

MICROPLASTIC

Microplastics in sediments on the Norwegian Continental Shelf

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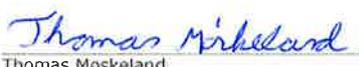
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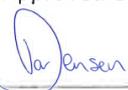
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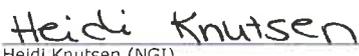

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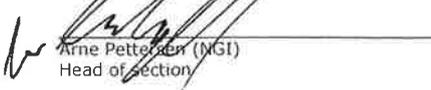
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1 ACKNOWLEDGEMENT

This study presents sampling and analysis of sediments for microplastics. The sampling area covers large scale geographical areas on the Norwegian Continental Shelf (NCS). The samples have been collected as an extra task during the regional offshore sediment monitoring on the NCS on behalf of the Oil & Gas industry. This study had therefore not been initiated without the good will from Oil & Gas operators, especially Statoil and ConocoPhillips Norway, which allowed use of some additional time during field work to take these samples.

The Norwegian Environment Agency saw this as a good opportunity to acquire knowledge of microplastic abundances from the NCS and therefore funded the project.

The Norwegian Geotechnical Institute (NGI) has put in own effort, assisted through the Skattefunn system (Project 266408), the projects FANTOM (RCN, 231736/F20) and JPI Oceans WEATHER-MIC (RCN, Project Grant 257433/E40), for the development of the analytical protocols and execution of the analysis and reporting. Through a relatively short period they have managed to deliver all results. A special thanks to Prof. Hans Peter Arp, Heidi Knutsen, Emma Jane Wade and Arne Petersen for the cooperation and all the work they have put in. Also, a special thanks to Øyvind Lilleeng at NMBU (Norwegian University of Life Sciences) whose substantial contribution to this report was done in partial fulfilment of the Masters Project tentatively entitled "The presence of microplastics on the Norwegian Continental shelf and coast of Havana".

Høvik 23th of February 2018

Thomas Møskeland – project manager DNV GL

2 EXECUTIVE SUMMARY

During the regional environmental sediment monitoring on the Norwegian Continental Shelf (NCS) on behalf of the Oil & Gas industry in 2017, 35 sediment samples were sampled from a large geographical area, covering the central North Sea, northern North Sea and the Barents Sea. The main purpose was to gain increased knowledge of abundance and distribution of microplastics in sediments on the NCS. The focus of the field work was not related to microplastic but to standard regional offshore monitoring, where sediment was sampled for analysis of soft bottom macro-fauna and selected chemicals. Hence, the sampling stations have not been especially designed for mapping of microplastics in sediments on the NCS.

The sediment samples were analysed for microplastic concentrations and number of items by an innovative analytical procedure. This report describes the analytical procedure and the analytical results.

The main conclusions are:

- ✓ A maximum¹ average of 60 ± 80 mg microplastics/kg dry sediment (corresponding to a maximum of $37\,000 \pm 50\,000$ items/m² sediment surface) of potential microplastics were found in the sediment samples from the Norwegian Continental Shelf.
- ✓ The sediment samples from the central North Sea had more microplastics than in the northern North Sea or Barents Sea areas, on maximum average 90 ± 100 , 30 ± 40 and 30 ± 20 mg microplastics/kg dry sediment. Further, the samples with the top 6 highest concentrations were all found in the central North Sea. The reason for this is uncertain but may be explained by large scale currents/gyres which accumulate debris in this part of the North Sea and influences from continental Europe through river run off among others. In addition, this area has relatively high shipping traffic, high fishing activity and high Oil & Gas activity which all may contribute to plastic emissions.
- ✓ It is emphasized that there are relatively large uncertainties related to the results. A duplicate analysis of one sample resulted in a variation of 55 %. At this time, the method quantifies the maximum weight and number of items that *could be* microplastic based on their density (lower than 1.53 g/mL), having a size range between 45 µm and 5mm, and resilience to an organic matter digestion process. Other materials fitting this profile such as soot, char, porous glass and porous ceramics would also be included in this quantification. Therefore, all data is presented as the potential "maximum" microplastic concentration. On the other side, plastic particles with higher density than 1.53 g/ml or with a size less than 45 µm will not be included in the numbers.
- ✓ Extrapolating the results for the entire North Sea (area 142 000 km²) and acknowledge large uncertainties this would imply there could be roughly 16 000 to 100 000 tonnes of microplastics in the North Sea.
- ✓ The highest maximum potential microplastics concentrations are in general found at locations close to Oil & Gas installations, though the aforementioned uncertainties should be considered.
- ✓ The results of this study have revealed relatively high concentrations of potential microplastics in the areas that were studied, which may confirm the widespread occurrence of microplastics in the marine benthic environment. The results above are subject to various uncertainties, which are described in detail in the report (in section 7.1), and should be interpreted with these in mind.

¹ The definition maximum is used because all foreign particles that are found in the sediments are assumed to be plastic.

- 
- ✓ No clear trends could be seen for the following parameters: sampling location and depth, density of ZnCl₂:CaCl₂-solution used, total organic carbon content and dry weight percent of the sediment samples.
 - ✓ Further investigations and developments are needed to draw clearer conclusions with regards to sources and distribution of microplastics on the NCS. For example, systematic sampling and the use of polymer identification techniques, for instance FT-IR microscopy, to verify if particles are microplastic and identify the polymer type will provide valuable information in this regard.
 - ✓ The transport path of microplastics from the surface of the ocean to the seabed is very complex. Therefore, it cannot be expected to see a clean gradient of microplastics from areas where they are emitted to areas farther away. Further, because microplastics can be quite buoyant, they may be able to travel vast distances from their source before settling in sediment.

For a better understanding of the presence of microplastics in sediment, it is important to further investigate deposition of plastics to sediments and to carry out more rigorous testing for processing and extraction of microplastics from different sediment matrices. It is also important to further investigate the potential influence of environmental variables on microplastics concentration and composition in sediments. The few studies available in the literature on microplastics in sediment indicate there are orders of magnitude more microplastics on the seabed than on the ocean surface (Hidalgo-Ruz et al. 2012). The environmental impact of microplastics on benthic ecosystems are unknown but need further investigation (Galloway et al. 2017). This is particularly the case because the anticipated concentrations of microplastics are expected to increase in the foreseeable future, potentially reaching levels where they become a planetary boundary threat (Jahnke et al. 2017).

3 INTRODUCTION

Production of plastic has increased almost exponentially since 1950 and according to UN about 280 million tons of plastic is produced globally each year (<http://www.unenvironment.org/news-and-stories/story/innovation-abounds-plastic-substitutes-its-behaviour-change-will-save-our>). Just a small amount of this production is reused or recycled. The world's oceans are receiving roughly 13 million tons of plastic yearly and it is claimed that within 2050 the amount of plastic in the world oceans will exceed the amount of fish (http://www3.weforum.org/docs/WEF_The_New_Plastics_Economy.pdf).

In recent years, knowledge and an acknowledgement that plastic pollution in the marine environment is a global challenge, has grown. Plastic is found in all parts of the world's oceans such as the littoral zone, water surface, water column in general, at the seafloor, frozen in sea ice and in biota. Negative effects of plastic have been documented through findings in dead marine mammals and sea birds. At the same time a growing concern related to abundance and effects of smaller plastic parts and particles, sorted under the term microplastic, has arisen. The definition of microplastic is based on size and in general have plastic particles less than 5 mm been incorporated in this term.

To understand the environmental effects of microplastic in the marine environment and to implement mitigating actions knowledge about distribution and abundance are crucial. In conjunction with regional monitoring of the Norwegian Continental Shelf, on behalf of the Oil & Gas industry, DNV GL has organised and sampled sediments over a large area to acquire knowledge of distribution and abundance of microplastic in offshore sediments in different geographical areas, covering the central North Sea in the south and the Barents Sea in the north. The samples have been analysed by the Norwegian Geotechnical Institute (NGI) through an innovative sampling procedure including density separation, extraction and quantification.

4 PLASTIC IN GENERAL

Plastic is a general term used for materials produced industrially for decades and which have contributed to an increased quality of life for people throughout the world. Plastic is a synthetic polymer which can be divided into different categories such as thermoplastic. Thermoplastic has been foreshortened to plastic in every day speech (UNEP 2016). Plastic is a material that at some point in the manufacturing process can be made liquid or soft so it can be shaped. The main difference between what is called thermoplastic and thermoset is that thermoplastic can be reshaped during re-heating while thermoset cannot.

There exist many different plastic types with different properties such as polyvinyl plastic (PVC), polyurethane (PUR), polystyrene, bio-based plastic, biodegradable plastic and more. The areas of application are diverse and vary from packaging of food and other consumer products, plastic bags, drinking cups, in plates and laminates, in foundations of road constructions, in clothes, cosmetics and hygiene articles, in surgical implants, prosthesis and more.

5 MATERIAL AND METHODS

5.1 Sampling locations

During the regional environmental sediment monitoring on the Norwegian Continental Shelf on behalf of the Oil & Gas industry in 2017, 35 sediment samples from a large geographical area were sampled, covering the central North Sea, northern North Sea and the Barents Sea. A detailed overview of the sampling locations is presented in Figure 5-1.

All samples were collected with a van Veen grab with surface area 0.15 m², except for one sample from the central North Sea (EKO-14), where surface area sampled was 0.10 m². The whole 0-1 cm surface area of a dedicated grab was taken for each sample. The samples were stored in glass jars in air temperature during field work and stored in refrigerators at DNV GLs Biolab after demobilization until they were shipped to the Norwegian Geotechnical Institute (NGI) for analysis.



Figure 5-1. Overview of sampling areas with indications of sampling stations. Central North Sea in the south, northern North Sea in mid Norway and Barents Sea in the north.

An overview of the general large scale currents in Norwegian waters are presented in Figure 5-2. The general pattern is that warm salty water from the north Atlantic current (a branch of the Gulf stream) is flowing along the coast of Norway and into the Barents Sea, following the topography of the seafloor (Norwegian trench). Some branches of the north Atlantic current are penetrating the North Sea and even Skagerrak. In Skagerrak, currents from the Baltic Sea and fresh water run-off from land are meeting and forming the Norwegian coastal current which flows north along the Norwegian coast as a wedge between the coast and the North Atlantic current and into the Barents Sea. The Norwegian coastal current is characterised by lower salinity compared to its surrounding water masses which is due to less salty water from the Baltic Sea and fresh water run-off. This salinity difference weakens as the current flows north and is mixed with more salty water from the north Atlantic current.

In the southern part of the shallow North Sea the current is to a large degree influenced by wind. There is an eastward flow against Jylland (Denmark) and Skagerrak. In general, a flow against the clock can be seen which also forms a gyre in the southern parts which to some degree keep water circulating in this part of the North Sea. This area is to a large degree influenced by fresh water run-off from continental rivers in Europe such as the river Rhine and Elbe.

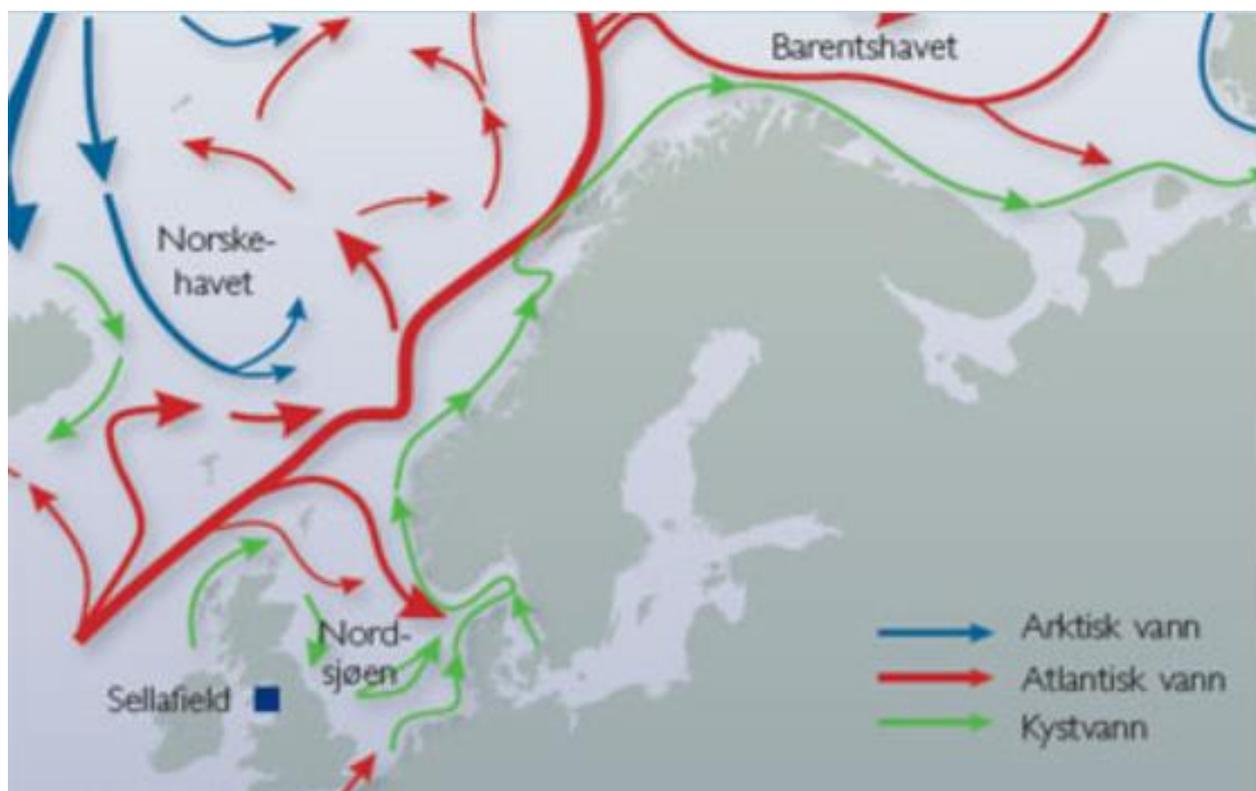


Figure 5-2. General large scale current patterns (picture from the Norwegian Institute for Marine Research, IMR).

5.1.1 Central North Sea

The central North Sea is a shallow area with a water depth of around 70 m. The sediments are mainly fine sand. Sediments were sampled at 20 stations in this area.

Ten of the samples were from so called regional stations. Regional stations are stations that represent the natural state in the area, meaning they are considered as not influenced by Oil & Gas activities, and as such can be considered as reference stations. The remaining 10 stations are sampled around Oil &

Gas fields, namely Ekofisk, Gyda, Valhall and the Ula field. Relevant station specific information is presented in Table 5-1 and a geographical overview is presented in Figure 5-3.

Table 5-1. Station information, central North Sea.

Sampling station	Field	Direction ¹⁾ (°)	Distance ²⁾ (m)	Depth (m)	Sediment characteristic	TOC (%)
Reg-01	Regional	n.r	n.r	73	Fine sand (MDΦ = 2.83)	0.30
Reg-02	Regional	n.r	n.r	68	Fine sand (MDΦ = 2.79)	0.31
Reg-03	Regional	n.r	n.r	68	Fine sand (MDΦ = 2.81)	0.37
Reg-04	Regional	n.r	n.r	71	Fine sand (MDΦ = 2.75)	0.32
Reg-06	Regional	n.r	n.r	72	Fine sand (MDΦ = 2.87)	0.33
Reg-07	Regional	n.r	n.r	73	Fine sand (MDΦ = 2.82)	0.36
Reg-08	Regional	n.r	n.r	70	Fine sand (MDΦ = 2.67)	0.32
Reg-09	Regional	n.r	n.r	66	Fine sand (MDΦ = 2.66)	0.19
Reg-11	Regional	n.r	n.r	71	Fine sand (MDΦ = 2.70)	0.29
Reg-14	Regional	n.r	n.r	80	Fine sand (MDΦ = 2.74)	0.24
EKO-12	Ekofisk	148	2500	78	n.a	n.a
EKO-14	Ekofisk	140	850	76	Very fine sand (MDΦ = 3.03)	0.48
EKO-21	Ekofisk	287	4000	71	n.a	n.a
Gyda-18	Gyda	135	250	67	Silt & clay (MDΦ = 5.12)	0.88
Gyda-21	Gyda	135	2000	67	n.a	n.a
VAL-02	Valhall	74	500	76	Fine sand (MDΦ = 2.82)	
VAL-04	Valhall	74	2000	72	n.a	n.a
VAL-05	Valhall	74	5000	70	n.a	n.a
VAL-15	Valhall	254	500	76	Fine sand (MDΦ = 2.80)	0.42
ULA-06	Ula	45	250	71	Fine sand (MDΦ = 2.86)	0.28

n.r: Not relevant

n.a: Not analysed

MDΦ = Median grain diameter (mm)

1) Direction/heading from Oil & Gas installation

2) Distance from Oil & Gas installation

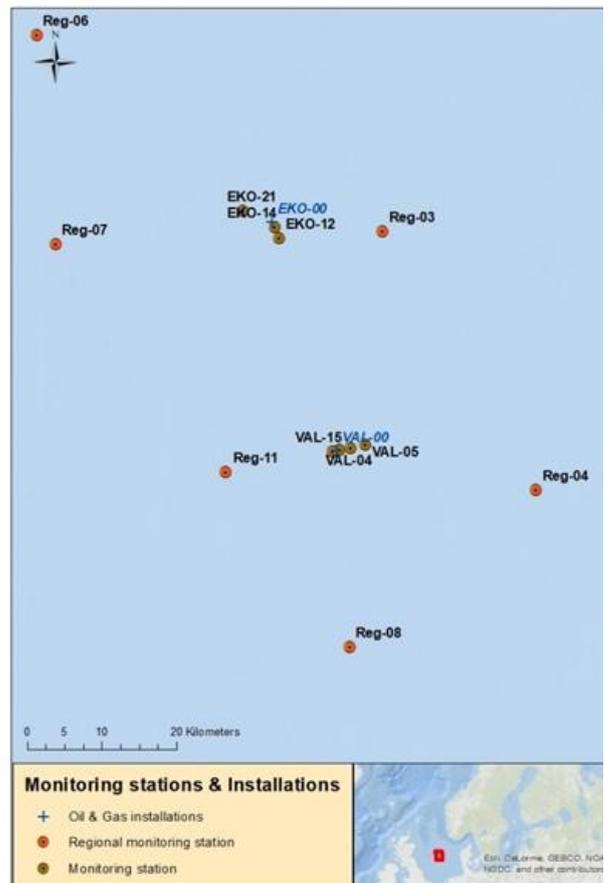


Figure 5-3. Overview of sampling stations in the central North Sea, southern part (left) and northern part (right).

5.1.2 Northern North Sea

The water depth in the sampling area is varying from 100 m to above 400 m in general. The varying water depth results in different sediments characteristics such as sand and gravel to finer material as clay and silt. Sediments were sampled at 10 stations in this region where 5 were regional/reference stations and 5 were stations in the vicinity to Oil & Gas fields namely the Kvitebjørn and the Visund fields. Relevant station specific information is presented in Table 5-2 and a geographical overview is presented in Figure 5-4.

Table 5-2. Station information, northern North Sea.

Sampling station	Field	Direction¹⁾ (°)	Distance m²⁾	Depth (m)	Sediment characteristic³⁾	TOC (%)⁴⁾
SNB-16R	Snorre B ref	315	10000	342	Silt and clay	-
VI-RB	Visund Ref	330	10000	330	Silt and clay	-
STC-06R	Statfjord C Ref	130	10000	137	Medium sand	-
Reg-12	Regional	n.r	n.r	400	Silt and clay	-
Vega-R	Vega Ref	-	-	380	Silt and clay	-
KV-14	Kvitebjørn	316	7224	187	Fine sand	-
KV-02	Kvitebjørn	140	500	185	Fine sand	-
VI-01	Visund	150	500	330	Silt and clay	-
VI-03	Visund	150	1000	330	Silt and clay	-
VI-30	Visund	330	250	316	Silt and clay	-

n.r: Not relevant

1) Direction/heading from Oil & Gas installation

2) Distance from Oil & Gas installation

3) Based on field description. Analytical results not available before March 2018

4) Analytical results not available before March 2018



Figure 5-4. Overview of sampling stations in northern North Sea.

5.1.3 Barents Sea

The samples taken in the Barents Sea were sampled in relation to baseline investigations covering relatively large areas in the northern area of the Norwegian Continental Shelf. The water depth in the region is variable, from 200 m to above 500 m. The varying water depth results in different sediments characteristics such as sand and gravel to finer material as clay and silt. Sediment were sampled at 5 stations in this region. Relevant station specific information is presented in Table 5-3 and a geographical overview is presented in Figure 5-5.

Table 5-3. Station information, Barents Sea.

Sampling station		Field	Direction (°) ¹⁾	Distance m ²⁾	Depth (m)	Sediment characteristic	TOC %
STT-2		Stangnestind	90	250	251	Silt and clay (MDΦ=5.31)	1.93
KF2-6		Korpfjell	85	900	242	Silt and clay (MDΦ=4.05)	1.76
SC3-4		Scarecrow3	270	100	461	Silt and clay (MDΦ=5.57)	1.56
KRT-14		Kråketind	n.r	n.r	440	Silt and clay (MDΦ=5.70)	1.34
GRS-2		Gråspett	90	250	508	Silt and clay (MDΦ=5.93)	2.09

n.r: Not relevant

MDΦ = Median grain diameter

1) Direction/heading from Oil & Gas installation

2) Distance from Oil & Gas installation



Figure 5-5. Overview of sampling stations in the Barents Sea.

5.2 Sample preparation and analytics

5.2.1 Sediment – microplastic separation

The 35 sediment samples from the Norwegian Continental Shelf (listed in Table 5-1 to 5-3) were received by NGI after delivery by DNV GL AS 2017-08-31. The samples were, preserved with 5% formaldehyde, processed and analysed for microplastics (45 µm – 5 mm) at NGI's certified Environmental laboratory.

Disclaimer: The method presented here is currently under development and at this time, the method quantifies the maximum weight and number of items that *could be* microplastic based on their density (lower than 1.53 g/mL), having a size range between 45 µm and 5mm, and resilience to an organic matter digestion process. Other materials such as soot, char, porous glass and ceramics would also be included in this quantification. Therefore, all data is presented as "maximum" microplastic concentrations within the aforementioned density and size range, based on the assumption that only microplastics are being separated.

5.2.2 Sample preparation

The samples were stored at NGI at 2-4 °C until processing. The first step of sample preparation was to decant the formaldehyde containing supernatant on top of the sample through a 45 µm steel mesh filter. The sediment was then transferred as quantitatively as possible from the glass containers to pre-weighed aluminium trays, and weighed in order to obtain the total sample wet weight.

The Percent dry matter (DM%) was obtained for all sediment samples by taking a subsample (ca 100 g wet weight) and drying it at 60 °C for two days or more, and using the following formula:

$$DM\% = \frac{\text{Dry weight (g)}}{\text{Wet weight (g)}} * 100\% \quad \text{Formula 1}$$

It is noted that a drying temperature of 60 °C was used instead of the more standard 110 °C, as a precautionary measure to prevent the potential melting of microplastics in the sub-sample.

The sediments were made into a homogenised slurry by adding ZnCl₂:CaCl₂-solution (ρ ~ 1.53). For some of the more cohesive sediment samples, having a paste like consistency, 0.1 % sodium dodecyl sulphate (SDS) surfactant was added to facilitate homogenisation.

5.2.3 Bauta Microplastic-Sediment Separator and extraction of sample

The Bauta microplastic-sediment separator (BMSS) was developed at NGI; its design was inspired by the Munich Plastic-sediment separator (MPSS) (Imhof et al, 2012). The BMSS is made up of four components: *the base* with a motorised propeller, the *sedimentation chamber* in which fluids are introduced and drained, a 650-mm tall *glass column* and *the sample separation chamber* with a ½" ball valve and a shut-off valve.

The purpose of the BMSS is to separate non-colloidal particles based on density, in which particles with a density less than the introduced fluid are collected in the separation chamber, and all denser particles are collected in the sediment chamber. Here the fluid used was filtered high-density ($\rho \sim 1.53$) $\text{ZnCl}_2:\text{CaCl}_2$ -solution (see section 5.5.1).

The BMSS was thoroughly cleaned, flushed with distilled water and inspected before each use, to ensure minimal particle contamination. Filtered high-density ($\rho \sim 1.53$) $\text{ZnCl}_2:\text{CaCl}_2$ -solution was added through the inlet valve, until the liquid level was within the constriction of the glass column. Approximately 700 g of homogenised sediment was introduced gradually from the top of the glass column and into the $\text{ZnCl}_2:\text{CaCl}_2$ -solution using a spoon or spatula, to allow sample break-up during descent in the glass column. The top of the glass column was then covered with aluminium foil. The sediment was stirred up by the bottom-fitted propeller at max speed (100 - 1000 rpm, depending on the Bauta) for 30 minutes and left over-night (for 15 hours or more) for density separation.

After separation, all floating materials (including microplastic, organic material and other debris with $\rho < 1.53$) were collected by first attaching the separation chamber to the top, and raising the level of the $\text{ZnCl}_2:\text{CaCl}_2$ -solution over the shut-off valve. The separation chamber was closed, dismantled and fastened in an inversed position for filtration. Filtration of sample in the separation chamber was done by rinsing out the sample onto a pre-weighed 45 μm stainless steel mesh (#300 Mesh - 0.045mm Aperture - 0.04mm Wire Diameter - SS316 Grade - Woven Wire, purchased from the Mesh Company, Warrington UK), using a vacuum pump and Milli-Q water. Particles that stuck to the walls of the Bauta were then rinsed off the walls into the $\text{ZnCl}_2:\text{CaCl}_2$ -solution by rinsing carefully with $\text{ZnCl}_2:\text{CaCl}_2$ -solution, and the filtration process was repeated until no particles in the Bauta were visible.

The steel mesh filter containing the combined filtrate sample from several rinsing's was carefully folded into a "tea-bag" like form, and tied up with a pre-weight steel wire. Finally, the samples were dried over-night at 60 °C and weighed before treated by chemical digestion.

5.2.4 Chemical digestion

A two-step chemical digestion process was performed for removal of organic material. The first step involves dissolving organic polymers, such as chitin and cellulose, using a mixture of NaOH, urea and thiourea (Zhang et al., 2013). During this step, some of the organic material dissolves and is rinsed through the "tea bag" filter, or is otherwise partially dissolved to facilitate oxidation in the second step.

The second step involves digesting the remaining sample with 30% H_2O_2 and NaOH. Initial tests with this digestion method indicated that it can successfully remove organic solids like paper and cotton ($98 \pm$

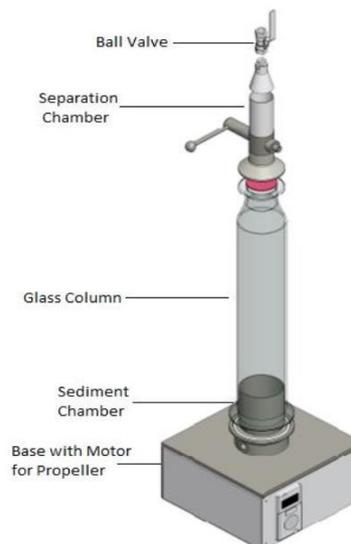


Figure 5-6. Schematic presentation of NGI's Bauta Microplastic-Sediment Separator (BMSS)

4 % sample digestion), yet is relatively harmless to the plastics tested (4% maximum sample digestion, for PET fibres). The digestion step was done at least once and repeated up to three times depending on the amount of organic matter present. The procedure is not described in further detail as it will soon be published (Olsen et al., in prep.).

5.3 Statistical analysis

Multivariate analysis was performed using principal component analysis (PCA) to describe the multivariate structure of the data (Sirius v. 8.1 ©1997-2007 by PRS, Norway). As the different variables investigated had different variance, the variables were scaled by standardization before analysis (Nortvedt et al., 1996).

To study variation among and between groups, ANOVA (analysis of variance) was used to analyse the differences among group means, followed by Tukey HSD post hoc test as the multiple comparison procedure. Both ANOVA and Tukey's test assume independence of samples, homogeneity of variance and normality of residuals (Zar, 1999). Box-Cox transformation was used to transform non-normalized data. In addition, both ANOVA and Tukey's test assumes approximately similar population sizes, even though both tests seem to be relatively robust against deviations from the assumptions (Zar, 1999, Osborne, 2010). The significance level was 0.05 (STATISTICA v. 13.1 ©1984-2016 by Statsoft, Tulsa, USA).

5.4 Quantification and quality control

The samples were quantified by weight and visually through light microscopy, as described in chapter 5.4.4. In both cases, precautions were taken to account for laboratory contamination through the use of blanks and keeping the sample closed to the laboratory atmosphere as much as possible. To reduce airborne contamination, several contamination prevention strategies were performed, such as thorough washing of the equipment with MilliQ or ultrasonic cleaning in MilliQ water, proper sealing of the samples to as much as possible etc. Two different types of blanks were used: method- and spiked blanks, as described below. Both method blanks and spiked blanks were performed for sediment samples from different sampling areas. Weights were recorded for both types of blanks. In addition, methods blanks were examined under a microscope.

5.4.1 Method blanks

The method blank sample protocol included the exact same steps as the sediment samples, except they were done without using any sediments. Blanks were controlled by weight and by microscopy. The method blank is used to evaluate contamination resulting from the preparation and analytical procedure.

The weight of materials in the blanks (m_{blank}) was calculated as:

$$m_{blank} = m_{total\ blank} - m_{filter} - m_{wire} \quad \text{Formula 2}$$

Where $m_{total\ blank}$ is the total weight of the dried filter containing the "blank" sample and steel wire after digestion and drying over-night at 60 °C, m_{filter} is the pre-weighed filter weight and m_{wire} is the pre-weighed wire weight.

5.4.2 Spiked blanks

The precision of the method was tested by adding a known amount of microplastics (granulates, fibres and/or micropowders) to sediment where organic matter and microplastics have already been removed by the BMSS separation step. After spiking the sediment sample, the spiked blank samples were then processed following the standard protocol for microplastics separation as described above (first separation with ZnCl₂:CaCl₂-solution, followed by digestion and then microscopy).

The weight of materials in the spiked blanks ($m_{spiked\ blank}$) was calculated similarly as the method blanks:

$$m_{spiked\ blank} = m_{total\ spiked\ blank} - m_{filter} - m_{wire} \quad \text{Formula 3}$$

Where $m_{total\ spiked\ blank}$ is the total weight of the dried filter containing the spiked "blank" sample. The spiked sample recovery was calculated as:

$$f_{recovery} = \frac{m_{spiked\ blank} - m_{blank}}{m_{spike}} \quad \text{Formula 4}$$

Where m_{spike} is the original amount of microplastics spiked into the sample.

5.4.3 Weight concentrations

The maximum weight of microplastic (m_{mMP}) in the processed sample was determined simply by weighing the steel-mesh and wire containing the digested sample, and subtracting the pre-weighed weight of the steel-mesh and wire. The resulting weight was corrected by subtracting the average weight from the method blanks, to account for weight contributions from lab or method contamination. In addition, the resulting average particle recoveries for the microplastic powder and fibre from the spiked blank were used as correction factors (recovery correction).

Thus, m_{mMP} was calculated through the formula:

$$m_{mMP} = \frac{m_{total\ sample} - m_{filter} - m_{wire} - m_{blank}}{f_{recovery}} \quad \text{Formula 5}$$

Preliminary tests indicated that the steel-mesh and filter weight are stable throughout the procedure; though, in some cases after sample drying, rust could form on the steel-mesh likely due to ZnCl₂ residue. Artefacts from rust formation were avoided by 1) weighing/re-digesting as soon as possible after drying, 2) storing processed samples in a desiccator, and 3) reprocessing samples with substantial rust evident. After obtaining m_{mMP} , concentrations of microplastic in sediment could be reported in units of mg/kg dry sediment or mg/m² sediment surface to allow comparison with other studies, using the following relationships.

Max weight concentration:

$$C_{microplastic\ d.w.} \left(\frac{mg}{kg_{d.w.}} \right) = \frac{m_{microplastic}}{m_{bauta\ sed.d.w.}} \quad \text{Formula 6}$$

Max area concentration:

$$C_{\text{microplastic area}} \left(\frac{\text{mg}}{\text{m}^2} \right) = \frac{m_{\text{microplastic}} * \frac{m_{\text{bauta sed.w.w.}}}{m_{\text{sed.w.w.}}}}{A_{\text{grab}}} \quad \text{Formula 7}$$

Where $m_{\text{bauta sed d.w.}}$ and $m_{\text{bauta sed w.w.}}$ are the dry weight and weight of sediments introduced into the BMSS, respectively, $m_{\text{sed w.w.}}$ is the total wet weight of the sample collected and A_{grab} is the area of the grab sample (here 0.15 m² for all samples except for one sample from the central North Sea (EKO-14), where a surface area of 0.10 m² were sampled).

It is noted that for both weight and visual analysis, there was no instrument available to objectively identify the plastic content or types of plastic in the processed samples, such as FT-IR, Raman or pyrolysis GC-MS. Without such objective quantification, the methods used here present a quantification of material with a density less than 1.53 in the sediments that can survive the applied digestion procedure. In addition to plastic, this can include other forms of low-density materials other than plastic that are resilient to the digestion method, like bitumen, charcoal and some forms of natural organic matter. Therefore, the data presented here represent a maximum level for microplastics, with the operator "≤".

5.4.4 Visual identification of microplastics

Identification and quantification of microplastics without automated software is a difficult and time-consuming method, based on subjective judgments and assumptions. The use of novel approaches that integrate objective methods of confirming plastic identity (e.g. FT-IR, Raman, pyrolysis GC-MS) and automated counting software are preferred, and are starting to become available (Bergmann et al., 2017); however, the scientific community is still some way from developing standard protocols. A limiting factor in developing standard protocols is that even automated software packages to conduct counting are based on key assumptions, and biases caused by these assumptions are often not straightforward to identify.

No objective methods of confirming plastic identity were available for this study. Rather, a visual microscopic analysis was included to collect descriptive and qualitative data and additional knowledge for the respective samples. The samples were stored in such a way that further analysis with an FT-IR microscope can be done later (the NGI will acquire such a device in February 2018).

The processed samples on the steel mesh filters were investigated using a microscope with a mounted NIKON Coolpix 950 camera. The microscope was set at x10 and x40 magnification. Before being examined under the microscope, each sample were unfolded and placed between two clear 10x10cm acryl plates that were each 3-mm thick (Clas Ohlson, Sweden), for flattening and to lock the sample in place. Each individual plate had a thin protective plastic cover which were removed from the plates before the plates were carefully placed over and under the samples in a way that collects little or no atmospheric particles on their surface. A transparent 5x5 cm gridded film were placed on top of each sample for systematic counting, starting from the left and moving to the right following a pattern as described in the MERIs (Marine & Environmental Research Institute) guide to microplastic identification (MERI, 2015). The outermost rows and columns were not used (Figure 5-7).

As a basis for discerning plastic from non-plastic/biological material during this counting approach, MERIs guide to microplastic identification (MERI, 2015) was used. In short, the particles that resembled microplastic were visually distinguished according to the following criteria: no cellular or organic structure visible, fibres should be equally thick throughout the entire length, and particles should present a clear and homogenous colour.

Each potential plastic particle was morphologically noted based on their size, shape (fibre (1D), layer (2D) or granule (3d)) and colour, according to the scheme presented in Table 5-4. For effective categorising, particles were subdivided into four size groups A, B, C and D, where group A are particles $\geq 45 \mu\text{m}$ to $100 \mu\text{m}$, group B are particles between $100\text{-}300 \mu\text{m}$, group C are particles between $300 - 1000 \mu\text{m}$ and group D are particles from 1 to 5 mm .

This counting procedure was conducted for all method blanks, all sediment samples with measured mass below the limit of detection, and for a selection of other samples. Not all samples could be included because many of them had too many particles to count. Results from the counting procedure are shown in Appendix C.

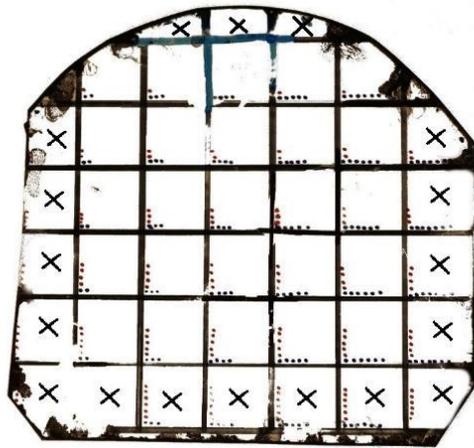


Figure 5-7. The transparent 5x5 cm grid system for systematic counting of possible microplastic items. Each cell is 1x1cm and is marked with a unique set of dots 1-1, 1-2... etc.

Table 5-4. Spreadsheet used to log data for visual analysis of microplastics during microscopy.

Colour	Fibre 1D				Layer 2D				Granule 3D			
	A	B	C	D	A	B	C	D	A	B	C	D
Clear/white												
Light brown	≥ 45 to $< 100 \mu\text{m}$	100-300 μm	300 - 1000 μm	1 - 5 mm								
Dark brown												
Black												
Blue												
Red												
Green												
Orange												
Yellow												

The maximum number of counted microplastic items in the sediment samples ($n_{\text{mMP sample}}$) were corrected due to the number of counted particles in the Method Blanks. The sample correction was done by subtracting the maximum number of possible MP items within an individual group (e.g. white granules, size class B) by the average number of particles within that group deriving from the Method Blanks ($n = 8$), as seen in Formula 8 – 9.

$$n_{mMP\ type} = n_{mMP\ type\ in\ sample} - n_{mMP\ type\ in\ Method\ Blank}$$

Formula 8

$$n_{mMP\ sample} = \sum n_{mMP\ type}$$

Formula 9

Where:

- n_{mMP} , = mMP items within an individual group in the sample
- $n_{mMP\ type\ in\ sample}$ = average mMP items (within an individual group) counted in the sample
- $n_{mMP\ type\ in\ Method\ Blank}$ = average number of items (within an individual group) counted in the method blanks
- $n_{mMP\ sample}$ = blank corrected mMP items in the sample

For samples where the particle numbers could not be determined because single particles could not be distinguished, the number of items were extrapolated from samples where $n_{mMP\ sample}$ was determined and their corresponding mMP weights (m_{mMP}) weights, provided that it was above the LOD, by using the following extrapolation factor (f_e):

$$f_e = \frac{n_{mMP\ sample}}{m_{mMP}}$$

Formula 10

Where:

- $n_{mMP\ sample}$ = average maximum number of MP items in the sediment samples
- m_{mMP} = average maximum weight of MP in the sediment samples (Formula 5)

Thereafter, the estimated number of mMP items was calculated by multiplying the extrapolation factor with the dry weight of sample material after digestion (m_{mMP}).

To allow comparison with other studies, the results could be reported in units of items/kg dry sediment and items/m² sediment surface, using the following relationships:

Max weight concentration:

$$C_{microplastic\ d.w.} \left(\frac{items}{kg\ d.w.} \right) = \frac{n_{mMP\ sample}}{m_{bauta\ sed.\ d.w.}}$$

Formula 11

Max area concentration:

$$C_{microplastic\ area} \left(\frac{items}{m^2} \right) = \frac{n_{mMP\ sample} * \frac{m_{bauta\ sed.\ w.w.}}{m_{sed.\ w.w.}}}{A_{grab}}$$

Formula 12

5.5 Chemicals and solvents

All chemicals used during solution preparation are listed below.

Table 5-5. List of chemicals.

Chemicals	Molecular formula	Manufacturer/ Distributor	Purity (%)
Zinc Chloride	ZnCl ₂	VWR International	97
Calcium Chloride	CaCl ₂	VWR International	90-98
Hydrogen peroxide	30 % H ₂ O ₂	VWR International	Analytical grade
Urea	CO(NH ₂) ₂	Sigma Aldrich	≥ 98
Thiourea	CH ₄ N ₂ S	Merck K GaA	≥ 98
Sodium hydroxide	NaOH	Merck K GaA	99 – 100
Sodium dodecyl sulphate	CH ₃ (CH ₂) ₁₁ OSO ₃ Na	Sigma Aldrich	≥ 99 (Chromatography)

Table 5-6. List of microplastics used for spiked blanks.

Form	Type	Manufacturer/ Distributor	Properties
Powder	Polyester (PET, PETP)	Goodfellow Cambridge Ltd. (UK) - catalogue nr. ES306030	Density 1.40 g/cm ³ Diameter 75 – 300 µm
Fibre	Polyethylene (LDPE)	Goodfellow Cambridge Ltd. (UK) catalogue nr. ET315710	Density: 0.92 g/cm ³ Length 5 – 10 mm
Granulate	Polyester (PET)	Goodfellow Cambridge Ltd. (UK) catalogue nr. ES306312	Density = 1.40 g/cm ³ Nominal size range 3 – 5 mm.

5.5.1 Preparation of ZnCl₂:CaCl₂-solution

The ZnCl₂:CaCl₂-solution was prepared by combining distilled water, ZnCl₂ and CaCl₂ in this exact order. The ratio by weight was 4.4: 3.6: 2 kg (ZnCl₂:CaCl₂:H₂O) (Hudgins, C. M., 1964). As the reaction is exothermic, the carboys were placed in a tub filled with ice under a fume hood. Precipitates and impurities were centrifuged and filtered out by first placing the salt solution in Nalgene centrifugation vials, rotating at 4000 RPM for 10 minutes and then filtering the supernatant through a Whatman GF/D filter using a high-pressure filtration apparatus. Finally, the density of the solution was controlled by dividing the weight of filtered ZnCl₂:CaCl₂ solution in a 100-mL volumetric flask by the volume. The density was recorded for each sample, and on average the density of the solution was on average 1.53 ± 0.02 g/mL.

5.5.2 Preparation of solutions used for chemical digestion

The NaOH, urea, thiourea solution was made according to Olsen et al. (in prep). The 30 % H₂O₂ was made by dilution of 50 % H₂O₂ with Milli-Q water.

6 RESULTS

6.1 Quality control

6.1.1 Method Blanks

In total, 11 method blanks were prepared and analysed for impurities due to the method used, as shown in Table 6-1. Raw data is provided in Appendix A.

Table 6-1. Calculated weight of impurities for method blanks.

Mehod Blank-ID	Date	Density of ZnCl ₂ :CaCl ₂ (g/mL)	Weight of collected impurities (g)	
			Before chemical digestion	After chemical digestion
20170922-Blank1	22.09.2017	1.54	0.0059	0.0001
20170922-Blank2	23.09.2017	1.57	0.0023	0.0003
20170922-Blank3	24.09.2017	1.55	0.0046	0.0015
20171019-Blank	19.10.2017	1.51	0.0126	0.0000
20171109-blank 5:1	09.11.2017	1.51	0.0196	0.0026
20171123-blank*	23.11.2017	1.52	-	-
20171121-Blank	22.11.2017	1.50	0.0119	0.0005
20171129-Blank1	29.11.2017	1.48	0.0038	0.0008
20171129-Blank2	29.11.2017	1.47	0.0027	0.0001
20171206-Test1	06.12.2017	1.52	0.0108	0.0013
20171312-Test2	13.12.2017	1.53	0.0136	0.0031
Average		1.52	0.0088	0.0010
Standard deviation		0.03	0.0055	0.0011

*No weight recorded, only used for visual identification of possible impurities

As earlier reported, the steel mesh used for filtration had a mass recovery rate of more than 100 % (Mahat, 2017). The impurities collected on the filters contributed to 0.001 g average additional weight after chemical digestion. Thus, the dry weights of sample microplastics were blank corrected. The limit of detection (LOD) and limit of quantification (LOQ) are commonly calculated as three times standard deviation and ten times standard deviation, respectively. However, common practice within microplastics research is to correct the results based on particles identified in method blanks. As such, the LOD was set to 0.001 g, as this was the average dry weight of impurities in the Method Blanks for this report. The LOQ was set to three times the LOD.

Even though some of the impurities were removed through chemical digestion, some ZnCl₂ crystals (< 45 µm) and fibres were retained on the steel mesh also after digestion, as shown in Figure 6-1. Fibres were also identified in method blanks, with the source being likely from clothes worn in the lab (e.g. blue and white lab coats). The average number of particles observed in the method blanks (n=8) were 55.5, as shown in three blanks (Blank 1-20171129, 20171206-Test1 and 20171312-Test2) were not included in the blanks correction for visual analysis due to abnormal amounts of suspected ZnCl₂ crystal impurities.

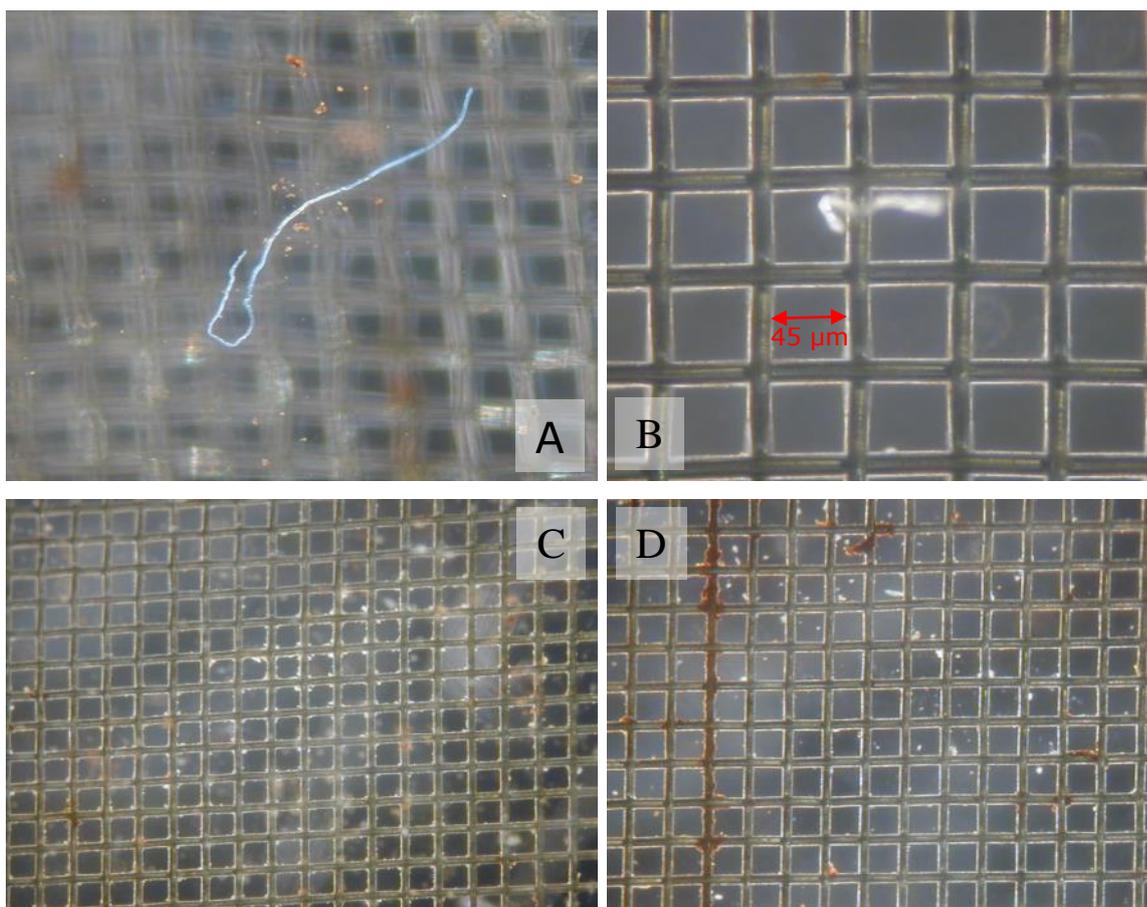


Figure 6-1. Impurities observed under on steel mesh (45 µm) after chemical digestion. A = blue fibre, B = white fibre, C and D = ZnCl₂ crystals.

Table 6-2. Abundance of particles within each individual group, based on 8 method blanks.

	Colour	Fibre 1D				Layer 2D				Granulat 3D			
		A	B	C	D	A	B	C	D	A	B	C	D
Average (n=8)	Clear/white	3.5	5.9	4.5	1.9	1.8	0.4	0.8	0.1	19.0	3.6	0.0	0.0
	Light brown	0.0	0.3	0.0	0.0	1.8	1.1	0.6	0.3	2.3	2.0	0.1	0.0
	Dark brown	0.0	0.1	0.0	0.0	0.3	0.0	0.0	0.0	0.9	0.3	0.3	0.0
	Black	0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.4	0.0	0.0	0.0
	Blue	0.1	0.6	0.5	0.6	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0
	Red	0.0	0.0	0.1	0.0	0.3	0.0	0.0	0.0	0.8	0.0	0.0	0.0
	Green	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Orange	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.3	0.1	0.0	0.0
	Yellow	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Sum*	3.8	6.9	5.1	2.5	4.0	1.6	1.4	0.4	23.5	6.0	0.4	0.0
	Fraction of total	18.3		32.9 %		7.4		13.3 %		29.9		53.8 %	
SUM									55.5				

* The SUM accounts for non-significant digits that are not displayed.

When correcting for method blanks, mainly clear/white particles and blue fibres were corrected for, as these were the most abundant particles.

6.1.2 Spiked Blanks

In total, eight spiked blanks were prepared and weighed to predict the recovery rates of microplastics from the environmental samples, as shown in Table 6-3. Raw data for the spiked blanks are given in Appendix A.

Table 6-3. Recovery rates for three types of microplastics used for spiking (PET powder, PE fibre, PET pellets)

Spiked Blank	Date	Density of ZnCl ₂ :CaCl ₂ (g/mL)	Weight recovery rate (%)		
			PET powder	PE fibre	PET pellets
20171002-R1-11-Blank	02.10.2017	1.51	43	-	-
20171004-Reg-03-Blank	04.10.2017	1.51	70	-	-
20171005-Reg-09-Blank	05.10.2017	1.51	-	95	100
20171009-Reg-01 Blank	06.10.2017	1.52	-	97	100
20171114-SNB-16R-Blank1	13.11.2017	1.55	-	83	-
20171114-SNB-16R-Blank2	13.11.2017	1.57	56	-	-
20171130-KRT-14-Blank 1	30.11.2017	1.48	-	89	-
20171130-KRT-14-Blank2	30.11.2017	1.47	84	-	-
Average		1.51	63	91	100
Standard deviation		0.03	18	6.4	0

Average recovery rate of PET pellets was 100 ± 0 %. 91 ± 6 % recovery rates were obtained for the PE fibres, whereas PET powder resulted in 63 ± 18 % average recovery rate.

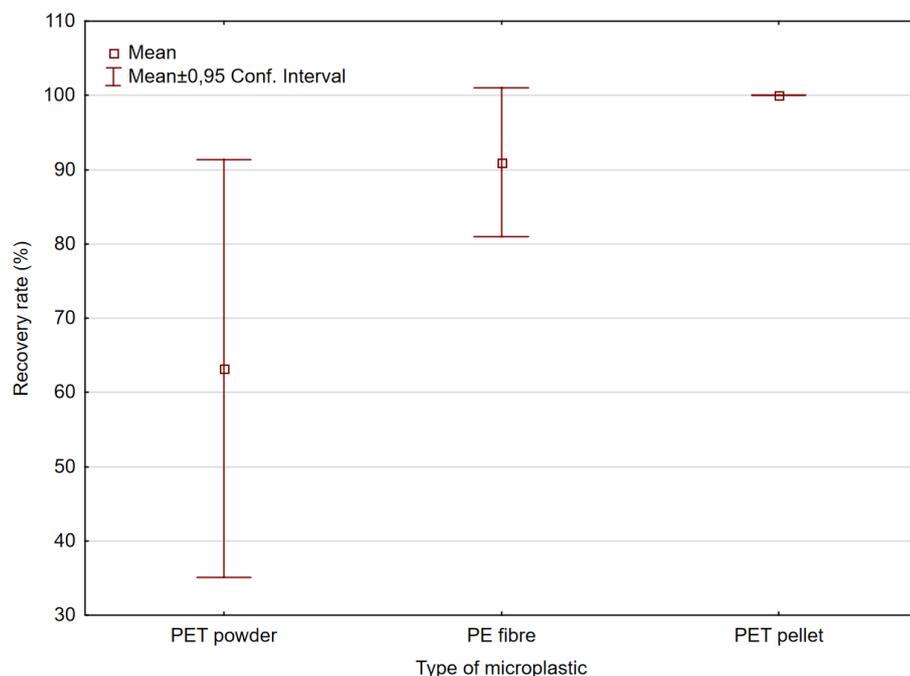


Figure 6-2. Recovery rates (%) with 95% confidence interval of PET powder, PET pellets and PE fibres. PET powder has the lowest recovery rates.

The causes of the sample loss could have been coming from each step of the method: 1) failure for the microplastic to separate from the sediment; 2) microplastic adhering to walls and surfaces of the glass column or separation chamber; 3) leakage from the steel mesh filters during filtration, digestion or drying; and 4) loss due to digestion. This is despite the effort, for each of these steps, that care was taken to be as quantitative as possible.

The total average recovery rate obtained for PE fibres and PET powder was $77 \pm 20 \%$. Thus, a fraction (f_{recovery} , see formula 4) of 0.77 was used for recovery correction for the dry weight of sample microplastics. Visual images of the different spiking material are shown in Figure 6-3.

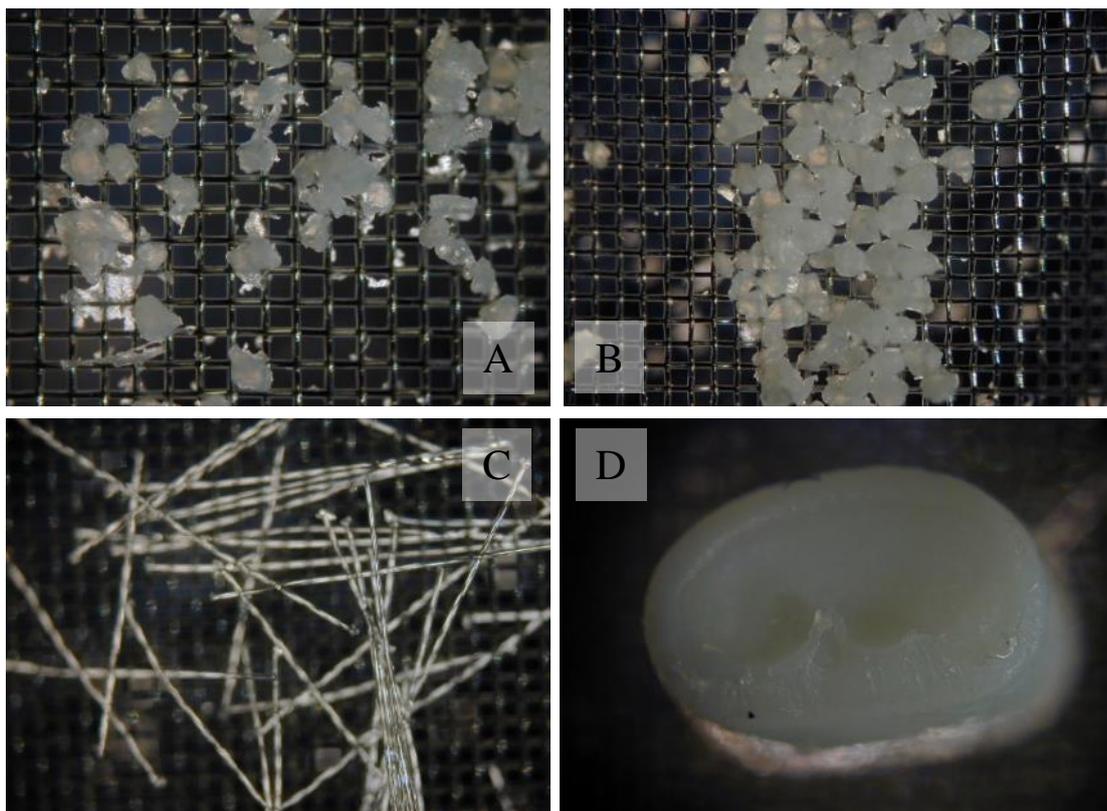


Figure 6-3. Photos of spiking material (x10 magnification). A and B: PET powder; C: PE fibre; D: PET pellet.

6.1.3 Deviation between parallels

As described in the method section, approximately 700 g homogenised sediment was introduced to the BMSS for density separation followed by chemical digestion, which corresponds to roughly 50 % of the total sample weight. The total sediment sample from station Reg-06 (regional station in the central North Sea) was divided in two parts for MP quantification. In the first replicate, the sample was found to contain 110 mg mMP/kg dry weight, and the second 190 mMP/kg dry weight, giving a deviation of 55 % or within a factor two. It is unclear if the cause is lack of sample homogenisation or other artefacts. Due to time constraints, parallel analysis was not performed for more than one sediment sample.

6.2 Sediment samples

6.2.1 Water Content

Average dry weight percent for all the sediment samples were 68 ± 14 %, ranging from 36 to 80 %. The sediment samples from the central North Sea had significantly higher dry weight percent (lower water content) than the sediment samples from northern North Sea and Barents Sea (Figure 6-4). The determined water contents and the resulting dry weights of the sediments were used for the analysis.

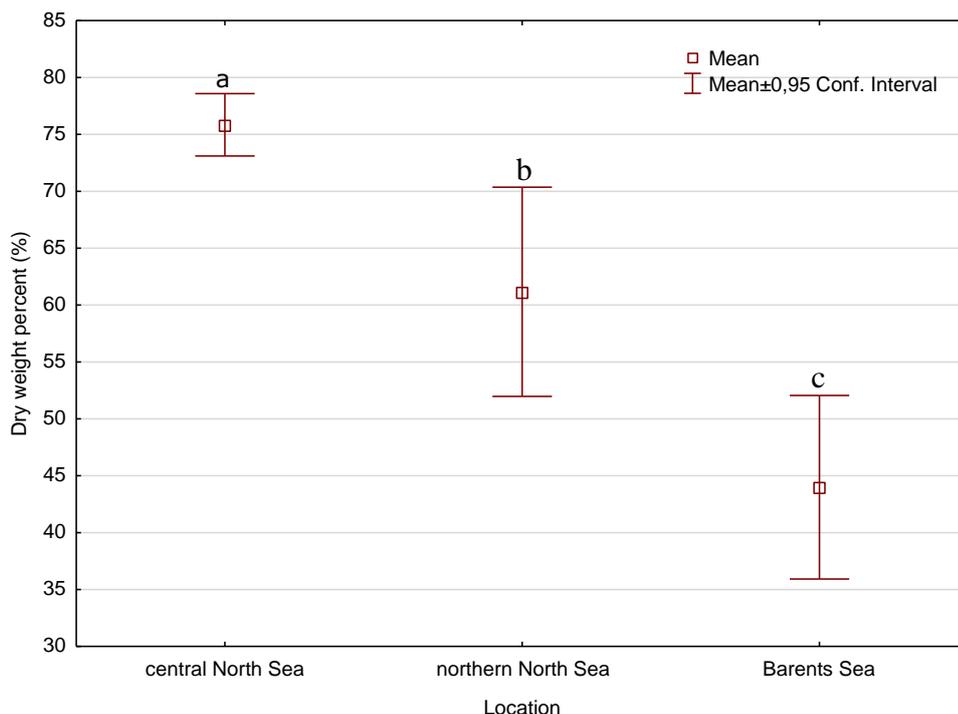


Figure 6-4. Average dry weight percent for the sediment samples. Different letters indicate statistical differences ($p < 0.05$). The results are shown as mean \pm 0.95 confidence interval.

6.2.2 Maximum microplastic concentration by weight and number of items

As it cannot be quantified at this current time how much of the low-density, chemically resistant materials are microplastics, data is interpreted as the "maximum microplastic" mMP.

In Table 6-4, the obtained mMP concentration values are listed. The concentrations are given as both mg and number of items per kg dry sediment and per m^2 sediment surface to allow comparison with other studies. As seen in the table, a substantial number of possible MP items were counted also for the samples with concentrations lower than LOD on a weight basis.

Table 6-4. Maximum microplastic concentrations (mMP) by weight and number of items. SD = standard deviation. LOD = 0.001 g corrected dry weight.

Station	Location area	mMP concentration			
		mg/kg	mg/m ²	Items/kg	Items/m ²
Reg-01*	central North Sea	61	500	4 600	38 000
Reg-02*	central North Sea	6.1	53	470	4 000
Reg-03*	central North Sea	16	130	1 200	10 000
Reg-04*	central North Sea	120	1 000	9 200	79 000
Reg-06*	central North Sea	150	1 200	11 000	93 000
Reg-07*	central North Sea	64	570	4 900	43 000
Reg-08*	central North Sea	6.4	58	490	4 400
Reg-09	central North Sea	4.6	21	730	3 400
Reg-11*	central North Sea	13	100	990	7 700
Reg-14	central North Sea	< LOD	< LOD	490	4 100
EKO-12*	central North Sea	91	710	6900	54 000
EKO-14*	central North Sea	22	260	1 700	20 000
EKO-21*	central North Sea	130	1 100	10 000	87 000
GYDA-18*	central North Sea	6.2	42	370	2 500
GYDA-21*	central North Sea	220	1 800	17 000	130 000
VAL-02*	central North Sea	130	960	9 700	73 000
VAL-04*	central North Sea	150	1 100	11 000	84 000
VAL-05*	central North Sea	94	740	7 200	56 000
VAL-15*	central North Sea	69	540	5 200	41 000
ULA-06*	central North Sea	410	3 200	31 000	250 000
Reg-12	northern North Sea	< LOD	< LOD	180	700
SNB-16R	northern North Sea	< LOD	< LOD	580	2 200
VI-RB	northern North Sea	< LOD	< LOD	640	1 300
STC-06R*	northern North Sea	11	110	810	8 200
KV-14*	northern North Sea	40	510	3 100	39 000
KV-02*	northern North Sea	64	650	4 900	50 000
VI-01*	northern North Sea	63	320	4 800	25 000
VI-03	northern North Sea	19	110	550	3 200
VI-30*	northern North Sea	120	380	8 800	29 000
Vega-R	northern North Sea	< LOD	< LOD	280	770
STT-2*	Barents Sea	36	90	2 700	6 900
KF2-6*	Barents Sea	18	83	1 400	6 300
SC3-4*	Barents Sea	41	130	3 200	9 600
KRT-14	Barents Sea	14	52	830	3 000
GRS-2*	Barents Sea	51	130	3 900	9 900
Average ± SD (min-max)		60 ± 80 (< LOD-410)	480 ± 660 (< LOD-3 200)	4 900 ± 6 200 (180-31 000)	37 000 ± 50 000 (700-250 000)

*Estimated number of possible MP items as the particle number could not be determined because single particles could not be distinguished. The extrapolation method was therefore used.

6.2.3 Maximum microplastic concentration by weight

Based on the corrected weights of the processed samples, low-density, chemically resistant materials were found above the LOD in 30 of the 35 analysed sediment samples. The average concentration of mMP separated from the sediment samples using the developed method were found to be 60 ± 80 mg mMP/kg dry sediment, ranging from concentrations under the limit of detection (LOD = 0.001 g mMP based on the results from the method blanks) to max 410 mg mMP/kg dry sediment. The highest microplastic concentrations are in general found at locations close to Oil & Gas installations; however, the method uncertainties should be kept in mind, such as the presence of other particles that are not plastic but have a similar density and chemical resistance profile as plastics (e.g. soots, chars, porous glass and ceramics).

The highest mMP concentrations were found in the samples from the central North Sea (Figure 6-5), where the sediment sample from station ULA-06 had the highest mMP concentration of 410 mg mMP/kg



dry sediment, followed by GYDA-21 with 220 mg mMP/kg dry sediment. Samples from northern North Sea and the Barents Sea had in general lower concentrations compared to the central North Sea. As seen in Figure 6-6, the average mMP concentration (mg/kg dry sediment) was approximately three times lower in the samples from northern North Sea (30 ± 40 mg mMP/kg) and the Barents Sea (30 ± 20 mg mMP/kg), compared to the central North Sea (90 ± 100 mg mMP/kg). However, the concentrations did not vary significantly between the locations ($p > 0.05$). The highest MP concentration found in the samples from northern North Sea was 120 mg mMP/kg (VI-30), whereas the highest mMP concentration in the samples from the Barents Sea was 51 mg mMP/kg (GRS-2).

Some of the "Regional" samples in central North Sea had higher concentrations than measured in northern North Sea and the Barents Sea, such as Reg-04 and Reg-06 (with 120 and 150 mg mMP/kg dry sediment, respectively). The reason for relatively high concentrations at some regional stations in the central North Sea is unclear. The patchy distribution is difficult to explain by sediment type (all fine sand), depth or TOC content. In addition to Oil & Gas activities, fishing and other related marine activities within the central North Sea, land based sources may be an important influencing factor. The central North Sea is influenced by continental Europe through ocean currents and water run-off from large rivers such as Elbe and Rhine. There is a gyre like circulation in this part of the North Sea which may accumulate plastic debris in this area. In this regards it is interesting that station Reg-14, which is the most northerly location in this area has concentrations $< \text{LOD}$.

For comparison with other studies, the mMP concentrations were converted to mg mMP per m^2 . The corresponding results are shown in part B in Figure 6-5. As shown in the figure, the concentrations varied from values under the LOD to max 3 200 mg mMP/ m^2 , with an average of 480 ± 660 mg mMP/ m^2 .

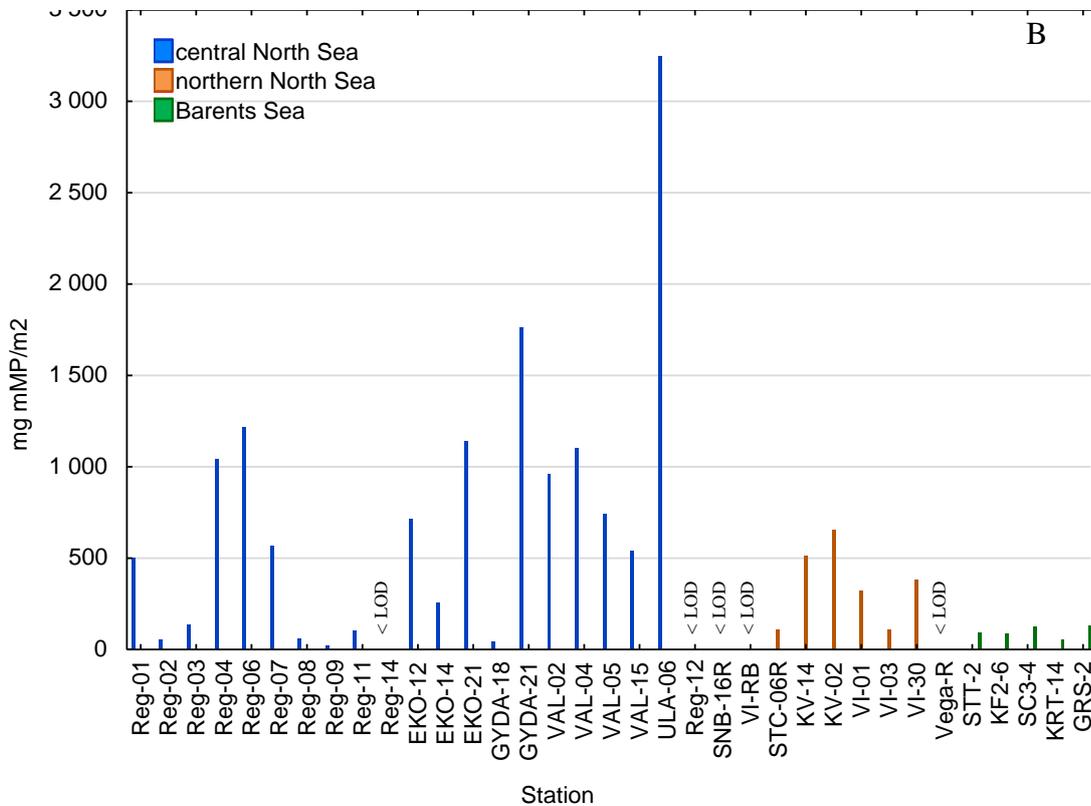
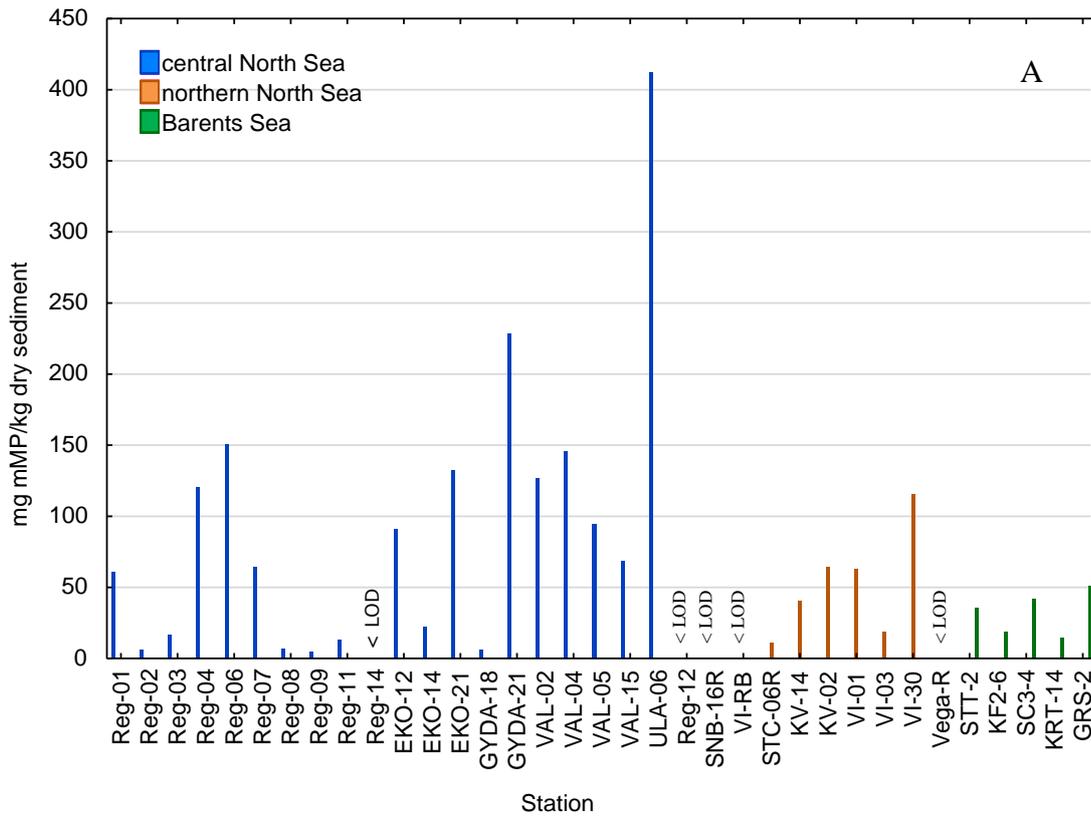


Figure 6-5. Blank- and recovery corrected maximum microplastic (mMP) concentration (A: mg mMP/kg sediment dry weight; B: mg mMP/m²) are shown. LOD = limit of detection.

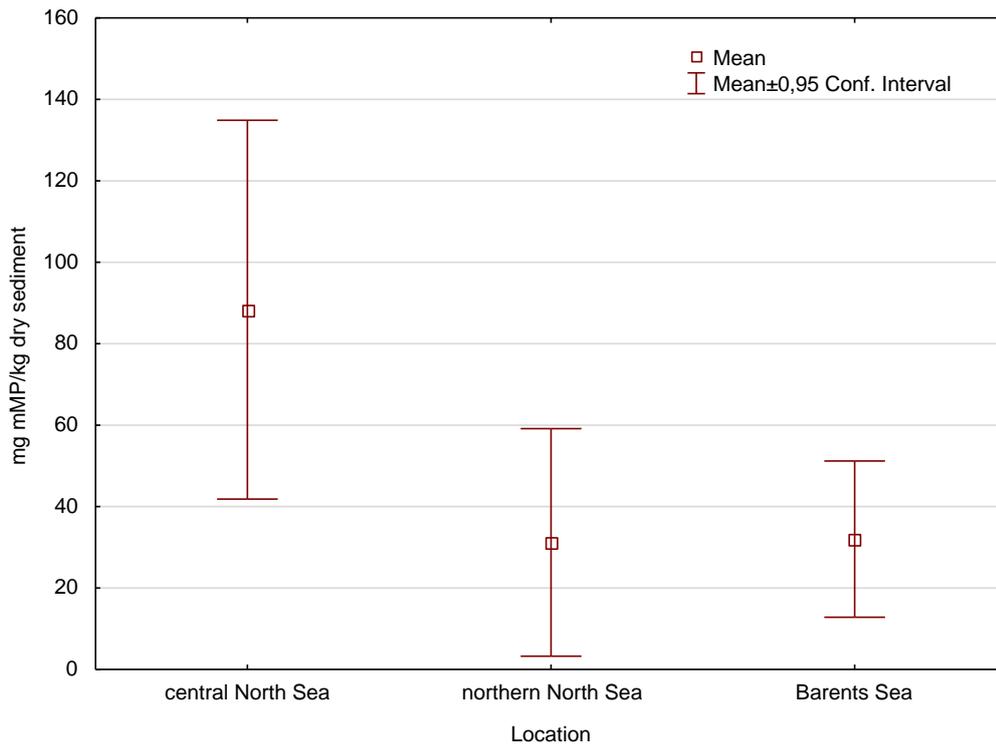


Figure 6-6. Mean maximum microplastic concentration (mg mMP/kg sediment) in sediment samples from central North Sea, northern North Sea and Barents Sea. The results are shown as mean \pm 0.95 confidence interval.

6.2.4 Maximum microplastic concentration by abundance

By visual quantification, low-density, chemically resistant materials were found in all investigated sediment samples, also for the samples with mMP weights lower than the limit of detection. For the samples with relatively high mMP concentration on a weight basis, the number of particles were too many to count, as the particle number could not be determined because single particles could not be distinguished. This is illustrated for three samples from the central North Sea in Figure 6-7 to Figure 6-9. Corresponding pictures of all analysed samples are shown in Appendix B.

Based on the actual and estimated number of items, the average concentration of mMP separated from the sediment samples using the developed method were found to be $4\,900 \pm 6\,200$ mMP items/kg dry sediment, ranging from 180 to a maximum of 31 000 mMP items/kg dry sediment. Converted to items per m^2 , this corresponds to approximately $37\,000 \pm 50\,000$ mMP items/ m^2 , ranging from 700 to 250 000 mMP items/ m^2 sediment area.

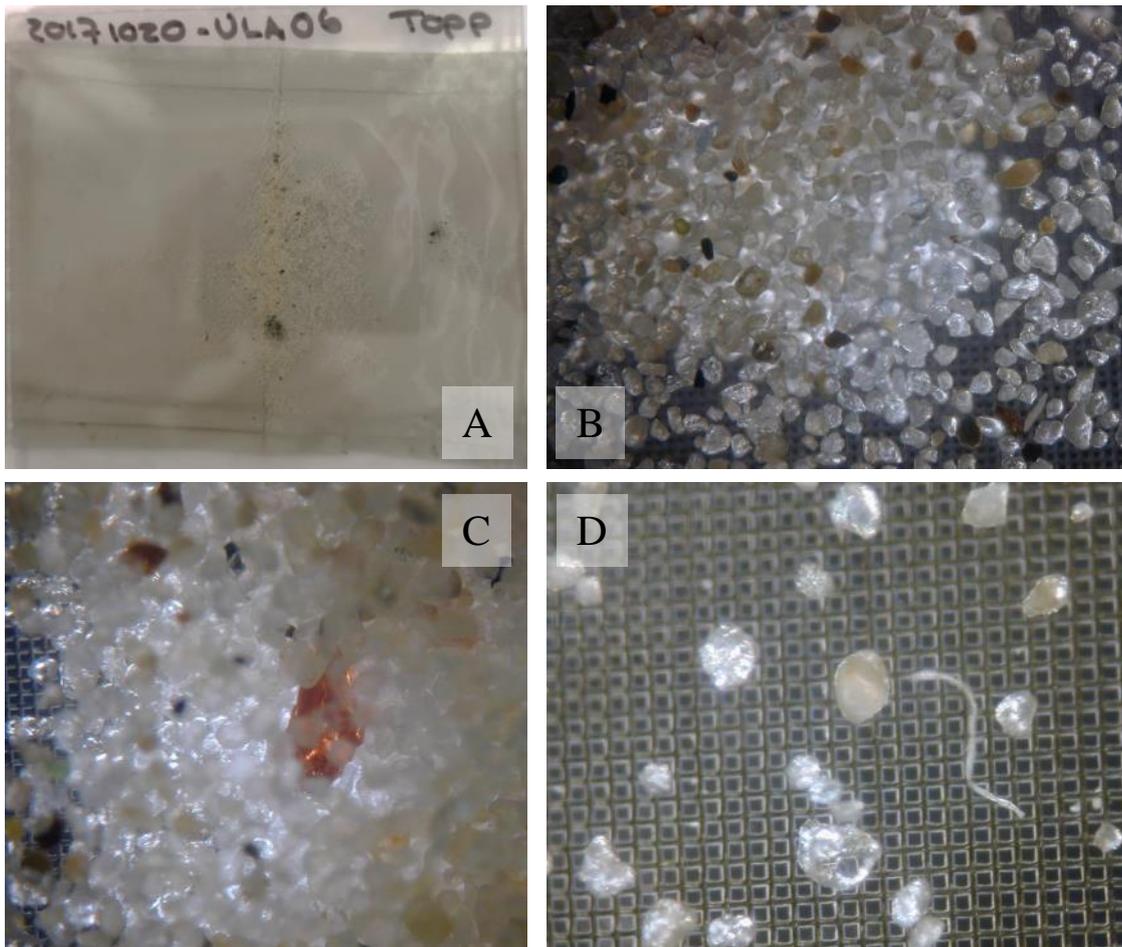


Figure 6-7. Panel showing pictures of possible microplastics for sediment sample ULA-06. A: overview of sample; B: representative picture of sample showing many granules (< 500 μm); C: observed, red granule (>1000 μm) among smaller granules (<500 μm); D: white fibre < 1000 μm .

As seen in Figure 6-7, the majority of particles in the sediment sample from ULA-06 were granules < 500 μm . Most of the granules were clear or white in colour, whereas some were light brown and black (it is possible the black granules were coal or charcoal), as well as a few green and red granules. In addition to granules, some fibres and films were observed.

The sediment sample GYDA-21, also from the central North Sea, had a similar particle distribution (and high MP concentration) as ULA-06. Pictures of GYDA-21 are shown in the following figure.

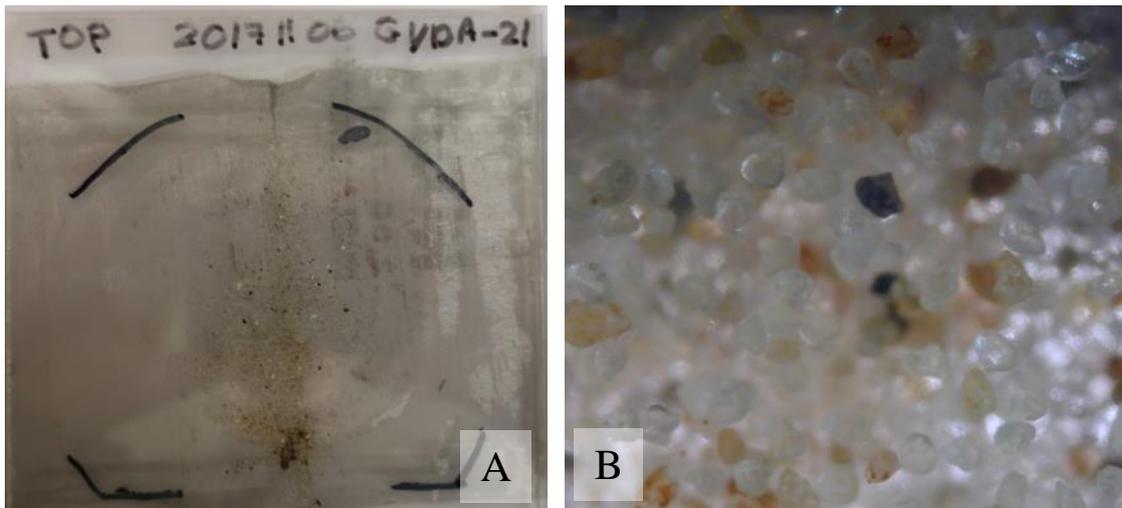


Figure 6-8. Panel showing pictures of possible microplastics for sediment sample GYDA-21. A: overview of sample; B: representative picture of sample showing many granules (< 500 µm).

The sample from VAL-05 had in agreement with samples above a relatively high mMP concentration. Observed by microscope, this sample also constituted of too many particles to count, mostly granules < 500 µm, but with a more heterogeneous colour composition. Compared to samples from northern North Sea and Barents Sea (Appendix B), however, the samples from the central North Sea had a more homogenous particle distribution, with a substantial amount of white and clear granules from 100 to 500 µm. All the samples constituted of a varying degree of black particles, which could be coal or charcoal.

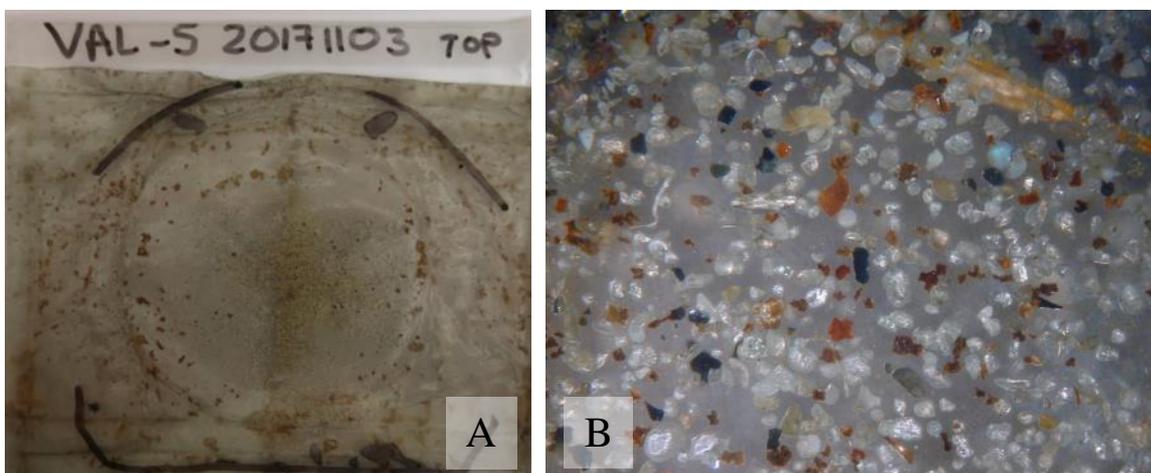


Figure 6-9. Panel showing pictures of possible microplastics for sediment sample VAL-05. A: overview of sample; B: representative picture of sample showing many granules with various colours (< 500 µm).

Corresponding to the weight concentrations (section 6.2.3), the samples from the central North Sea generally had higher concentration of potential microplastics due to the number of low-density, chemically resistant materials counted, as illustrated in Figure 6-10. The highest concentrations were found in the sediment sample from station ULA-06, with a maximum MP concentration of 31 000 mMP items/kg dry sediment, followed by GYDA-21 with 17 000 mMP items/kg dry sediment. The highest MP



concentration found in the samples from northern North Sea was 8 800 mMP items/kg dry sediment (VI-30) whereas the highest mMP concentration in the samples from Barents Sea was 3 900 mMP items/kg dry sediment (GRS-2).

Also, the average maximum MP number per kg sediment and per m² sediment area was higher in the samples from central North Sea compared to northern North Sea and the Barents Sea (6 800 ± 7 600, 2 500 ± 2 900 and 2 400 ± 1 300 mMP items/kg dry sediment, respectively). The average maximum number of potential microplastics per kg dry sediment for the different locations are shown in Figure 6-10. As evident from this figure, the average concentration was approximately three times higher in the sediment samples from central North Sea compared to northern North Sea and the Barents Sea. However, due to the wide range in sample concentrations, the concentrations cannot be considered statistically significant between the three major sampling areas ($p > 0.05$), as shown in Figure 6-11.

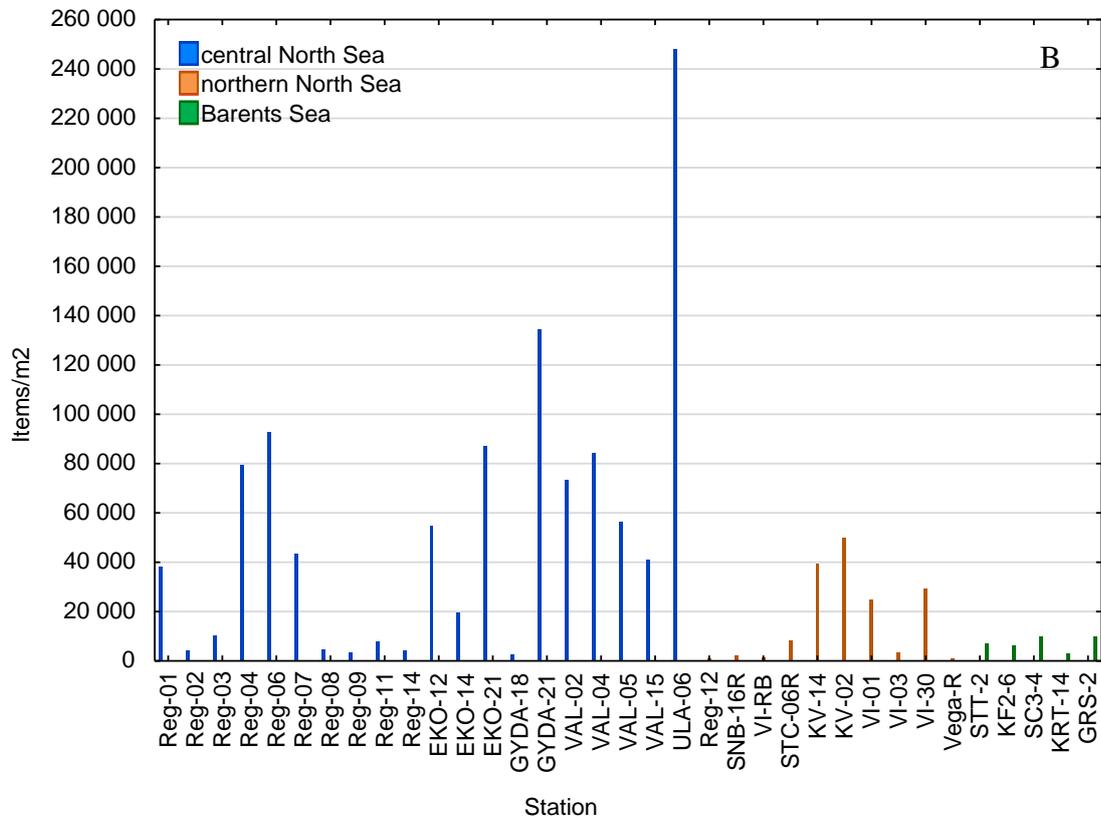
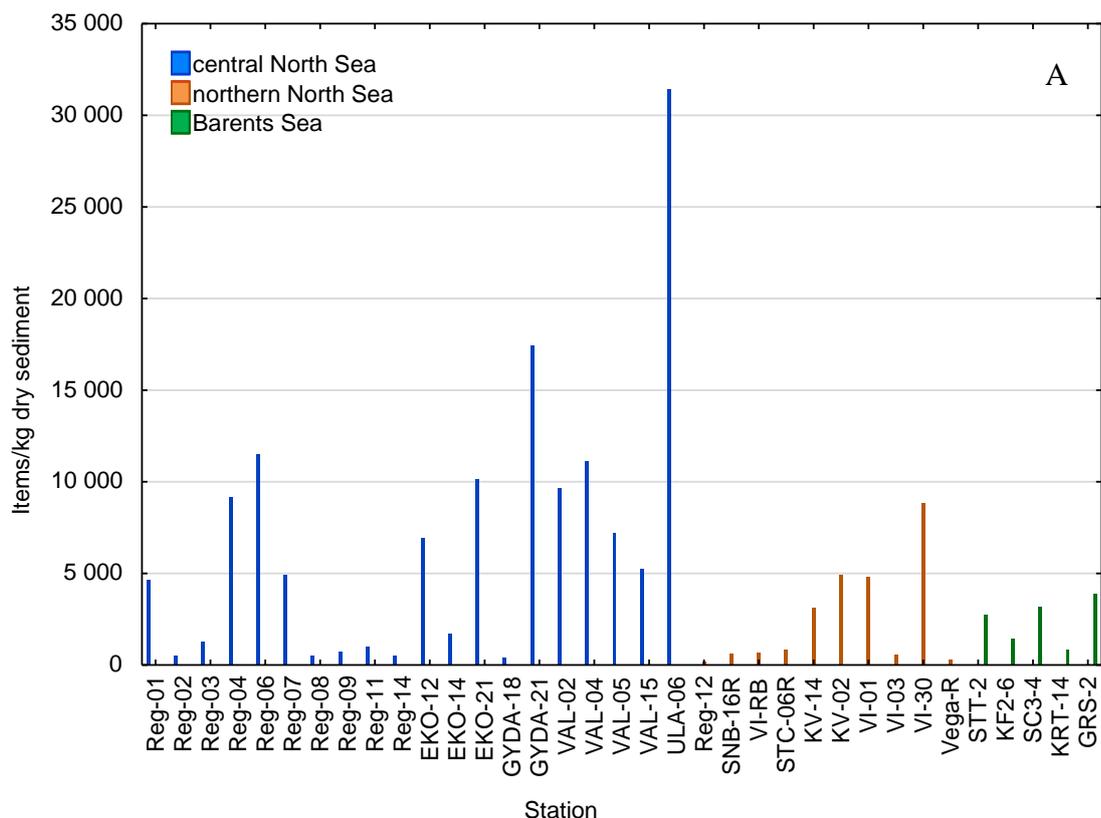


Figure 6-10. Blank corrected maximum microplastic (mMP) concentration (A: mMP items/kg sediment dry weight; B: mMP items/m²) are shown.

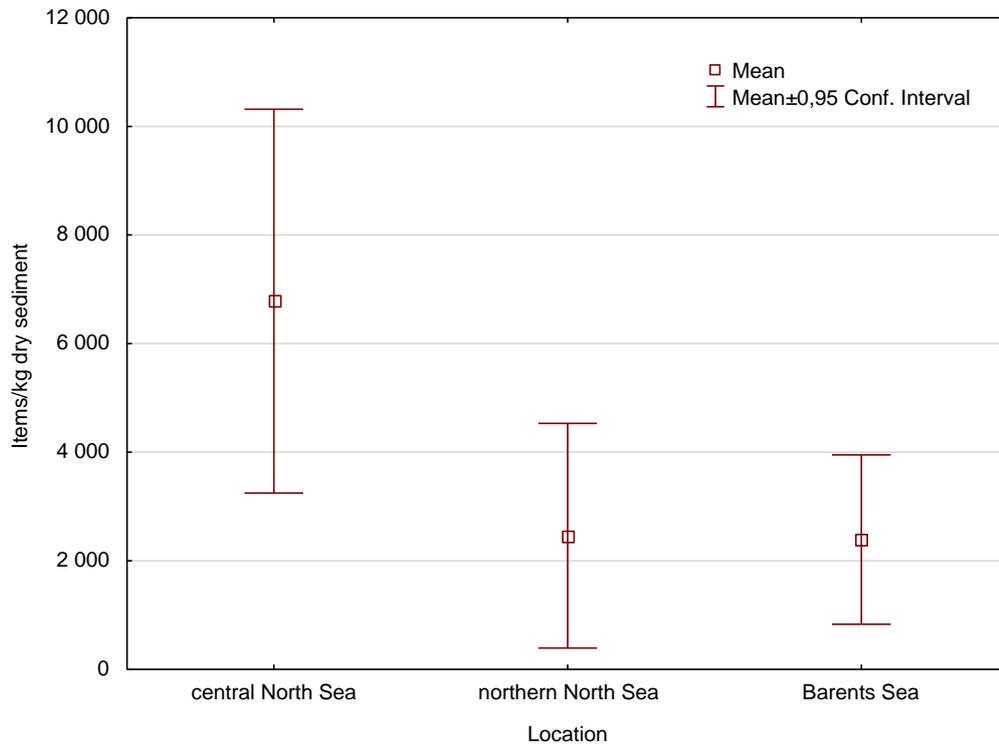


Figure 6-11. Mean, maximum microplastic concentration (mMP items/kg sediment) in sediment samples from central North Sea, northern North Sea and Barents Sea. The results are shown as mean ± 0.95 confidence interval.

7 DISCUSSION

7.1 Uncertainties and evaluation of method

7.1.1 Density limitation

Typical densities for sand or other sediments, including carbonate sea shells, are approximately 2.6 g/cm³, whereas density values for virgin plastic resins range from 0.8 to 1.4 g/cm³ (Hidalgo-Ruz et al., 2012). Density separation with ZnCl₂:CaCl₂-solution ($\rho \sim 1.53$ g/cm³) will thus separate the lighter plastic particles from the heavier sediment grains. However, there are some types of plastic with densities higher than this, such as pure Teflon and mixtures of polymers and glass, polymers and minerals or polymers and metals. By that means, plastics with a density higher than 1.53 g/cm³ are not extracted from the sediment, which might have led to an underestimation of the total microplastics concentration present in the sample. However, the density of the ZnCl₂:CaCl₂-solution ($\rho \sim 1.53$ g/cm³) is higher than the density of saturated sodium chloride solution, which is used in many other sediment surveys (ten of 13 sediment studies applied a concentrated saline solution with a density of 1.2 g/cm³, and other solutions applied were a sodium polytungstate solution with a density of 1.4 g/cm³) (Hidalgo-Ruz et al., 2012).

7.1.2 Particle size limitation

Due to the filter size of 45 μ m, the results in this report may have been underestimated, as Bergmann et al. (2017) reported that a significant amount of the counted MP particles were smaller than 25 μ m in their study. Thus, some of the smaller MP (including nanoplastics) would have gone unnoticed in this study.

7.1.3 Digestion limitation

The digestion method used here was optimised to be as destructive as possible to organic matter (including cellulose, chitin, proteins, lipids, etc.), while leaving synthetic polymers intact. In the development of this method, it was systematically tested for this and found to digest recalcitrant organic matter like cellulose at $98 \pm 4\%$, while not causing any weight change to granules and only a minimum weight change to plastic fibres (0-4%), as described in Olsen et al. (in prep).

Most of the material separated in the BMSS was $\ll 1$ mm. Thus, it is likely that the digestion removed most of the organic matter, as it is more difficult and time consuming to completely digest larger organic matter particles than smaller ones. However, it cannot be ruled out that some organic matter did survive. Further, other low density carbonaceous materials like coal, charcoal, bitumen, etc., or possibly non-carbonaceous low-density materials used by the oil and gas processing industry (e.g. porous glass, ceramics) were present, which would have been unaffected by digestion. Another concern for some of the samples was that rust was observed on the steel filter after digestion, which may have occurred from residual ZnCl₂ reacting with water in the atmosphere. Digestion of plastic materials themselves is to some extent accounted for by the correction using spiked blanks. Thus, overall these digestion limitations represent a bias that would increase the reported concentrations of mMP.

7.1.4 Characterisation by microscopy

Visual identification of microplastics is difficult due to the similarity to other materials such as organic debris (animal parts, died algae etc.) and other items (for instance metal paint, coatings, tar, charcoal etc.). Also, the identification may be influenced by subjectivity. To reduce this error source, comparison



of counts from different operators and blank samples were performed. The identification was based on guidelines given in MERI's guide to microplastic identification (MERI, 2015) as closely as possible, as mentioned in the method section. The potential microplastics requires chemical classification before they can be accepted, as earlier studies have shown that a substantial amount of visually identified potential microplastics did not confirm to be plastics by FT-IR spectroscopy (Hidalgo-Ruz et al., 2012). As the samples that had few enough particles to count were used to extrapolate to the samples with too many particles to count, any error in the counting would have gotten extrapolated further. Thus, errors from this procedure could have caused both positive and negative biases on the sample concentration.

7.2 Correlation and trends between microplastic concentration and other parameters

To check for trends in the dataset, principal component analysis (PCA) was performed. Parameters used for the PCA was the obtained microplastics concentrations for all the sediment samples (objects), as well as the following variables: dry weight percent and total organic carbon content in the sediment samples, placement properties for the sampling stations, water depth as well as the density of ZnCl₂:CaCl₂-solution used for each sample (see Appendix A).

The results from principal component analysis (PCA) showed that the obtained, maximum concentrations of microplastics were weakly, negatively correlated with the variables direction and distance ("retning" and "avstand", respectively, in Figure 7-1), which describes the location of the sampling stations with respect to an Oil & Gas installation. However, the correlations were not significant ($r \leq |0.4|$ and $p \geq 0.09$).

Further there was no consistent effect of total organic carbon content (TOC), dry weight percent, density of ZnCl₂:CaCl₂-solution or location (central North Sea, northern North Sea and Barents Sea) on the MP concentrations.

To partly account for the lack of correlation with TOC, the TOC concentrations for the samples we have data for (22 of 35 samples) range from 0.19 to 2.1 %, and the MP concentrations range from 0 – 0.004 %. This implies that MP can at best be only a minor part of the TOC, which can also contain algae, dense petroleum residues etc.

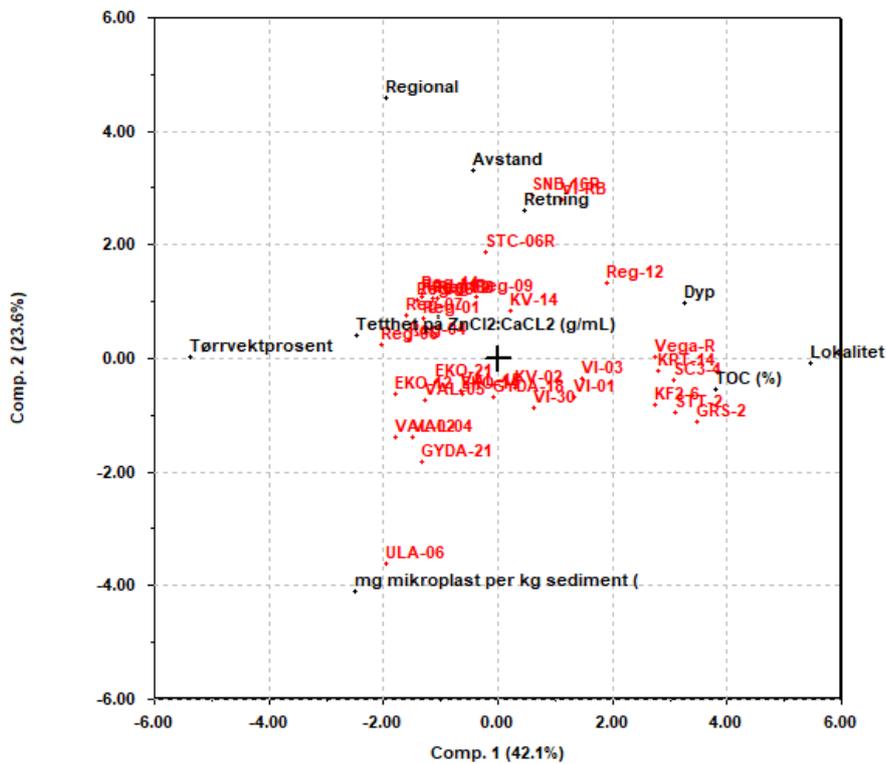


Figure 7-1. PCA biplot of microplastic concentrations (mg MP/kg dry sediment) in 35 sediment samples based on the variance in 9 variables. 66 % of the variance is explained by PC1 and PC2.

Approximately 66 % of the variance in the dataset was explained by two principal components (Comp. 1 and Comp 2. In Figure 7-1). The PCA biplot must therefore be used exploratively, subjected to the associated uncertainty. Further, the closer to the center of the plot a variable is, the less important it is for the first two components.

Note that a clear gradient in MP concentration from a point source to farther away is not necessarily expected, as this would depend on a) other sources of MP including long range transport, and b) under water currents that can transport MP, which at this time are poorly understood in the scientific literature. Regarding the latter point, it is expected based on Stoke's law that the smaller the MP, the slower it's sinking rate. Long fibres and very small microplastics (near 1 µm or smaller) behave as colloids and will not settle but remain suspended. This trend can be seen in the following graph.

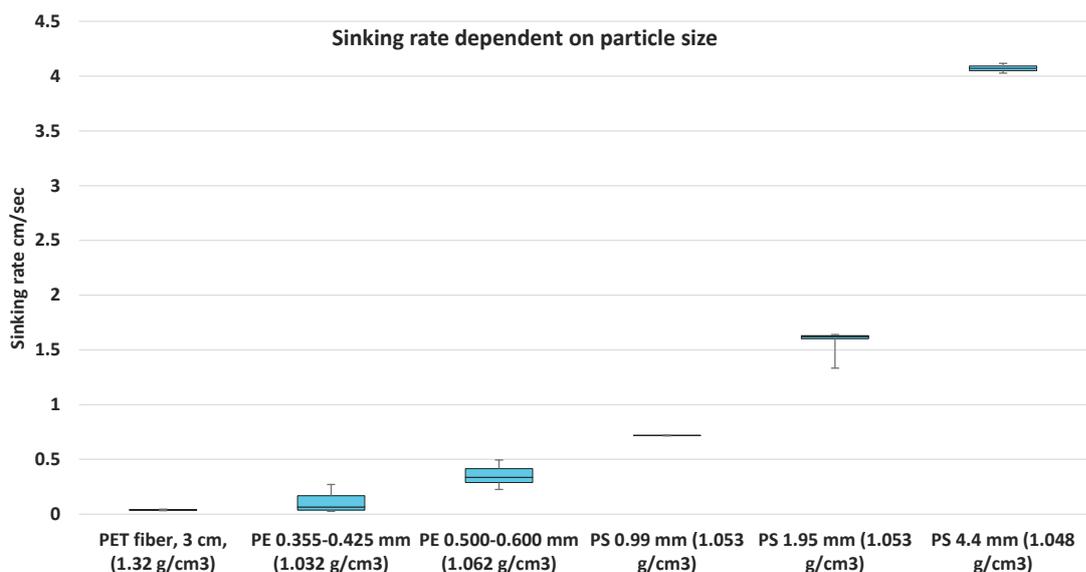


Figure 7-2. The sinking rate decreases with decreasing particle size, regardless of density, until the particles become colloidal (Source: Arp et al. WEATHER-MIC project).

Firstly, if the microplastic is capable of sinking because it is dense enough or has accumulated sufficient algae, it can be extremely slow, with an approximate maximum sinking rate of 1 cm/s for plastics under 1 mm (see Figure 7-2). If emitted as microplastic and during this slow sinking over several hundreds of meters, 3D currents and turbulences can cause the microplastics to spread far from the point of emissions. Further, microplastics on the sea bed could potentially be re-suspended through bioturbation and deep sea currents. Research on this is in its infancy. Therefore, it is unlikely at the present time to account for the fate path of the sampled microplastics.

Even though the highest mMP concentrations were found in the sediment samples from the central North Sea, compared to northern North Sea and the Barents Sea, there was no other clear trend in the dataset, and no significant correlations were found between mMP concentrations and other parameters investigated. It is possible that some trends were overlooked due to some missing variable values in the dataset (e.g. TOC values only given for 22 of 35 samples, as well as the placement properties direction, distance and depth was lacking/not relevant for some of the samples – see Appendix A for further details).

7.3 Literature comparison

While differences in sampling and analytical methodologies make comparisons with previous studies difficult, magnitude-scale comparisons may be reasonable. In Table 7-7, there is a list of reported MP abundances in sediments worldwide.

Table 7-7. Abundance of microplastics in sediments worldwide. The concentrations are expressed as mg or items per kg dry sediment, as well as mg or items per m² sediment. The maximum microplastic concentrations obtained in this report are listed in the last row for comparison.

Location	Location specification	Particle size	Measured concentration	Reference
Brazil	Beach	2 – 5 mm	60 items/kg	Ivar do Sul et al., 2009
Chile	Beach	1 - 4.75 mm	<1-805 items/m ²	Hidalgo-Ruz & Thiel, 2013
India	Ship-breaking yard	1.6µm – 5 mm	81.4 mg/kg	Reddy et al., 2006
India	Beach	1 – 5 mm	10 – 180 items/m ²	Jayasiri et al., 2013
Singapore	Mangrove	1.6 µm – 5 mm	36.8 items/kg	Nor & Obbars, 2014
NW Pacific	Deep sea trench	300 µm – 5 mm	60 – 2 020 items/m ²	Fisher et al., 2015
South Korea	Beach	50 µm – 5 mm	56 – 285 673 items/m ²	Kim et al., 2015
Belgium	Continental Shelf	38 µm – 1 mm	97.2 items/kg	Claessens et al., 2011
Italy	Subtidal	0.7 µm – 1 mm	672 – 2 175 items/kg	Vanello et al., 2013
Worldwide	Deep sea	5 µm – 1 mm	50 items/m ²	Van Cauwenberghe et al., 2013
Slovenia	Beach	0.25 – 5 mm	177.8 items/kg	Laglbauer et al., 2014
Arctic	Deep sea	10 µm – 5mm	42 - 6 595 items/kg dry	Bergmann et al., 2017
Norway	Oslo beach	45 µm – 5 mm plus fibres	500 – 9800 mg/kg	Mahat (2017)
Norway	Oslo sediment	45 µm – 5 mm plus fibres	20 – 90 mg/kg	Mahat (2017)
Norway	Reference areas in the Norwegian coastal shelf	unknown	1 – 400 items/kg	Mareano, 2018
Norway (this study)	Norwegian Continental Shelf	45 µm – 5 mm plus fibres	< LOD – ≤ 410 (60) max mg /kg < LOD – ≤ 3 200 (480) max mg /m ² ≤ 180 – ≤ 31 000 (4 900) max items/kg ≤ 700 – ≤ 250 000 (37 000) max items/m ²	<i>This report</i>

As seen in Table 7-7, reported MP concentrations in sediments vary widely. The maximum concentrations reported in this study are in general within the range or higher than values reported in the literature. For instance, the concentration of MP in the intertidal sediments of the world's largest ship-breaking yard at Alang-Sosiya in India reported a maximum MP concentration of 89 mg small plastic fragments per kg of sediment (Reddy et al., 2006). This concentration is in the range of what was found in this study (60 ± 80 mg mMP/kg sediment), which may indicate relatively high MP concentrations along the Norwegian Continental Shelf, as the plastics contribute to 40 – 50 % of the ship-breaking waste that enters the marine environment, according to Reddy et al. (2006).

A study of microplastics in Arctic deep-sea sediments from the HAUSGARTEN Observatory (2 340 – 5 570 m depth) recorded concentrations of microplastics from 42 to 6 595 MP items/kg sediment dry weight, with an overall mean number of 4 356 (± 675 standard error) items/kg (Bergmann et al., 2017). With disregard of one station with relatively lower MP concentration (41.76 items/kg dry sediment), the concentration values from the Arctic deep-sea sediments are somewhat lower, but in the same range as the results in this report. However, it should be kept in mind that HAUSGARTEN study was able to quantify microplastics less than 10 µm, and found the majority of particles to be less than 25 µm (which is below the 45 µm cut off of this study). The study by Mareano (2018), which is in preparation though with some information available, reported from a pilot study of remote regions in the Norwegian

Continental area, including the Barents Sea, though with one coastal location near Ålesund, reported with 200 – 400 plastic items in three areas (near Ålesund and outwards), and 1 – 200 items/kg in other areas. This is much smaller than the number reported here; this may be due to both differences in the methodology used and sampling in more remote locations. Mahat (2017) measured microplastics on Bygdoy sjøbad Beach sand before preparation for the swimming season and found 500 – 9800 mg/kg, which is substantially more than in this study; but only 20 – 90 mg/kg in sediments near the outflow of Bekkelaget water treatment plant, which is comparable to the range reported in this study.

As mentioned above, some of the observed differences in quantified microplastic concentrations may be due to the use of different methodologies, as there is a wide variety of approaches used to identify and quantify microplastics. For meaningful comparisons, it is important to define specific methodological conditions, such as the density on the density-solution used and the size range of microplastics quantified.

The relatively high mMP concentrations found in the sediment sample from central North Sea, northern North Sea and the Barents Sea may confirm the widespread occurrence of microplastics in the marine environment. According to a review paper of microplastics in the marine environment (Hidalgo-Ruz et al., 2012), values for abundances ranged from 0.21 to more than 77 000 items per m² in sediment, which is several orders of magnitude higher than in the sea surface. This shows that a substantial amount of microplastics can be found in sediments, as also is indicated by this study. Sediments are proposed as the final destination of microplastics and other pollutants in the environment. This is due to natural processes such as biofouling, the buoyancy and density changes, which eventually can lead to deposition of microplastics in sediments.

7.4 Extrapolation for the North Sea

It is worthwhile to extrapolate the findings in this study to estimate the mass of microplastics in larger areas, however it is important to bear in mind the large uncertainties in such an extrapolation. In Table 7-8 some examples of extrapolation of mass of microplastics in the North Sea and globally are shown, based on average and median values in this study. The difference between the average and median values is in general relatively large, which indicates a skewed distribution of the data, with the majority of measurements below the average value.

Table 7-8. Extrapolation of the findings in this study to mass of microplastics in the North Sea and globally.

	Median mg/m²	Average mg/m²	North Sea (tonnes)¹	Global (tonnes*10⁶)²
All data	130	480	18 000 – 68 000	46 – 171
central North Sea	550	700	78 000 – 100 000	200 - 254
central North Sea – regional stations only	115	360	16 000 – 52 000	41-130

1: 142 000 km² (<http://www.norskipetroleum.no/utbygging-og-drift/aktivitet-per-havomrade/>)

2: Area used: 360*10⁶ km²

There are very few similar estimates of this kind to be found in the literature. A report by Eunomia (2016) did a very rough estimation that the estimated worldwide average weight of plastic (both micro and macroplastic) is 70-180 kg/km² (0.07 – 0.18 tonnes/km²) on the ocean floor. Extrapolating these values to the North Sea yields 10 000 – 25 000 tonnes. This is lower but within the range of our estimate, considering that we are reporting maximum microplastics concentrations, and that the results



might have been influenced by sampling near possible hotspots (the North Atlantic Gyre area and near platforms).

7.5 Speculation on the origin of the microplastics in some samples

The appearance of relatively homogenous, clear granules with a particle size of roughly 100 – 500 µm in the samples with the highest concentrations, such as ULA-06 (Figure 6-7) and GYDA-21 (Figure 6-8) is indicative of a primary microplastic source (i.e. a microplastic product) rather than a secondary microplastic source (i.e. microplastic formed from the breakdown of larger plastics). Whether these granules can be linked to products used by the Oil & Gas industry such as for example drilling polymers or other particles used for lubrication (e.g. hollow glass particles), or possibly sandblasting plastic grit, is to the authors knowledge unknown. It is known that plastic particles can be included in oil based drilling fluids, but these are in little use on the NCS and not allowed to be released.

According to a report written by Mepex (2014) very few commercial-use products with primary microplastics are documented, and only brief mentions of some unspecified use in petroleum industry are reported. In oil, gas or other kinds of rock drilling, drilling fluids based on plastic microbeads have been used for a few decades (Skall et al., 1999), as well as Teflon strengthened particles have been patented and marketed heavily for drilling purposes internationally for the last ten years. If these are used along the Norwegian Coast, the releases directly to the ocean may be in tonnes (Mepex, 2014). A recent report by the European Commission (2017) stated that, "In conclusion, there is some evidence that suggests the use of microplastics in offshore oil and gas could be substantial, in the magnitude of hundreds of tonnes. However, sufficient data was not available to make a precise quantitative estimate. It is therefore recommended that this potential source be kept under review."

Further information on the origin of the microplastics can be obtained by the use of a chemical analysis (for example by use of FT IR microscopy).

8 CONCLUSION AND FURTHER RECOMMENDATIONS

This report provides a basis for assessing microplastics in sediments from the Norwegian Continental Shelf. The main conclusions are:

- ✓ A maximum average of 60 ± 80 mg microplastics/kg dry sediment (corresponding to a maximum of $37\,000 \pm 50\,000$ items/m² sediment surface) of potential microplastics were found in the sediment samples from the Norwegian Continental Shelf.
- ✓ The central North Sea had more MP than in the northern North Sea or Barents Sea areas, on maximum average 90 ± 100 , 30 ± 40 and 30 ± 20 mg microplastics/kg dry sediment. Further, the samples with the top 6 highest concentrations were found in central North Sea. The reason for this is uncertain but may be explained by large scale currents/gyres which accumulates debris in this part of the North Sea and influence from continental Europe through river run off among others. In addition, this area has relatively high shipping traffic and fishing activity which may contribute to plastic emissions. This area has also the longest history of Oil & Gas activity but the influence from this is unknown considering large geographical areas and the few samples analysed among others.
- ✓ It is emphasized that there are relatively large uncertainties related to the results. A duplicate analysis of one sample resulted in a variation of 55 %. At this time, the method quantifies the maximum weight and number of items that *could be* microplastic based on their density (lower than 1.53 g/mL), having a size range between 45 µm and 5mm, and resilience to an organic matter digestion process. Other materials fitting this profile such as soot, char, porous glass and porous ceramics would also be included in this quantification. Therefore, all data is presented as the potential "maximum" microplastic concentration. On the other side, plastic particles with higher density than 1.53 g/ml or with a size less than 45 µm will not be included in the numbers.
- ✓ Based on the analytical methodology used and the inherent uncertainties the highest maximum potential microplastics concentrations are in general found at locations close to Oil & Gas installations.
- ✓ The results of this study have revealed relatively high maximum concentrations of potential microplastics, which may confirm the widespread occurrence of microplastics in the marine environment. The results above are subject to various biases, and should be interpreted with these in mind.
- ✓ No clear trends could be seen with the following parameters: sampling location and depth, density of ZnCl₂:CaCl₂-solution used, total organic carbon content and dry weight percent of the sediment samples.
- ✓ Before clearer conclusions can be made regarding sources and distribution of microplastics in sediments on the NCS more detailed investigations, such as systematic sediment sampling and use of for instance a FT-IR microscope, will provide valuable information.
- ✓ The transport path of MP from the surface of the ocean to the seabed is very complex. Therefore, it cannot be expected to see a neat gradient of microplastics from areas where they are emitted and farther away. Further, because microplastics can be quite buoyant, they may be able to travel vast distances from their source before settling in sediment.

For a better understanding of the presence of microplastics in sediment, it is important to further investigate deposition of plastics to sediments and to carry out more rigorous testing for processing and extraction of microplastics from different sediment matrices. It is also important to further investigate the potential influence of environmental variables on microplastics concentration and composition in



sediments. The few studies available in the literature on microplastics in sediment indicate there are orders of magnitude more microplastics on the seabed than on the ocean surface (Hidalgo-Ruz et al. 2012). The environmental impact of this on benthic ecosystems are unknown but need further investigation (Galloway et al. 2017). This is particularly the case because the anticipated concentrations of microplastics are expected to increase in the foreseeable future, potentially reaching levels where they become a planetary boundary threat (Jahnke et al. 2017).

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APPENDIX A

Raw data



A1 Raw data for the Sediment Samples

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Table A 1 Raw data for the sediment samples from the Norwegian Continental Shelf (location areas "central North Sea", "northern North Sea" and "Barents Sea"). DW% = dry weight percent, TOC = total organic carbon.

Sample-ID	Location	Comment/Station	Date	DW% (%)	ZnCl ₂ :CaCl ₂ density (g/cm ³)	Measured weight of sample after digestion (g)	TOC (%)	Direction (°)	Distance (m)	Depth (m)
20171006-Reg-01	central North Sea	Regional	06.10.2017	78	1.52	0.0312	0.3	-	-	73
20171211-Reg-02	central North Sea	Regional	06.10.2017	78	1.52	0.0031	0.31	-	-	68
20171004-Reg-03	central North Sea	Regional	04.10.2017	78	1.54	0.0098	0.37	-	-	68
20171005-Reg04	central North Sea	Regional	05.10.2017	78	1.53	0.0639	0.32	-	-	71
20170926-Reg-06	central North Sea	Regional	26.09.2017	76	1.55	0.0538	0.33	-	-	72
20170929-Reg-07	central North Sea	Regional	29.09.2017	77	1.55	0.0368	0.36	-	-	73
20171206-Reg-08	central North Sea	Regional	04.10.2017	76	1.52	0.0034	0.32	-	-	70
20171005-Reg-09	central North Sea	Regional	05.10.2017	52	1.53	0.0025	0.19	-	-	66
20171002-Reg-11	central North Sea	Regional	02.10.2017	76	1.54	0.0066	0.29	-	-	71
20171002-Reg-14	central North Sea	Regional	02.10.2017	80	1.53	< LOD	0.24	-	-	80
20171026-EKO-12	central North Sea	Ekofisk	26.10.2017	77	1.57	0.0515	-	148	2500	78
20171206-Eko-14	central North Sea	Ekofisk	20.10.2017	77	1.52	0.0056	0.48	140	850	76
20171026-EKO-21	central North Sea	Ekofisk	26.10.2017	78	1.51	0.0704	-	287	4000	71
20171103-GYDA-18	central North Sea	Gyda	03.11.2017	71	1.51	0.0037	0.88	135	250	67
20171106-GYDA-21	central North Sea	Gyda	06.11.2017	79	1.53	0.1289	-	135	2000	67
20171027-VAL-02	central North Sea	Valhall	27.10.2017	76	1.59	0.0662	0.45	74	500	76
20171030-VAL-04	central North Sea	Valhall	30.10.2017	78	1.56	0.0835	-	74	2000	72



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20171102-VAL-05	central North Sea	Valhall	02.11.2017	79	1.55	0.0534	-	74	5000	70
20171102-VAL-15	central North Sea	Valhall	02.11.2017	76	1.51	0.0374	0.42	254	500	76
20171019-ULA-06	central North Sea	Ula	19.10.2017	79	1.51	0.2287	0.28	45	250	71
20171117-Reg-12	northern North Sea	Regional	17.11.2017	46	1.52	< LOD	-	-	-	400
20171113-SNB-16R	northern North Sea	Snorre B ref/regional	13.11.2017	63	1.56	< LOD	-	315	10000	342
20171117-VI-RB	northern North Sea	Visund ref/regional	17.11.2017	62	1.52	< LOD	-	330	10000	330
20171121-STC-06R	northern North Sea	Statfjord C ref/regional	21.11.2017	73	1.54	0.0052	-	130	10000	137
20171121-KV-14	northern North Sea	Kvitebjørn	21.11.2017	77	1.54	0.0180	-	316	7224	187
20171120-KV-02	northern North Sea	Kvitebjørn	20.11.2017	75	1.52	0.0218	-	140	500	185
20171213-VI-01	northern North Sea	Visund	09.11.2017	59	1.53	0.0092	-	150	500	330
20171120-VI-03	northern North Sea	Visund	20.11.2017	61	1.52	0.0057	-	150	1000	330
20171120-VI-30	northern North Sea	Visund	20.11.2017	60	1.57	0.0269	-	330	250	316
20171116-Vega-R	northern North Sea	Vega	16.11.2017	36	1.51	< LOD	-	-	-	380
20171122-STT-2	Barents Sea	Stangnestind	22.11.2017	41	1.53	0.006	1.93	90	250	251
20171122-KF2-6	Barents Sea	Korpefjell	22.11.2017	54	1.51	0.0046	1.76	85	900	242
20171122-SC3-4	Barents Sea	Scarecrow3	22.11.2017	43	1.51	0.0075	1.56	270	100	461
20171127-KRT-14	Barents Sea	Kråketind	30.11.2017	45	1.52	0.0036	1.34	-	-	440
20171124-GRS-2	Barents Sea	Gråspett	24.11.2017	37	1.51	0.0086	2.09	90	250	508
20170926-Reg-06-P1	central North Sea	Regional	26.09.2017	76	1.55	0.0413	0.33	-	-	72
20170926-Reg-06-P2	central North Sea	Regional	26.09.2017	76	1.55	0.0662	0.33	-	-	72
20171113-SNB-16R #1	northern North Sea	Snorre B ref/regional	13.11.2017	63	1.55	< LOD	-	315	10000	342
20171113-SNB-16R #2	northern North Sea	Snorre B ref/regional	13.11.2017	63	1.57	< LOD	-	315	10000	342

A2 Raw data for the Method Blanks

Table A 2 Raw data for the Method Blanks. SD = standard deviation.

Sample-ID	ZnCl ₂ :CaCl ₂ density (g/cm ³)	Measured weight of sample after digestion (g)
20170922-Blank1	1,54	0,0001
20170922-Blank2	1,57	0,0002
20170922-Blank3	1,55	0,0015
20171019-Blank	1,51	-0,0002*
20171121-Blank	1,50	0,0005
20171123-blank****	1,52	-
20171129-Blank1	1,48	0,0008
20171129-Blank2	1,47	0,0001
20171109-blank 5:1	1,51	0,0026
20171206-Test1	1,52	0,0013
20171312-Test2	1,53	0,0031
Average		0,0010**
SD		0,0011

*Due to measurement uncertainty

**Used as blank correction for sample weight after digestion

*** No weight recorded, only used for visual identification of possible impurities

A3 Raw data for the Recovery Blanks

Table A 3 Raw data for Spiked Blanks and calculated recovery rate for different microplastic spiking materials

Sample-ID	DW% (%)	ZnCl ₂ :CaCl ₂ density (g/cm ³)	Measured weight of spiked MP-material (± 0,0001 g)			Measured weight of sample after digestion (g)
			PET powder	PE fibre	PET pellet	
20171004-Reg-03-Blank	78	1,51	0,2000	-	-	0,1408
20171002-Reg-11-Blank	76	1,51	0,4298	-	-	0,1858
20171005-Reg-09-Blank	52	1,51	-	0,1318	0,1007	0,2270
20171114-SNB-16R-Blank1	63	1,55	-	0,0527	-	0,0445
20171114-SNB-16R-Blank2	63	1,57	0,1074	-	-	0,0615
20171009-Reg-01 Blank	78	1,52	-	0,1308	0,094	0,2213
20171130-KRT-14-Blank1	45	1,48	-	0,0532	-	0,0484
20171130-KRT-14-Blank2	45	1,47	0,1022	-	-	0,0873
Average weight recovery rate ± SD (%)*			63 ± 18	91 ± 6,4	100 ± 0	-

*Calculation based on (method blank) corrected, measured weight of sample after digestion

APPENDIX B

Visual identification of potential microplastic



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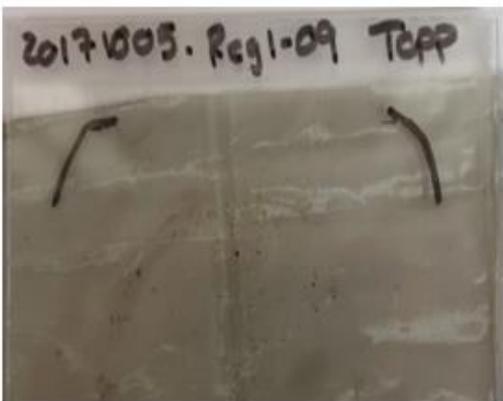
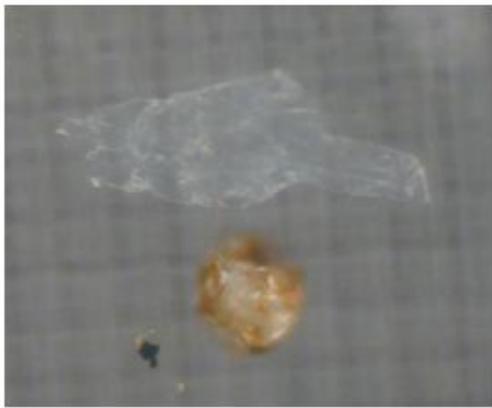
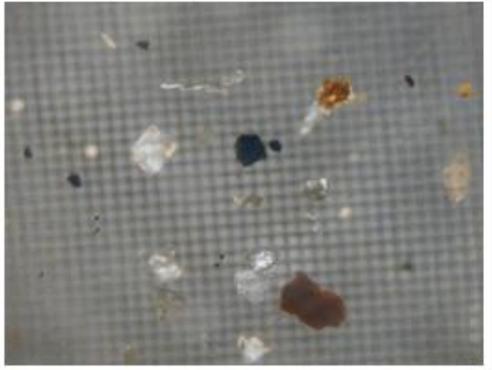
The sediment samples listed in the table below were investigated under a microscope for detection of possible microplastic concentrations. To confirm if the particles are microplastics, one should perform FT-IR-analysis.

Table B 1 List of samples used for visual identification of potential microplastics

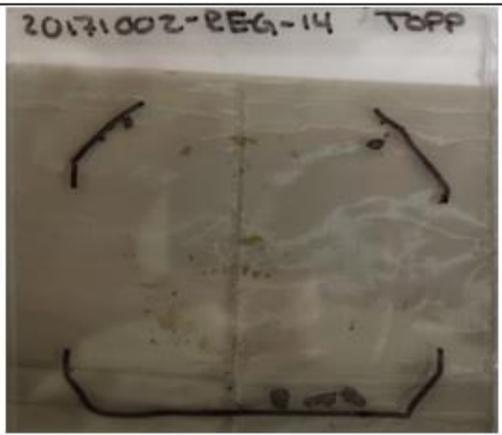
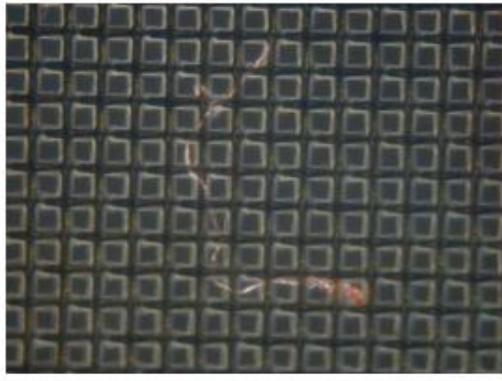
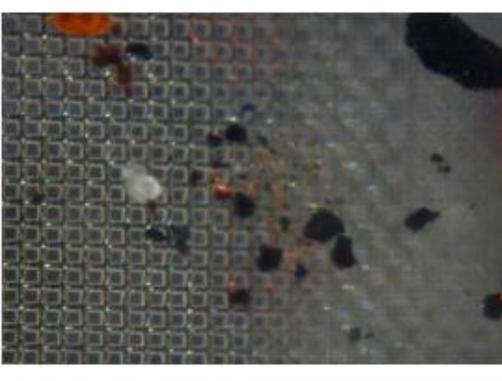
Sample-ID	Location	Station	MP concentration (mg/kg dry weight)
20171005-Reg-09	central North Sea	Regional	5
20171002-Reg-14	central North Sea	Regional	< LOD
20171026-EKO-12	central North Sea	Ekofisk	91
20171103-GYDA-18	central North Sea	Gyda	6
20171106-GYDA-21	central North Sea	Gyda	228
20171102-VAL-05	central North Sea	Valhall	94
20171019-ULA-06	central North Sea	Ula	412
20171117-Reg-12	nortern North Sea	Regional	< LOD
20171113-SNB-16R	nortern North Sea	Snorre B ref/regional	< LOD
20171121-KV-14	nortern North Sea	Kvitebjørn	40
20171120-KV-02	nortern North Sea	Kvitebjørn	64
20171120-VI-03	nortern North Sea	Visund	19
20171117-VI-RB	nortern North Sea	Visund ref/regional	< LOD
2017116-Vega-R	nortern North Sea	Vega	< LOD
20171122-STT-2	Barents Sea	Stangnestind	36
20171122-KF2-6	Barents Sea	Korpefjell	18
20171122-SC3-4	Barents Sea	Scarecrow3	41
20171127-KRT-14	Barents Sea	Kråketind	14
20171124-GRS-2	Barents Sea	Gråspett	51

Pictures taken during microscopy are given in the following.

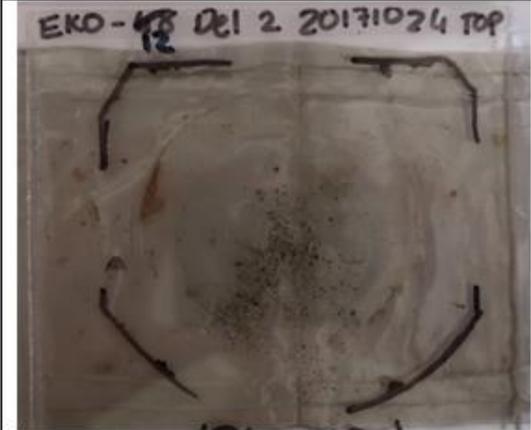
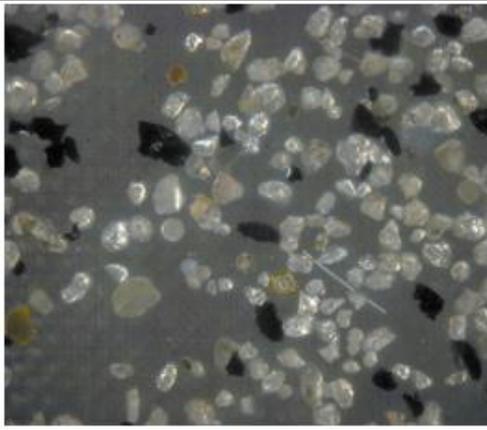
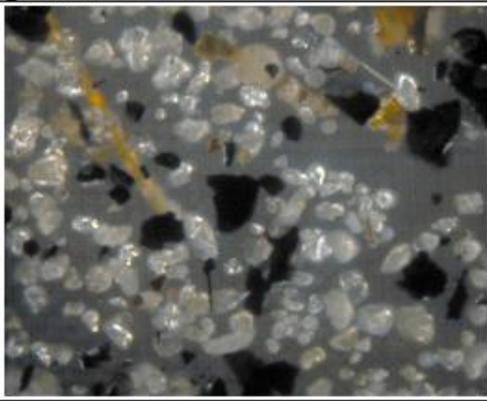
B1 20171005-Reg-09

	
<p>Overview</p>	<p>Clear layer (μm)</p>
	
<p>White fibre ($> 500 \mu\text{m}$)</p>	<p>Heterogenous distribution of particles</p>

B2 20171002-Reg-14

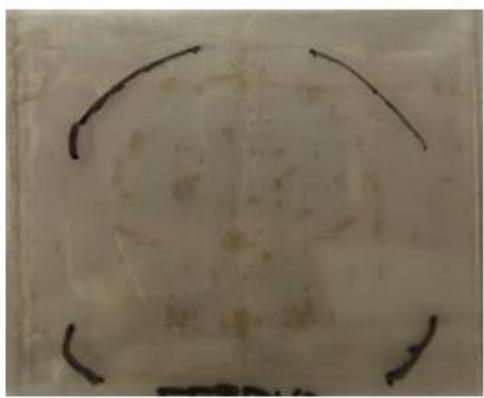
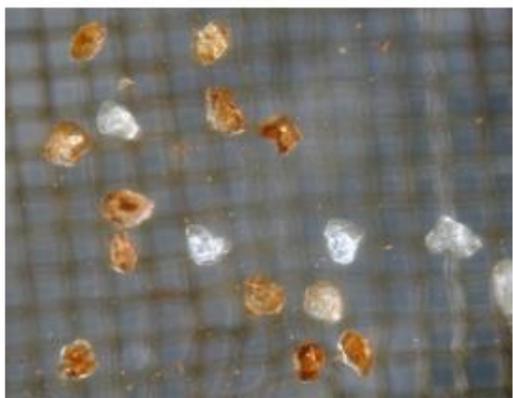
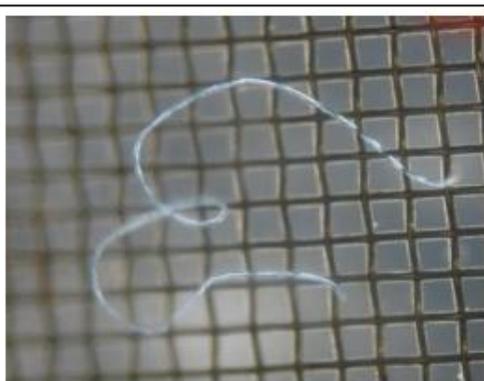
	
<p>Overview</p>	<p>Blue fibre</p>
	
<p>Red fibre</p>	<p>Mostly black particles</p>

B3 20171026-EKO-12

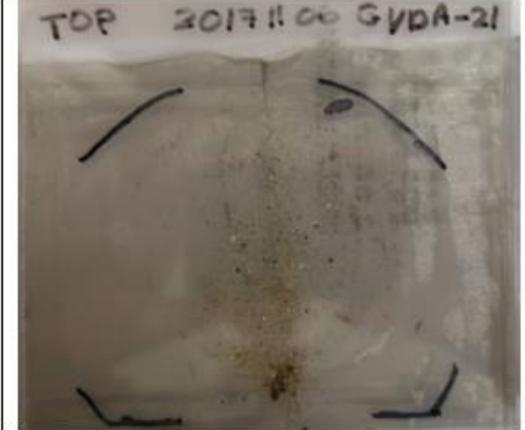
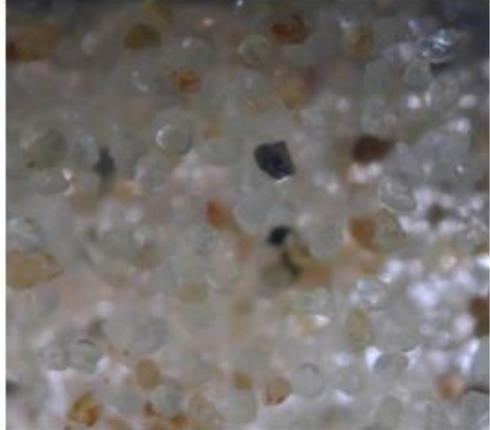
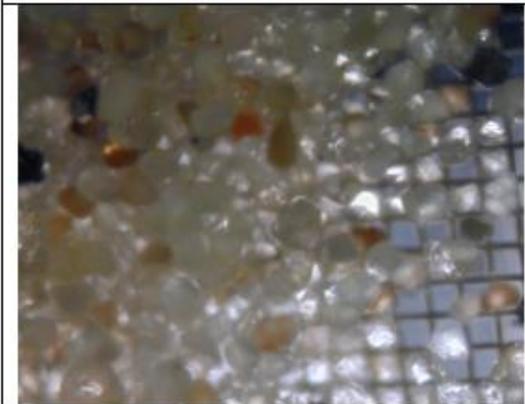
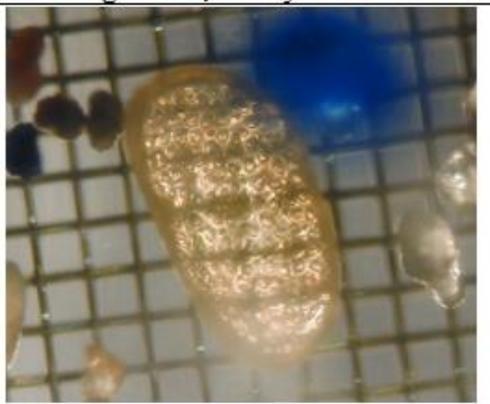
	
<p>Overview</p>	<p>Spherical and irregularly shaped granules</p>
	
<p>Red, spherical granule</p>	<p>Spherical and irregularly shaped granules</p>

Relatively homogenous composition of white/clear granules of approximately the same size (100-300 µm).

B4 20171103-GYDA-18

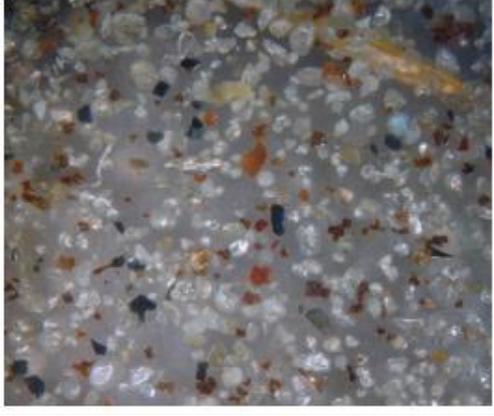
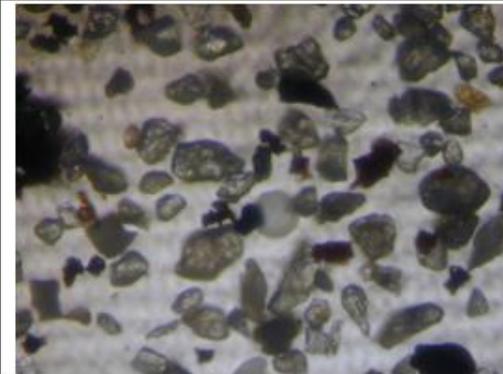
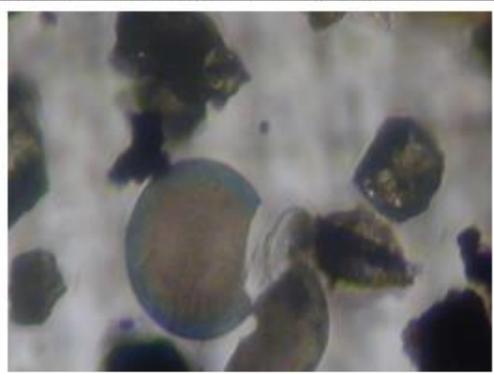
	
<p>Overview</p>	<p>Mostly clear and brown granules (100-300 μm)</p>
	
<p>Blue fibre</p>	<p>Brown granule with sharp edges</p>

B5 20171106-GYDA-21

	
<p>Overview</p>	<p>Several granules, mostly white/clear.</p>
	
<p>Several granules, mostly white/clear.</p>	<p>Probably organic matter due to cellular structure</p>

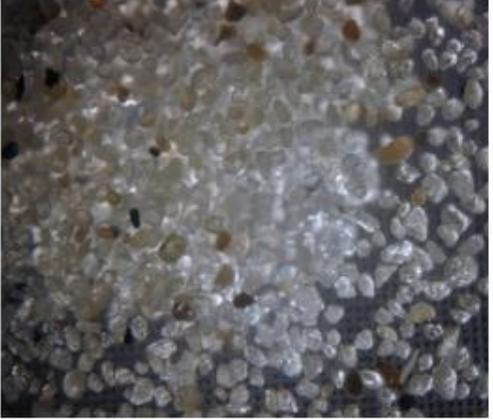
Relatively homogenous composition of white/clear granules of approximately the same size (100-300 µm).

B6 20171102-VAL-05

	
<p>Overview</p>	<p>Heterogeneous particle distribution</p>
	
<p>Several grey/black particles – charcoal?</p>	<p>Example of granule with sharp edges</p>

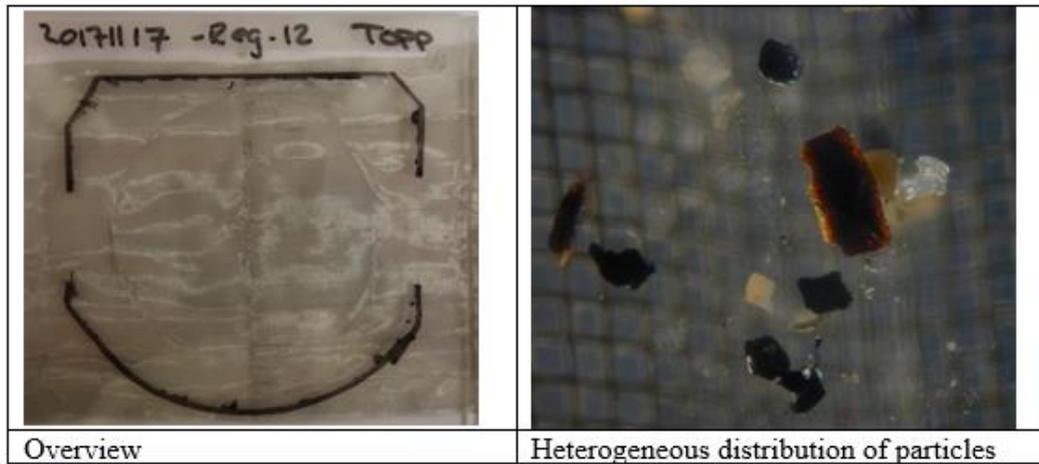
Relatively homogenous composition of white/clear granules of approximately the same size (100-300 µm).

B7 20171019-ULA-06

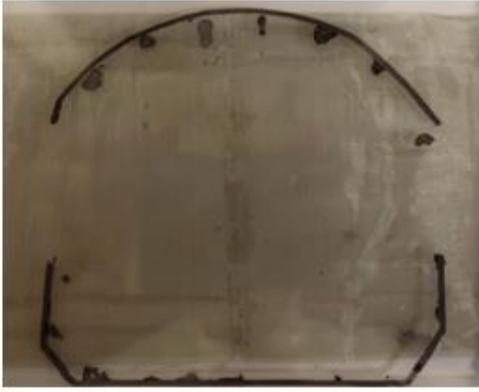
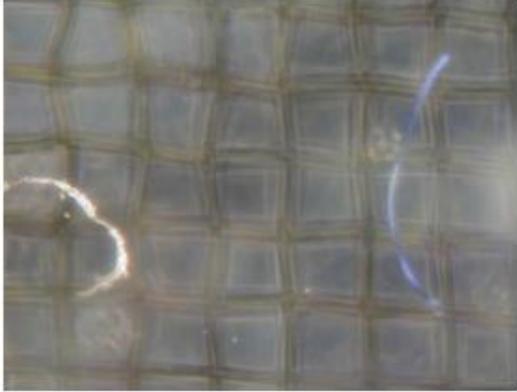
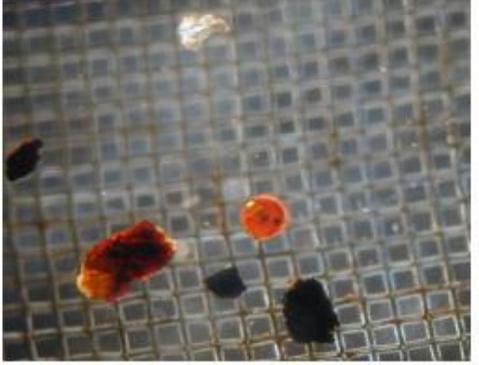
	
<p>Overview</p>	<p>Homogenous distribution of white/clear granules</p>
	
<p>Mostly white/clear granules</p>	<p>White fibre</p>

Relatively homogenous composition of white/clear granules of approximately the same size (100-300 µm).

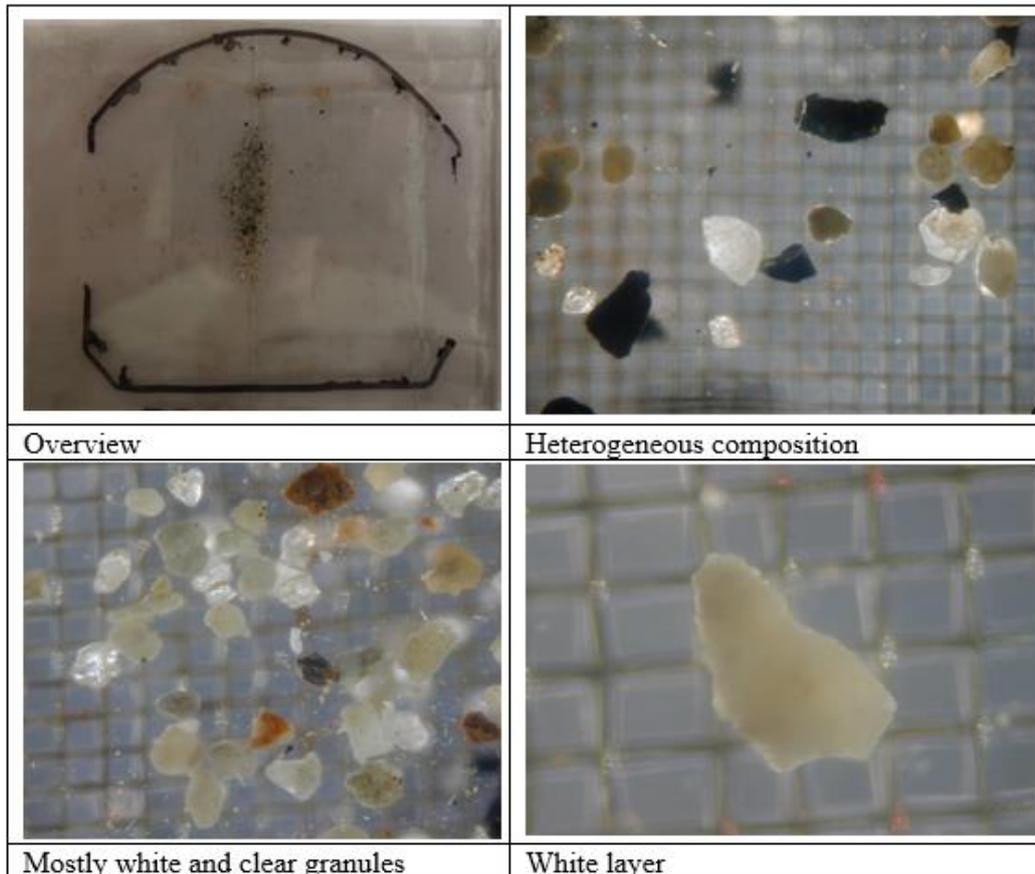
B8 20171117-Reg-12



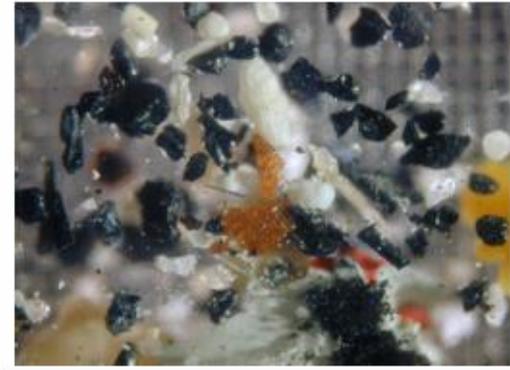
B9 20171113-SNB-16R

	
<p>Overview</p>	<p>White and blue fibre</p>
	
<p>Some black, clear and red/orange granules, one spherical.</p>	<p>White fibre</p>

B10 20171121-KV-14

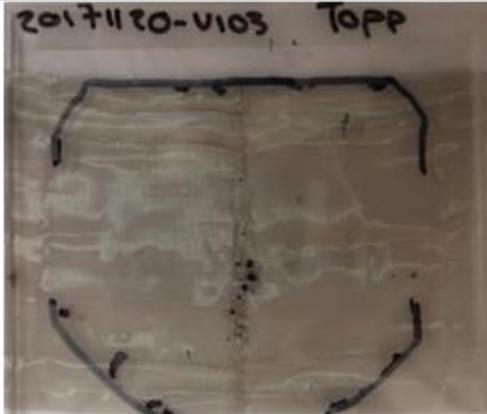
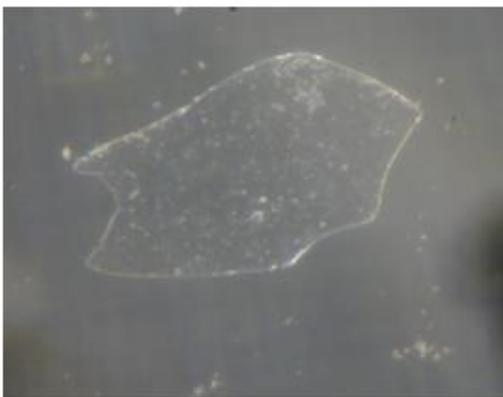
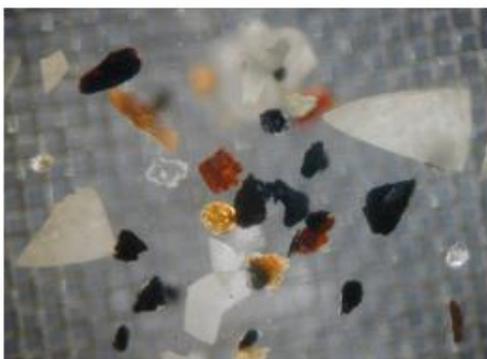


B11 20171120-KV-02

	
<p>Overview</p>	<p>Unknown structure</p>
	
<p>Several black granules and other particles</p>	<p>White layer</p>
	
<p>Cluster of white fibres</p>	<p>Yellow/white foam?</p>

This sample contained several particles not found in any other of the sediment samples, as shown in the photos above. The uppermost right pictures shows several black fibres clustered together with something that looks like glue or slime. Layers of different colours (white, blue and red) were also found, as well as white clusters of white fibres and a foam-looking particle. In agreement with other samples, a heterogeneous composition of granules was identified.

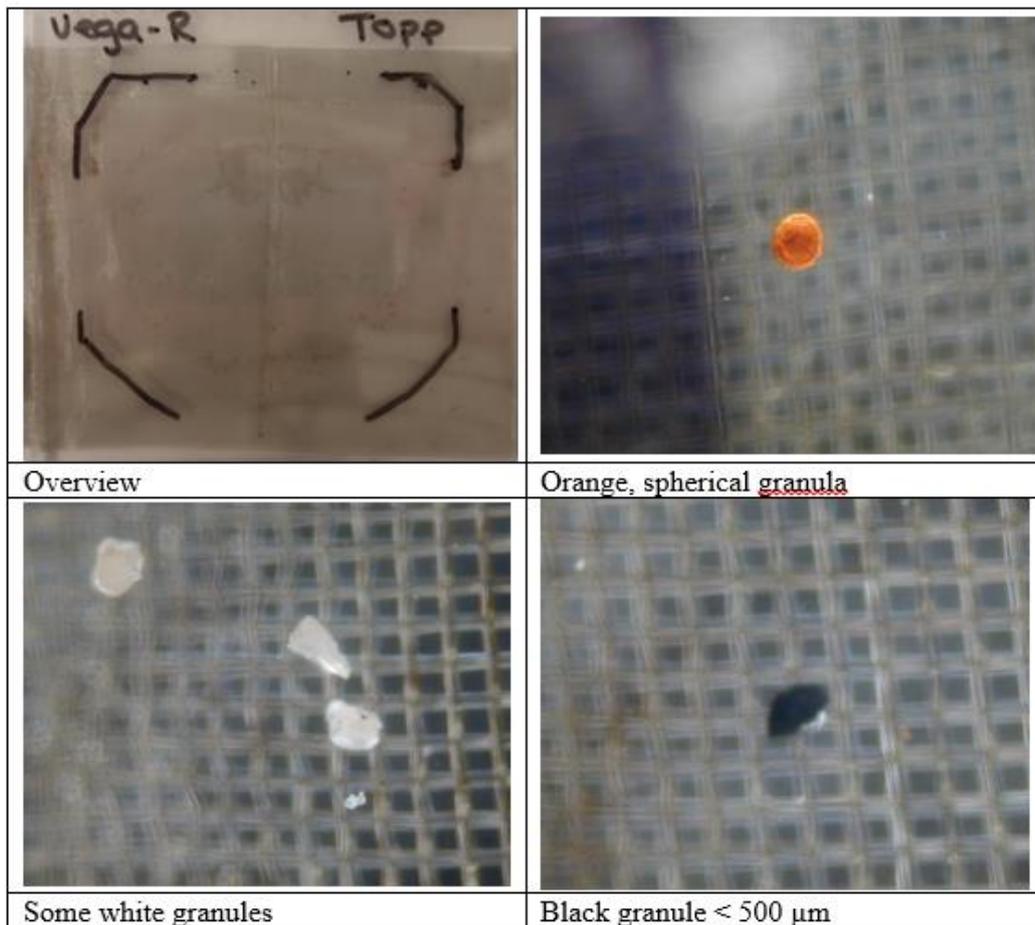
B12 20171120-VI-03

	
<p>Overview</p>	<p>Clear layer</p>
	
<p>Heterogeneous composition of particles. A substantial amount of white layers</p>	<p>White layers, black and clear granules</p>

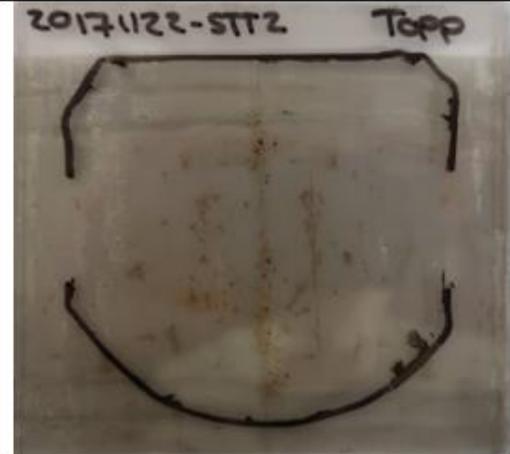
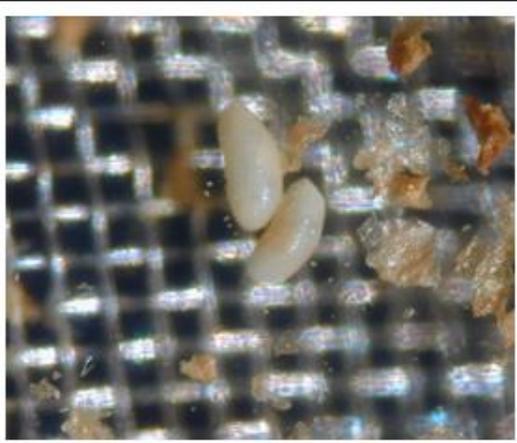
B13 20171117-VI-RB

<p>Overview</p>	<p>Heterogeneous distribution of particles – one spherical, red granule</p>
<p>Heterogeneous distribution of particles</p>	<p>Clear layer</p>

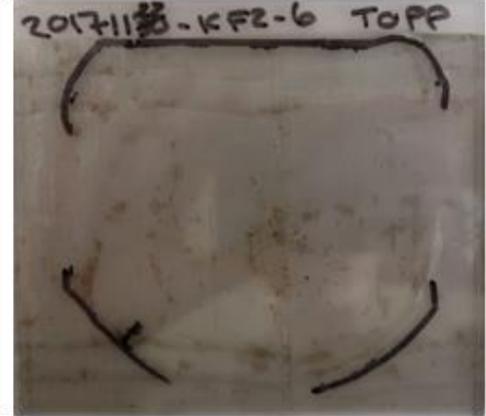
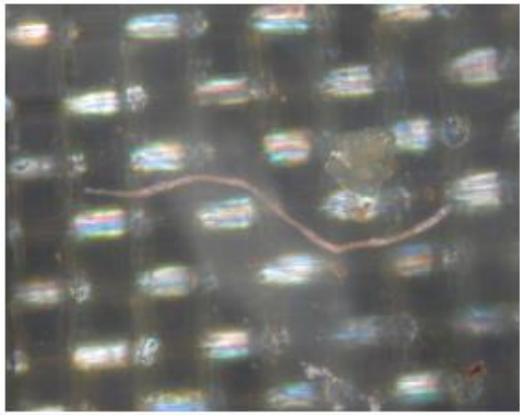
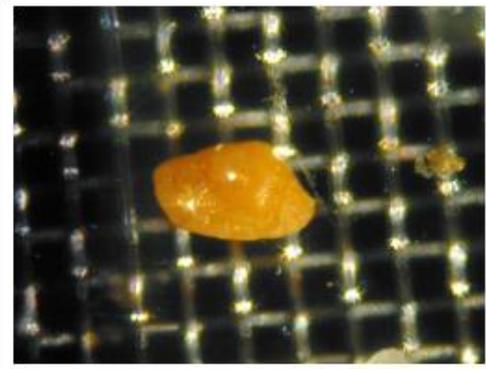
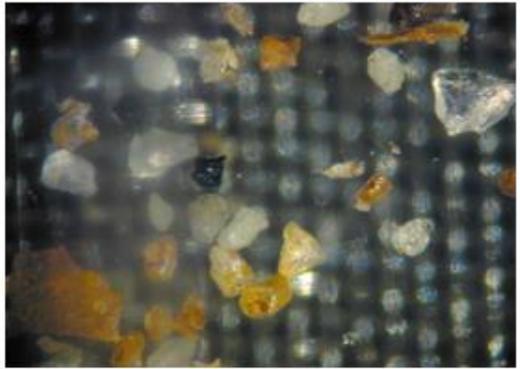
B14 2017116-Vega-R



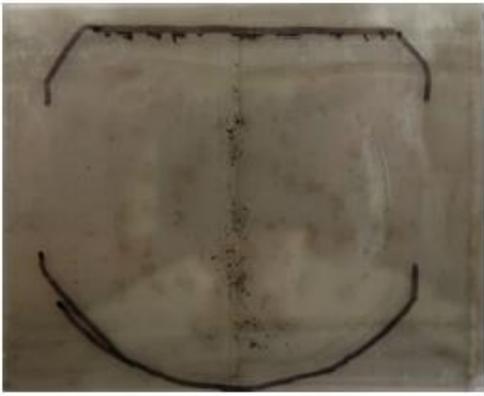
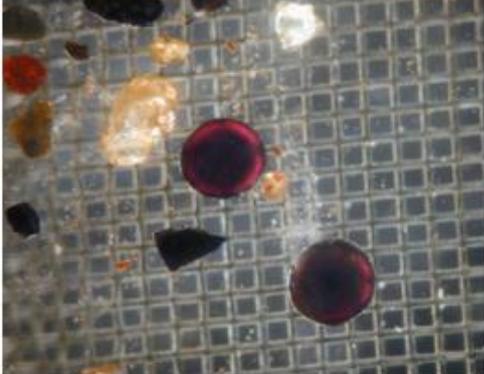
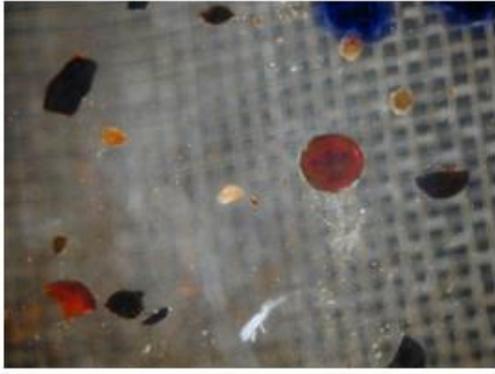
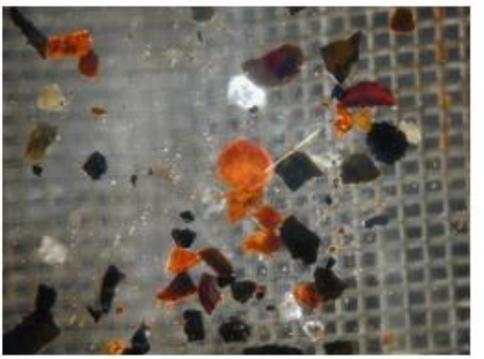
B15 20171122-STT-2

	
<p>Overview</p>	<p>White, bean-looking granules</p>
	
<p>White fibre</p>	<p>Area with rust and ZnCl₂-crystals</p>

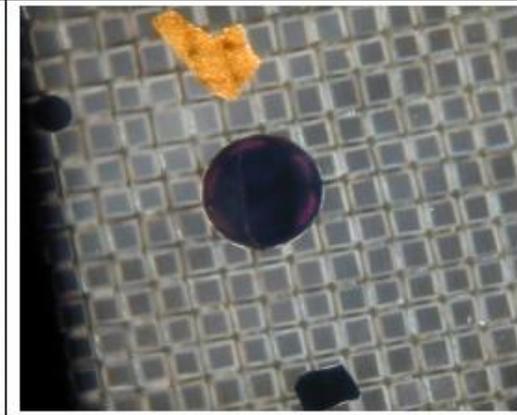
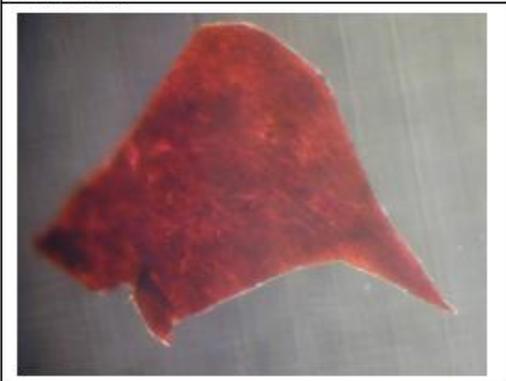
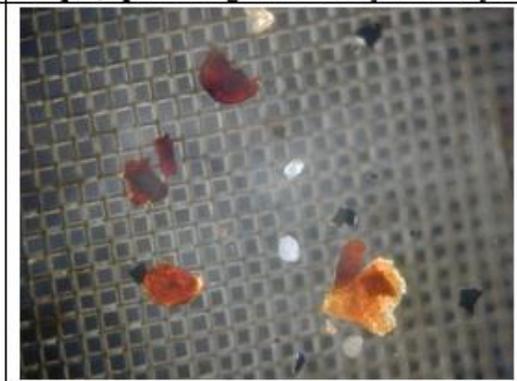
B16 20171122-KF2-6

	
<p>Overview</p>	<p>Red fibre</p>
	
<p>Orange granule</p>	<p>Clear, white, black and orange granules</p>

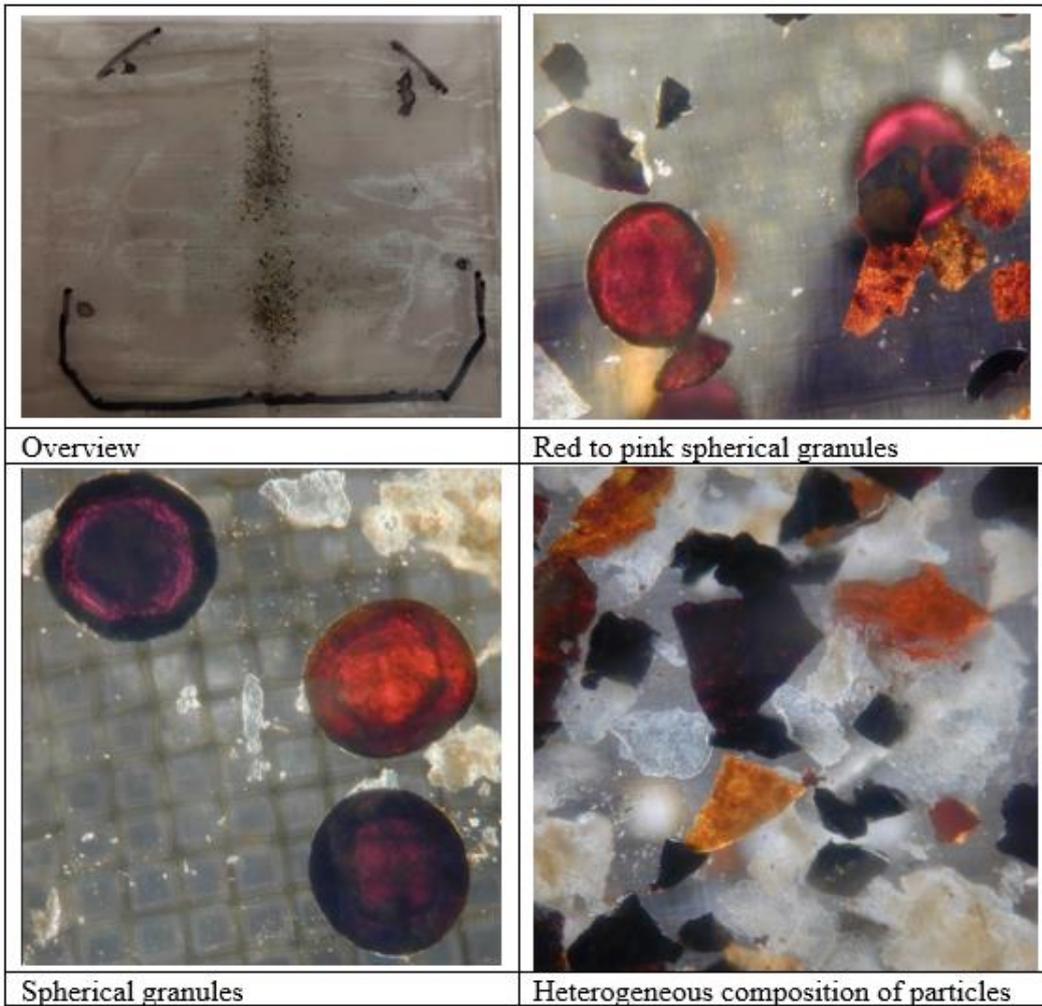
4 B17 20171122-SC3-4

	
<p>Overview</p>	<p>Red fibre</p>
	
<p>Spherical, purple granules</p>	<p>Spherical orange to red granule and red granule with sharp edges</p>
	
<p>Heterogeneous composition of particles. NB! Orange, spherical granule</p>	<p>White fibre</p>

B18 20171127-KRT-14

	
<p>Overview</p>	<p>Purple, spherical granule and yellow layer</p>
	
<p>Red layer with sharp edges</p>	<p>Heterogeneous composition of particles, some with sharp edges</p>

B19 20171124-GRS-2



APPENDIX C

Abundance of potential microplastic

Visual analysis for method blanks

Date examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				No further comment
			A	B	C	D	A	B	C	D	A	B	C	D	
04.12.2017	Blank 1 (20170922)	Clear/white	6	9	1	1	2	1			12	1			No further comment
		Light brown		1			2				3	9			
		Dark brown					2				7	2			
		Black									1				
		Blue		1											
		Red													
		Green													
		Orange													
		Yellow													
		Total		6	11	1	1	6	1	0	0	23	12	0	
Fraction		19				7				35			57,4 %		

Date examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				Damage on filter.
			A	B	C	D	A	B	C	D	A	B	C	D	
05.12.2017	Blank 2 (20170922)	Clear/white	1	2	2		2				1	1			Damage on filter.
		Light brown													
		Dark brown													
		Black	1												
		Blue		2	1	3									
		Red					1				2				
		Green													
		Orange													
		Yellow													
		Total		2	4	3	3	3	0	0	0	3	1	0	
Fraction		12				3				4			21,1 %		

Visual analysis for method blanks

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				No further comment	
			A	B	C	D	A	B	C	D	A	B	C	D		
05.12.2017	Blank 3 (20170922)	Clear/white	2	2												No further comment
		Light brown														
		Dark brown														
		Black														
		Blue	1													
		Red												3		
		Green														
		Orange														
		Yellow														
					3	2	0	0	0	0	0	0	0	0	4	
			5										4		44,4 %	
															9	

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				Rust and damage on filter. Holes observed.		
			A	B	C	D	A	B	C	D	A	B	C	D			
05.12.2017	Blank (20171019)	Clear/white	15	7	12	1					1						Rust and damage on filter. Holes observed.
		Light brown															
		Dark brown					2				3						
		Black															
		Blue															
		Red													1		
		Green															
		Orange															
		Yellow															
					15	8	13	2	3	0	3	1	8	0	0	0	
			38				7			13,2 %			8		15,1 %		
															53		

Visual analysis for method blanks

Date examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				Possible cross-contamination of micropowder detected.						
			A	B	C	D	A	B	C	D	A	B	C	D							
05.12.2017	Blank (20171123)	Clear/white																			
		Light brown																			
		Dark brown																			
		Black																			
		Blue																			
		Red																			
		Green																			
		Orange																			
		Yellow																			
					0	3	1	0	10	0	0	0	0	0	0	3	1	0	0		
			4			22,2%	10						4							22,2%	

Date examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				Damage to filter, but no holes.						
			A	B	C	D	A	B	C	D	A	B	C	D							
05.12.2017	Blank (20171121)	Clear/white																			
		Light brown																			
		Dark brown																			
		Black																			
		Blue																			
		Red																			
		Green																			
		Orange																			
		Yellow																			
					2	13	3	1	0	2	5	0	0	0	21	4	0	0	0		
			20			37,7%	8						25							47,2%	

Visual analysis for method blanks

Colour	Fibre 1D				Layer 2D				Granulat 3D				% based on color
	A	B	C	D	A	B	C	D	A	B	C	D	
	Clear/white	3,5	5,9	4,5	1,9	1,8	0,4	0,8	0,1	19,0	3,6	0,0	
Light brown	0,0	0,3	0,0	0,0	1,8	1,1	0,6	0,3	2,3	2,0	0,1	0,0	15,0 %
Dark brown	0,0	0,1	0,0	0,0	0,3	0,0	0,0	0,0	0,9	0,3	0,3	0,0	3,1 %
Black	0,1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,4	0,0	0,0	0,0	0,9 %
Blue	0,1	0,6	0,5	0,6	0,0	0,1	0,0	0,0	0,0	0,0	0,0	0,0	3,6 %
Red	0,0	0,0	0,1	0,0	0,3	0,0	0,0	0,0	0,8	0,0	0,0	0,0	2,0 %
Green	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0 %
Orange	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,3	0,1	0,0	0,0	0,7 %
Yellow	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0 %
Sum	3,8	6,9	5,1	2,5	4,0	1,6	1,4	0,4	23,5	6,0	0,4	0,0	
Fraction of total	18,3		32,9 %		7,4		13,3 %		29,9		53,8 %		55,5
SUM													

Average (n=8)

Visual analysis of MP-samples (not corrected)

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				
			A	B	C	D	A	B	C	D	A	B	C	D	
22.12.2017	VI-03 (20171120)	Clear/white	1	7	10	3			23	17			25		
		Light brown						1					3		
		Dark brown											12		
		Black									2	27	36	14	
		Blue				1									
		Red											6	1	
		Green													
		Orange												5	
		Yellow												8	
		Total			1	7	10	4	0	0	24	17	2	86	37
Fraction			22		10,9%	41		20,3%	139		68,8%	202			

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				
			A	B	C	D	A	B	C	D	A	B	C	D	
03.01.2018	KRT-14 (20171130)	Clear/white	1	5	10	4			2				28		
		Light brown											1		
		Dark brown			1								30	9	
		Black									4	55	13	4	
		Blue			2										
		Red								1		14	3	3	
		Green													
		Orange											8	2	
		Yellow									2	1	11	2	
		Total			1	5	13	4	0	0	4	2	5	147	29
Fraction			23		10,6%	6		2,8%	188		86,6%	217			

Visual analysis of MP-samples (not corrected)

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				See SNB-16R #1 and SNB-16R #2 for separate tables
			A	B	C	D	A	B	C	D	A	B	C	D	
18.12.2017	SNB-16R (AVG)	Clear/white	14,5	40,0	9,5	2,5	13,0	14,0			12,5	30,0			
		Light brown					2,5	3,5			1,5	3,0			
		Dark brown					1,5	2,0				2,5			
		Black					18,0	9,0			2,5	14,5	1,0		
		Blue		2,0											
		Red						1,5			1,0	5,5	1,0		
		Green													
Orange															
Yellow															
Total			14,5	42,5	10	2,5	35	30	0	0	18	56	2	0	
Fraction			69,5		33,0 %	65		30,9 %		76		36,1 %		210,5	

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				TMC = Too Many to Count
			A	B	C	D	A	B	C	D	A	B	C	D	
12.12.2017	VAL-5 (20171103)	Clear/white	23	13	1	8	9	2			TMC	TMC	1		
		Light brown													
		Dark brown					1	5				1			
		Black													
		Blue	1		1	2	1	1	1		TMC	73			
		Red									3	3			
		Green													
Orange										2					
Yellow															
Total			1	23	15	10	0	11	8	0	8	77	1	0	
Fraction			49		31,8 %	19		12,3 %		86		55,8 %		154	

Visual analysis of MP-samples (not corrected)

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D								
			A	B	C	D	A	B	C	D	A	B	C	D					
20.12.2017	Reg-12 (20171117)	Clear/white	1	14	2														
		Light brown					2												
		Dark brown						1											
		Black														5	5	2	
		Blue	1		2														
		Red																	
		Green																	
		Orange																	
		Yellow																	
		Total			2	14	4	0	2	10	0	0	0	13	16	2	0		
Fraction			20		31,7 %	12		19,0 %			31		49,2 %						

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D								
			A	B	C	D	A	B	C	D	A	B	C	D					
19.12.2017	Reg1-09 (20171005)	Clear/white		2	5	3	13	11	1	1	19	124	11						
		Light brown			1			11		2		18							
		Dark brown										2							
		Black																	
		Blue						5			10	70	17	8					
		Red										1							
		Green										3							
		Orange																	
		Yellow														2	3	2	1
		Total			0	2	6	3	13	27	1	3	221	30	10				
Fraction			11		3,2 %	44		12,7 %		292		84,1 %							

Visual analysis of MP-samples (not corrected)

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D					
			A	B	C	D	A	B	C	D	A	B	C	D		
10.01.2018	Reg-14 (20171002)	Clear/white			10	16	10	6	24	7	77	55	2			
		Light brown							5	1						
		Dark brown										1				
		Black						6	3			21	19	3		
		Blue		2									3	1		
		Red			1											
		Green														
		Orange						3								
Yellow																
Total			0	12	17	11	15	32	8	102	77	6	0	280		
Fraction			40		14,3%		55		19,6%	185		66,1%				

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D					
			A	B	C	D	A	B	C	D	A	B	C	D		
10.01.2018	Vega-R (20171116)	Clear/white	1	6	8	2					4	34				
		Light brown										2				
		Dark brown														
		Black									1	7				
		Blue														
		Red														
		Green														
		Orange											1			
Yellow																
Total			1	6	8	2	0	0	0	5	44	0	0	66		
Fraction			17		25,8%		0		0,0%	49		74,2%				

Visual analysis of MP-samples (not corrected)

Dato examined	Sample ID	Colour	Fibre ID				Layer 2D				Granulat 3D							
			A	B	C	D	A	B	C	D	A	B	C	D				
10.01.2018	VI-RB (20171117)	Clear/white	1	3	3	2					5				6	39	2	
		Light brown													1	5	2	
		Dark brown													6	2		
		Black													17	75	7	
		Blue																
		Red														1		
		Green															8	
		Orange																
		Yellow								1								
		Total			1	3	3	2	0	5	1	0	0	30	130	11	0	186
Fraction			9			4,8%	6			3,2%		171			91,9%			

Dato examined	Sample ID	Colour	Fibre ID				Layer 2D				Granulat 3D					
			A	B	C	D	A	B	C	D	A	B	C	D		
10.01.2018	GYDA-18 (20171103)	Clear/white			4	5			1				3	92		
		Light brown												101		
		Dark brown												8		
		Black												6		
		Blue														
		Red														
		Green														
		Orange											1	1		
		Yellow											2	3		
		Total			0	4	5	1	0	1	1	0	6	211	0	0
Fraction			10			4,4%	2			0,9%	217			94,8%		

Visual analysis of MP-samples (not corrected)

Dato examined	Sample ID	Colour	Fibre ID				Layer 2D				Granulat 3D			
			A	B	C	D	A	B	C	D	A	B	C	D
18.12.2017	SNB-16R #1	Clear/white	29	67	11		26	28			20	21		
		Light brown					5	7			3	6		
		Dark brown					3	4						
		Black		1			36	18				1		
		Blue		4							1			
		Red						1						
		Green												
		Orange												
		Yellow												
Fraction			29	72	11	0	70	58	0	0	24	28	0	0
			112				128				52			
			38,4 %				43,8 %				17,8 %			
			292											

Dato examined	Sample ID	Colour	Fibre ID				Layer 2D				Granulat 3D			
			A	B	C	D	A	B	C	D	A	B	C	D
09.01.2018	SNB-16R #2	Clear/white												
		Light brown		13	8	5					5	39		
		Dark brown											5	
		Black									5	28	2	
		Blue			1									
		Red							2		2	11	2	
		Green												
		Orange									1			
		Yellow												
Fraction			0	13	9	5	0	2	0	0	12	84	4	0
			27				2				100			
			20,9 %				1,6 %				77,5 %			
			129											



Visual analysis of MP-samples (corrected)

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				sum	
			A	B	C	D	A	B	C	D	A	B	C	D		
22.12.2017	VI-03 (20171120)	Clear/white	1	6	1				22	17			21			Sample contained unusual amounts of crystal formation
		Light brown											1			
		Dark brown											12			
		Black										2	27	36	14	
		Blue														
		Red											6	1		
		Green														
Orange												5				
		Yellow										8				
															180	

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				sum	
			A	B	C	D	A	B	C	D	A	B	C	D		
03.01.2018	KRT-14 (20171130)	Clear/white		6	2					1				24		No further comment
		Light brown														
		Dark brown		1										30	9	
		Black										4	55	13	4	
		Blue			2											
		Red									1		14	3	3	
		Green														
Orange												8	2			
		Yellow							2	1		11	2			
															197	

Visual analysis of MP-samples (corrected)

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				sum	
			A	B	C	D	A	B	C	D	A	B	C	D		
18.12.2017	SNB-16R (AVG)	Clear/white	11	34	5		11	14					26			An average of SNB-16R#1 og SNB-16R#2
		Light brown					2						1			
		Dark brown					1	2					2			
		Black					18	9				2	15	1		
		Blue		1												
		Red						2					6	1		
		Green														
Orange																
Yellow																
															164	

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				sum	
			A	B	C	D	A	B	C	D	A	B	C	D		
12.12.2017	VAL-5 (20171103)	Clear/white	17	9	6		9	1			TMC	TMC	1			An underestimate. Rust around crystal formation. Round transparent particles observed: (278 pcs A-B). Damage to filter observed.
		Light brown			1											
		Dark brown					1	5								
		Black									TMC	73				
		Blue						1			3	3				
		Red									2					
		Green														
Orange																
Yellow										2						
															135	

Visual analysis of MP-samples (corrected)

Date examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				sum	
			A	B	C	D	A	B	C	D	A	B	C	D		
10.01.2018	Reg-14 (20171002)	Clear/white	4	12	8	4	24	6	58	51	2					4 round transparent particles observed.
		Light brown					4									
		Dark brown														
		Black														
		Blue	1			6	3			21	19	3				
		Red									3	1				
		Green									2					
		Orange				3										
		Yellow														

Date examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				sum	
			A	B	C	D	A	B	C	D	A	B	C	D		
10.01.2018	Vega-R (20171116)	Clear/white				4										No further comment
		Light brown														
		Dark brown														
		Black														
		Blue														
		Red														
		Green														
		Orange														
		Yellow													1	

Visual analysis of MP-samples (corrected)

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				sum	162		
			A	B	C	D	A	B	C	D	A	B	C	D				
10.01.2018	VI-RB (20171117)	Clear/white													35	2	Multiple orange spheric particles.	
		Light brown					5								3	2		
		Dark brown									5	2						
		Black									17	75	7					
		Blue													1			
		Red																
		Green													8			
Orange								1										
Yellow																		
																	sum	162

Dato examined	Sample ID	Colour	Fibre 1D				Layer 2D				Granulat 3D				sum	208		
			A	B	C	D	A	B	C	D	A	B	C	D				
10.01.2018	GYDA-18 (20171103)	Clear/white													88		Most of the light brown particles looks like weathered beads.	
		Light brown													99			
		Dark brown													8			
		Black													6			
		Blue																
		Red																
		Green																
Orange												1	1					
Yellow												2	3					
																	sum	208



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