MICROPLASTIC POLLUTION IN THREE RIVERS IN SOUTH EASTERN NORWAY















M-1572 | 2020

]

MICROPLASTIC POLLUTION IN THREE RIVERS IN SOUTH EASTERN NORWAY

Claudia Lorenz¹, Jane K. Dolven^{2,3,*}, Nina Værøy², Diana Stephansen¹, Stein B. Olsen² and Jes Vollertsen¹

¹ Aalborg University, Thomas Manns Vej 23, 1-243, 9220 Aalborg, Denmark

² COWI AS, Karvesvingen 2, 0579 Oslo, Norway

³ University of South-Eastern Norway, Kjølnes ring 56, 3901 Porsgrunn, Norway

* Project manager and corresponding author

Front page photo: Jane K. Dolven Photos in text: Claudia Lorenz (labwork) and Jane K. Dolven (fieldwork)

PROJECT NO.	DOCUMENT NO.				
A125334	01				
VERSION	DATE OF ISSUE	DESCRIPTION	PREPARED	CHECKED	APPROVED
01	2020.02.18	Report	JKD & CL	JV & SBOL & DS	JKD

CONTENTS

Summ	hary	7
1	Introduction	11
2	Material and methods	12
2.1	Choice of rivers	12
2.2	Sample collection	16
2.3	Sample preparation and analysis	20
3	Results and discussion	24
3.1	Microplastic in filtered water samples and sediment ("full analysis")	24
3.2	Results from water samples analyzed by ALS and Eurofins	31
3.3	Microplastic in the >300µm fraction	34
3.4	Evaluation of methods: Strengths and weaknesses (limitations)	37
4	Conclusions	43
5	References	45
6	Acknowledgements	47
7	Appendix	48
7.1	Results from ALS	48
7.2	Results from Eurofins	50
7.3	Results from AAU	52

Summary

The objective of this study was to investigate the occurrence of microplastic pollution in three differently influenced rivers in Norway, how the microplastic concentration vary with water flow and to test different methods for sampling and analysis in order to suggest the most suitable method for future river investigations.

To achieve these goals, water samples were collected monthly from May to October 2019 in three rivers: Akerselva (city influenced), Hobølelva (agriculture influenced) and Gryta (pristine river) in the southeastern part of Norway. Water flow was continuously recorded upstream each sampling site by the Norwegian Water Resources and Energy Directorate (NVE). About 1000 liters (1 m³) of water were filtered through 300 μ m- and 10 μ m-filters in each river at each sampling time (eight in total). During the May sampling additional unfiltered water samples were collected and sent to ALS and Eurofins (the two largest commercial laboratories in Norway) for analysis. From August to October additional water samples were collected using a manta trawl (300 μ m mesh size). Sediment traps were deployed at the river floor for 2-3 months (May-August) and grab samples were collected in June. The analysis of the filtered water samples and sediments were done at Aalborg University using ATR-FT-IR, μ FT-IR imaging and water samples were analyzed with Py-GC-MS at NORCE.

The water analyses show that the concentration of microplastic particles (no. MP/m³ between 10-5000 µm, blanc corrected) was highest in the city influenced river Akerselva (1069 MP/m³), followed by the pristine river Gryta (140 MP/m³) and the agricultural impacted river Hobøl (138 MP/m³). Akerselva also had the most diverse composition of polymers (6 types), followed by Hobøl (5) and Gryta (2). At Akerselva polypropylene (PP) was the most abundant polymer type, followed by polyethylene (PE), polyamide (PA), polystyrene (PS), polyurethane (PUR) and some other less common polymer types (e.g. acrylics). Results from the Py-GC-MS analysis of the water samples taken at Akerselva, Hobølelva and Gryta in May 2019 (provided by NORCE) show that 0.50 µg PVC per L was detected in Hobølelva. For Gryta, PE and PVC were found in mass concentrations of 0.17 µg/L and 0.28 µg/L, respectively. In the sample from Akerselva PE and PVC were detected with mass concentrations of 0.47 µg/L and 2.27 µg/L a signal of D-limonene was detected indicating the presence of natural rubber or car tire particles in a mass concentration of 1.8 µg/L. The latter was not found in Hobølelva and Gryta.

A comparison of number of particles in the two investigated fractions 10-300 μ m and >300 μ m in the May water samples, reveals that the number was much higher (150-530 times) in the smaller fraction than in the larger. Eleven different sampling times spread out from May to October, under various water flow conditions, showed that the microplastic concentrations (>300 μ m) in the three rivers were low, ranging from 0-4.0 particles per m³. This shows that investigating the larger

fraction alone does not give a realistic image of what is transported in the rivers. To get the full picture one also need to investigate the smaller fraction, especially when correlating with water flow.

Regarding sediments, Akerselva has the highest number of microplastic particles (in the 10-300 μ m fraction), i.e. 1.5×10^4 (4.6 mg/kg) in the grab sample and 1.1×10^5 (18.4 mg/kg) microplastics per kg (dw) in the sediment trap sample. In comparison, the microplastic concentration at Hobølelva was 2.5×10^3 microplastics per kg (dw) (5.1 mg/kg). For the sediment trap sample from Akerselva polypropylene (PP), polyethylene (PE), cellulose acetate (CA), and polystyrene (PS) were the most common polymers, while for the grab sample PP was making up more than half of the sample followed by PS and PE and with minor contributions from polyamide (PA), polyester (PEST), polyvinyl chloride (PVC), acrylics, CA and some other polymers (e.g. acrylonitrile butadiene styrene (ABS)). For Hobølelva there were fewer polymers present, with PEST, PE and PP being the most common. The sediment trap sample from Gryta was not analyzed due to a complex matrix that hampered the sample preparation. Our result for the sediments in combination with our results for the water analysis reveal that the city influenced river, which had the highest anthropogenic impact, thus a magnitude of potential sources, has the highest microplastics concentration.

To gain a full overview on the microplastic pollution in the river environment all compartments (surface water, water column and sediments) need to be investigated. What strategy to use regarding sampling methods and analyses, depends on the focus. A microplastic-screening to address the baseline of the amounts and type of microplastics is useful before studying long term trends.

To allow for quantitative analysis of the smaller fraction (10-300 µm) we recommend using a filtration unit and filter 1 m³ of water. If the focus is to analyze the larger fraction, a manta trawl allows to filter a large volume of water which is needed to provide representative data for the larger microplastics. Due to accumulation over time microplastic concentrations are higher in sediments. Sampling and analysis of suspended particles in the water column by use of sediment traps and river sediments by use of grabs, allow for monitoring of denser particles than those found in the surface water.

To allow for a reliable quantification of microplastics a spectroscopic imaging approach is recommended (e.g. FT-IR). This would enable polymer type identification and would provide the necessary data on microplastic concentrations and size distribution. This can be supplemented by thermal chemometric analyses (e.g. Py-GC-MS) for identification of rubber. However, whichever analysis method is chosen, it relies on an efficient sample preparation to reduce matrix effects.

Sammendrag

Formål med undersøkelsen har vært 1) å kartlegge tilstedeværelse og konsentrasjoner av mikroplast i vann og sedimenter i tre elver med ulike typer nedbørsfelt, 2) undersøke om det er en sammenheng mellom mengde mikroplast i elvevannet og vannføring (nedbørsmengder), samt 3) teste ulike typer prøvetakings- og analysemetoder for å vurdere hvilken metodikk som egner seg best for undersøkelse og overvåking av mikroplast i Norske elver fremover.

Vannprøver ble samlet inn månedlig fra mai til oktober (2019) i Akerselva (bypåvirket elv), Hobølelva (jordbrukspåvirket elv) og Gryta (upåvirket referanseelv), alle tre lokalisert i sørøst-Norge. Vannføring ble kontinuerlig målt oppstrøms prøvetakingspunkter av Norges Vassdrags- og energidirektorat (NVE). Under hver vannprøvetaking ble det filtrert ca. 1000 liter (1 m³) vann gjennom 300 µm- and 10 µm-filter. I mai ble det samtidig samlet inn vannprøver som ble sendt til analyse hos ALS og Eurofins (de to største kommersielle laboratoriene i Norge). I perioden august til oktober ble det foretatt ytterligere prøveinnsamlinger med manta-håv (maskevidde 300 µm). Sedimentfeller ble satt ut på bunnen i alle tre elvene for å samle suspendert materiale i 2-3 måneder. Det ble også innhentet overflatesedimentprøver med liten grabb. Analyser av filtrerte vannprøver (inklusive manta-prøver) og sedimentprøver ble gjennomført med bruk av ATR-FT-IR, µFT-IR av Aalborg Universitet og Py-GC-MS ble utført hos NORCE.

Undersøkelse av mikroplastpartikler i vann (10-5000 µm) i mai viser at konsentrasjonen (antall mikroplast partikler/m³, blank korrigert) var høyest i den bypåvirkede Akerselva (1069 partikler/m³), etterfulgt av upåvirkede Gryta (140 partikler/m³) og deretter den den jordbrukspåvirkede Hobølelva (138 partikler/m³). Det var også Akerselva som hadde den høyeste diversiteten av polymerer (6 forskjellige typer), fulgt av Hobøl (5 typer) og Gryta (2 typer). Følgende polymerer (i avtagende konsentrasjoner) ble funnet i Akerselva: Polypropylene (PP), polyethylene (PE), polyamide (PA), polystyrene (PS), polyurethane (PUR) og andre mindre vanlige typer (f.eks. akryl). Resultater fra Py-GC-MS analysen (utført av NORCE) viser at det i prøven fra Akerselva ble detektert 0.47 µg/L PE and 2.27 PVC µg/L. I Hobølelva ble det funnet 0.50 PVC µg/L, og i Gryta 0.17 PE µg/L and 0.28 PVC µg/L. I Akerselva-prøven ble det også detektert 1.80 µg/L naturlig gummi/bildekk basert på tilstedeværelsen av indikatoren D-limonene. Sistnevnte ble ikke detektert i Hobøl og Gryta prøvene.

En sammenlikning av de to undersøkte størrelsesfraksjonene i vann, 10-300 µm og >300 µm, i mai viser at antallet partikler i den minste fraksjonen var mye høyere (150-530 ganger) enn i den største fraksjonen. I perioden mai til oktober ble det tilsammen gjennomført 8-11 vannprøvetakinger under ulike vannføringer i de tre elvene. Mikroplastkonsentrasjonen for den største fraksjon (>300 µm) var lav (varierte fra 0-4 partikler/m³), og for lav til å korrelere mot vannføringen i elvene. Dette understreker viktigheten av å analysere hele fraksjonen (dvs. også de minste partiklene) dersom man ønsker å korrelere mikroplast i elvevann mot vannføring.

Undersøkelse av sedimenter viser at det i Akerselva i 10-300 µm fraksjonen (blank korrigert) ble funnet 1.5×10^4 mikroplast partikler per kg tørrvekt (4.6 mg/kg) i bunnsedimentet (grabbprøve) og 1.1×10^5 (18.4 mg/kg) i sedimentfelleprøven (begge resultater ekstrapolert fra subprøver). Til sammenlikning var mikroplast-konsentrasjonen i grabbprøven fra Hobølelva 2.5 × 10³ mikroplast partikler per kg tørrvekt (5.1 mg/kg) (data ekstrapolert fra subprøve).

I sedimentfelle prøven fra Akerselva var polypropylene (PP), polyethylene (PE), cellulose acetate (CA), og polystyrene (PS) de mest vanlige polymertypene. I grabbprøven utgjorde PP mer enn halvparten av mikroplastinnholdet i Akerselva, etterfulgt av PS og PE samt mindre konsentrasjoner av PA, PEST, PVC, acrylics, CA og noen andre polymertyper (e.g. acrylonitrile butadiene styrene, ABS). Antallet polymerer i sedimentfellen i Hobøl var lavere enn i Akerselva, med polyester (PEST),

polyethylene (PE) og polypropylene (PP) som de vanligste. Sedimentet i Gryta ble ikke analysert pga. vanskelig matriks.

Valget av prøvetakingsmetode og analyser, avhenger av formål med undersøkelsen. Det anbefales at en kartlegging av mengde og typer av mikroplast i elvesystemet bør gjennomføres før en overvåking av trender/variasjoner igangsettes. For å få en full oversikt over mikroplastforurensningen, bør det under kartleggingsundersøkelsene tas prøver fra både overflatevann, vannsøyle og sedimenter. For å gjøre en kvantitativ analyse av den mindre fraksjonen (10-300 µm) anbefales det bruk av en eller flere filtreringsenheter og filtrere 1 m³ vann. Hvis ønsket er å analysere den større fraksjonen (>300 µm) vil en manta-håv kunne filtrere et stort volum vann relativt raskt, og med det øke representativiteten for dataene (redusere LOQverdien). Men manta-håven samler ofte mye organisk materiale som gjør at preparering av prøvene før analysene blir tidkrevende og dyre. Prøvetaking og analyse av suspenderte partikler i vannsøylen ved bruk av sedimentfeller og prøvetaking av bunnsediment ved å bruke grabb (evt. kjerneprøvetaking dersom mulig), bør gjennomføres for å kartlegge mikroplast partikler med større tetthet enn de som transporteres i overflaten. Det ble i inneværende studie testet ulik analysemetodikk (ATR-FT-IR, µFT-IR, Py-GC-MS og SEM). Våre erfaringer viser at µFT-IR gir de beste resultatene. Metoden har høy nøyaktighet for identifikasjon av polymerer, kan detektere en rekke forskjellige typer polymerer ned til 10 µm samt brukes til å estimere massen av hver polymer i prøven. Metoden er ikke egnet for deteksjon av naturlig gummi/bildekk (pga. "carbon black"), så informasjon om dette bør undersøkes med f.eks. Py-GC-MS. Andre studier har vist at Raman-spektroskopi også en egnet metode for analyse av mikroplast, men denne ble ikke testet i inneværende studie.

1 Introduction

Plastic is an important part of our everyday life. It is used for packaging (39.9%), in buildings and constructions (19.8%), automotive parts (9.9%), electrical and electronics (6.2%), households, leisure, and sports (4.1%), farming (3.4%) and other products (including appliances, furniture, makeup etc. 16.7%). According to PlasticEurope (2019) almost 360-million-ton plastic was produced globally in 2018. A lot of the waste plastic is either recycled or used as an energy source when burned, but unfortunately a large undefined proportion is also spread into the environment as macro- and microplastics. The amount of miss-managed waste is increasing with an increasing world population. The demand for proper recycling- and waste management is therefore urgently growing, particularly in undeveloped countries where such systems are partly or completely lacking.

Much of the plastic ends up in the ocean where it poses a threat to the marine ecosystem as well as others (e.g. seabirds, humans) that harvest from the ocean. Rivers have been found to be one of the main modes of transportation of plastic from urban areas to the ocean. It is therefore of great importance to investigate and monitor the plastic concentration in rivers and take actions where needed.

Microplastic pollution has been given increased attention lately as it has been found ubiquitous in aquatic environments, soil, biota, snow, air, drinking water and sewage sludge (Bergmann et al., 2019; Piehl et al., 2018; Mintenig et al., 2019; Vianello et al., 2019; Vollertsen and Hansen, 2017). The sources for microplastics are vast. Microplastics are commonly understood as synthetic solid particles of polymeric matrix, with size ranging from 1 µm to 5mm, regular or irregular shape, of either primary or secondary manufacturing origin, which are insoluble in water (Frias and Nash, 2019). However, no internationally recognized definition of microplastics exists. It is characterized by size, shape, texture, color and chemical composition (Rezania et al., 2018; Noren, 2007), and is often divided into primary microplastics (pellets, flakes and powder) and secondary microplastics (derived from large particles due to breakdown/weathering).

The knowledge about microplastic sources, concentration, and fate within the fluvial system is still limited (Blettler et al., 2018). Recent studies focusing on microplastics in river water and sediments revealed that microplastics in the Rhine River, one of the largest rivers in Europe, result from fragmentation of plastic debris, pellets or components of consumer- and industrial products (Mani et al. 2015). Mani et al. (2019) showed that microplastics were temporarily or permanently inhibited from migrating downstream by retention in sediments or ingestion by organisms.

A standardized methodology for sampling, sample preparation and analysis does not yet exist. This makes it very difficult to compare results from different studies. Studies have also shown that visual identification approaches using morphological criteria alone often have led to significant errors, which underlines the importance of using chemical structure-based identification methods (Käppler et al. 2018). A standardized methodology is therefore urgently needed. Such a standardization should be based on critical comparison and testing of different sampling and analysis methods. Data presented in the present study are limited, but may be one small step in the right direction of formalizing future national river monitoring programs in Norway.

2 Material and methods



2.1 Choice of rivers

Figure 1. Locations of the investigated rivers.

All three investigated rivers (Akerselva, Hobølelva and Gryta) are located in the southeastern part of Norway (Figure 1). They were selected due to their different surroundings and potential sources of microplastic particles. Akerselva is an urban river, while Hobøl is possibly impacted by surrounding farming activities. Gryta was selected as a pristine reference river. A more detailed description of the three rivers can be found below.

2.1.1 Akerselva

The river Akerselva receives water from "Nordmarksvassdraget", a 230 km² large drainage area upstream of lake Maridalsvannet (the main drinking source of Oslo). The outlet of Maridalsvannet makes the start of Akerselva. The river winds through Oslo, the largest city of Norway, hosting about 685 000 inhabitants. On its way down to the Inner Oslofjord, Akerselva receives runoff water from roads and dense surfaces. According to the municipality of Oslo there are about 100 storm-drain-outlets to Akerselva, as well as 40 combined-sewer-overflow-outlets (from the Oslo sewages systems).

The fishing spot next to "National scene of Dance" was chosen as the sampling site (Figure 2). This site is downstream from two of the most trafficked roads in Oslo (i.e. Ring 2 and Ring 3). Runoff water from roads and dense surfaces as well as wastewater are known transport mediums for

microplastics. The sampling site was chosen upstream of any saltwater intrusion from the inner Oslofjord.

The Norwegian Water Resources and Energy Directorate (NVE) has two stations where water flow is measured in the river, i.e. one upstream the sampling site at the dam of Maridalsvannet and one about 100 meters downstream from the sampling site (Figure 3). The water flow in Akerselva is partly controlled by the dam.



Figure 2. Sampling site in Akerselva located next to the "National scene of Dance".



Figure 3. Akerselva in Oslo and the location of sampling site (red star). The arrows show the locations of the NVE's "water flow" stations.

2.1.2 Hobølelva

The river "Hobølelva" has its origin in Mjær in Enebakk and ends up in lake Vansjø. The drainage area to the river is about 336 km² and covered by glacial deposits and exposed bedrock. The river is meandering an area mainly used for forestry and agriculture (19%). The grown crops consist mainly of grain (90%). Sewages sludge is often used as a fertilizer and soil conditioner. Hobølelva contains a lot of fine particles (silt and clay smaller than 10 μ m) suspended in the water giving the river a brownish color (Figure 4). More information about Hobølelva can be found in Bioforsk Rapport Vol. 2 Nr. 129-2007.

The Norwegian Water Resources and Energy Directorate (NVE) is continuously measuring the water flow at Høgfoss a couple of kilometers upstream of the sampling site (Figure 5).



Figure 4. Hobølelva close to the sampling site at Kurefossen.



Figure 5. Hobølelva and the sampling site (red star). The arrow shows the position of the NVE's "water flow" station at Høgfoss.

2.1.3 Gryta

Gryta is a small river/creek that originates in Nordmarka north of Oslo and ends up in the lake Maridalsvannet (Figures 6-7). Since Maridalsvannet is the drinking water reservoir for 90% of Oslo's population there are severe restrictions for use of the surrounding area to prevent pollution of the drinking water. Gryta was therefore chosen as a reference river in this project, as it is very little influenced by runoff from industry, sewage systems, landfills and roads/dense surfaces. The drainage area to Gryta is only 7 km². The Norwegian Water Resources and Energy Directorate (NVE) is measuring the water flow upstream the sampling site in Gryta. The water flow record goes back to 1967.



Figure 6. The sampling site in Gryta with NVE's "water flow" station in the background.



Figure 7. Location of sampling site in Gryta (red star). The arrow shows the position of the NVE's "water flow" station.

2.2 Sample collection

A list of sampling dates and methods used to collect the different matrixes (water and sediment) in the three rivers are shown in Table 1.

Table 1. Sampling dates and methods used to collect the different matrixes (water and sediment) in the three investigated rivers (Akerselva, Hobøl and Gryta).

AAU - filtered water samples

Date	Sampling and analysis	Akerselva	Hobøl	Gryta
22-23.05.2019	300 & 10 µm filters: ATR FT-IR/µFT-IR/Py-GC-MS	х	х	х
05.06.2019	300 µm filter: ATR FT-IR	х	х	
26-27.06.2019	300 μm filter: ATR FT-IR	х	х	х
8-9.07.2019	300 μm filter: ATR FT-IR	х	х	
12-13.08.2019	300 μm filter: ATR FT-IR	х	х	х
10-11.09.2019	300 µm filter: ATR FT-IR	х	х	х
9-10.10.2019	300 µm filter: ATR FT-IR	х	х	

Other water samples

Date	Sampling and analysis	Akerselva	Hobøl	Gryta
22-23.05.2019	Water sample ALS: SEM	х	х	х
22-23.05.2019	Water sample Eurofins: Py-GC-MS	х	х	х
12-13.08.2019	Manta 300 µm: ATR FT-IR	х	х	х
29.08.2019	Manta 300 µm: ATR FT-IR	х	х	х
20.09.2019	Manta 300 µm: ATR FT-IR	х	х	х
22-23.10.2019	Manta 300 µm: ATR FT-IR	х	х	х

Sediment samples

Date	Sampling and analysis	Akerselva	Hobøl	Gryta
26-27.06.2019	Grab sampling: ATR FT-IR/µFT-IR	х	х	х
22.05.2019	Start of sediment trap sampling			х
26-27.06.2019	Start of sediment trap sampling	х	х	
12-13.08.2019	End of sediment trap sampling: ATR FT-IR/µFT-IR	х	х	х

2.2.1 Water sampling: AAU system

Water sampling was done by filtering 1 m³ of water through a filtering system made by Aalborg University (AAU; Figure 8). The filtering setup consisted of three filter containers and a flow meter to log the flow. The water was first lead through a container holding a 300 μ m filter and then split and forwarded to the next two containers holding one 10 μ m filter each. Filtering 1 m³ took about 3 hours at each site. The 10 μ m filters were changed after filtering about 500 liters to prevent clogging. Sampling blanks for collection of possible airborne microplastic particles were applied when opening the filter containers (changing filters) and used as a control.



Figure 8. Filtering setup with three filtering containers and a flow meter (system made by AAU)

2.2.2 Water sampling: Manta trawl

Additional water samples were collected using a manta trawl (300 µm mesh size; Figure 9). The Manta trawl is used for taking samples from the water's surface (0 to ca. 10 cm depth). The trawl contained a mechanical flow meter to be able to calculate the filtered water volume. The manta trawl was deployed for 30-45 minutes at each site each time. Number of revolutions was noted and the filtered volume (i.e. the volume passing through the plankton net) was calculated using the following equation:

Water volume = number of revolutions x 0.3 x net opening area (m²) x 1000



Figure 9. Manta trawling in Gryta.

2.2.3 Water sampling: Un-filtered (untreated)

Both ALS and Eurofins, the two largest commercial labs in Norway, are offering microplastic analysis. Their respective analysis are described below. One 250 mL glass bottle and four 1-liter glass bottles were filled with water from each sampling site, for the analysis at ALS and Eurofins respectively. The bottles were rinsed in river water, before held upstream and fully submerged during the water collection. The lids were screwed back on immediately after sampling to reduce the risk of contamination. One empty bottle was kept open during the water sampling at each site and used as a control (blank) for the Eurofins analysis.

2.2.4 Sediment sampling: Grab sampling

A small Van Veen grab (126 cm²; Figure 10) was used to collect the upper 3-5 cm of the river sediments. At each sampling site 10 grab samples were collected at different spots within a radius of about 10 m. This was done to include samples from different types of sedimentation environment (to get a variety of particle sizes), in order to increase the chance of retrieving a representative sediment sample from the rivers. All 10 grab samples were gathered and mixed in a bowl and transferred to three glass jars for transportation to the lab.



Figure 10. Van Veen grab used for sampling river sediments.

2.2.5 Sediment sampling: Trap sampling

COWIs sediment traps, specially designed for rivers, were used to collect suspended particles (Figure 11-12). In the smallest river (Gryta) it was difficult to find a spot deep enough to place the sediment trap below water. Due to varying water level in the Gryta river (and possibly periods without sampling sediments due to shallow water), this trap was therefore kept in the field for one month longer (May-August) than the traps in the other two rivers (June-August).



Figure 11. The sediment trap placed in Gryta.



Figure 12. Sediment trap in Gryta after harvesting sediment for about three months.

2.3 Sample preparation and analysis

2.3.1 Target size of particles

For the AAU analysis (filtered water and sediments) the target size for the microplastic particles ranged from 10 to 300 μ m as quantitative analysis and >300 μ m as qualitative analysis. Below 300 μ m the analysis was done by μ FT-IR imaging while microplastic particles larger 300 μ m were analyzed by ATR-FT-IR upon selecting potential plastic particles from the matrix.

Most microplastic materials can be identified by the FT-IR method, however, rubber from for example car tires cannot be detected due to their content of carbon black. Indicating the presence of rubber from car tires has therefore been done by Py-GC-MS analysis.

2.3.2 Level of quantity (LOQ)

The microplastic concentration in water from heavily polluted rivers can be expected to be below 100-1000 µg/m³. These figures are deducted from the fact that such concentrations have been reported in stormwater runoff and treated wastewater discharges, and it seemed reasonable to assume lower concentrations in river water. In pristine waters, the concentration will probably be 2-3 orders of magnitude lower. This corresponds to number of particles that we expect to find, between 1x10¹ and 1x10⁴ pieces of plastic per m³ of water when analyzing for the small particle sizes down to 10 µm. The number of particles found, grows exponentially when looking at smaller sizes, because - by nature - there are always many more small particles than large particles. If particles down to 300 µm are counted, we can expect a factor of 100 to 1000 more particles in the smaller fraction (<300 µm). This knowledge of what can be expected of plastic in the samples determines how much water we need to analyze to obtain a reasonable statistical certainty, as one must find a reasonable number of plastic particles to be able to express for example which polymer groups and sizes are present. With respect to the LOQ in our analyses, sample volume is also important. The unavoidable contamination of a sample happens during the different process steps. For example, a plastic particle could deposit on the filter from the surrounding atmosphere during the sampling. If one for example takes only 1 L of sample, this would correspond to a contamination of 1 particle per liter. However, if one takes 1000 L, the LOQ becomes 1000 times lower because now the contamination corresponds to 0.001 particles per L. Regarding sediments we would expect concentrations of at least 1×10^3 particles per kg or $1 \times 10^2 \mu g/kg$ (Liu et al. 2019) which would thus result in a similar LOQ.

2.3.3 Filtered water samples (AAU)

Due to very few particles on the 300 μ m-filters only a short version of the sample preparation was done with a surfactant, sodium dodecyl sulfate (SDS), and an oxidation with hydrogen peroxide (H₂O₂). After this the particles were picked out directly under a stereo microscope, measured, weighted and analyzed using an ATR-FT-IR. If possible three spectra were acquired for each particle. The obtained IR spectra were then compared against an openly available spectral library (https://simple-plastics.eu/download.html). The identification was accepted if the similarity score was more than 70%. If the match was between 60–70% an expert judgement was needed to approve or reject the results (Hanke et al., 2013). Below 60% the results were rejected, and a new analysis had to be done.

For the particles 10–300 µm the water samples taken in May 2019 were treated first with a surfactant, sodium dodecyl sulfate (SDS), followed by enzymes, protease and cellulase, oxidized with Fenton's reagent and density separated (Figure 13). A protocol for this sample treatment is described in Löder et al. (2017). Additionally, a procedural blank was run together with the water samples following the same treatment steps to estimate in-lab contamination and ensure quality of the received data. To minimize this contamination all sample preparation was performed in a laminar flow cabinet or a laboratory fume hood (for the oxidation).

The cleaned sample was concentrated in 5 mL of 50% ethanol, and suitable sub-samples (see Appendix 7.3 Table A1) were deposited on an infrared transmissive window and scanned using a μ FT-IR imaging system (Figure 14), i.e. a microscope equipped with a focal plane array (FPA) detector consisting of 128x128 detector elements and integrated with a FT-IR spectroscope. The spectroscope sends the IR light through the microscope, and the either absorbed or reflected IR light is gathered by the FPA. The FPA is built up of pixels, which then each obtain an IR-spectrum. In a typical operation modus, the resolution per pixel is 5.5 μ m. One scan hence creates a map of 128x128 pixels representing an area of (for example) 704 μ m. The machine then repeats this for the neighboring area, creating a mosaic of pixels. Typically, a 10x10 mm scan, creates more than 3.2 million pixels, each holding an IR-spectrum. At the same time, it obtains a visual image of the area scanned. A visual image of a scan of a circular substrate of 10 mm diameter is shown in Figure 14 (middle photo, with right photo showing a zoomed-in part).



Figure 13. Sample preparation steps.

The IR spectra were transferred to a software developed at Aalborg University and Alfred Wegener Institute, called siMPle (https://simple-plastics.eu/index.html). This software compares all the spectra with an extended library of reference spectra, and identifies which particles are actually microplastics and which are natural materials. The illustration in the lower right of Figure 14 shows part of an outcome of such an analysis. The program also calculates the size of each particle in 2 dimensions, its surface area and estimates its volume. The particle mass was deducted from the IR identification of the polymer type and the calculated volume. This was done for each particle on the substrate. This resulted in a list of particles, their size, volume and mass, and polymer type. For

each sample the microplastic concentration as numbers per sample/m³ and mass per sample/m³ was found. The polymer distribution onto mass and particle numbers, as well as the size distribution were also calculated.

Another approach to get mass related data on microplastics is Py-GC-MS. Therefore, sub-samples of the same three purified and concentrated samples as well as the procedural blank were filtered onto muffled 1.2 μ m glass fiber filters (see Appendix 7.3 Table A1). These filters were folded into tin cups and send to NORCE for Py-GC-MS analysis according to Gomiero et al. 2019. An additional muffled filter was run to account for any background contamination from the instrument. Mass concentrations in μ g/L were provided for six different polymer types (PE, PP, PS, PVC, PA66, and PMMA) and natural/car tire rubber.



Figure 14. Identifying the microplastics in a prepared sample.

2.3.4 Sediment samples (AAU)

The collected sediment samples from each river were treated with peroxide (5% H₂O₂-solution) to oxidize organic material. The sample was then dried and transferred to a density separation column where a solution of sodium polytungstate (SPT, density: $1.7-1.8 \text{ g/cm}^3$) was added. After the separation process the sample was filtered on 300 µm- and 10 µm-stainless steel filters. Then the >300 µm fraction was treated with H₂O₂. The fraction 10–300 µm was treated with SDS, enzymes and an oxidation was performed followed by another small scale density separation with SPT (Figure 15). As quality control a procedural blank was run together with the sediment samples and sample preparation was performed in laminar flow cabinet or laboratory fume hood whenever possible. The larger fraction (>300 µm) was analyzed using ATR-FT-IR method as described earlier, while the 10–300 µm fraction was concentrated in 5 mL of 50% ethanol, sub-sampled (see Appendix 7.3 Table A1) and scanned using a µFT-IR imaging system. A detailed sample preparation protocol is found in Liu et al. (2019).



Figure 15. Sediment trap sample from Akerselva (left), after pre-oxidation (middle), added to a density separation funnel (right).

2.3.5 Water samples (ALS)

ALS has developed a method for analysis of microplastics in water. The amount of water needed for the analysis is only 250 mL. Before the analysis organic material is degraded using HNO₃ (COWI note; with the risk of also degrading the plastic). The water is then filtered through a polycarbonate filter, covered with gold before applying to a scanning electron microscope (SEM). The SEM is equipped with an energy dispersive detector for the identification of elements with an atom number >5. Particles between 10 μ m and 1 mm are identified, and number of particles per liter are calculated and reported according to the following groups of polymers. Example given:

- > org particle e.g. PP, PE, PS
- > org particle e.g. PMMA, PUR, PET
- > org particle with silicon e.g. rubber
- > org particle with chlorine e.g. PVC
- > org particle with fluorine e.g. PTFE

2.3.6 Water samples (Eurofins)

The methods used by Eurofins are described in Eurofins (2019). Microplastic particles were filtered using a filtration setup and vacuum pump. Between 250 ml and one liter of river water from each site was filtered. The filtration was done through a 27 μ m steel filter or a 0.2 μ m silicon dioxide filter. The choice of filter was depending on the amount of suspended particles in the water sample (i.e. the "cleaner" water the smaller mesh size was used). The analysis was performed using Py-GC-MS (600° C in a helium atmosphere).

Eurofins report-template includes:

- The mass concentration (µg/L) of individual polymer type (PE, PP, PS, PVC, PET, PC, PMMA, PA6) and the sum microplastic.
- > The mass concentration (μ g/L) of polyisoprene and polybutadiene.
- > Comment regarding qualitative identification of rubber (due to the presence of the indicators polyisoprene and polybutadiene.

Water samples analyzed by AAU (FT-IR), ALS (SEM) and Eurofins (Py-GC-MS) were collected the same day and can therefore be regarded as parallel samples.

3 Results and discussion

3.1 Microplastic in filtered water samples and sediment ("full analysis")

3.1.1 Water

The water samples (1000 L) taken in May 22-23, 2019 with the AAU filtration unit were analyzed for microplastics in the size fraction of 10-300 μ m as well as >300 μ m. Microplastic concentrations of the smaller size fraction were extrapolated based on the analyzed sub-sample which ranged from 3% to 15%, representing 30 L (Akerselva), 50 L (Gryta) and 150 L (Hobøl), respectively (see Appendix Table A1). Microplastic numbers and masses were blank corrected by subtracting the number or mass of each polymer type detected in the procedural blank from the respective polymer type detected in the samples (for uncorrected raw data see Appendix Table A2). As can be seen in Table 2 microplastic concentrations for the smaller size fraction ranged from 138 particles per m³ (Hobøl), to 140 particles per m³ (Gryta), to 1067 particles per m³ (Akerselva). At Akerselva polypropylene (PP) was the most abundant polymer type, followed by polyethylene (PE), polyamide (PA), polystyrene (PS), polyurethane (PUR) and some other less common polymer types (e.g. acrylics) (Table 2, Figure 16). Polymer type composition was different at the different sampling sites with Akerselva being the most divers (6), followed by Hobøl (5) and least at Gryta (2). In the larger size fraction no polymers were encountered in the sample from Gryta while two particles each were detected in samples from Akerselva and Hobøl, being one PE fragment and one polyester/polyethylene terephthalate (PEST) fiber (Table 2). In the procedural blank, which was treated simultaneously with the three river samples no microplastics were detected in the larger size fraction while concentration in the small size fraction reached 15 particles per m³ being comprised only of PEST. In terms of mass of the microplastics for the larger size fraction, the two particles identified in the sample from Hobøl was three times higher than for the two particles from Akerselva, weighing 102 µg and 32 µg, respectively. The mass of the microplastics identified in the smaller size fraction was calculated based on the volume of the particles, assuming an ellipsoid shape, and the density of the polymer type. The extrapolated and blank corrected mass of the microplastics per m³ ranged from 11.99 μg (Gryta) over 18.63 μg (Hobøl) to 54.79 μg (Akerselva). For uncorrected raw data see Appendix Table A2. In the sampling blank taken at Akerselva in May four PEST particles were identified resulting in a concentration of 40 particles per sample. This blank was not used for blank correction since it is only representative for the water sample taken at Akerselva in May 2019.

Table 2. Microplastics analyzed in water samples taken in May 22-23, 2019 divided into two size fractions (10-300 μ m and >300 μ m). Results were blank corrected by subtracting the number or mass of each identified polymer type in the procedural blank from the respective polymer types detected in the samples. Microplastic concentrations are provided for both fractions as number of particles per sample as well as mass estimation in μ g per sample which refers to 1 m³ sampled at each river.

				micro	plastic	s 10–30	00 μm pe	er san	nple		micro	plastic	s > 300 μm	per sample
sample	PE	PP	PS	PA	PEST	PUR a	crylics	CA	∑ number	∑mass [µg]	PE	PEST	∑ number	∑mass [µg]
Akerselva	267	467	67	100	0	100	33	33	1067	54.79	1	1	2	32
Hobøl	53	20	0	27	32	7	0	0	138	18.63	1	1	2	102
Gryta	120	0	0	20	0	0	0	0	140	11.99	0	0	0	0



Figure 16. Polymer composition of microplastics based on number of particles identified in the 10-300 µm fraction of samples taken at Akerselva, Hobøl and Gryta on 22-23 May 2019. Polymer composition of a procedural blank and a sampling blank taken at Akerselva are shown as well.

The results provided by NORCE for the Py-GC-MS analysis of the water samples taken at Akerselva, Hobøl and Gryta in May 2019 are presented in Figure 17 and Table A4 in the Appendix. Subsamples representing 100 L (Akerselva) or 200 L (Hobøl and Gryta) respectively (Table A1) were analyzed and detected mass concentrations per identified polymer type are extrapolated to the total sample volume and presented as mass concentration in μ g/L (Figure 17). In the sample from Hobøl PVC was detected in a mass concentration of 0.05 μ g/L. For Gryta PE and PVC were detected in a mass concentrations of 0.17 μ g/L and 0.28 μ g/L, respectively. In the sample from Akerselva PE and PVC were detected with mass concentrations of 0.47 μ g/L and 2.27 μ g/L, respectively. Furthermore, in the sample from Akerselva a signal of D-limonene was detected, which indicates the presence of natural rubber or car tire particles (Lachowicz et al. 2012), in a mass concentration of 1.8 μ g/L. D-limonene was not found in Hobøl or Gryta. No amounts of any of the targeted polymer types could be detected in the procedural blank which was processed as contamination control during the whole procedure of sample preparation (Figure 17). Also no microplastics or car tire was detected in the filter blank run to determine possible contamination from the instrument.





Figure 17. Results from the Py-GC-MS analysis of the water samples taken at Akerselva, Hobøl and Gryta in May 2019 provided by NORCE. Subsamples of 10-20% were analyzed and mass concentrations of the individual polymer types detected are extrapolated to the total sample volume and reported in μ g/L. PE = polyethylene, PVC = polyvinyl chloride, car tire (indicated by the presence of D-limonene).

When looking at the size distribution of the microplastics identified in the with FT-IR analyzed water samples it is apparent that most particles (>70%) are between 10–100 μ m, followed by particles 100–300 μ m (13–28%) and only a few or none >300 μ m (Figure 18).



Figure 18. Microplastic concentration in particles per m³ (left) and relative proportion (right) of the three different size fractions: 10-100 μ m (blue), 100-300 μ m (orange), and >300 μ m (grey) for the water samples taken at Akerselva, Hobøl and Gryta in May.

3.1.2 Sediment

The separation of the two sediment trap samples (Akerselva and Hobøl) as well as one sediment grab sample (Akerselva, 1.29 kg dry weight) was successful. The sediment trap sample from Gryta (50 g dry weight) contained a very high amount of organic content (40.6% of dry weight) and fine sediment which hampered the following treatment steps and was therefore not processed further. The >300 µm fraction from the Gryta sample was still analyzed, but no microplastics were detected (Table 3). The two microplastics particles detected in the sediment trap sample from Hobøl (124 g dry weight) were identified as polytetrafluoroethylene (PTFE) which might be a contamination from the stopcock of the separatory funnel which is made out of PTFE. The sediment trap from Akerselva contained a relatively high amount of sediment (357 g dry weight). Thus, this sample was divided into two fractions, one processed like the other sediment trap samples in the separatory funnel (115 g dry weight) and the other like the grab sample in the separation column (242 g dry weight). Both samples, processed with the separation column, contained a very high number of particles >300 µm (Figure 19), thus only a subsample of each was analyzed (Table 3). The subsample of the Akerselva trap sample contained 17 microplastic particles with seven of it being PE, three each of PS and acrylates/polyurethanes and four other plastics consisting of a blend of polymers (e.g. polyvinyl chloride (PVC) and polyurethane (PUR)). Examples of some microplastic particles >300 µm detected in the sediment trap sample of Akerselva can be seen in Figure 20. In the fraction of the Akerselva sediment trap sample processed in a separatory funnel two PE and one PP particle were identified (Table 3). For the analysis of the smaller size fraction only this part of the Akerselva sediment trap sample was analyzed further. Two subsamples of 50 µl, representing 1% of the total sample volume, were analyzed and results combined to increase representativeness of the final data. A visual image of one of these subsamples as well as the corresponding false color image derived from the analysis with siMPle of the µFT-IR scanning data is shown in Figure 21.



Figure 19. Fraction >300 μ m of the Akerselva sediment grab sample (left) and the Akerselva sediment trap sample (middle) and a subsample of it (right) after density separation.



Figure 20. Examples for particles > $300 \mu m$ detected in the sediment trap sample from Akerselva with the sample spectrum in red and the best matching database spectrum in blue provided below the picture taken with a stereo microscope.



Figure 21. Visual image (left) and false color image (right) of a subsample (1%) of the Akerselva sediment trap sample, analyzed using μ FT-IR imaging. The different colors correspond to different polymer types.

The blank corrected results from the 10-300 μ m fraction of the sediment samples (Table 4, for uncorrected and raw data see Appendix Table A2 and Table A3) show that Akerselva has the highest number of microplastic particles, i.e. 2.0×10^4 microplastics (6.0 mg) in the grab sample and 1.3×10^4 microplastics (2.1 mg) in the trap sample resulting in 1.5×10^4 (4.6 mg/kg) and 1.1×10^5 (18.4 mg/kg) microplastics per kg (dw) when extrapolating the data. In comparison to that, the microplastic concentration at Hobøl was 315 particles per sample (0.6 mg) extrapolated to 2.5×10^3 microplastics per kg (dw) (5.1 mg/kg). The processing of the Gryta sediment trap sample was not completed and therefore no data could be provided for the smaller size fraction. The procedural blank processed with the sediment samples only showed low numbers (90 microplastics) and mass (14.27 μ g).

Table 3. Concentrations of microplastics >300 μ m detected in the sediment samples taken at Akerselva, Hobøl and Gryta. Sediment samples were taken with a sediment grab at Akerselva on June 26th as well as with sediment traps installed from May-August (Gryta) or June-August (Akerselva, Hobøl). The results can be considered as blank corrected since no microplastics >300 μ m were detected in the procedural blank. Concentrations are provided as numbers and weight in μ g per kg dry weight (dw) sediment. For the sediment grab sample of Akerselva (*) a subset of approximately 10% of the subsample weight was analyzed for particles >300 μ m because of the high particle load. PE = polyethylene, PP = polypropylene, PA = polyamide, PVC = polyvinyl chloride, PUR = polyurethane. The polymer type "others" includes compound materials and polytetrafluoroethylene (PTFE).

sample	PE	РР	PA	PVC	PUR	acrylics	others	∑number	∑mass [µg]
Akerselva (grab) *	8	0	8	23	8	8	31	85	138641
Akerselva (trap)	17	9	0	0	0	0	0	26	569
Hobøl (trap)	0	0	0	0	0	0	16	16	1868
Gryta (trap)	0	0	0	0	0	0	0	0	0

micro	nlastics >	300	ıım	ner	kσ	wh)
THICLU	μ iastics \sim	300	μπ	per	ng i	uvv.

Table 4. Concentrations of microplastics 10-300 μ m detected in the sediment samples taken at Akerselva, Hobøl and Gryta. Sediment samples were taken with a sediment grab at Akerselva on June 26th as well as with sediment traps installed from May-August (Gryta) or June-August (Akerselva, Hobøl). *Results were blank corrected by subtracting the number or mass of each identified polymer type in the procedural blank from the respective polymer types detected in the samples.* Concentrations are provided as extrapolated numbers of microplastics per sediment dry weight (dw) and mass estimation in μ g per kg (dw). *PE = polyethylene, PP = polypropylene, PS = polystyrene, PA = polyamide, PEST = polyester/polyethylene terephthalate, PVC = polyvinyl chloride, PUR = polyurethane, CA = cellulose acetate. The polymer type "others" includes polyurethane based paints, alkyds and polyacrylonitrile fibers.*

	microplastics 10–300 μm per kg (dw)											
sample	PE	PP	PS	PA	PEST	PVC	PUR	acrylics	CA	others	∑ number	∑mass [µg]
Akerselva (grab)	1775	8202	3023	233	442	78	0	698	233	752	15435	4643.55
Akerselva (trap)	17755	39861	13490	2611	8877	0	1741	6527	22193	1044	114099	18372.40
Hobøl (trap)	624	40	101	101	1670	0	0	0	0	0	2534	5101.73
Gryta (trap)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.



Figure 22. Polymer composition of microplastics based on number of particles identified in the 10-300 μ m fraction of samples from sediment traps taken at Akerselva and Hobøl in June-August 2019, from a sediment grab sample taken at Akerselva in June 2019 and from a procedural blank processed with the sediment samples. PE = polyethylene, PP = polypropylene, PS = polystyrene, PA = polyamide, PEST = polyester/polyethylene terephthalate, PVC = polyvinyl chloride, PUR = polyurethane, CA = cellulose acetate. The polymer type "others" includes polyurethane based paints, alkyds and polyacrylonitrile fibers.

For the sediment trap sample from Akerselva polypropylene (PP), polyethylene (PE), cellulose acetate (CA), and polystyrene (PS) are the most common polymers found using the µFT-IR analysis. For the grab sample from Akerselva PP was making up more than half of the sample followed by PS and PE and with minor contributions from PA, PEST, PVC, acrylics, CA and some other polymers (e.g. acrylonitrile butadiene styrene (ABS)). For Hobøl there are fewer polymers present, with polyester (PEST) and polyethylene (PE) being the most common (Figure 22). The procedural blank processed along with the sediment samples contained very few particles, one PE, two PP, three polyester and three acrylic fibers.



Figure 23. Microplastic concentration in particles per kg dry weight (left) and relative proportion (right) of the three different size fractions: 10-100 μ m (blue), 100-300 μ m (orange), and >300 μ m (grey) for the sediment trap samples taken at Hobøl and Akerselva between June and August and the sediment grab sample taken at Akerselva in June.

The size distribution of the microplastics identified in the analyzed sediment trap samples revealed that microplastics 10-100 μ m contributed more than 50% and the size fraction 100-300 μ m around 40% leaving the size fraction >300 μ m with a minor contribution (Figure 23).

3.2 Results from water samples analyzed by ALS and Eurofins

Results from the SEM-analysis at ALS and the Py-GC-MS analysis at Eurofins are respectively shown in Table 5 and Table 7. Full reports from both laboratories are shown in Appendix 1 (ALS) and 2 (Eurofins). Note that the results from ALS are presented as number of particles per liter (no/L) while the results from Eurofins are reported as mass concentration (μ g/L). The amount of water treated in the lab was 250 mL for the SEM analysis and about 1 liter for the Py-GC-MS analysis. The water samples for both analyses were collected on the 22-23th of May i.e. at the same time as the AAU-samples were collected. The sampling was done at the same time to be able to compare results.

COWI 32 M-1572|2020

Parameter	Unit	Gryta 2019-05-22	Akerselva 2019-05-22	Hobølelva 2019-05-23
Amount of water filtered	ml	250	250	187
org particles e.g. PP, PE, PS*	no. of particles /L	16	8	27
org particles e.g. PMMA, PUR, PET*	no. of particles /L	<4	4	27
org particles with silicon e.g. rubber*	no. of particles /L	<4	<4	<5
org particles with chlorine e.g. PVC*	no. of particles /L	<4	<4	<5
org particles with fluorine e.g. PTFE*	no. of particles /L	<4	4	<5
* > 10 μm	total no. of particles /L	16	16	54

Table 5. Results from the SEM analysis at ALS. Note that the results are presented as number of particles per liter.

The results from the SEM-analysis showed that the highest concentration (number of particles per liter) of PP, PE and PS was found in Hobøl, followed by Gryta and then Akerselva in a proportion of 3.4:2:1, respectively (Table 5). For the poly(methyl) methacrylate (PMMA), polyurethane (PUR) and polyethylene terephthalate (PET) the highest concentration was found in Hobøl, followed by Akerselva and none in Gryta (proportion 7:1:0). Teflon/PTFE was only recorded in Akerselva. No PVC or rubber were detected by the SEM-analysis in any of the rivers.

Table 6. A comparison of particle concentration (no of particles /1m³ or no of particles /L) in samples collected in the three rivers on the 22-23th of May 2019, as revealed by different methods of analysis. The FT-IR analysis include the MP sizes between 10-5000 μ m. The SEM-analysis include all MP particles >10 μ m.

Unit	Akerselva	Hobøl	Gryta	Method
no particles/1m ³	1069	156	140	FT-IR
no particles/1m ³	16000	54000	16000	SEM

Unit	Akerselva	Hobøl	Gryta	Method
no particles/L	1,1	0,2	0,1	FT-IR
no particles/L	16	54	16	SEM

Converting the SEM-results to number of particles per 1 m³ or the FT-IR results to number of particles per liter make the results comparable (Table 6). In all three rivers there is a substantially higher concentration found by the SEM-analysis compared to the FT-IR analysis. Taken into consideration the small amount of water collected (only 250 mL) for the SEM-analysis compared to the FT-IR analysis (1000 liters) the former results seem neither representative nor realistic for the investigated rivers.

Parameter	Unit	Gryta 2019-05-22	Akerselva 2019-05-22	Hobølelva 2019-05-23
Fraction	μm	> 0,2	> 27	> 27
Polyetylen (PE)	μg/l	<1	<3	<3
Polypropylene (PP)	μg/l	<0,1	<1	<1
Polystyren (PS)	μg/l	<0,1	<1	<1
Polyvinylklorid (PVC)	μg/l	<0,1	<1	<1
Polyetylentereftalat (PET)	μg/l	<0,1	<1	<1
Polyamid 6 (PA6)	μg/l	<1	<1	<1
Polymetylmetakrylat (PMMA)	μg/l	<1	<1	<1
Polykarbonat (PC)	μg/I	<0,1	<1	<1

Table 7. Results from the Py-GC-MS analysis at Eurofins. Note that the results are reported as mass concentration (μ g/L).

Py-GC-MS results from Eurofins revealed no mass concentration (μ g/L) above the quantification limits (Table 7). The quantification limit (LOQ) is the lowest concentration that can be quantified with a given measurement uncertainty with a given probability. It depends on the amount of water filtered. In the Eurofins-analysis a maximum of 1 liter was filtered from the three river samples compared to 100 liter (Akerselva) and 200 liters (Hobøl and Gryta) for the AAU/NORCE-analysis. This gives a hundred times lower LOQ for the latter.

In addition to analyzing a much larger volume, the AAU/NORCE samples have been treated before analysis, removing organic material. This will help to reduce the signal/noise-ratio in the analysis results making it easier to detect and define the different polymers.

3.3 Microplastic in the >300µm fraction

Microplastic concentrations in the water of the three rivers, analyzed using ATR-FT-IR, were low ranging from 0-4.0 particles per m³ In most of the samples no microplastics >300 μ m were detected (Figure 24, Appendix Table A5).



Figure 24. Concentrations of microplastics >300 µm and polymer type distribution detected in Akerselva, Gryta and Hobøl. Samples were taken from May to October with the AAU filtration unit and from August to October additionally with a Manta net (highlighted in grey). Samples marked with an * were not taken or not analyzed.

For Akerselva seven samples were taken between May and October with the AAU filtration unit, where 1 m³ of water was filtered (see Appendix Table A5). Four additional samples were taken between August and October with a manta net of 300 μ m mesh size, where 11-42 m³ were filtered. In three of the samples from the filtration unit microplastic concentrations between 2-4 particles >300 μ m per m³ were detected while no microplastics were detected in the other four samples (Figure 24). Microplastics were detected in all samples taken at Akerselva with the manta net with concentrations ranging between 0.3 and 0.7 particles per m³ as can be seen in Figure 24. This is due to the amount of water filtered, being 10-57 times more using the manta net than using the AAU filtration unit therefore allowing for a higher accuracy. However, the concentration (i.e number of microplastic particles per m³) found using both methods are about the same level and very low (Appendix Table A5).



Figure 25. Examples for particles >300 μ m detected in the manta net sample from Akerselva (29th of August) with the sample spectrum in red and the best matching database spectrum in blue provided below the picture taken with a stereo microscope.

At Hobøl the filtered water volume taken with the filtration units was six times 1 m³ and one time (27th of June) only 250 L due to fast clogging of the filters because of a high particle load of fine sediments (see Appendix Table A5). The filtered volume for the four manta net samples ranged from 1 to 30 m³. Only in one sample (taken with the filtration unit) a concentration of 2 microplastic particles per m³ was detected, being one PE fragment and one PEST fiber (Figure 24). No other microplastic particles were identified in the other six filtration unit samples nor the four manta net samples in Hobøl.

For Gryta, four samples, of 1 m³ filtered water volume each, were taken with the filtration unit with no microplastic particles >300 μ m detected. For the manta net samples only one of four samples contained microplastic, being one PEST and one polyvinyl acetate fragment (Figure 24). The concentration in this sample was only of 0.1 microplastics per m³, in spite that the filtered water volumes were high ranging from 17-57 m³ (Figure 24, Appendix Table A5).



Figure 26. Examples for particles >300 µm of a natural origin that could be mistaken for microplastics

Examples of two microplastic particles identified in the manta net samples taken at Akerselva (29th of August) are shown in Figure 25. One of the particles had the appearance of a grass fiber but was identified as PP. On the other hand, Figure 26 shows some examples for particles of natural origin which were identified in the >300 μ m fraction which were sorted out as potential microplastics based on their visual appearance. This highlights the need to validate the data with spectroscopic methods to avoid false positive identification of microplastics but also to underestimate microplastic concentrations. Pie charts showing the results for the composition of microplastics >300 μ m in terms of color and shape provided for the two different matrices sampled can be found in the Appendix Figure A1 and Figure A2.

3.3.1 Concentration of microplastics compared to river flow

The numbers of microplastic particles >300 μ m per m³ in the three rivers are very low. The highest concentration was found in Akerselva (Appendix Table A5), and these results are plotted against waterflow measured by NVE (data subtracted from the online database Sildre) in Figure 27. Hobøl and Gryta only contained microplastics (>300 μ m) in one sample each (Appendix Table A5). These graphs are therefore not shown. The low number of microplastic particles/m³ in the larger fraction makes it difficult to compare with water flow in the river. One will need to include the smaller fraction (10-300 μ m) where there is a higher number of microplastic particles to be able to compare datasets.



Figure 27. Number of microplastic particles >300 μ m/m³ plotted against waterflow (m³/sec) measured at the dam of Maridalsvannet (data from NVE: Sildre.nve.no).

3.4 Evaluation of methods: Strengths and weaknesses (limitations)

One of the goals with the present study was to describe the strengths and weaknesses of the different sampling and analysis methods that have been used in this study. These are summarized in Table 8 (sampling methods) and Table 9 (analysis methods) as well as the text below. Our experiences from this project may be useful when planning methodology and sampling strategies in future national river-monitoring-programs.

3.4.1 Sampling methods

T , , , , , , , , , , , , , , , , , , ,	
Table 8. Advantages and limitations of the	ested sampling methods
5	1 3

Sampling method	Advantages	Limitations
"No-treatment"	Quick sampling.	Snapshot.
water sampling	Only 250 mL or 1-2 L needed.	Not representative sample (need several
(filling of a glass		replicates to become more representative).
bottle and sending it		Need a high concentration (i.e. no of MP/L)
for analysis at		to be able to find MP (high detection limits).
commercial labs).		
Filtering water 1m ³	Representative sample.	Snapshot.
(e.g. filtering unit	Quantitative approach when	Time consuming to filter a large amount of
AAU)	including the smallest fraction	water.
	(MP ≥10 µm).	Heavy equipment.
	Can separate several fractions	
	(e.g. 10-300 µm and 300-5000	
	um).	
	Do not have to transport a lot of	
	water to the lab (only the filters)	
	Use of sampling blanks in the	
	field gives a better contamination	
	control.	
	≥10 um-fraction can be	
	correlated with waterflow.	
Manta trawling	A large amount of water can be	Snapshot.
······································	filtered quickly.	Loose the heavier microplastic particles.
	Sampling restricted to the surface	Only fractions > 300µm (lower limit depends
	therefore targeting the lighter	on mesh size, possibly down to $>100 \mu m$)
	particles only.	In most cases a rather qualitative approach
		due to few particles in the larger fraction.
		Collects a lot of organic material (branches
		and leaves), which is time consuming and
		difficult to remove (chemically) later in the
		lab.
		Large net, particles get easily stuck in the
		net (difficult to get the whole sample into
		the glass jar).
		Difficult to clean the manta-net well enough
		between sampling sites (potential source of
		cross-contamination).
		The manta net contains plastic parts.
Sediment trap	Sampling suspended material	The design of the sediment trap would need
placed at the	over a longer period.	to be adopted to each river.
riverbed	Representative sample for what is	Need very large containers (e.g. glass jars)
	transported in the river.	to reduce the risk of losing suspended
	Quantitative approach when	particles.
	including the smallest fraction	Difficult to measure sediment accumulation
	(MP ≥10 μm).	rates.

		Possible risk of losing the particles with higher densities (that are transported just above the river bed/floor), as well as the lower density particles (floating in the surface water).
Grab samples (bulk samples)	Sampling material accumulated usually more than 1 year. Fits best for rivers where water flow is not too high allowing all sizes to settle to the bottom.	Need to sample many subsamples that represent the types of "sedimentary environments" within the river to get a representative sample. Cannot be directly compared to water flow
	microplastic particles.	The amount and composition of microplastic particles in the sediments may be impacted during river flushes.

Water sampling depicts a snapshot of the situation in the river. Snapshots are useful when wanting to study the polymer content/concentration related to water flow e.g. floods- or droughts events. Generally, the more water is collected (filtered) the better, as large water volume gives a more representative sample and a lower detection limit.

Collecting only ≤ 1 liter of water for analysis will probably not give a representative picture of what is in the water. In our study the SEM seems to overestimate the number of microplastic particles being present (at least compared to the FT-IR-method).

Sampling about 1 000 liters of water by filtering devices or between 1 400-57 000 liters using a manta trawl, collected only a few microplastic particles in the >300 μ m fractions. However, for the smallest fraction (10-300 μ m) there were many microplastic particles present (in the 1 000 liters). This reveals the importance of including the smaller fraction and to sample a high enough water volume to get a representative sample that can be used to document the presence of microplastic concentration at the time of sampling.

Furthermore, as LOQ can be quantified as 10 times the contamination determined by the blanks, the large volumes sampled allow lowering of the LOQ by a factor 100 when comparing the sampling of 1 liter versus the sampling of 1000 L.

Sediment samples can be done with e.g. sediment traps, which accumulate suspended particles in transport during the time of sampling and give thus a good indication of what is found in the river during e.g. a month, season or year depending on deployment time of the trap. Grab samples collect particles that have accumulated on the river floor during a longer time period, usually > 1 year (depending on accumulation rates, water flow, flood events etc.). The study of polymer contamination in grab samples works best for rivers where the current is not too strong, allowing the full range of particle sizes to settle and being stored in the river sediments. Large river flushes have been found to resuspend and remove microplastics (Hurley et al. 2018). Unlike the water samples (only showing a snapshot of the situation), the sediment matrix gives a more complete picture of what types of polymers are in transport or have been deposited in the river system. But we once again emphasize the importance of investigating the total size range as our investigations revealed a much higher number of particles in the smaller fraction (10-300 μ m) than the larger fraction (>300 μ m) in the sediments, as well as a much larger range of polymer types.

To get a representative image of microplastics in the river sediment one will need to sample different sediment accumulation areas i.e. sedimented in low- to high-energy environment, and make a bulk sample of the different sediment types. This will not directly be comparable with flow variations but give a good indication of what microplastic types have been added to the river system through time.

3.4.2 Analysis methods

The SEM method of ALS used HNO_3 for removing the sample matrix. However, this is a rather harsh treatment, which can possibly remove both organic material as well as a lot of the plastics. ALS have now replaced the SEM-method with FT-IR instead (ALS, 2020).

The Py-GC-MS method of Eurofins did not remove the organic matrix before analysis. In case of high content of particulate organic matter this could lead to a strong matrix effect on the pyrograms and hence rather poor detection limits. However, an efficient sample preparation will reduce these matrix effects. Furthermore, this method allows for detecting car tire rubber. Eurofins is now working on developing/improving their method, both sample preparation and analysis (Joakim Skovly, personal communication, January 2020).

The limitation of the μ FT-IR imaging method to relatively small subsamples can be overcome by analyzing replicates to increase the representativeness of the results as has been shown for the sediment trap sample from Akerselva.

Generally, Py-GM-MS and µFT-IR imaging can be considered as being complementary analytical methods one providing mass-related the other particle (number/size/shape) related data.

Analysis method	Advantages	Limitations
	Very high accuracy of polymer	Need extensive sample preparation to avoid
uFT-IR imaging	identification.	matrix interference.
F	Can detect a very wide range of	Cannot go above roughly 500 µm in size.
	polymers.	Some materials, e.g. rubber from car tires,
	Can detect size and shape down	cannot be detected due to their content of
	to 10 μm.	carbon black.
	Can estimate mass of plastics.	
	Very high accuracy of polymer	Cannot go below roughly 300 µm in size.
ATR-FT-IR	identification.	Needs manual sorting and manual analysis
	Can detect a very wide range of	of particles.
	polymers.	Handling of small, single particles might
	Combined with microscopy this	result in loss of particles.
	method can quantify size and	
	mass.	
Py-GC-MS without	Can detect mass of polymers	Cannot detect particle size.
sample preparation	directly without estimations.	Can only detect a limited number of
(Eurofins) Can detect car tire rubbers		polymers.
		Very much affected by matrix effects if high
		concentrations of organic matter are
		present.

Table 9. Advantages and limitations of tested analysis methods in this study.

Py-GC-MS with Can detect mass of polymers		Need extensive sample preparation to avoid
sample preparation	directly without estimations.	matrix interference.
(NORCE)	Can detect car tire rubbers	Cannot detect particle size.
		Can only detect a limited number of
		polymers.
SEM (ALS)	M (ALS) Can detect small particles. Cannot positively identify plastics	
		identifies relative concentrations of
		elements).

Another widely used analysis technique, which has however not been tested within this study, is Raman spectroscopy. This technique also provides substance characteristic spectra considered to be complementary to FT-IR spectroscopy (Käppler et al. 2016). This method has the advantage of allowing an identification of polymer types down to a size limit of 1 μ m and providing additional information on added fillers or pigments (Käppler et al. 2016). The disadvantages are that there might occur some interference from auto-fluorescence from the organic matrix (e.g. from algae) which would hamper the identification and most notably the longer measurement time in comparison to μ FT-IR imaging (Käppler et al. 2016, Cabernard et al. 2018).

3.4.3 Quality assurance of methods

Limit of detection (LOD) and limit of quantification (LOQ) are well-defined quantities when addressing dissolved substances, and there exist best practices for how they should be defined. For particles that are seen as pollutants, like microplastics, this is however not the case, and practices valid for dissolved substances cannot readily be transferred here to. LOD and LOQ must hence be defined to match this. For the LOD, one particle identified in a scan, or set of scans, will be the LOD, as this is the lowest unit that can be detected. With respect to its mass, the corresponding LOD would be the mass of the smallest particle that can be detected. In the present case this is a $10 \times 5 \times 5 \mu m$ particle. Most plastics have densities not much below 1 g cm⁻³ and assuming this density and a rectangular particle will hence lead to a LOD of 2.5 10^{-10} g. LOQ for particles cannot be defined in the same way as best practices recommend for LOQ for dissolved substances. Instead a choice must be made based on the LOD value. In the present case the LOQ was defined as ten times the LOD. Table 10 indicates the so-determined LOD and LOQ values for the tested matrices and compares them to the blank corrected concentrations measured in the analyzed samples.

Table 10. Estimated limit of detection (LOD) and limit of quantification (LOQ) values and their comparison to
measured concentrations. The LOD was defined as one particles of the smallest detectable size
(10 μ m \times 5 μ m \times 5 μ m) and a density of 1 g/cm ³ and therefore a particle mass of 2.5 10 ⁻¹⁰ g. The LOQ is
defined as ten times the LOD.

	sample volume	concentrate	sub-sample	analyzed	LOD	LOD	LOQ	LOQ	sample concentration	
water	L	mL	mL	L	items/L	μg/L	items/L	μg/L	items/L	μg/L
Akerselva	1000	5	0.15	30	0.03	8.33E-06	0.33	8.33E-05	1.07	5.48E-02
Hobøl	1000	5	0.75	150	0.01	1.67E-06	0.07	1.67E-05	0.14	1.86E-02
Gryta	1000	5	0.25	50	0.02	5.00E-06	0.20	5.00E-05	0.14	1.20E-02
sediments	kg	mL	mL	kg	items/kg	μg/kg	items/kg	µg/kg	items/kg	µg/kg
Akerselva (grat	o) 1.26	5	0.05	0.01	79.37	0.02	793.65	0.20	15434.71	4643.55
Akerselva (trap) 0.11	5	0.10	0.00	454.55	0.11	4545.45	1.14	114099.22	18372.40
Hobøl (trap)	0.12	5	0.40	0.01	104.17	0.03	1041.67	0.26	2534.46	5101.73
Gryta (trap)	0.05	n.a	n.a	n.a	n.a	n.a	n.a	n.a	n.a.	n.a

Table 10 clearly shows that all the measured concentrations, concerning numbers of particles, lie above the LOD and most of them also clearly above the LOQ. Exception to this are the sediment trap sample from Hobøl, which falls into the same range as the LOQ but is still above the LOQ value, and the water sample from Gryta, which lies within the range of the LOQ but the value is slightly lower. For mass concentrations all samples lie clearly above the LOQ by factor 10³ for water and 10⁴ for sediments. This difference might be explained by the fact that the LOD/LOQ assumes an average density of 1 g/cm³ for the particles but the microplastics in the surface water of a river are most likely to have a lower density while the ones in the sediment might have a higher density. This just underlines that the LOD/LOQ discussion for this kind of analysis is far more complex. It also shows that theoretically each polymer type would need to be considered separately. Thus, the blank correction performed per polymer type in this study presents a very conservative approach, resulting in reliable data. The methods used in this study for extracting and identifying microplastics (10-5000 µm) in freshwater and sediment samples followed well established methods published by Olesen et al. (2019) and Liu et al. (2019). The recovery rates of these methods were 96% for freshwater and 64% or 66%, respectively, for the sediment protocol (Olesen et al. 2019, Liu et al. 2019).

4 Conclusions

Several different methods for sampling and analysis have been tested in this study. But due to a very limited time to conduct this complex study (just 7 months) and a restricted budget, many of the tests could only be performed once or a few times. Still, the results reveal some important issues from which we conclude:

- In general, sampling frequency and sampling methodology should be based on knowledge of the local riverine conditions. E.g. if the input (from diffuse and point sources of pollution) varies through time one will need a more frequent sampling program compared to areas where the situation is more static. We recommend a microplastic-screening to address the baseline of the amounts and type of microplastics before studying long term trends. When studying suspended microplastics in a previously unmonitored river we suggest filtering water samples close to the surface (1 m³) in a monthly interval. This sampling interval is in agreement with OSPARs guidelines (2015) for sampling nutrients and pollutions (e.g. heavy metals) in rivers. Sediment traps may be applied to investigate/monitor the lower part of the water column in a quarterly interval. Despite the time-consuming collection and sample preparation, this will give a reliable result on polymer types and abundances in the rivers at the time of sampling, particularly for the denser microplastic particles. Bulk grab samples on the other hand give a good image on what types of polymers there are in the river system that have accumulated in the river over a longer time period. However, the sampling points for the subsamples must be chosen with care to ensure that sediments are indeed collected in an accumulation zone and not in an area of transient sediment deposition, and that the bulk sample gives a representative sample of the varying grain sizes (sedimented in the different range from low- to high-energy environment). One need to be aware that a recent river flush event will influence the amount of microplastic particles found in the sediments. The Manta trawls are suitable for filtering a large volume of water when aiming for microplastics $>300 \ \mu m$ (i.e. the manta net mesh size). In our study the larger than 300 µm fraction only contained a few microplastic particles. If lowering the mesh size of the manta net to 100 µm this would allow to analyze also the fraction from 100-300 µm quantitatively by using µFT-IR imaging. However, one will still miss the more abundant 10-100 µm fraction. Furthermore, our experience with use of the manta net showed that a lot of organic material like branches and leaves where collected together with the microplastic particles. The procedures (in the lab) for removing the organic material without damaging the microplastics were extremely time consuming and costly (due to chemicals and the tedious lab work). When the mesh size is reduced to 100 μ m, the amount of this organic matrix would also increase, resulting in even longer sample preparation times.
- Analyzing particles by "sorting and collecting plastic candidates" works well down to a size of roughly 300 µm. Going below this value this approach is increasingly prone to human bias, as small plastic particles are easily overlooked or the particles may be difficult to handle (pick out, measure and transfer to e.g. the ATR-FT-IR machine). There is a risk of losing the particle or getting an incomplete measurement.
- In our study the largest amount of microplastics were detected in the smallest fraction (10-300 µm). We therefore stress the importance of including the smallest fraction in future monitoring program analysis in order to obtain a representative (and quantitative) result on what types of polymers and the abundance of microplastic particles are present in river water/sediments. It is furthermore worthwhile keeping in mind, that it is hypothesized that the smallest particles cause the most harm towards aquatic organisms.

- In the present study the concentration of microplastic particles larger than 300 µm were too low for correlation with the waterflow in the rivers. In future studies we recommend including the smallest fraction (10-300 µm) as well in the correlation.
- A higher temporal resolution of data on the 10-300 µm fraction in combination with the existing high-resolution data on water flow might also help to identify the most suitable periods for sampling (due to improving the sampling strategy).
- Representative samples are of great importance. A large water volume gives a more representative sample and a lower detection limit than small water volumes. The microplastic size fraction, which is aimed to be analyzed, determines the appropriate water volume that should be sampled. With increasing particle sizes, the analyzed water volume must also be increased. A sampled water volume of 1 m³ proved sufficient to gather representative data on microplastics 10–300 µm but not on microplastics >300 µm.
- Our study shows that a visual identification of the larger fraction (>300 µm) is not an acceptable approach for microplastic analysis of this fraction alone, as can be seen by the high amount of non-plastic particles which were sorted out during this assessment. Spectroscopic analysis must be conducted to verify that the collected particles are indeed microplastic particles.
- An efficient sample preparation to reduce the amount of organic and inorganic matter of natural origin is of great importance when analyzing the smaller size fraction (10-300 µm) as well as the >300 µm size fraction, to increase the ratio of microplastics to particles of natural origin and overcome interferences with the matrix during analysis.
- There are many sources of errors and contaminations when studying microplastics (during sampling, lab-preparation and analysis). The use of control samples (blanks), non-plastic (wool and cotton) cloths and sampling/lab equipment with a minimal amount of plastic is therefore of great importance. It is also paramount that a laboratory can document the accuracy of their analysis.

5 References

Bergmann, M., Mützel, S., Primpke, S., Tekman, M.B., Trachsel, J. and Gerdts, G. (2019) White and wonderful? Microplastics prevail in snow from the Alps to the Arctic. Science Advances 5(8), eaax1157. 10 pp.

Blettler, M.C.M., Abrial, E., Khan, F.R., Sivri, N. and Espinola, L.A. (2018) Freshwater plastic pollution: Recognizing research biases and identifying knowledge gaps. Water Research 143, 416-424.

Cabernard, L., Roscher, L., Lorenz, C., Gerdts, G. and Primpke, S. (2018) Comparison of Raman and Fourier Transform Infrared Spectroscopy for the Quantification of Microplastics in the Aquatic Environment. Environmental Science & Technology 52(22), 13279-13288.

Frias, J.P.G.L. and Nash, R. (2019) Microplastics: Finding a consensus on the definition. Marine Pollution Bulletin 138, 145-147.

Gomiero, A., Øysæd, K.B., Agustsson, T., van Hoytema, N., van Thiel, T. and Grati, F. (2019) First record of characterization, concentration and distribution of microplastics in coastal sediments of an urban fjord in south west Norway using a thermal degradation method. Chemosphere 227, 705-714.

Hanke, G., Galgani, F., Werner, S., Oosterbaan, L., Nilsson, P., Fleet, D., Kinsey, S., Thomposon, R.C., Palatinus, A., Van Franeker, J.A., Vlachogianni, T., Scoullos, M., Veiga, J.M., Matiddi, M., Alcaro, L., Maes, T., Korpinen, S., Budziak, A., Leslie, H., Gago, J. and Liebezeit, G. (2013) MSFD GES technical subgroup on marine litter. Guidance on Monitoring of marine litter in European Seas, 128 pp., European Commission, Luxembourg.

Hurley, R., Woodward, J. and Rothwell, J.J. (2018) Microplastic contamination of river beds significantly reduced by catchment-wide flooding. Nature Geoscience 11(4), 251-257.

Käppler, A., Fischer, D., Oberbeckmann, S., Schernewski, G., Labrenz, M., Eichhorn, K.-J. and Voit, B. (2016) Analysis of environmental microplastics by vibrational microspectroscopy: FT-IR, Raman or both? Analytical and Bioanalytical Chemistry 408(29), 8377-8391.

Käppler, A., Fischer, M., Scholz-Böttcher, B.M., Oberbeckmann, S., Labrenz, M., Fischer, D., Eichhorn, K.-J. and Voit, B. (2018) Comparison of μ -ATR-FT-IR spectroscopy and py-GCMS as identification tools for microplastic particles and fibers isolated from river sediments. Analytical and Bioanalytical Chemistry 410(21), 5313-5327.

Lachowicz, T., Zieba-Palus, J. and Kościelniak, P. (2012) Analysis of rubber samples by PY-GC/MS for forensic purposes. Problems of Forensic Science 91, 195-207.

Liu, F., Vianello, A. and Vollertsen, J. (2019) Retention of microplastics in sediments of urban and highway stormwater retention ponds. Environmental Pollution 255, 113335. 8 pp.

Löder, M.G.J., Imhof, H.K., Ladehoff, M., Löschel, L.A., Lorenz, C., Mintenig, S., Piehl, S., Primpke, S., Schrank, I., Laforsch, C. and Gerdts, G. (2017) Enzymatic Purification of Microplastics in Environmental Samples. Environmental Science & Technology 51(24), 14283-14292.

Mani, T., Hauk, A., Walter, U. and Burkhardt-Holm, P. (2015) Microplastics profile along the Rhine River. Scientific Reports 5, 17988. 7 pp.

Mani, T., Primpke, S., Lorenz, C., Gerdts, G. and Burkhardt-Holm, P. (2019) Microplastic pollution in benthic midstream sediments of the Rhine river. Environmental Science & Technology 53(10), 6053-6062.

Mintenig, S.M., Löder, M.G.J., Primpke, S. and Gerdts, G. (2019) Low numbers of microplastics detected in drinking water from ground water sources. Science of the Total Environment 648, 631-635.

Norén, F. (2007) Small Plastic Particles In Coastal Swedish Waters, 11 pp., KIMO Sweden.

Olesen, K.B., Stephansen, D.A., van Alst, N. and Vollertsen, J. (2019) Microplastics in a Stormwater Pond. Water 11(7), 1466 pp.

Piehl, S., Leibner, A., Löder, M.G.J., Dris, R., Bogner, C. and Laforsch, C. (2018) Identification and quantification of macro- and microplastics on an agricultural farmland. Scientific Reports 8(1), 17950. 9 pp.

PlasticEurope (2019). Plastics – the Facts 2019: An analysis of European plastics production, demand and waste data. 42 pp.

https://www.plasticseurope.org/application/files/9715/7129/9584/FINAL_web_version_Plastics_the _facts2019_14102019.pdf

Rezania, S., Park, J., Md Din, M.F., Mat Taib, S., Talaiekhozani, A., Kumar Yadav, K. and Kamyab, H. (2018) Microplastics pollution in different aquatic environments and biota: A review of recent studies. Marine Pollution Bulletin 133, 191-208.

Vianello, A., Jensen, R.L., Liu, L. and Vollertsen, J. (2019) Simulating human exposure to indoor airborne microplastics using a Breathing Thermal Manikin. Scientific Reports 9(1), 8670. 11 pp.

Vollertsen, J. and Hansen, A.A. (2017) Microplastic in Danish wastewater: Sources, occurrences and fate. Vollertsen, J. and Hansen, A.A. (eds), 55 pp., The Danish Environmental Protection Agency

Software and webpages

ALS (2020) https://www.alsglobal.no/miljo/mikroplast

Eurofins (2019) https://cdnmedia.eurofins.com/european-east/media/2852407/mikroplast-web.pdf

NVE <u>http://sildre.nve.no/sildre/</u> (online hydrological real time data provided by the Norwegian Water Resources and Energy Directorate from more than 2000 stations in Norway)

OSPAR (2015) Riverine Inputs and Direct Discharges Monitoring Programme (RID). HASEC 14/14/1, Annex 8, 31 pp. <u>https://www.ospar.org/documents?d=33689</u>

siMPle https://simple-plastics.eu/index.html

6 Acknowledgements

This project "Kartlegging av mikroplast i elver" (#19087229), was funded by Miljødirektoratet and lasted from May-November 2019. We thank Eivind Farmen in Miljødirektoratet for useful comments and feedback during the project and on the report. Acknowledgements to Aquateam COWI who provided lab space and help to Nina during preparations of the manta samples. We would very much like to thank Henrik Koch who made the AAU sampling device and was very helpful during the first sampling in May. Thanks also to Fan Liu (AAU) who helped during the May sampling and Alvise Vianello (AAU) for preparing some of the samples for Py-GC-MS. Further thanks go to Helena Hjørringgaard, Camilla Jungklas Nybo and Elena Alexandra Bouros-Bandrabur (AAU) for their support in analyzing larger microplastic particles. We also would like to thank Alessio Gomiero and his team at NORCE for the Py-GC-MS analysis on the water samples. Many thanks also to Fred Wenger in NVE for helping us getting access to water flow data from the three rivers.

7 Appendix

7.1 Results from ALS

Rapport

Sida 1 (2)

Ankomstdatum 2019-05-31 Utfärdad 2019-06-14

N1909675

T1919010



1NL50DUEJUU

ALS Scandinavia

Postboks 643 N-0214 OSLO Norge Norway

Projekt Bestnr

Analys av vatten

Er beteckning	N00662328 N1909675					
Labnummer	O11147590					
Parameter		Resultat	Enhet	Metod	Utf	Sign
filtrerad mängd		250	ml	1	1	EH
org partiklar t.ex. PP, PE, PS		16	antal/l	1	1	EH
org partiklar t.ex. PMMA, PUR, PET		<4	antal/l	1	1	EH
org partiklar med kisel t.ex. plast, gummi		<4	antal/l	1	1	EH
org partiklar med klor t.ex. PVC		<4	antal/l	1	1	EH
org partiklar med fluor t.ex. PTFE		<4	antal/l	1	1	EH

Er beteckning	N00662329 N1909675					
Labnummer	O11147591					
Parameter		Resultat	Enhet	Metod	Utf	Sign
filtrerad mängd		250	ml	1	1	EH
org partiklar t.ex. PP, PE, PS		8	antal/l	1	1	EH
org partiklar t.ex. PMMA, PUR, PET		4	antal/l	1	1	EH
org partiklar med kisel t.ex. plast, gummi		<4	antal/l	1	1	EH
org partiklar med klor t.ex. PVC		<4	antal/l	1	1	EH
org partiklar med flu	or t.ex. PTFE	4	antal/l	1	1	EH

Er beteckning	N00662330 N1909675						
Labnummer	O11147592						
Parameter		Re	esultat	Enhet	Metod	Utf	Sign
filtrerad mängd			187	ml	1	1	EH
org partiklar t.ex. PP, PE, PS			27	antal/l	1	1	EH
org partiklar t.ex. PMMA, PUR, PET			27	antal/l	1	1	EH
org partiklar med kisel t.ex. plast, gummi *			<5	antal/l	1	1	EH
org partiklar med klor t.ex. PVC			<5	antal/l	1	1	EH
org partiklar med f	luor t.ex. PTFE		<5	antal/l	1	1	EH

ALS Scandinavia AB Box 700 182 17 Danderyd Sweden Webb: <u>www.alsqlobal.se</u> E-post: <u>info.ta@alsqlobal.com</u> Tel: + 46 8 52 77 5200 Fax: + 46 8 768 3423

Dokumentet är godkänt och digitalt signerat av Eli-Aálenius 2019.06.14 14:56:26 ALS Scandinavia AB Client Service elke.halenius@alsglobal.com

Rapport

Sida 2 (2)

T1919010

1NL50DUEJUU



* efter parameternamn indikerar icke ackrediterad analys.

	Metod					
1	Paket A-4	hB.				
	Analys av	mikroplaster i avlopps/lakvatten				
	Vatten behandlas för att lösa upp naturlig organiska partiklar. Provet filtreras genom ett pc filter (porstorlek 10 µm) och filtret guldbeläggs. Analysen genomförs med ett svepelektronmikroskop som är utrustat med en energidispersiv detektor för identifiering av element med atomnummer >5. Antal mikroplastpartiklar/1000 ml beräknas. Partiklar med en kornstorlek >10 µm analyseras. Observera att det kan finnas andra plaster i gruppen än dem som har nämnts som exempel.					
	Förkortnir	idar.				
	PE	Polyeten				
	PP	Polypropen				
	PS	Polystyren				
	PMMA	Polymetylmetakreylat, plexiglas				
	PUR	Polyuretan				
	PET	Polyetentereftalat				
	PVC	Polyvinylklorid, vinylplaster				
	PTFE	Polytetrafluoreten, Teflon				
		-02-18				

GodkännareEHElke Hålenius

1

Utf¹ För mätningen svarar ALS Scandinavia AB, Box 700, 182 17 Danderyd.

Denna rapport får endast återges i sin helhet, om inte utfärdande laboratorium i förväg skriftligen godkänt annat. Resultaten gäller endast det identifierade, mottagna och provade materialet. Beträffande laboratoriets ansvar i samband med uppdrag, se aktuell produktkatalog eller vår webbplats <u>www.alsglobal.se</u>

Den digitalt signerade PDF filen representerar originalrapporten. Alla utskrifter från denna är att betrakta som kopior.

¹ Utförande teknisk enhet (inom ALS Scandinavia) eller anlitat laboratorium (underleverantör).

ALS Scandinavia AB Box 700 182 17 Danderyd Sweden Webb: <u>www.alsqlobal.se</u> E-post: <u>info.ta@alsqlobal.com</u> Tel: + 46 8 52 77 5200 Fax: + 46 8 768 3423

Dokumentet är godkänt och digitalt signerat av



7.2 Results from Eurofins

🔅 eurofins

COWI AS Torget 6 3256 Larvik Attn: Jane Dolven Eurofins Environment Testing Norway AS (Bergen) F. reg. 965 141 618 MVA Sandviksveien 110 5035 Bergen

Tlf: +47 94 50 42 42 bergen@eurofins.no

AR-19-MX-003648-01

EUNOBE-00034555

Prøvemottak: Temperatur: Analyseperiode: Referanse: 05.07.2019 05.07.2019-30.07.2019 A125334 / jane Dolven

ANALYSERAPPORT

	Prøvenr.: Prøvetype: Prøvemerking:	441-2019-0705-052 Rent vann Gryta-2019-05-22	Prøvetakingsdato: Prøvetaker: Analysestartdato:		22.05.2019 Oppdragsgiver 05.07.2019		
4	Analyse		Resultat	Enhet	LOQ	MU	Metode
	Mikroplast >0,2µm (8 µ	polymere)					
	Polyetylen (PE)		<1	µg/l	1		Py-GC-MS
	Polypropylene (PP)		<0.1	µg/l	0.1		Py-GC-MS
	Polystyren (PS)		<0.1	µg/l	0.1		Py-GC-MS
	Polyvinylklorid (PVC)		<0.1	µg/l	0.1		Py-GC-MS
	Polyetylentereftalat (PB	ET)	<0.1	µg/l	0.1		Py-GC-MS
	Polyamid 6 (PA6)		<1	µg/l	1		Py-GC-MS
	Polymetylmetakrylat (P	MMA)	<1	µg/l	1		Py-GC-MS
	Polykarbonat (PC)		<0.1	µg/l	0.1		Py-GC-MS
	Sum kvantifiserte polyr	nere	<3.5	µg/l	3.5		Py-GC-MS

	Prøvenr.: Prøvetype: Prøvemerking:	441-2019-0705-053 Overflatevann Akerselva-2019-05-22		Prøvetakingsdato: Prøvetaker: Analysestartdato:		22.05 Oppdr 05.07	.2019 ragsgiver .2019
A	nalyse		Resultat	Enhet	LOQ	MU	Metode
	Mikroplast >27µm (8 p	olymere)					
	Polyetylen (PE)		<3	µg/l	3		Py-GC-MS
	Polypropylene (PP)		<1	µg/l	1		Py-GC-MS
	Polystyren (PS)		<1	µg/l	1		Py-GC-MS
	Polyvinylklorid (PVC)		<1	µg/l	1		Py-GC-MS
	Polyetylentereftalat (PE	ET)	<1	µg/l	1		Py-GC-MS
	Polyamid 6 (PA6)		<1	µg/l	1		Py-GC-MS
	Polymetylmetakrylat (P	MMA)	<1	µg/l	1		Py-GC-MS
	Polykarbonat (PC)		<1	µg/l	1		Py-GC-MS
	Sum kvantifiserte polyr	nere	<10	µg/l	10		Py-GC-MS

Tegnforklaring:

<: Mindre enn >: Større enn nd: Ikke påvist. Bakteriologiske resultater angitt som <1,<50 e.l. betyr 'ikke påvist'.

Måleusikkerhet er ikke tatt hensyn til ved vurdering av om resultatet er utenfor grenseverdi/-området og er angitt med dekningsfaktor k=2.

For mikrobiologiske analyser oppgis konfidensintervallet. Ytterligere opplysninger om måleusikkerhet fås ved henvendelse til laboratoriet.

Rapporten må ikke gjengis, unntatt i sin helhet, uten laboratoriets skriftlige godkjennelse. Resultatene gjelder kun for de(n) undersøkte prøven(e).

^{*} Ikke omfattet av akkrediteringen LOQ: Kvantifiseringsgrense MU: Måleusikkerhet

Side 1 av 2

AR-19-MX-003648-01

EUNOBE-00034555

🛟 eurofins

Prøvenr.: Prøvetype: Prøvemerking:	441-2019-0705-054 Overflatevann Hobølelva-2019-05-22		Prøvetakingsda Prøvetaker: Analysestartdat	ito:	22.05.2 Oppdra 05.07.2	.2019 ragsgiver .2019		
Analyse		Resultat	Enhet	LOQ	MU	Metode		
Mikroplast >27µm (8 p	oolymere)							
Polyetylen (PE)		<3	µg/l	3		Py-GC-MS		
Polypropylene (PP)		<1	µg/l	1		Py-GC-MS		
Polystyren (PS)		<1	µg/l	1		Py-GC-MS		
Polyvinylklorid (PVC)		<1	µg/l	1		Py-GC-MS		
Polyetylentereftalat (Pl	ET)	<1	µg/l	1		Py-GC-MS		
Polyamid 6 (PA6)		<1	µg/l	1		Py-GC-MS		
Polymetylmetakrylat (F	PMMA)	<1	µg/l	1		Py-GC-MS		
Polykarbonat (PC)		<1	µg/l	1		Py-GC-MS		
Sum kvantifiserte poly	mere	<10	µg/l	10		Py-GC-MS		

Bergen 30.07.2019

Omme Tommie Christensen

LOQ: Kvantifiseringsgrense MU: Måleusikkerhet									
<: Mindre enn >: Større enn nd: Ikke påvist. Bakteriologiske resultater angitt som <1,<50 e.l. betyr "ikke påvist".									
Måleusikkerhet er ikke tatt hensyn til ved vurdering av om resultatet er utenfor grenseverdi/-området og er angitt med dekningsfaktor k=2.									
For mikrobiologiske analyser oppgis konfidensintervallet. Ytterligere opplysninger om måleusikkerhet fås ved henvendelse til laboratoriet.									
Rapporten må ikke gjengis, unntatt i sin helhet, uten laboratoriets skriftlige godkjennelse. Resultatene gjelder kun for de(n) undersøkte prøven(e). Side 2 av 2									
	LOQ: Kvantifiseringsgrense MU: Måleusikkerhet ävist. Bakteriologiske resultater angitt som <1,<50 e.l. betyr 'ikke påvist'. rdering av om resultatet er utenfor grenseverdi/ -området og er angitt med dekningsfaktor k=2. insintervallet. Ytterilgere opplysninger om måleusikkerhet fås ved henvendelse til laboratoriet. het, uten laboratoriets skriftlige godkjennelse. Resultatene gjelder kun for de(n) undersøkte prøven(e).	LOQ: Kvantifiseringsgrense MU: Måleusikkerhet àvist. Bakteriologiske resultater angitt som <1,<50 e.l. betyr 'ikke påvist'. rdering av om resultatet er utenfor grenseverdi/-området og er angitt med dekningsfaktor k=2, insintervallet. Ytterligere opplysninger om måleusikkerhet fås ved henvendelse til laboratoriet. het, uten laboratoriets skriftlige godkjennelse. Resultatene gjelder kun for de(n) undersøkte prøven(e). Side 2 av 2							

ASM Kundesupport Berge

7.3 Results from AAU

Table A1. Volume and mass of the initial samples taken in May 2019 (surface water), June 2019 (sediment grab) and from June to August 2019 (sediment traps) as well as the volume of the analyzed sub-samples.

	initially sampled	concentrated sample after	sub-sample analyzed	sub-sample analyzed with
sample	volume/mass unit	purification unit	t with μFT-IR uni	t pyrolysis-GC-MS unit
Akerselva - surface water	1 m ³	5 mL	0.15 mL	0.5 mL
Hobøl - surface water	1 m ³	5 mL	0.75 mL	1 mL
Gryta - surface water	1 m ³	5 mL	0.25 mL	1 mL
Procedural blank - surface water		5 mL	1 mL	2 mL
Sampling blank Akerselva		5 mL	0.5 mL	n.a.
Akerselva sediment grab	1.29 kg (dw)	5 mL	0.05 mL	n.a.
Akerselva sediment trap	0.11 kg (dw)	5 mL	0.1 mL	n.a.
Hobøl sediment trap	0.12 kg (dw)	5 mL	0.4 mL	n.a.
Gryta sediment trap	0.05 kg (dw)	n.a.	n.a.	n.a.
Procedural sediment		5 mL	0.5 mL	n.a.

Table A2. Number of polymer types detected in surface water samples taken in May 2019 and sediment samples taken between June and August 2019, in the procedural blanks processed simultaneously with these samples and the accompanying sampling blank taken at Akerselva in May 2019. The polymer type "others" includes polyurethane based paints, alkyds and polyacrylonitrile fibres. N.a. = sample has not been analyzed.

			initial sample	proportion of analyzed sub-	concentration of microplastics 10–300 μm									
sample	PE	PP	PS	PA	PEST	PVC	PUR	Acrylic	CA	others∑r	number	amount unit	sample	∑ number unit
Akerselva - surface water	8	14	2	3	0	0	3	1	1	0	32	1 m ³	0.03	1067 m ³
Hobøl - surface water	8	3	0	4	7	0	1	0	0	0	23	1 m ³	0.15	153 m ³
Gryta - surface water	6	0	0	1	0	0	0	0	0	0	7	1 m ³	0.05	140 m ³
Procedural blank - surface water	0	0	0	0	3	0	0	0	0	0	3		0.2	15
Sampling blank (Akerselva)	0	0	0	0	4	0	0	0	0	0	4		0.1	40
Akerselva - sediment grab	23	106	39	3	6	1	0	9	3	10	200	1.29 kg (dw)	0.01	15504 kg (dw)
Akerselva - sediment trap	41	92	31	6	21	0	4	15	51	3	264	0.11 kg (dw)	0.02	114883 kg (dw)
Hobøl - sediment trap	7	2	1	1	19	0	0	0	0	0	30	0.12 kg (dw)	0.08	3017 kg (dw)
Gryta - sediment trap	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.05 kg (dw)	n.a.	n.a. kg (dw)
Procedural blank - sediment	1	2	0	0	3	0	0	0	0	3	9		0.1	90

Table A3. Mass of polymer types detected in surface water samples taken in May 2019 and sediment samples taken between June and August 2019, in the procedural blanks processed simultaneously with these samples and the accompanying sampling blank taken at Akerselva in May 2019. The polymer type "others" includes polyurethane based paints, alkyds and polyacrylonitrile fibres. N.a. = sample has not been analyzed.

			micr	initial sample	proportion of analyzed sub-	concentration of microplastics 10–300 μm								
sample	PE	PP	PS	PA	PEST	PVC	PUR	Acrylic	CA	others	∑mass unit	amount	sample	∑mass unit
Akerselva	0.34	0.83	0.10	0.06	0	0	0.07	0.24	0.01	0	1.64 µg	1 m ³	0.03	0.05 μg/L
Gryta	0.58	0	0	0.02	0	0	0	0	0	0	0.60 µg	1 m ³	0.05	0.01 µg/L
Hobøl	0.26	0.10	0	0.13	2.36	0	0	0	0	0	2.85 μg	1 m ³	0.15	0.02 μg/L
Procedural blank	0	0	0	0	0.07	0	0	0	0	0	0.07 µg		0.2	0.00 µg
Sampling blank (Akerselva)	0	0	0	0	3.41	0	0	0	0	0	3.41 μg		0.1	0.03 µg
Akerselva - sediment grab	6.83	28.72	18.91	0.43	0.08	0.11	0	0.52	0.05	4.40	60.04 µg	1.29 kg (dw)	0.01	4654.61 µg/kg(dw)
Akerselva - sediment trap	12.18	14.76	2.30	0.16	6.44	0	0.06	2.62	3.83	0.16	42.51 μg	0.11 kg (dw)	0.02	18496.56 µg/kg(dw)
Hobøl - sediment trap	0.20	0.02	0.02	0.00	50.76	0	0	0	0	0	51.01 µg	0.12 kg (dw)	0.08	5130.30 µg/kg(dw)
Gryta - sediment trap	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.05 kg (dw)	n.a.	n.a.
Procedural blank - sediment	1.22	0.03	0	0	0.07	0	0	0	0	0.10	1.43 µg		0.1	14.27 μg

Table A4. Results from the Py-GC-MS analysis of the water samples taken at Akerselva, Hobøl and Gryta in May 2019 provided by NORCE. Subsamples of 20%, representing 200 L, (Hobøl and Gryta) or 10%, representing 100 L, (Akerselva) were analyzed and are presented as mass in μ g.

n.d. = no substantial amount detected.

	Filter	Procedural Hobøl		Gryta	Akerselva	
parameter	blank	blank	23-05-2019	22-05-2019	22-05-2019	unit
PP	n.d.	n.d.	n.d.	n.d.	n.d.	μg
PS	n.d.	n.d.	n.d.	n.d.	n.d.	μg
PE	n.d.	n.d.	n.d.	33	470	μg
PVC	n.d.	n.d.	10	56	227	μg
PA66	n.d.	n.d.	n.d.	n.d.	n.d.	μg
PMMA	n.d.	n.d.	n.d.	n.d.	n.d.	μg
car tire	n.d.	n.d.	n.d.	n.d.	180	μg

Table A5. Concentrations of microplastics >300 μ m detected in Akerselva, Hobøl and Gryta from samples taken with AAUs filtration (highlighted in gray) unit and with a Manta net of 300 μ m mesh size. n.a.=not analyzed

			# potential	# natural	# particles not								others (e.g.	
	sampling	water volume	plastics per	particles	identified per	# microplastics						acrylates	compound	#microplastics
river	date	filtered [m ³]	sample	per sample	sample	per sample	PE	PP	PS	PEST	PVC	and PUR	materials)	per m ³
Akerselva	22-05-2019	1.0	8	1	5	2	1	0	0	1	0	0	0	2.0
Akerselva	08-06-2019	1.0	28	12	12	4	0	0	1	0	0	2	1	4.0
Akerselva	26-06-2019	1.0	4	4	0	0	0	0	0	0	0	0	0	0.0
Akerselva	08-07-2019	1.0	11	4	4	3	1	0	0	0	1	1	0	3.0
Akerselva	13-08-2019	1.0	5	2	3	0	0	0	0	0	0	0	0	0.0
Akerselva	13-08-2019	13.6	10	4	3	3	2	1	0	0	0	0	0	0.2
Akerselva	29-08-2019	42.2	41	4	6	31	7	13	1	3	0	2	5	0.7
Akerselva	11-09-2019	1.0	1	0	1	0	0	0	0	0	0	0	0	0.0
Akerselva	30-09-2019	10.7	7	2	2	3	0	1	0	0	0	2	0	0.3
Akerselva	09-10-2019	1.0	1	0	1	0	0	0	0	0	0	0	0	0.0
Akerselva	23-10-2019	10.6	10	2	6	2	0	1	0	1	0	0	0	0.2
Hobøl	23-05-2019	1.0	7	0	5	2	1	0	0	1	0	0	0	2.0
Hobøl	08-06-2019	1.0	3	0	3	0	0	0	0	0	0	0	0	0.0
Hobøl	27-06-2019	0.3	4	0	4	0	0	0	0	0	0	0	0	0.0
Hobøl	09-07-2019	1.0	21	4	17	0	0	0	0	0	0	0	0	0.0
Hobøl	12-08-2019	1.0	2	1	1	0	0	0	0	0	0	0	0	0.0
Hobøl	12-08-2019	29.8	5	2	3	0	0	0	0	0	0	0	0	0.0
Hobøl	29-08-2019	9.3	0	0	0	0	0	0	0	0	0	0	0	0.0
Hobøl	10-09-2019	1.0	1	0	1	0	0	0	0	0	0	0	0	0.0
Hobøl	30-09-2019	1.4	0	0	0	0	0	0	0	0	0	0	0	0.0
Hobøl	10-10-2019	1.0	9	6	3	0	0	0	0	0	0	0	0	0.0
Hobøl	22-10-2019	13.5	0	0	0	0	0	0	0	0	0	0	0	0.0
Gryta	22-05-2019	1.0	2	0	2	0	0	0	0	0	0	0	0	0.0
Gryta	26-06-2019	1.0	3	1	2	0	0	0	0	0	0	0	0	0.0
Gryta	12-08-2019	1.0	5	1	4	0	0	0	0	0	0	0	0	0.0
Gryta	12-08-2019	57.4	4	1	3	0	0	0	0	0	0	0	0	0.0
Gryta	29-08-2019	17.2	2	0	0	2	0	0	0	1	0	0	1	0.1
Gryta	10-09-2019	1.0	0	0	0	0	0	0	0	0	0	0	0	0.0
Gryta	30-09-2019	25.2	0	0	0	0	0	0	0	0	0	0	0	0.0
Gryta	23-10-2019	17.6	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.



Figure A1: Composition of colors for microplastic particles >300 μ m detected in the sediment (left) and water samples (right).



Figure A2: Composition of shapes for microplastic particles >300 μ m detected in the sediment (left) and water samples (right).