would give similar results. Two aeration intensities were therefore tested; low ( $20 \text{ L min}^{-1}$ ) and high ( $40 \text{ L min}^{-1}$ ) for their significance. Each aeration intensity level was performed three times giving a total of six experiments.

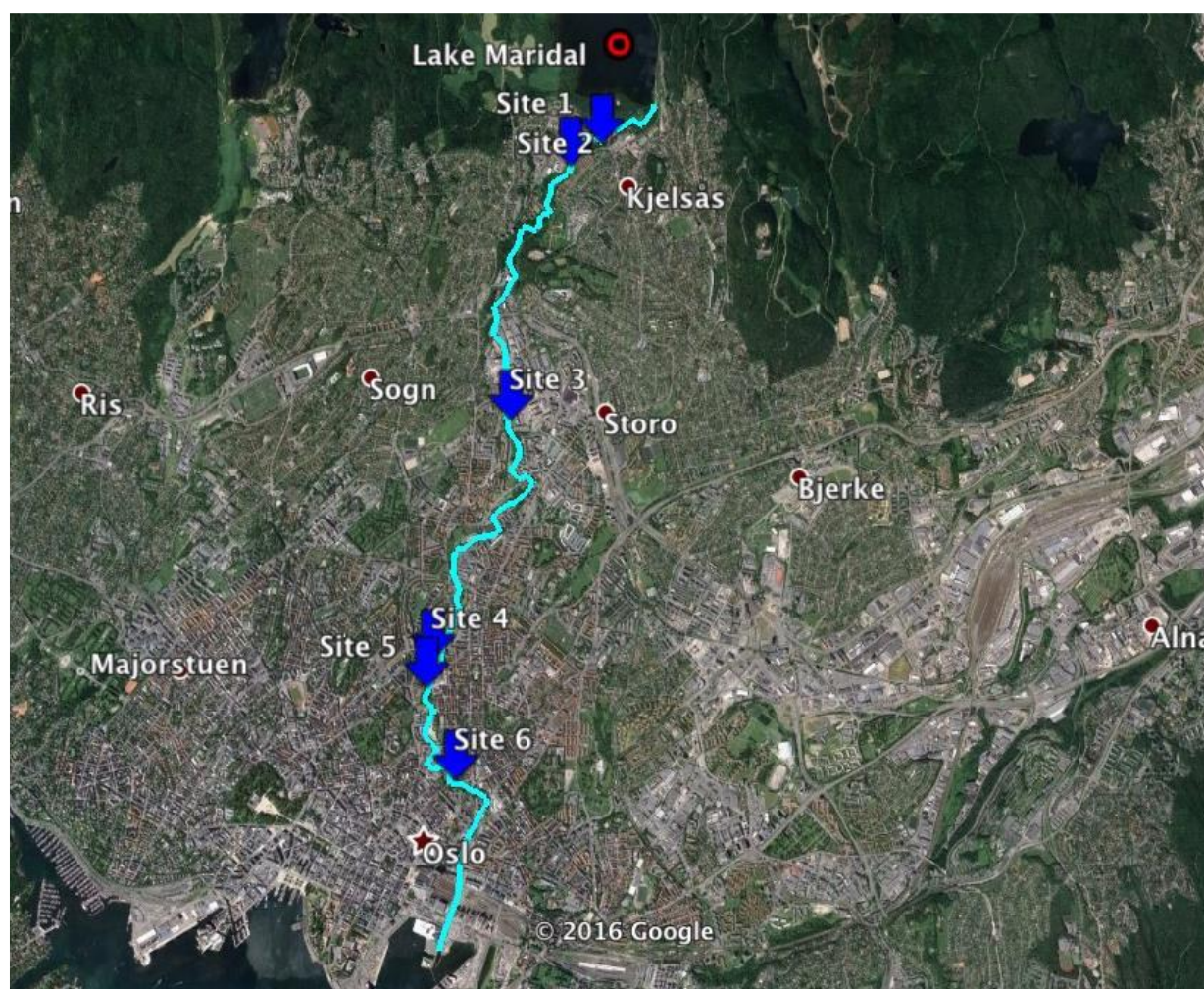
The following experiments were therefore conducted; 50 ml of artificial sediment was spiked with 10 virgin microplastic pellets of one type HDPE; LDPE; PP; PET; PC and PS. During these trial experiments with artificial sediments, each plastic type was individually tested but with the same procedure as the previous experimental setup with low and high aeration intensities. Each aeration intensity was duplicated and this lead to a total of 24 experiments. The run time for these experiments however was changed to 25 minutes.

## 2.4 Microplastics from River Sediment – A Case Study

River shore sediment samples using the same corer as described above, were collected between July 11<sup>th</sup>–14<sup>th</sup> and were taken downstream of Lake Maridal, starting at the closest site to Lake Maridal and ending at a site favourable for sampling sediments as far downstream as possible (Table 3 and Fig. 5). Sites were numbered, named, grain size of the sediments collected were roughly estimated and GPS coordinates were noted

**Table 3.** Site numbers, name of location, sediments grain size and GPS coordinates of areas where samples were taken.

Site no.	Name	Sediment Type	GPS Coordinates
Site 1	Brekdammen	-fine sand, sand, gravel, org. matter	N 59°57.977' E 10°46.798'
Site 2	Kjellsåsveien bro	-fine sand, sand, gravel, org. matter	N 59°57.975' E 10°46.511'
Site 3	Bro i Kristoffer Aamotsvei (Ny dalen)	-sand, gravel, org. matter	N 59°56.718' E 10°45.945'
Site 4	Aamot bru	-fine sand, sand, gravel, org. matter	N 59°55.642' E 10°45.227'
Site 5	Oslo National Academy of the Arts (KHiO)	-sand, gravel, some org. matter	N 59°55.516' E 10°45.168'
Site 6	Eventyrbrua	-silt, fine sand, sand, gravel, org. matter	N 59°55.100' E 10°45.422'



**Figure 5.** Map of Akers River highlighted in light blue and sample sites (GPS coordinates) along the river. Map modified from Google Earth.

Six river sediments samples were collected from Akers River where three replicate subsamples were taken at each site, such that total of 18 samples were taken.

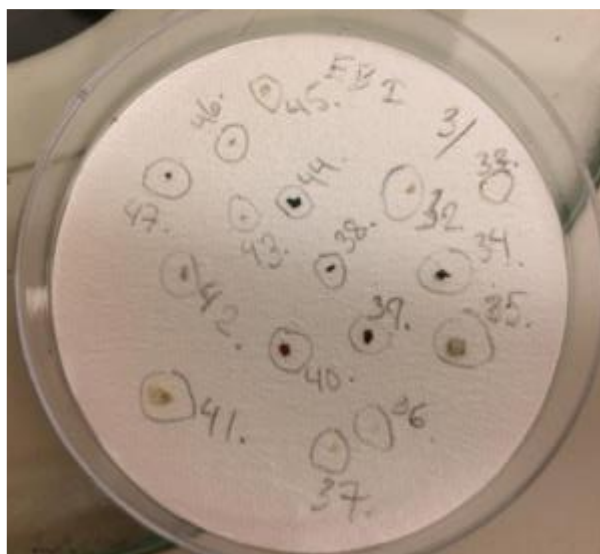
Three virgin LDPE pellets were added to each subsample as a positive control to ensure that the elutriation device was working properly. Based on the findings from the previous experiments the river sediment extraction was done with the following settings:

- 500 ml of Akers River sediment
- Three LDPE virgin pellets added to each sample as positive controls
- Low aeration intensity ( $20 \text{ L min}^{-1}$ )
- Water flow ( $300 \text{ L h}^{-1}$ )
- Run time 25 minutes

All floating matter that was collected by the  $500 \mu\text{m}$  mesh sieve was transferred to a 5.5 mm diameter petri dish (and plastic bags when the petri dishes were not large enough), sealed, named and stored in a freezer for further analysis.

## 2.5 Microscopy

Due to limited time, only 10 subsamples could be analysed under a stereomicroscope (Nikon SMZ 745T): Site 1, three subsamples; Site 2, three subsamples; Site 3, one subsample; Site 4, one subsample; Site 5, one subsample; and Site 6, one subsample. Visual sorting of the obtained matter and particles were done on a glass petri dish, and the particles suspected of being plastics were then transferred to another petri dish containing a Whatman<sup>®</sup> Glass Microfiber Filter (GF-F). The filters were named and numbered according to site and subsample (Fig. 6). Fragments or items that were chosen to undergo FT-IT analysis were photographed through the stereomicroscope using Lumenera Infinity Analyze Software v6.5.2 and described after their appearance (fibre, granule, film, softness, hardness, surface roughness/smoothness), size (length/width/breadth in mm), colouration (including gloss/shiny/matt) and numbered. Size was determined by measuring the longest stretch of the particle.



**Figure 6.** Petri dish containing a Whatman<sup>®</sup> GF-F with particles suspected of being plastic polymers.

## 2.6 FT-IR Analysis

Following visual identification, chemical characterisation of the particles thought to be plastic was performed by using a Fourier transform infrared spectroscopy (FT-IR) Attenuated Total Reflection (FTIR-ATR) (ThermoScientific Nicolet iS50 FT-IR). The infrared absorption spectrum was recorded by subjecting the particle to a beam of infrared light ( $4000-400\text{ cm}^{-1}$ ) at surface contact with a diamond crystal. The FT-IR analysis underwent 32 scans with a resolution of 8. With several spectral libraries, an automatic comparison was performed to obtain the chemical characterisation of the particles.

## 2.7 Data Analysis

All data handling, Chi Squared analysis, line charts and bar charts were performed in Microsoft Office Excel 2016. Data analysis of a Two-Way Analysis of Variance (ANOVA) and boxplot was achieved by the open source statistical program R Console, version R 3.3.2 GUI 1.68.

## 2.8 Minimising Contamination of Microplastics to Samples

There is a well-known problem within the field of microplastic research, that it is easy to contaminate samples with synthetic fibres from our surroundings. Therefore, several contamination precautions were performed. Cotton clothing was chosen during the collection of sediment samples, and samples were kept in a glass jar with tin foil between the lid and the samples. Lab coats comprising of cotton were also used during elutriation and visual inspection and a lint roller was used on the lab coat to remove excess fibres and particles before handling the samples. Tools used to handle sediments were of steel, such as a steel spatula and a metal bowl. The metal bowl also functioned as a lid for the ED and was placed on top to minimise atmospheric contamination to the samples whilst the ED was running. There was no lid to cover the second opening of the ED where the floating matter exited. There was however high traffic in the room where the ED was set up and where the samples underwent elutriation. Before and after each experiment, the ED and all other equipment that could be in contact with the samples were thoroughly washed to minimise plastic contamination. During visual sorting the laboratory room was closed to other personnel to minimise atmospheric contamination. The workbench, tweezers and needles were wiped and washed with ethanol and deionised water and were inspected for particles prior to use. Before and whilst undergoing visual inspection with the stereomicroscope, a control petri dish was placed next to the samples and checked for fibre contamination before, during and after handling.

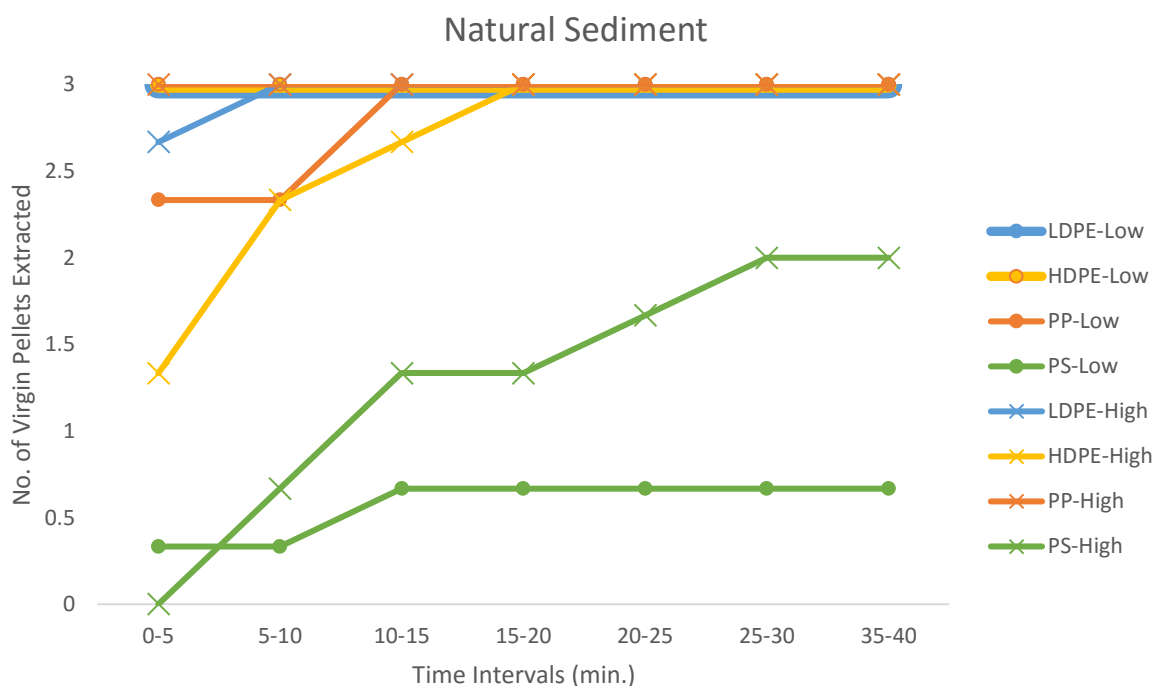
Three other personnel entered the laboratory at the time when Site 6, subsample 1 was open for visual inspection. Although the lids to the petri dishes were rapidly placed on top of both the samples and the control, fibres from the atmosphere still contaminated the control GF-F. It was therefore determined that all fibres from all sites would be excluded from the results.

### 3 Results

#### 3.1 Experiments of Microplastic Extraction with Natural Sediment

At low aeration intensity, all pellets for LDPE and HDPE were extracted within the first five minutes. All PP pellets were recovered within 10-15 minutes, while none of PET and PC were recovered (Fig. 7). PS had an average of 0.67 particles between 10-15 minutes.

High aeration gave a quick extraction of all pellets consisting of PP within five minutes, and LDPE between 10-15 minutes. All HDPE were recovered between 15-20 minutes, whilst PS had an average of 2 pellets at 25-30 minutes. PET and PC were not recovered and are thus excluded from the graph (Fig. 7).



**Figure 7.** Cumulative pellet extractions with natural sediments from Aker River spiked with virgin plastic pellets ( $n = 3$ ) and water flow at  $300 \text{ L h}^{-1}$ . LDPE-Low, HDPE-Low and PP-High plastic types were all extracted within the five minutes and are overlapping. PET and PC not included in this graph. Circle markers are low ( $20 \text{ L min}^{-1}$ ) and X markers are high ( $40 \text{ L min}^{-1}$ ) with one type of plastic polymer given one colour.

From the data obtained from experimenting with low and high aeration, it is shown that LDPE and HDPE are extracted quicker at aeration intensity low than at high. High aeration lead to an increase of water flow. When the water and other floating materials exited, some bounced off the outer filter or did not land on the filter but outside of its diameter. No such observation was seen during the low setting. The extraction time plateaued at time interval 25-30 minutes at high intensity. It was thus decided that each experiment should have a time frame of 25 minutes.

### 3.2 Experiments of Microplastic Extraction with Artificial Sediment

A Chi Squared test was performed to investigate significant differences between the two replicate experiments (Table 4). The results show that there is no significant difference between the two low replicates, P-value > 0.05.

**Table 4.** A Chi Squared analysis was performed to distinguish a significant difference between the two replicates: Low aeration intensity.

Chi square Analysis of Low1 and Low2 Aeration

Plastic type	Low1	Low2	Row Totals
LDPE	10	10	20
HDPE	10	10	20
PET	0	0	0
PC	0	0	0
PP	10	10	20
PS	10	7	17
<b>Colum Tot.</b>	40	37	77
Degrees of Freedom	5		
Chi Squared	0.431		
P-value at 0.05	0.994		

A Chi Squared analysis was also performed to investigate significant differences between the two high experimental parallels. With a P-value > 0.05 there is no significance between the two high experiments (Table 5).

**Table 5.** A Chi Squared analysis was performed to distinguish a significant difference between the two replicates: High aeration intensity.

Chi Square Analysis of High1 and High2 Aeration

Plastic type	High1	High2	Row Totals
LDPE	10	10	20
HDPE	10	10	20
PET	0	0	0
PC	1	0	1
PP	10	10	20
PS	7	8	15
<b>Colum Tot.</b>	38	38	76
Degrees of Freedom	5		
Chi Squared	1.0667		
P-value at 0.05	0.957		

Since no significant differences occurred between the two parallels of low and high, respectively, the data was then pooled to perform another Chi Square analysis to investigate significant differences between the two intensities low and high (Table 6). The results show

that there was no significant difference in microplastic retrieval between low and high aeration intensities ( $P > 0.05$ ).

**Table 6.** Chi Squared analysis of Low and High aeration intensities.  
Chi Square Analysis of Combined Low and Combined High Aeration

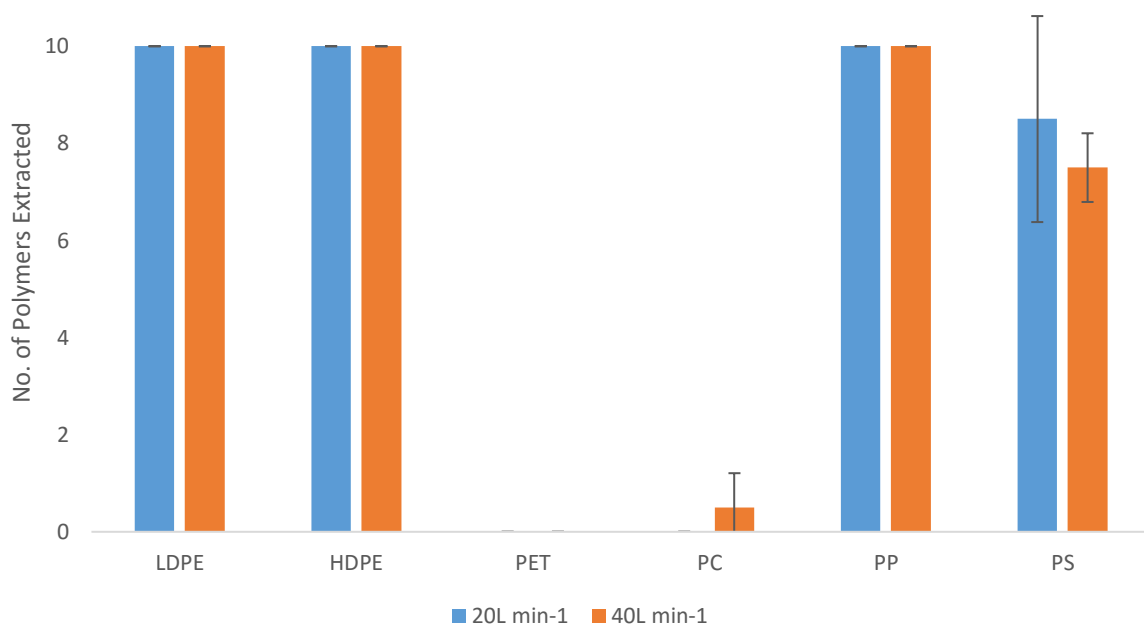
Plastic type	Low Both	High Both	Row Totals
LDPE	20	20	40
HDPE	20	20	40
PET	0	0	0
PC	0	1	1
PP	20	20	40
PS	17	15	32
<b>Colum Tot.</b>	77	76	153

Degrees of Freedom	5
Chi Squared	1.1185
P-value at 0.05	0.952

Cumulative pellet extraction from artificial sediment yielded all of LDPE, HDPE and PP pellets for both low and high aeration intensities. All PET were retained in the ED, thus none were extracted. At high aeration intensity, one PC pellet was extracted amongst all experiments and was therefore perceived as an outlier. The average pellet extraction for PS at low aeration was 8.5 (SD = 2.12), and at high aeration intensity 7.5 (SD = 0.70) pellets (Fig. 8).

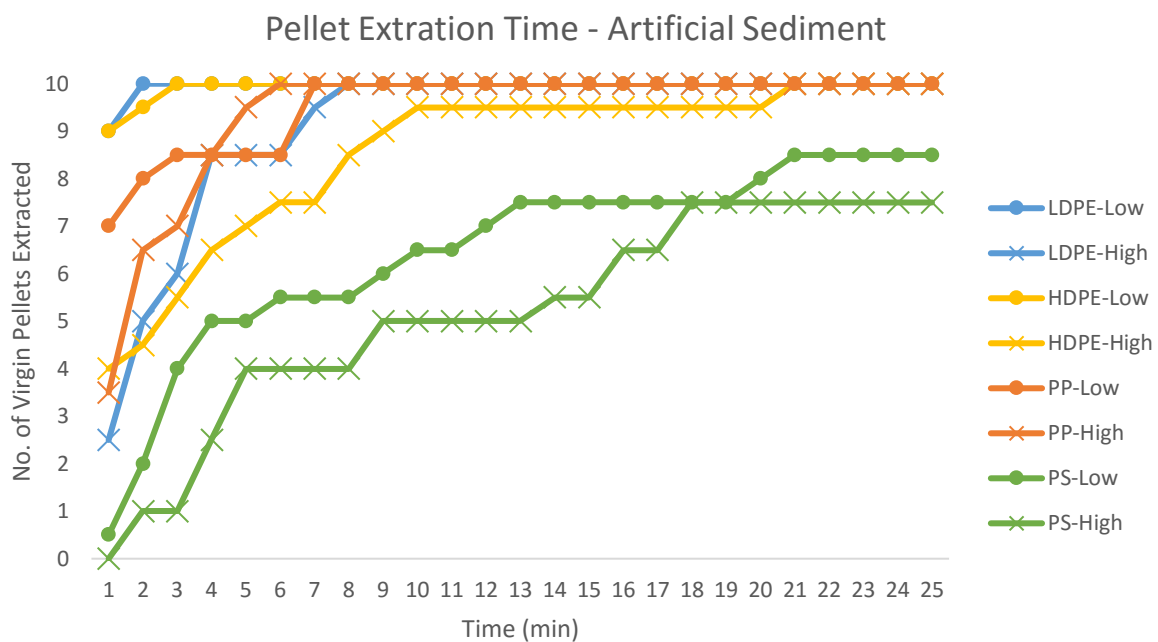
#### Pellet Extraction - Artificial Sediment



**Figure 8.** Cumulative pellet extraction from artificial sediment illustrating pellet extraction from low (20 L min<sup>-1</sup>) aeration intensity (blue bars) and high (40 L min<sup>-1</sup>) (orange bars) with tested plastic polymer types.



Extraction time for each individual pellet were recorded and the cumulative results showed that the quickest yield of pellets was with low aeration intensity (Fig. 9). At 2 minutes, all LDPE pellets in the low setting were extracted, and after 3 minutes all HDPE pellets were obtained. In comparison, in the high setting, all LDPE were extracted after 8 minutes and all HDPE after 21 minutes. PP yielded all pellets at low after 7 minutes, and at high setting all pellets after 6 minutes. The highest cumulative extraction number for PS was 8.5 pellets at low setting after 21 minutes, and at high setting 7.5 pellets extracted after 18 minutes. Results with total number of pellets < 10 showed that not all pellets were extracted and that the pellets were retained in the ED.



**Figure 9.** Cumulative number of recovered particles against time, demonstrating recovery time of the virgin plastic pellets at low and high aeration intensities. Circle markers are low ( $20 \text{ L min}^{-1}$ ) and X markers are high ( $40 \text{ L min}^{-1}$ ) aeration intensities. Each individual plastic type is give one colour,  $n = 10$ . PET (average = 0) and PC (average = 0.5) are excluded from the graph.

A Two-Way Analysis of Variance (ANOVA) was performed on the control experiments. The response variable was the amount of pellets extracted (Pellet.extracted) and factor variables was aeration intensities of low and high (Aeration) and type of plastic (Type.of.Plastic). The results show that there were significant differences amongst plastic types, but no differences between the two aeration intensities, nor any significant interaction between type of plastic and aeration (Table 7).

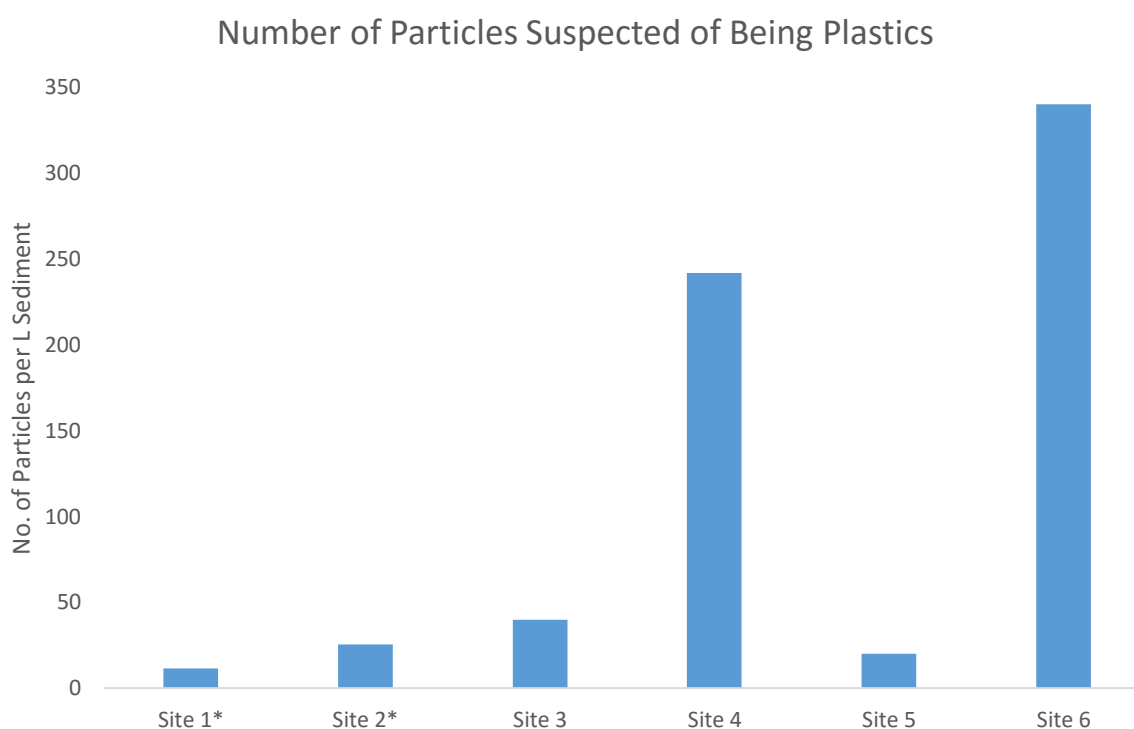
**Table 7.** A summary of a Two-Way ANOVA performed on pellets extracted against aeration, type of plastic and their interactions.

Response: Pellet.extracted	Sum Sq	Df	F value	Pr(>F)
Aeration	0.04	1	0.0909	0.7682
Type.of.Plastic	480.88	5	209.8364	3.022e-11 ***
Aeration:Type.of.Plastic	1.21	5	0.5273	0.7517
Residuals	5.50	12		

Signif. codes: 0 '\*\*\*' 0.001 '\*\*' 0.01 '\*' 0.05 '.' 0.1 ' ' 1

### 3.3 Microplastics from Akers River Sediment – A Case Study

A total estimate of 679 particles were suspected of being plastic from the sediment samples collected from Akers River. Site 1 had 11 particles; Site 2 had 25; Site 3 an estimate of 40; Site 4 an estimate of 242; Site 5 an estimate of 20; and Site 6 had an estimated particle count of 340 per L sediment (Fig. 10).

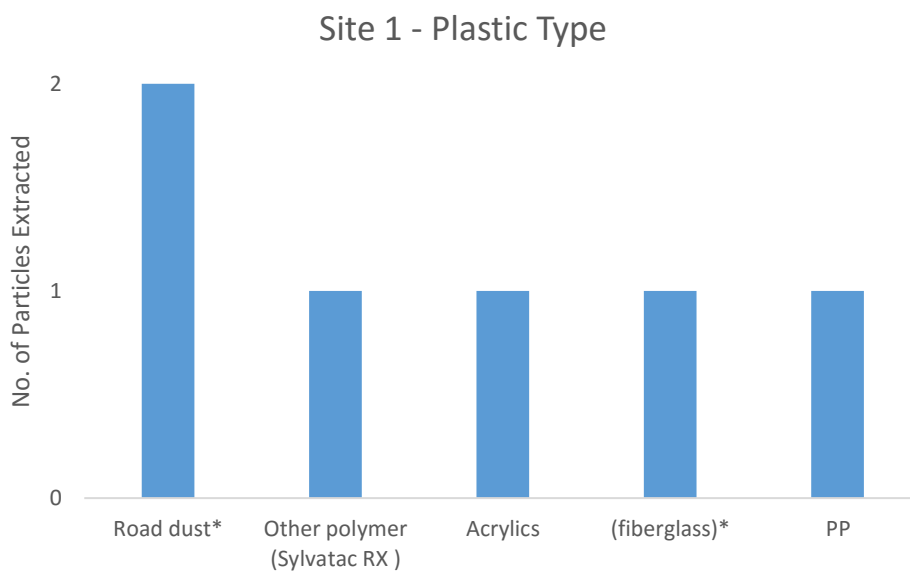


**Figure 10.** Particles suspected of being plastics from all six sites per L. Site 1\* and Site 2\*, all three subsamples underwent visual inspection, whilst Site 3, 4, 5 and 6 only had one subsample each visually inspected.

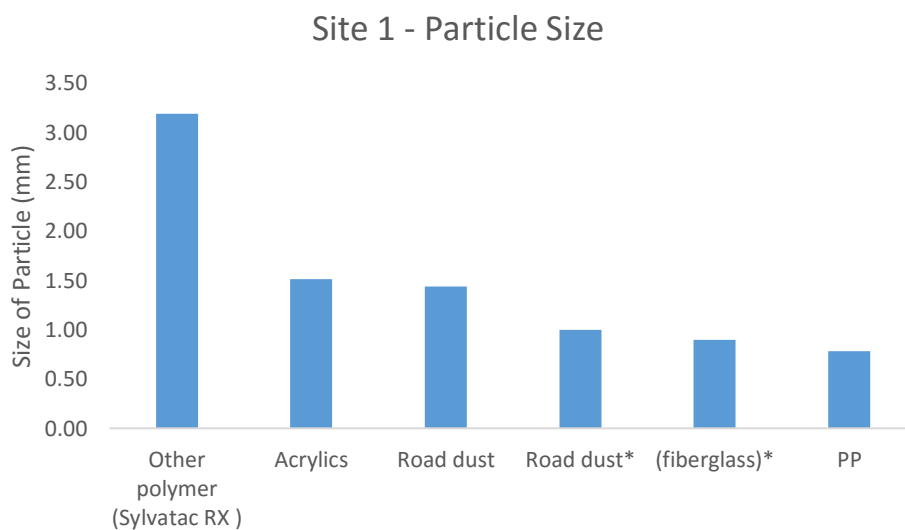
Due to time constraints and the large number of particles that were found at Site 6, only Site 1 (all three subsamples) and Site 6 (one subsample) underwent FT-IR analysis. A total of 187 particles were suspected of being plastics from Site 1 and Site 6 (one subsample); 13 fibres were found and excluded from FT-IR analysis; 35 particles were made of natural materials; 15 particles either broke in the process or were too small/thin to get a reading; six were lost while transferring the particles from the petri dish to the FT-IR machine or were not found/identified; and 11 particles were of anthropogenic material but not plastic. The

remaining 107 particles were identified as plastic polymers by FT-IR analysis and ranged from 0.58 to 7.70 mm in size. Out of the 107 particles, 105 were microplastics and two were mesoplastics.

Out of the 6 particles extracted from Site 1, a total of 5 different plastic types were found. Road dust (road film from auto paint, usually comprised of polyurethane (PUR)) had the highest occurrence of two particles, following one particle of other polymer (Sylvatac RX) containing plastics, one acrylics, one fiberglass containing plastics, and one polypropylene (PP) (Fig. 11). All six particles that were extracted from Site 1 were classified as microplastics (Fig. 12). One fibre was found but was excluded from FT-IR analysis.

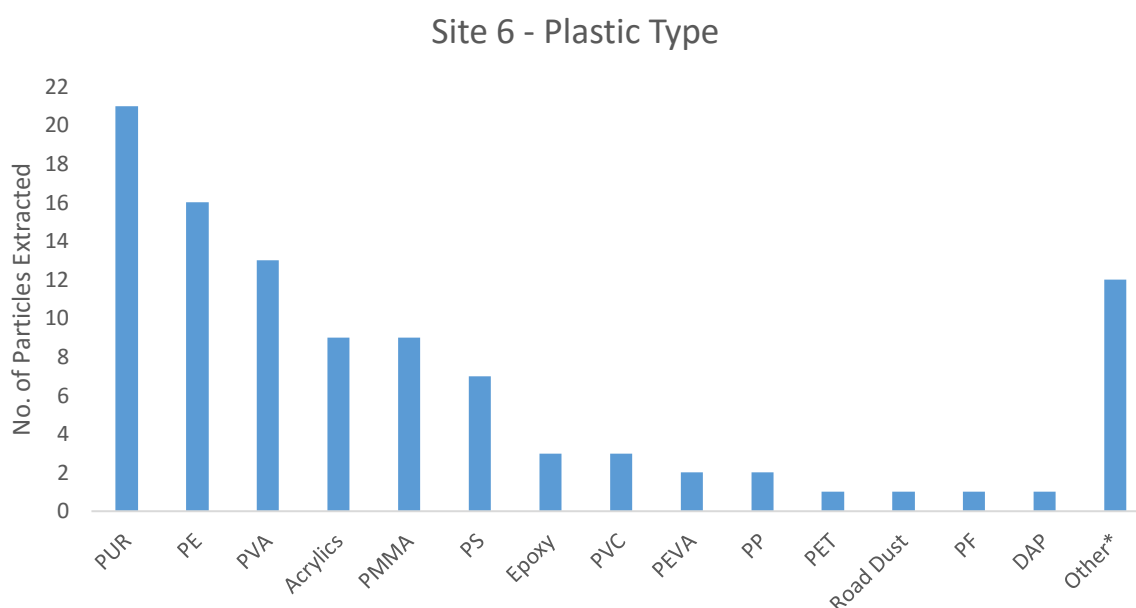


**Figure 11.** Number of plastic particles extracted from Site 1 (all three subsamples) and plastic type identified by FT-IR analysis (n = 6). \* FT-IR match <60 %. Polymer is plastic, but the amount of plastic in particle is < 60 %.



**Figure 12.** Microplastic sizes at Site 1 (all three subsamples) of all six particles extracted. \* FT-IR match <60 %. Polymer is plastic, but amount of plastic in particle is < 60 %.

Fourteen plastic polymer types were identified at Site 6 from subsample 1 (Fig. 13); PUR yielding 21 particles all originated from paint fragments, except two that were found in oil enamel; 16 PE; 13 polyvinyl alcohol/acetate (PVA) originated from amongst others paint, hair spray, glue and masking tape; nine acrylics, mostly from paint; nine polymethyl methacrylate (PMMA) all stemmed from Plexiglas; seven PS; three epoxy; three PVC; two poly(ethylene-vinyl acetate) (PEVA); two PP; one PET as polyester from plastic sheet; one road dust (road film from auto paint); one phenolic resin (PF); one diallyl phtalalte (DAP); and 12 particles with a plastic content < 60% under a pooled category named “Other”. The former group comprises of four acrylics, all from paint; six PUR, all from paint except for one that originated from roofing foam; and two PVA particles.

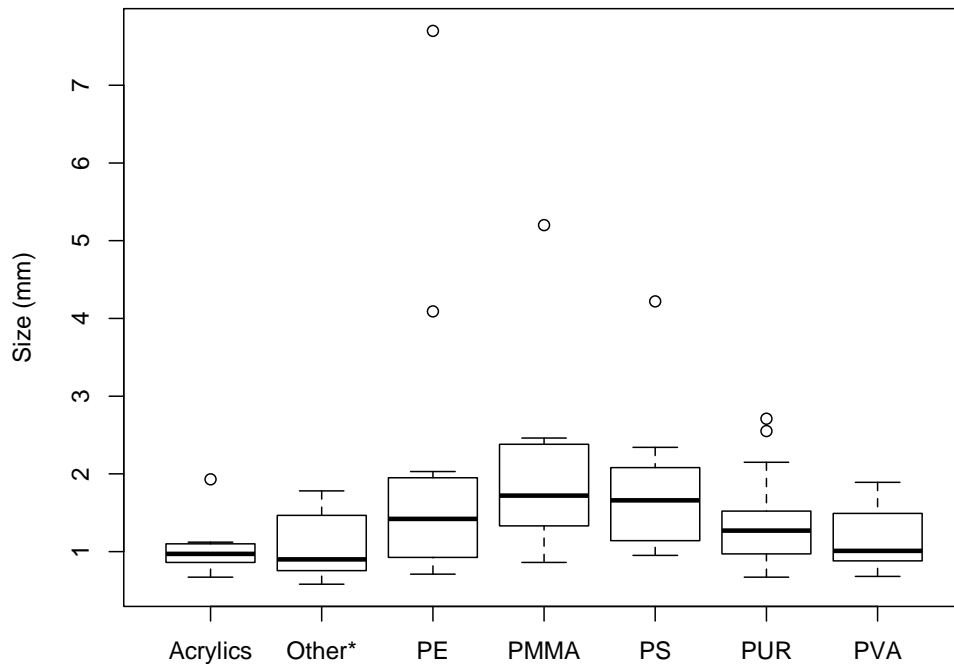


**Figure 13.** Plastic types (n = 14) identified by FT-IR analysis from Akers River and the number of particles (total of 101 particles) found at site 6 (subsample 1). Other\*, FT-IR match < 60 %. Polymer is plastic, but amount of plastic in particle is < 60 %.

At site 6 – subsample 1, the microplastics ranged from 0.58 to 7.70 mm in size (Fig. 14). From all microplastics found, 99 of the particles were classified as microplastics from 0.58 – 4.22 mm, while two were mesoplastics 5.20 (PMMA) and 7.70 mm (PE). The average sizes for the different plastic types are; Acrylics 1.08 mm; Epoxy 2.00 mm; PE 1.88 mm; PMMA 2.10 mm; PS 1.90 mm; PUR 1.37 mm; PVA 1.17 mm; and Other 1.08 mm.

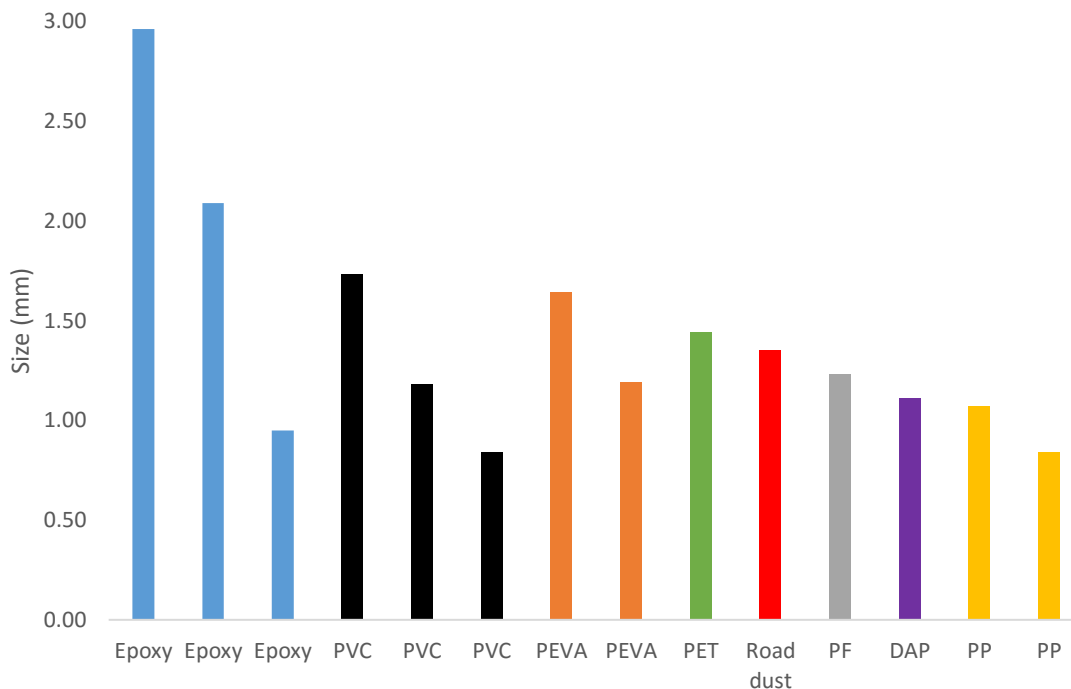
Epoxy had an average of 2 mm; and PVC 1.25 mm, three particles of each were extracted from the sample. PEVA had an average of 1.42 mm; and PP 0.96 and two of each were extracted. Only one particle of DAP 1.11 mm; PET 1.44 mm; PF 1.23 mm; and road dust 1.35 mm were extracted (Fig. 15).

### Site 6 - Particle Size



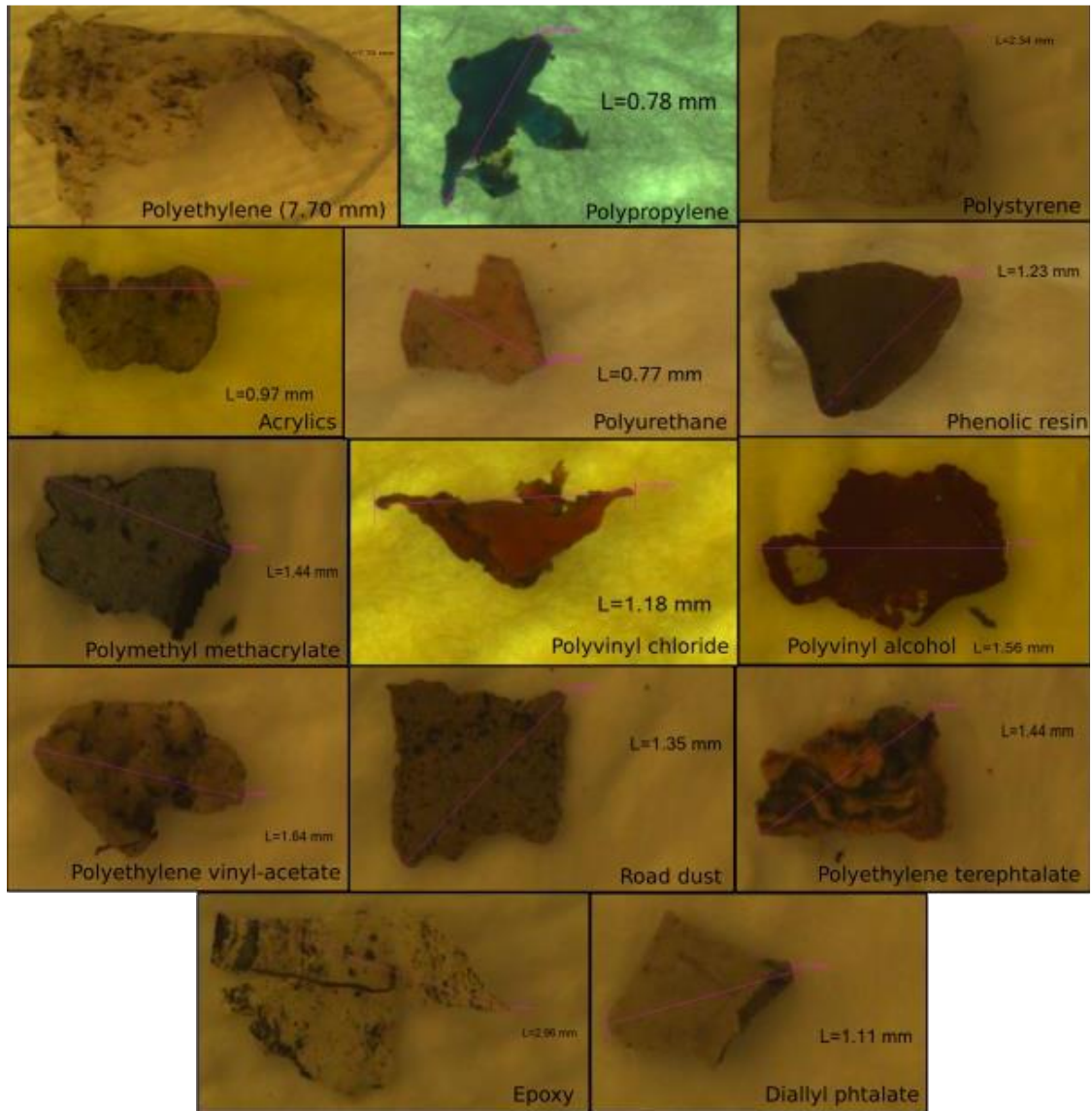
**Figure 14.** Size range for different plastic polymers found at Site 6 (subsample 1). The line in the boxes are the median and the boxes illustrate the upper and lower 50 % sizes of the plastic types. The whiskers represent the maximum and minimum sizes outside the 50 % box. Circles are particles with sizes that do not fit within the standard size range of the specific plastic type and are called outliers. Epoxy, PVC, PEVA, PET, Road Dust, PF, DAP and PP are excluded from this boxplot as their total  $n < 5$  particles.

### Site 6 - Particle Size, $n < 5$

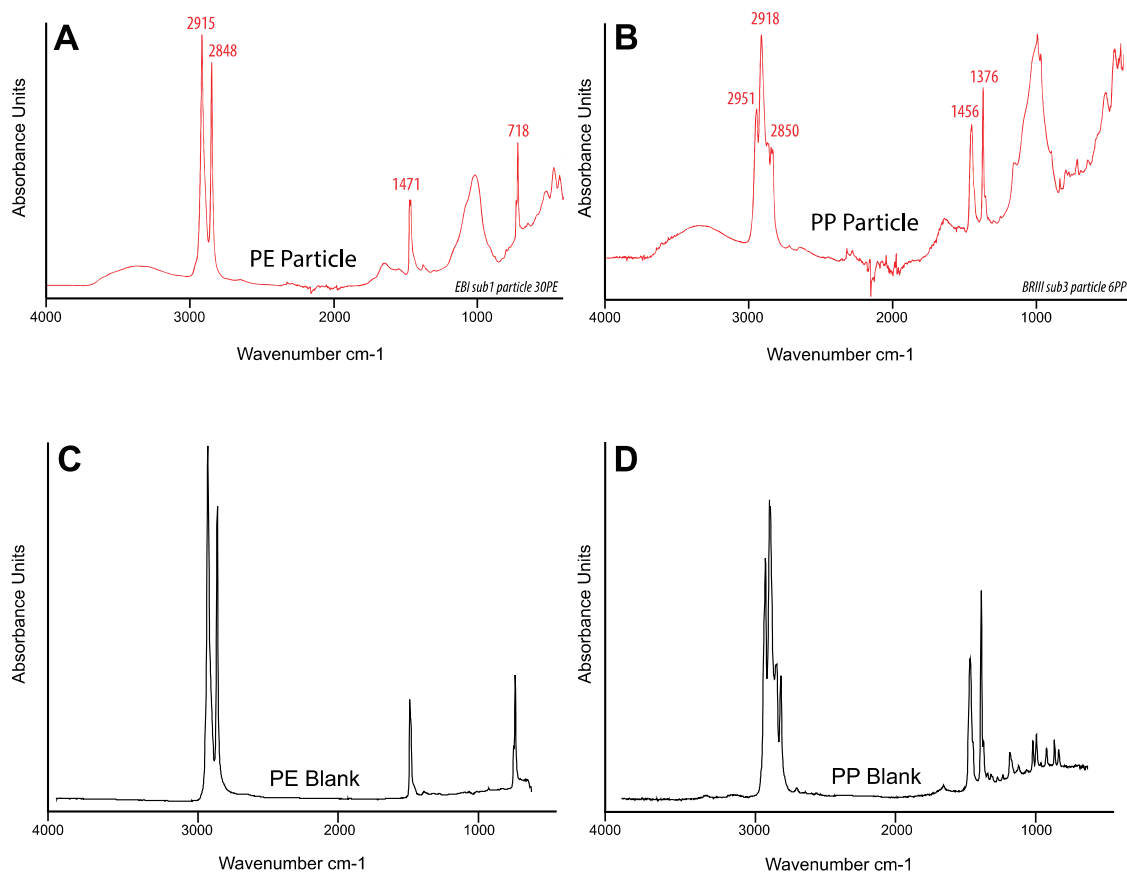


**Figure 15.** Plastic types with  $n < 5$  illustrating the size ranges of each particle found at Site 6 (subsample 1) where each plastic type has been give one specific colour.

All particles from Site 1 were < 3.5 mm (Fig. 11), and disregarding the outliers (Fig. 14 and Fig. 15), most of the particles collected by the ED at Site 6 are < 3 mm. A compilation of the different plastic polymers extracted are pictures below (Fig. 16) and FT-IR images of PE and PP extracted from Akers River including a blank/reference sample of PE and PP (Fig. 17).



**Figure 16.** A selection of meso- and microplastic particles extracted from Akers River sediments.



**Figure 17.** A compilation of FT-IR images of PE and PP where **a)** is the PE particle found in Fig. 15 with peak numbers at 2915.16, 2847.96, 1471.27 and 718.29  $\text{cm}^{-1}$ ; **b)** is the PP particle found in Fig. 15 with peak numbers at 2951.28, 2917.80, 2850.58, 1455.94 and 1376.27  $\text{cm}^{-1}$ ; **c)** FT-IR spectrum from a PE blank with reference peak numbers (from left to right) at 2916, 2849, 1471, 729 and 717  $\text{cm}^{-1}$ ; **d)** FT-IR spectrum from a PP blank with reference peak numbers (from left to right) at 2951, 2919, 2869, 2838, 1456 and 1358  $\text{cm}^{-1}$ . Image a) and b) are personal, and images c) and d) with their subsequent reference numbers are modified from Corcoran et al. (2015).

## 4 Discussion

### 4.1 Assessment of the Elutriation Device – Trial Experiments

#### 4.1.1 Density-Dependence of Extraction

An elutriation device (ED) was built for particle separation to specifically extract microplastic particles from freshwater sediments. The device is based on Claessens et al. (2013), who originally designed their ED for application to marine sediment samples (Fig. 3) and as the first step in a two-step separation technique. This thesis however only focused on the first step of extraction by elutriation to assess if and what types of plastics could be extracted by the ED with the use of freshwater. Many trial experiments were performed to become familiar with assembling and disassembling the device, but most importantly how the device performed with elutriation of freshwater sediment before commencing the case study from Akers River.

From the trial experiments, the recovery rate of virgin plastic pellets was found to be strongly density-dependent. Low-density polyethylene (LDPE), high-density polyethylene (HDPE) and polypropylene (PP) (all with densities  $< 1.0 \text{ g cm}^{-3}$ ) had a 100 % recovery rate. By contrast, polystyrene (PS) (density  $1.04\text{-}1.1 \text{ g cm}^{-3}$ ) had a maximum recovery rate of 85 %, and none of the virgin pellets with densities  $> 1.1 \text{ g cm}^{-3}$  were recovered, such as polyethylene terephthalate (PET) and polycarbonate (PC) (one obtained but regarded as an outlier).

Density-dependence was thus strongly focused at  $1.0 \text{ g cm}^{-3}$ , or the approximate density of freshwater ( $\approx 1.0 \text{ g cm}^{-3}$ ) used in the ED, as expected. Some plastics with density ranging between  $1.04\text{-}1.1 \text{ g cm}^{-3}$  can also be expected, but not all particles of those densities will be recovered. Our experimental setup is therefore well-suited to extract low-density plastics, but is expected to significantly under-extract heavy plastics; plastics  $\geq 1.0 \text{ g cm}^{-3}$  may not be extracted at all, as also reported by Bottolfsen (2016).

#### 4.1.2 Effects of Water Flow and Aeration Intensity

The water flow used in this investigation was approximately  $300 \text{ L h}^{-1}$ , after Claessens et al (2013). The effect of alternative water velocities on efficiency of particle separation was not investigated in this study, but investigations by Bottolfsen (2016) showed that a velocity of  $200 \text{ L h}^{-1}$  would decrease the recovery rate of some plastic types. Velocities at  $300 \text{ L h}^{-1}$  and  $400 \text{ L h}^{-1}$  had a higher recovery rates, however the two settings showed little significance between them (Bottolfsen, 2016). A study by Zhu (2015) determined that the optimum flow rate was  $385 \text{ L h}^{-1}$  and that velocities above this would inhibit the extraction of microplastics (Zhu, 2015). The author had however a smaller diameter ( $5.06 - 10.16 \text{ cm}$ ) and a shorter length ( $50 \text{ cm}$ ) for the ED than the one used in this thesis and in Bottolfsen (2016). Altering the length and the inner diameter of the columns may influence the effect of water flow and aeration particle selection, so it may not be appropriate to consider these flow rates more generally. A smaller ED would be easier to handle, however the volume of sediment that may be inserted into the device will also be reduced. The column that was used in this thesis could easily handle  $500 \text{ ml}$  of sediment, and future studies could investigate if the ED is still efficient at extracting microplastics from samples with a greater volume. The result could be higher volumes of samples being investigated for microplastics in an area with less time used, thus being more cost effective and time efficient.

Manipulation of the aeration intensity yielded no statistically different results in the character of plastics recovered, however the rate of pellet extraction was significantly faster when aeration was kept low ( $20 \text{ L min}^{-1}$ ) (Fig. 9), and the handling of exited materials was more convenient. This low aeration intensity was thus used in the Akers River case study.



#### 4.1.3 Suggested Improvements

During the course of the experiments, several issues were encountered that could be mitigated or resolved through adaptations to the experimental setup:

1. The use of tap water only allowed extraction of plastic particles  $< 1.0 \text{ g cm}^{-3}$ , giving an underestimate of the population of heavy plastic particles in sediment samples
2. The second opening of the ED was not enclosed, thus recovered materials on the sieve may have been contaminated by atmospheric fibres
3. The outer sieve only had a mesh size of  $500 \mu\text{m}$  and most likely failed to retain particles below  $500 \mu\text{m}$
4. Samples with high organic content increased the chance for water overflow on the sieve that may have led to further underestimation of plastic particles

The use of tap water is an inexpensive and safe way to conduct experiments, especially in such high volumes as used in this experimental setups, however this will exclude all particles with densities  $> 1.0 \text{ g cm}^{-3}$ . To mitigate this issue, Kedzierski et al. (2016) developed an enclosed elutriation system based on the same device used in this thesis. The improvements by Kedzierski et al. (2016) included:

- An injection and flow system controlling the water velocity and aeration in the elutriation column
- A storage and filtration system comprising a 30 L reservoir tank for water flowing in to the column, and a 30 L settling tank to allow particles to settle out of the water before being re-used; between the two tanks are sieves to prevent contamination
- Water temperature control

Although Kedzierski et al. (2016) also used tap water as their medium, their ED system is more long-term cost effective because it re-uses liquids and could allow extraction of heavier plastics if used with salt solutions such as NaCl (which would allow recovery of plastics up to  $1.2 \text{ g cm}^{-3}$ ). Other solutions such as NaI or  $\text{ZnCl}_2$  will yield even denser particles but are very expensive and may not be affordable in such high volumes. The Kedzierski et al. (2016) system is also much larger (and thus initially costly) than the Claessens et al. (2013) column but solves a number of issues encountered in this thesis (specifically points 1-3 above). Also the use of an enclosed system will minimise atmospheric contamination (Kedzierski et al., 2016).

A high amount of water was used for trial and the case study experiments, although the water temperature was set to cold, but the temperature of the water was not recorded. As water temperature changes, so might the density. Kedzierski et al. (2016) solved these issues by including a thermometer and heater for controlling the temperature of the water. The authors also included several filtration steps to separate particles in accordance with their grain size prior to elutriation (Kedzierski et al., 2016). However, this step might be difficult with sediment samples containing high volumes of organic matter, such as continental samples (Eerkes-Medrano et al., 2015); such issues are further discussed in the next section.

## 4.2 Issues with Samples Containing High Organic Matter and Visual Inspection

The elutriation device (ED) of Claessens et al. (2013) was specifically designed for extracting plastic particles from marine sediments which generally contain less organic materials than continental sediments. In many of the samples collected for the trial experiments, problems occurred with clogging of the sieve due to high volumes of low-density organic material occurred and led to overflow that may have resulted on loss of extracted plastic particles. Performing elutriation to extract microplastics from sediment samples may be less efficient if the content of organic matter is high. Several oxidative agents have been used to remove organic materials from samples such as strong acids like nitric acid (HNO<sub>3</sub>) and hydrochloric acid (HCl) (Claessens et al., 2013). Use of such strong acids that were applied to remove organic materials should be avoided as they may affect and degrade plastic polymers in the samples (Dris et al., 2015b). However, from the preliminary results of Imhof et al. (2013), a mixtures of sulfuric acids (H<sub>2</sub>SO<sub>4</sub>) with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) showed no damage to the plastic polymers tested (Imhof et al., 2013). Similarly, a solution of 30 % H<sub>2</sub>O<sub>2</sub> and 35 % H<sub>2</sub>O<sub>2</sub> was applied to samples with high bioorganic matter with no, or very little alteration to the plastic polymers, as demonstrated by FT-IR analysis (Nuelle et al., 2014, Tagg et al., 2015). Hidalgo-Ruz et al. (2012) however states that the use of acids only prove to be efficient in separating known plastics spiked into specific sediments, and not in the separation of mixed polymers from more diverse natural sediments and organic matter (Hidalgo-Ruz et al., 2012). Another approach to counteract problems with high organic matter clogging the sieve that was encountered in Akers River sediments, is to fit a net similar to a zooplankton net to the second opening. This net will retain all small particles with little chance of water flowing over whilst, protecting the recovered materials in the net from atmospheric contamination.

Visual inspection and sorting of the samples was the second step after the samples had gone through the ED. In this step, the inspector sorts out microplastics form organic, inorganic and other anthropogenic materials. It was apparent once the process of visual sorting started that it was difficult at times to distinguish natural from anthropogenic particles unless they had a bright colouration. Additionally, particles that appear to be stone or mineral, were in fact soft once handled by needles and forceps. Thus, visual sorting does not solely rely on visual considerations, but the texture of the particles also. This step is crucial as it can give an underestimate of microplastic particles in samples, yet it is very subjective. Dekiff et al. (2014) attempted to test the robustness of such visual separation of microplastics. They had three independent inspectors and obtained three separate quantitative records from the same sample extract (Dekiff et al., 2014). Because separation and identification of microplastics is a necessary but subjective step, there is a need to develop standardised techniques, protocols and methodologies to avoid misidentification and underrepresentation of microplastic particles in samples (Rocha-Santos and Duarte, 2015).

#### 4.3 Limitations of the Elutriation Device – Akers River Sediment

From the results obtained during the trial experiments on natural and artificial sediments, the plastic particles expected to be extracted from Akers River sediments mostly have densities  $< 1.0 \text{ g cm}^{-3}$ , with some of densities ranging between  $1.04\text{-}1.1 \text{ g cm}^{-3}$  (as seen on polystyrene (PS) extraction), and no plastics with densities above PS. Thus again, these results show that the experimental setup has limitations with respect to the extraction of high-density plastics and most likely lead to underestimation of the quantity of high-density plastic particles in natural samples.

In contrast, the plastic types expected to be found in Akers River sediments are of high-density because in order to be collected from a sample, they must have first been sedimented, in turn implying that they were sufficiently dense to settle through the (freshwater) water column. Low-density plastics will thus not be expected to be found in sediment samples and will most likely be expected to be found in the water column. Importantly, these expectations are formulated on the assumption that the plastics are in pure virgin form without additives and/or chemical and physical changes, excluding the formation of biofilm that may occur in the aquatic environment. These virgin pellets used in the trial experiments have round or are cylindrical in form with smooth surfaces. Consequently, the plastics that are expected to be in the sediment samples are exactly those with lowest chance of being extracted by the ED. However, as size, shape and the structure of plastic litter vary, the effect of the aquatic environment may also play a role in the extraction on the particle.

#### 4.4 Extracting Microplastics from Akers River Sediment – A Case Study

The total particles suspected of being plastics along the Akers River was 679 particles extracted from 9 litres of sediment distributed across 6 sampling sites. The 6 sampling sites were distributed down the length of Akers River, with Site 1 located 0.6 km of Lake Maridal, the source of the river, and site 6 located approximately 7 km from the mouth of the river, where it empties into the Oslo Fjord. Lake Maridal, being the drinking water reservoir of Oslo, is extensively protected. Settlements along the banks of the river upstream of Site 1 are also limited, the visible impact on the river is recreation, and the park from where Site 1 was collected is a popular bathing area. Downstream from Site 1 the river banks become progressively more developed, with both expanding residential areas but also industry. A number of major roads also run parallel to or cross the river downstream from Site 1, and notable bridge crossings occur between Site 1 and Site 2; close to Site 3; and near Site 4. Sites 4, 5 and 6 are located close to the urban centre of Oslo, and are therefore expected to be heavily influenced by the expansive residential and industrial areas, as well as the dense network of roads. Consequently, the Akers River profile presents a rather ideal natural laboratory, as the first sites near the source of the river should be unpolluted, whereas the last few sites are close to the urban centre of a European capital city, and are accordingly expected to be marked by significant pollution.

An increase of anthropogenic particles was found from Akers River sediments from the first to the last location (Fig. 10). This corresponds well with expectations, that the upstream

parts of Akers River, close to Lake Maridal, receive little microplastic pollution. Furthermore, microplastic pollution increases further downstream as the river flows through the city. In reality, from Site 1 -three subsamples (1.5 L) yielded 6 microplastic particles, whilst Site 6-subsample 1 (500 ml) generated 101 plastic particles. Thus, despite only processing one subsample from the site furthest downstream, one subsample from Site 6 contained much more plastics than all three subsamples from Site 1.

'Road dust' particles were found in Akers River sediments with the highest amount being at Site 1 with two particles, and one at Site 6. This is a small amount considering that car tyres and road paint is thought to be the highest microplastic contributor in Norway, according to Sundt et al. (2016). Both sites were in the proximity of roads and could therefore be the source to 'road dust' particles. The number of particles found at Site 1 was low per L, despite the areas high anthropogenic activity. One of the main contributory factors is due to its proximity to Lake Maridal. Site 2 was chosen because of the visible large and high anthropogenic litter content on the riverbed. This area is usually not a place where people stop and rest, but rather walk past, so the litter most likely comes from upstream. Nevertheless, 25 particles L<sup>-1</sup> in the sediment samples were suspected of being plastics. Site 3 and 4 were selected as these were along settlements. Site 3 had an estimate of 40 plastic particles L<sup>-1</sup> whilst Site 4 had 242 L<sup>-1</sup>. This sudden increase in particles suspected to be plastics at Site 4 may be due to accumulation and settling of particles from upstream. At Site 5, the estimated particles number dropped down to 20 L<sup>-1</sup>. The samples were taken not far from Øvre foss (also called Seilduksfossen), a waterfall. This waterfall will likely increase the turbidity and water velocity and may be the reason why the particle numbers decreased at this site. All the sites had similar grain size and similar organic matter content except for Site 5 (Table 3). At this site, most were of the size class gravel with some sand, and little organic matter compared to the other sites. The larger grain sizes in Site 5 indicate faster water flow resulting in less particle sedimentation. Thus, contributing to less particles suspected of being plastics in samples collected from Site 5. Although there was a decrease in particle numbers at Site 5, Site 6 had an estimate of 340 particles L<sup>-1</sup> suspected of being plastics. Most of the plastics (21 polyurethane (PUR) and nine acrylic particles) originated from paint fragments. As Site 5 were next to Oslo National Academy of the Arts (KHiO) where students frequently have exhibitions in and along the river, it can be speculated that their installations may to some extent contribute to microplastic pollution in Akers River.

The highest particle count was PUR which exclusively was found at Site 6. The second most abundant polymer was PE (low- and high-density grouped) followed by polyvinyl alcohol/acetate (PVA); acrylics; polymethyl methacrylate (PMMA); and polystyrene (PS) from both Site 1 and Site 6. The polymers found in Aker River, except PP, PE (high- and low-density) are denser than water, and theoretically these plastics should not have been extracted by the ED. As the virgin plastics that were extracted during the trial experiments were < 1.0 g cm<sup>-3</sup> and a maximum of 85 % PS with density ranging from 1.04-1.1 g cm<sup>-3</sup>, the expected plastics that could float out of the ED were all particles of LDPE, HDPE, PP and some PS. Furthermore, as low-density plastics are expected to float, those light plastics were not expected to be found in Akers River sediments. In this thesis however, it was shown that polymers such as LDPE, HDPE, PP and PS that have densities ≤ 1.1 g cm<sup>-3</sup> are found in river sediments. Additionally, it was shown also that plastics with density ≥ 1.1 g cm<sup>-3</sup> can be extracted with the ED. PUR, PVA, Acrylics and PMMA amongst others, were recovered from

Akers River samples. The heaviest known polymer extracted by elutriation from Akers River was one polyethylene terephthalate (PET) as polyester from a plastic sheet with a density ranging between 1.32-1.45 g cm<sup>-3</sup>. Although these plastics were found in Akers River sediment, it cannot be sure that these particles were quantitatively extracted. One possible explanation for why these plastics were extracted with the ED is that known densities of plastic polymers are determined from virgin pellet, meaning that they are pure and clean without additives. Plastics in general however, usually contains additives, making it difficult to know their true density, because density may change due to the additives. Moreover, chemical and physical alterations along with biofouling has shown to alter densities of plastics in aquatic environments.

The accumulation of sediments/particles depends on the river morphology and velocity of water at a particular site. However, since plastic may change over time, the sedimentation rate of plastic is unknown. Additionally, particles with densities > 1.0 g cm<sup>-3</sup> may have been extracted by chance or by flocculation with the lighter organic materials in the samples and thus floated to the surface and exited the ED. All these factors mentioned above may contribute to explaining why low-density plastics may have settled in the Akers River sediment in spite of theoretically being “lighter” than water, and why high-density plastics were extracted by elutriation. Furthermore, a study by Ryan (2015) suggest that macrolitter such as long and thin plastic bags show rapid sedimentation than smaller items. Size is thus more important than shape where surface area to volume ratios increases dramatically for particles with a diameter < 5 mm. The particles size affects movement and sedimentation rate independent of buoyancy/density (Ryan, 2015). Nizzetto et al. (2016) modelled a theoretical assessment of microplastic transport in river catchments and river sediments. One of the model’s outcome showed that the transport of microplastics in a stream is strongly dependent by the streams hydrological properties, and that size of the particles also contribute to their retention or mobilisation. For instance, larger and high-density microplastics mobilisation occurs during intense flow regimes and flooding periods. Slower flow periods however, may inhibit transportation of particles ranging between 0.3-0.5 mm and particles denser than water. Moreover, particle size may have a significant role in microplastic transport dynamics and that density may have a smaller effect on retention of plastic particles. Thus supporting the idea of Ryan (2015) that dimension affects particle transport (Nizzetto et al., 2016, Ryan, 2015).

A study by Morét-Ferguson et al. (2010) brought up the issue of the uncertainty of a plastic particles density when it has been subjected to chemical and physical alterations. The chemical and physical properties of particles changes in the ocean most likely because of the attachment of microbial biofilm (Morét-Ferguson et al., 2010). This increases its density and possibly increases its chances of sedimentation (Imhof et al., 2012, Morét-Ferguson et al., 2010, Ye and Andrady, 1991) . Other studies confirm that density is not the only contributory factor deciding the fate of plastic particles. Wang et al. (2017) found not only low-density plastics but also 18 PET (density 1.32-1.45 g cm<sup>-3</sup>), five nylon (1.15 g cm<sup>-3</sup>) and two PS (1.04-1.1 g cm<sup>-3</sup>) in urban surface waters in lakes and rivers in China (Wang et al., 2017). Additionally, Mani et al. (2011) reported that PS was the most dominant component of the estimated 191 million microplastics that are transported by the river Rhine to the North Sea (Mani et al., 2015). Another study conducted in the river Rhine was done by Klein et al. (2015) with most of the plastic particles comprising of PS followed by PE and PP, all three

making up more than 75 % of the total plastics found in the sediment (Klein et al., 2015). These three studies by Wang et al. (2017), Mani et al. (2011) and Klein et al. (2015) show that high-density plastics can be found floating in surface waters and that low-density plastics can be found in sediments. This is in accordance with the results presented here finding heavy particles in sediments, but unexpectedly extracting them. Furthermore, surprisingly finding lighter plastics in the sediments and extracting those polymers, showing that lighter and smaller plastics can settle in sediments yet still be extracted by elutriation.

Horton et al. (2017) investigated the possibility of microplastics in sediments in tributaries of the River Thames where they found a total of 34 plastic particles that could be identified to a specific plastic polymer. The plastic polymers found in the sediments were 14 PET, five PP, five polyarylsulphone thermoplastic, two PE, one PS, and one PVC. They also found PC, PUR and PMMA. Particles that they did not manage to extract from sediment samples by flotation were identified as composites of road-marking paints, aggregates, a painted coating on a dense particle or high-density mineral-polymer mixture. (Horton et al., 2017). These plastic types, except for polyarylsulphone thermoplastic, were also found in Akers River sediments. Horton et al. (2017) findings of road paint is according to Sundt et al. (2016) along with synthetic rubber dust, the largest sources of microplastic contribution in Norway. Only three 'road dust' particles were identified from Akers River sediments. The second largest source is thought to stem from plastic polymers in paint by either weathering or removal of paints on surfaces, contributing to be > 1000 tonnes yearly (Sundt et al., 2016). A total of 30 plastic particles associated with paint was found in sediments taken from Akers River which is not surprising according to Sundt et al. (2016) technical report on paint pollution.

A maximum estimate of 113 particles L<sup>-1</sup> thought to be microplastics were found in Akers River sediments, and four particles L<sup>-1</sup> were extracted from Alna River sediment (Bottolfsen, 2016). These are relatively small numbers compared to for example what Castañeda et al. (2014) found in the Canadian St. Lawrence River sediment of approximately 1000 particles L<sup>-1</sup>, only considering microbeads (Castañeda et al., 2014). During collection of sediment samples in Akers River, the water flow was approximately 1.5 m<sup>3</sup> s<sup>-1</sup>. Low water flow is associated with storage, retention and deposition of microplastics (Nizzetto et al., 2016) and plastic particles of both low- and high-density may settle and sediment along the river.

## 5 Concluding Remarks

This case study on microplastic pollution in Akers River confirms that both low-density and high-density plastic types are being retained in river sediments in Norway. Further research should be conducted on several issues related to microplastics. As this field is still in its starting point, it is expected that there are many techniques used to attempt to extract microplastics from sediments, however as techniques and units vary, the issue of comparing results becomes difficult. It is thus important to standardise methods used for sampling microplastics. The use of Claessens et al. (2013) elutriation device (ED) was first and foremost due to its simple yet effective extraction of known plastic densities, that it was easily replicated and that most institutions could afford a such device. It was also important

that the medium used was not dependent on using costly salt solutions, but tap water that can be used in high volumes.

The ED was successful in extracting virgin plastics with densities lower than freshwater, and extracting some plastics close to freshwater density; however, particles heavier than freshwater were not recovered with the ED during trial experiments. During the case study from Akers River, non-virgin plastic particles of both low- and high-density plastics were extracted from sediment samples, which was surprising as the extraction of plastics were strongly density-dependent when using the ED during the trial experiments. The ED can be seen as a small-scale in-lab river. Where it simulates sedimentation of particles, particles suspended in the water column and particles floating on the surface, eventually escaping the river environment by the aid of the upwards water flow and turbulence by aeration. By using in situ sediments and investigating the presence/absence of plastics by elutriation, this thesis may give an idea of what types of plastics are retained in the river sediment and, although it is likely that the quantity of microplastics are an underestimate, an indication of the numbers of microplastics can be a starting point for further research as more information is needed regarding sedimentation of microplastics in rivers in Norway. The findings from the river sediments coincides with common plastics used all over the world (Plasticseurope, 2016). Furthermore, as plastics associated with paint were found in high numbers in Akers River sediments, special attention to paint litter as they can act as contaminants transporting toxic chemicals in their surrounding environment (Imhof et al., 2016).

Although it is important to have an idea of what type of plastics could be found based upon their density in relation to water, it is also important to factor in other variables that may have significant roles on their chemical and physical properties. This thesis, Bottolfsen (2016) thesis and other published literature found high-density plastic polymers in surface water and low-density plastics in sediments (Mani et al., 2015, Morét-Ferguson et al., 2010, Wang et al., 2017) which is unexpected in their plastic virgin forms. It can thus be said that virgin plastics pellets released in the aquatic environments are expected to behave similar to the trial experiments results from this thesis. But the longer they are subjected to mechanical abrasion and other factors altering their chemical and physical attributes, the pellets behaviour and position in the aquatic environment may change.

This thesis has shown that i) plastic particles with a theoretical density lighter than water can be found in river sediments, and ii) plastic particles with a theoretical density heavier than water can be extracted from river sediments using the ED; however, these are likely not extracted quantitatively. The following parameters are likely to explain these phenomena:

- Hydrological dynamics of the river
- Seasonal variability relating to water flow
- Surface area to volume ratios (size and shape)
- Surface structure, smoothness/roughness of particle
- Changes to the chemical and physical properties of particle which also affects the rate of biofouling and degradation

The research conducted in this thesis could be a stepping stone for further investigations on the amount of microplastics in freshwater environments in Norway. Additionally, how rivers not only contribute to marine plastic pollution, but may act as sinks for plastic particles. It should also be emphasised that more attention on preventing plastic pollution and more efficient waste/recycle management should be of high priority. There has also been positive feedback from marine clean-up arrangements, and if similar attention was focused on rivers and lakes less plastic pollution could reach marine habitats.



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ANNEX 3PREPARATION OF FORMULATED SEDIMENTSediment composition

The composition of the formulated sediment should be as follows:

Constituent	Characteristics	% of sediment dry weight
Peat	Sphagnum moss peat, as close to pH 5.5-6.0 as possible, no visible plant remains, finely ground (particle size $\leq 1$ mm) and air dried	4-5
Quartz sand	Grain size: > 50% of the particles should be in the range of 50-200 $\mu\text{m}$	75-76
Kaolinite clay	Kaolinite content $\geq 30\%$	20
Organic carbon	Adjusted by addition of peat and sand	2 ( $\pm 0.5$ )
Calcium carbonate	$\text{CaCO}_3$ , pulverised, chemically pure	0.05 – 0.1
Water	Conductivity $\leq 10$ $\mu\text{S/cm}$	30 – 50

Preparation

The peat is air dried and ground to a fine powder. A suspension of the required amount of peat powder in deionised water is prepared using a high-performance homogenising device. The pH of this suspension is adjusted to  $5.5 \pm 0.5$  with  $\text{CaCO}_3$ . The suspension is conditioned for at least two days with gentle stirring at  $20 \pm 2$  °C, to stabilise pH and establish a stable microbial component. pH is measured again and should be  $6.0 \pm 0.5$ . Then the peat suspension is mixed with the other constituents (sand and kaolin clay) and deionised water to obtain an homogeneous sediment with a water content in a range of 30–50 per cent of dry weight of the sediment. The pH of the final mixture is measured once again and is adjusted to 6.5 to 7.5 with  $\text{CaCO}_3$  if necessary. Samples of the sediment are taken to determine the dry weight and the organic carbon content. Then, before it is used in the chironomid toxicity test, it is recommended that the formulated sediment be conditioned for seven days under the same conditions which prevail in the subsequent test.

Storage

The dry constituents for preparation of the artificial sediment may be stored in a dry and cool place at room temperature. The formulated (wet) sediment should not be stored prior to its use in the test. It should be used immediately after the 7 days conditioning period that ends its preparation.

Sample ID	Area	Colour	Size (mm)	FT-IR match	Polymer	Abbr.
EBI - 16	Site 6-a	beige	1,19	78,14	Ethylene/vinyl acetat copolymer 28 wt%	<b>PEVA</b>
EBI - 122	Site 6-a	dirty yellow	1,64	75,72	Ethylene/vinyl acetat copolymer 40 wt%	<b>PEVA</b>
BrII - 4	Site 1-b	purple/brown	1,44	74,65	Road film from suto paint surface	<b>Road dust</b>
EBI - 161	Site 6-a	grey/brown, matt	1,35	63,52	Road film from auto paint -note: Paint Protection Film is a thermoplastic urethane	<b>Road dust</b>
EBI - 75	Site 6-a	grey/light green	2,34	84,49	Poly(methyl methacrylate); Plexiglass Plate; Acrylic Solution Lacquer	<b>PMMA</b>
EBI - 86	Site 6-a	light brown film	1,33	89,8	Poly(methyl methacrylate); Plexiglas	<b>PMMA</b>
EBI - 105	Site 6-a	light grey	2,46	72,1	MBX-12,5%, 12um Polymethyl Methacrylate delustering Spheres, Plexiglas	<b>PMMA</b>
EBI - 126	Site 6-a	green	2,38	73,83	Plexiglas Plate; 69,63% Poly(methyl methacrylate) which is a type og thermoplastic used in plexi glass	<b>PMMA</b>
EBI - 128	Site 6-a	white/grey	1,17	75,68	MBX-12,5%, 12um Polymethyl Methacrylate delustering Spheres, Plexiglas	<b>PMMA</b>
EBI - 130	Site 6-a	dark grey	0,86	70,34	MBX-60,5%, 12um Polymethyl Methacrylate delustering Spheres, Plexiglas	<b>PMMA</b>
EBI - 131	Site 6-a	grey/green	1,44	90,41	Poly(methyl methacrylate), Plexiglass	<b>PMMA</b>
EBI - 142	Site 6-a	grey	5,20	76,86	Poly(methyl methacrylate); 75,65% Plexaglas Plate	<b>PMMA</b>
EBI - 158	Site 6-a	grey	1,72	82,69	Poly(methyl methacrylate), Plexiglas	<b>PMMA</b>
BrIII - 6	Site 1-c	turquoise	0,78	88,4	polypropylene, isotactic	<b>PP</b>



EBI - 10	Site 6-a	white with small dark particles	1,07	96,73	Polypropylene, isostatic (+ poly(ethylene:propylene)	PP
EBI - 159	Site 6-a	brown transparent	0,84	94,88	Duct Tape, Adhesive, Clear, Colorless, Oriented Polypropylene, Tyco	PP
EBI - 1	Site 6-a	grey	1,73	79,37	Polyvinylchloride carboxylated	PVC
EBI - 67	Site 6-a	red/red-yellow	1,18	73,65	Velon, Poly Vinylidene Chloride + PVC & PVA, (plasticized) Daiper Cover,	PVC
EBI - 129	Site 6-a	light grey	0,84	71,33	Remelon Plastic, Clay filled w/Raybo 68 HydroSlip, PVC&PolyVinylidene Chloride	PVC
EBI - 3	Site 6-a	light grey	1,52	62,77	Paint,PolyUrethane Oil Enamel, Alkyd Enamel w/pigments. Ov	PUR
EBI - 11	Site 6-a	grey	2,71	68,78	Paint,PolyUrethane Oil Enamel, Alkyd Enamel w/pigments. Ov	PUR
EBI - 24	Site 6-a	dirty white/light grey	1,36	80,42	PolyUrethane Oil Enamel, Alkyd Enamel w/pigments. Ov	PUR
EBI - 26	Site 6-a	red/purple	1,70	73,94	Paint,PolyUrethane Oil Enamel, Alkyd Enamel w/pigments. Ov	PUR
EBI - 32	Site 6-a	shiny, flat white/grey	1,23	72,54	Paint,PolyUrethane Oil Enamel, Alkyd Enamel w/pigments. Ov	PUR
EBI - 38	Site 6-a	red	1,14	75,18	Paint,PolyUrethane Oil Enamel, Alkyd Enamel w/pigments. Ov	PUR
EBI - 39	Site 6-a	red	1,27	70,65	Paint PolyUrethane	PUR
EBI - 40	Site 6-a	light red	1,44	78,49	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI - 42	Site 6-a	grey	1,51	69,82	Paint,PolyUrethane Enamel, Light Blue, red Devil, Indoor/Outdoor	PUR
EBI - 65	Site 6-a	dirty yellow	1,04	78,49	Paint, PolyUrethane	PUR
EBI - 107	Site 6-a	grey w/red part in middle	2,55	63,85	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI - 113	Site 6-a	grey	1,59	81,88	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/o Pigment, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI -	Site 6-a	light beige	0,77	69,37	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments,	PUR

115					Overnight 65C Dry, Krylon, Satin Black Resin	
EBI - 116	Site 6-a	light beige	0,67	69,59	Paint, PolyUrethane Enamel, Red Devil, Indoor/Outdoor	PUR
EBI - 120	Site 6-a	light grey/dirty yellow	1,21	71,23	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI - 138	Site 6-a	light grey	1,36	77,85	Paint, PolyUrethane Enamel, Light Blue, Red devil, Indoor/Outdoor; 77,1% Propylene, glycol ricinoleate, SPP	PUR
EBI - 147	Site 6-a	grey	2,15	63,73	PolyUrethane Oil Enamel, Alkyd Enamel w/pigments. Ov	PUR
EBI - 163	Site 6-a	dark red	0,97	74,24	Paint, PolyUrethane Enamel, Red Devil, Indoor/Outdoor Unpigmented Resin	PUR
EBI - 165	Site 6-a	light grey	0,82	69,11	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI - 166	Site 6-a	dark grey	0,88	82,8	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI - 170	Site 6-a	grey	0,91	66,17	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI - 9	Site 6-a	red/fushia	0,88	76,76	Paint extender, white (vinyl acetate/ethylene/vinyl chloride)	PVA
EBI - 17	Site 6-a	back and white spots	1,49	78	Polyvinyl acetate	PVA
EBI - 51	Site 6-a	beige	1,01	68,45	PVA/Ethylene copolymer	PVA
EBI - 79	Site 6-a	whitw/light grey	1,71	72,11	Poly(vinyl acetate) found in paint Extender, Hair Spray, Glue, Padding, Masking tape	PVA
EBI - 102	Site 6-a	yellow	1,89	83,47	Poly(vinyl acetate) found in paint Extender, Hair Spray, Glue, Padding, Masking tape	PVA
EBI - 114	Site 6-a	grey, forlded in half	0,90	81,31	Paint extender, white (vinyl acetate/ethylene/vinyl chloride)	PVA
EBI - 136	Site 6-a	yellow/light brown	1,24	68,74	Poly(vinyl acetate) found in paint Extender, Hair Spray, Glue, Padding, Masking tape	PVA

EBI - 148	Site 6-a	dirty white/light grey	0,95	90,64	Poly(vinyl acetate) found in paint Extender, Hair Spray, Glue, Padding, Masking tape	PVA
EBI - 149	Site 6-a	yellow	0,81	71,84	Glue, Elmer's School (PVA+)	PVA
EBI - 156	Site 6-a	yellow	0,72	74,19	Glue, Elmer's School (PVA+)	PVA
EBI - 164	Site 6-a	grey/light brown	0,68	71,77	Paint extender, white (vinyl acetate/ethylene/vinyl chloride)	PVA
EBI - 167	Site 6-a	transparent	1,71	89,01	NEOCRYL A-655, chem. comp. Polyvinyl, Acrylic/Styrene	PVA
EBI - 168	Site 6-a	light grey	1,25	73,88	Paint extender, white (vinyl acetate/ethylene/vinyl chloride)	PVA
EBI - 30	Site 6-a	white	7,70	95,98	Polyethylene (Mn 6500)	PE
EBI - 44	Site 6-a	dark green	1,73	63,66	Polyethylene, chlorinated 48 wt%	PE
EBI - 46	Site 6-a	white/dirty	0,93	70,61	Polyethylene, chlorinated 48 wt%	PE
EBI - 57	Site 6-a	grey, matt	0,98	66,73	Polyethylene, chlorinated 48 wt%	PE
EBI - 91	Site 6-a	dirty whitw/beige	2,02	86,86	Polyethylene KR 16 (additives and plasticizers)	PE
EBI - 95	Site 6-a	dirty white	1,43	88,55	Polyethylene KR 16 (additives and plasticizers); 86,2% Polyethylene High Density	PE
EBI - 99	Site 6-a	green with yellow spots	1,52	61,81	Polyethylene, chlorinated 36wt% ( see notes)	PE
EBI - 101	Site 6-a	red	2,03	60,49	Polyethylene, chlorosulfonated 43wt%	PE
EBI - 104	Site 6-a	yellow metallic	4,09	68,94	Polyethylene (Mn 900); Type F; KR16; (Mn6500), this was on second FTIR analysis-104.1	PE
EBI - 106	Site 6-a	white	0,71	67,63	Polyethylene	PE
EBI - 137	Site 6-a	beige	1,41	70,32	Polyethylene, chlorinated 42 wt%	PE

EBI - 139	Site 6-a	white, is dirty	1,00	73,72	Polyethylene, chlorinated 25 wt%	PE
EBI - 140	Site 6-a	beige	0,89	67,12	Poly(Ethylene) 25% Chlorinated w/talc, SPP	PE
EBI - 143	Site 6-a	white/dirty white	0,92	75,43	Polyethylene, chlorinated 25 wt%; 69,81% Poly(vinyl stearate)	PE
EBI - 150	Site 6-a	grey	1,88	94,38	Polyethylene (Mn 1800); 91,98% PE-HD	PE
EBI - 169	Site 6-a	white/beige	0,83	65,81	Polyethylene, chlorinated 48 wt%	PE
EBI - 125	Site 6-a	yellow	1,44	65,08	Poly(ethylene terephthalate); Polyester, Plastic sheet	PET
EBI - 19	Site 6-a	red, rectangular	1,10	81,95	Polyester, Mod, MT153, Trevira 350	PS
EBI - 35	Site 6-a	light grey	2,34	95,48	Microscopial Diamond ATR, Polystyrene, Nicolet Ref Mtl #ADP	PS
EBI - 93	Site 6-a	green/light green in middle	4,22	87,4	Polystyrene	PS
EBI - 103	Site 6-a	brown/red	1,18	63,12	Poly(styrene), atactic; 67,18% Alkyd, 35% triglyceride + 26% POLYANHYDRIDE *	PS
EBI - 144	Site 6-a	beige	1,66	66,92	Poly(Styrene Acrylonitrile), Copolymer, Polysciences, masking tape	PS
EBI - 145	Site 6-a	red	0,95	63,26	Styrene/allyl alcohol copolymer 5-6% Hydroxyl	PS
EBI - 154	Site 6-a	white, grey, brown	1,82	88,79	Polystyrene	PS
BR I -5	Site 1-a		3,19	85,82	Sylvatac RX, rosin esters used in high and low vinyl acetate EVA, acryls, polyurethanes	Other polymer

BrIII - 4	Site 1	white/beige	1,51	66,12	NACRYLIC 78-6368	<b>Acrylics</b>
EBI - 66	Site 6-a	grey	0,68	80,85	Paint, Clear Coat, Acrylic Styrene Urethane	<b>Acrylics</b>
EBI - 76	Site 6-a	white/yellow/light grey	1,10	63,86	NACRYLIC 78-6368	<b>Acrylics</b>
EBI - 77	Site 6-a	greyish-transparent	0,97	93,87	CAFA_250 (Evap'd), High mol.wt. Polyacrylate surface tension modifier	<b>Acrylics</b>
EBI - 78	Site 6-a	dark grey	1,93	76,28	One Time Light Weight Spackling Complound: consists of Acrylic/Vinyl Acrylic Emusion Blend/Acrylamide	<b>Acrylics</b>
EBI - 85	Site 6-a	grey/dirty whitw	1,12	79,66	One Time Light Weight Spackling Complound: consists of Acrylic/Vinyl Acrylic Emusion Blend/Acrylamide	<b>Acrylics</b>
EBI - 89	Site 6-a	white	0,98	64,1	NACRYLIC 78-6368	<b>Acrylics</b>
EBI - 100	Site 6-a	green with yellow spots	0,86	64,27	Acrylic Polymer A, DuPont, From Base Coat Resin System8975S, 66,44% Duct Tape, impregnated paper	<b>Acrylics</b>
EBI - 135	Site 6-a	grey/white	0,67	77,62	One Time Light Weight Spackling Complound, Acrylic/Vinyl Acrylic Emusion Blend/Acrylamide	<b>Acrylics</b>
EBI - 146	Site 6-a	white/light grey	0,97	68,5	Styrene Acrylic	<b>Acrylics</b>
EBI - 41	Site 6-a	yellow/beige	2,96	85,58	Epoxy Polymer, Duro/Locklite2h, 84,53%Phenoxy Resin, 82,02% Epoxy Resin, bisphenolA+epichlorohydrin	<b>Epoxy</b>
EBI - 141	Site 6-a	grey/glittery	2,09	85	Phenoxy resin	<b>Epoxy</b>
EBI - 160	Site 6-a	grey, matt	0,95	80,68	Phenoxy Resin; 80,02% Epoxy Polymer, Duro/Locktite 2hr	<b>Epoxy</b>
EBI - 118	Site 6-a	dark grey	1,23	63,71	Phenolic Resin, hard cured (synthetc polymers, first commercial synthetic resins (plastics)	<b>PF</b>

EBI - 8	Site 6-a	light grey	1,11	71,2	Poly(Diallyl Phtalate) from melt (SPP Std#010 (o-phtalate )(atr)	DAP
				<60 %		
BrIII - 1	Site 1-c	black	1,00	55,87	Road film from auto paint surface	Road dust
BrIII - 5	Site 1-c	transparent/brown	0,90	43,45	fiberglass	Other polymer
EBI - 82	Site 6-a	light red/pink	0,72	56,75	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI - 94	Site 6-a	grey	1,78	59,45	Paint, PolyUrethane Enamel, Red Devil, Indoor/Outdoor, Unpigmented Resin	PUR
EBI - 108	Site 6-a	beige	0,58	55,08	Paint, PolyUrethane Enamel, Light Blue, Red Devil, Indoor/Outdoor	PUR
EBI - 110	Site 6-a	grey	1,11	54,2	Paint, PolyUrethane Oil Enamel, Alkyd Enamel w/Pigments, Overnight 65C Dry, Krylon, Satin Black Resin	PUR
EBI - 112	Site 6-a	grey-brown-ish	1,78	56,61	Roofing Foam, Poly Urethane, poly(ester urethane) type, 1141	PUR
EBI - 117	Site 6-a	yellow/beige	0,91	56,06	Paint, PolyUrethane Enamel, Ligh Blue, Red Devil, Indoor/outdoor	PUR
EBI - 7	Site 6-a	red	1,56	57,92	PVA/Ethylene copolymer	PVA
EBI - 20	Site 6-a	light grey/white	1,37	57,41	Polyvinyl acetate	PVA
EBI - 133	Site 6-a	yellow	0,89	59,16	One Time Light Weight Spackling Complound: consists of Acrylic/Vinyl Acrylic Emusion Blend/Acrylamide	Acrylics
EBI - 134	Site 6-a	yellow	0,79	50,79	Applique embroidery Acrylate/Acrylamide paint on frabric	Acrylics
EBI - 152	Site 6-a	beige/light yellow/greyish	0,64	43,95	Paint, Light Grey Primer, Styrene Acrylic Urethane w/Zirconia, TiO2, BaSO4, Talc	Acrylics
EBI - 157	Site 6-a	white	0,87	56,84	Paint, Blue Base/Color Coat, Styrene modedefied Acrylic -50% Alkyd w/ Urethane, Phthalocyanine Blue	Acrylics

Sample ID	Area	Colour	Size(mm)	Non-Plastics
BR 1 -1	Site1-a	shiny,metallic	1,23	mineral
BR 1 -3	Site1-a	off-white/beige	2,09	mineral, albite
BR 1 -4	Site1-a	black	1,89	analcime
BrII - 5	Site1-b	Brown/white	2,84	analcime, mineral
BrIII - 2	Site1-c	transparent/shiny	1,89	Mica
BrIII - 3	Site1-c	transparent/shiny	1,82	mica
EBI - 2	Site6-a	yellow/brown	1,20	organic
EBI - 6	Site6-a	yellow/beige	0,73	unknown (dolomite, mineral?)
EBI - 12	Site6-a	grey	1,86	organic, protein
EBI - 18	Site6-a	yellow oval shaped	0,79	Mica (most likely organic) #1 (Biotite) 0,081 wt%
EBI - 21	Site6-a	transparent	1,58	Organic, oli
EBI - 23	Site6-a	dark grey	1,35	unidentified organic
EBI - 28	Site6-a	white/light grey	1,09	unidentified organic
EBI - 33	Site6-a	shiny,metallic, dark	1,01	Biotite, Mica, Potassium Magnesium Aluminium Silicate
EBI - 36	Site6-a	white/beige	0,94	Montmorillonite, clay
EBI - 37	Site6-a	white/beige	1,07	Montmorillonite, clay
EBI - 45	Site6-a	transparent, shiny	1,35	Mica
EBI - 47	Site6-a	transparent,metallic, shiny	1,19	Mica
EBI - 52	Site6-a	transparent, metallic	1,14	Mica
EBI - 54	Site6-a	red	0,91	Kaolinite
EBI - 56	Site6-a	beige/yellow	1,15	Hollandite
EBI - 59	Site6-a	red, matt	1,92	Talc Desi, soapstone
EBI - 64	Site6-a	light brown	2,53	plant, rauwfina serpentina
EBI - 68	Site6-a	brown/transparent	1,44	plant matter
EBI - 83	Site6-a	light red, oval	0,82	Mica
EBI - 84	Site6-a	grey	1,04	Clay

EBI - 109	Site6-a	grey/dark grey white, w/smaller darker spots	1,05	Clay
EBI - 111	Site6-a	multi coloured	1,19	glass, silica
EBI - 97	Site6-a	metallic, yellow, grey	0,87	mica (biolite)
EBI - 121	Site6-a	dark grey/black	1,93	Soil Fines, Bentonite
EBI - 123	Site6-a	metallic, yellow, grey	1,92	mineral, jarlite
EBI - 124	Site6-a	bright green	1,50	mineral/metal
EBI - 127	Site6-a	transparent, glass	0,88	mica
EBI - 155	Site6-a	dark grey	0,44	Soil Fines, Albite
EBI - 162	Site6-a		0,85	Analcime
BR 1 -2	Site1-a		0,95	fiber, exlude from FTIR analysis
EBI - 15	Site6-a	fibre, red and transparent	7,27	fiber, exlude from FTIR analysis
EBI - 31	Site6-a	fibre, white/transparent	x	fiber, exlude from FTIR analysis
EBI - 62	Site6-a	blue fibre transparent, blue and red fibres	5,35	fiber, exlude from FTIR analysis
EBI - 63	Site6-a	blue/green fibre	7,07	fiber, exlude from FTIR analysis
EBI - 69	Site6-a	red fiber	7,53	fiber, exlude from FTIR analysis
EBI - 70	Site6-a	fibres, transparent	5,91	fiber, exlude from FTIR analysis
EBI - 72	Site6-a	fibre, transparent	5,54	fiber, exlude from FTIR analysis
EBI - 73	Site6-a	fibre, transparent	19,61	fiber, exlude from FTIR analysis
EBI - 74	Site6-a	fiber, transparent		fiber, exlude from FTIR analysis
EBI - 87	Site6-a	teal/blue-green fibre	10,26	fiber, exlude from FTIR analysis
EBI - 96	Site6-a	blue/teal fibre	1,03	fiber, exlude from FTIR analysis
EBI - 151	Site6-a		0,43	fiber, exlude from FTIR analysis



Brll - 1	Site1-b	Brown	1,89	too thin to sample
Brll - 2	Site1-b	white	1,52	too hard to sample, broke under pressure
Brll - 3	Site1-b	brown	2,26	nothing
Brll - 6	Site1-b	transparent	1,03	particle broke into too small fragments, too low match
EBI - 4	Site6-a	yellow/beige	1,56	nothing
EBI - 13	Site6-a	white/beige	0,95	broke in process
EBI - 14	Site6-a	metallic, silver/black shiny	1,46	nothing
EBI - 25	Site6-a	white	0,38	unknown, too small sample
EBI - 27	Site6-a	grey	0,43	Sample too small
EBI - 29	Site6-a	white w/two dark particles	0,38	not analysed, too small?
EBI - 43	Site6-a	white/beige	0,41	too small to get a reading
EBI - 50	Site6-a	metallic, dark/transparent	0,65	too small to get a reading
EBI - 60	Site6-a	white/beige	0,37	too small to get a reading
EBI - 61	Site6-a	white	0,94	broke while under compression, did not get a reading
EBI - 90	Site6-a	beige	0,52	too small to get a reading
EBI - 98		grey	0,43	too small to read
EBI - 22	Site6-a	yellow/brown	2,93	Gum?
EBI - 34	Site6-a	black and white clusters	1,54	Frankincense Resin, Gum Thus
EBI - 48	Site6-a	white on one side	1,36	paper, processed cellulose
EBI - 49	Site6-a	beige/tan	2,01	Bag Fibre, Rauwolfine Serpentina, Tobacco Marlboro Lights
EBI - 55	Site6-a	grey, matt	1,06	Black pigment, dmc2#10201, complex FE(II)
EBI - 80	Site6-a	white/yellow	1,07	Sand-Tex (Sand Finish Paint Additive)
EBI - 88	Site6-a	tan, light brown	6,06	Bag Fibre, Rauwolfine Serpentina, Tobacco Marlboro Lights
EBI - 132	Site6-a	red/pink w/yellow spots	1,63	Adhesive, Gasket Sealer, Non-Hardening, Clay Filled Polymer
EBI - 153	Site6-a	red w/yellow circles	0,69	Adhesive, Gasket Sealer, Non-Hardening, Clay Filled Polymer

EBI - 5	Site6-a	silver/metallic	2,17	lost
EBI - 53	Site6-a	grey	0,79	lost
EBI - 58	Site6-a	dirty yellow/beige	0,81	lost
EBI - 81	Site6-a	transparent, metallic	0,95	lost
EBI - 92	Site6-a	dark red	0,40	lost
EBI - 119	Site6-a	light grey/diry yellow	0,70	lost
EBI - 71	Site6-a	yellow	1,28	Poly(pentadecyl isocyanate)

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