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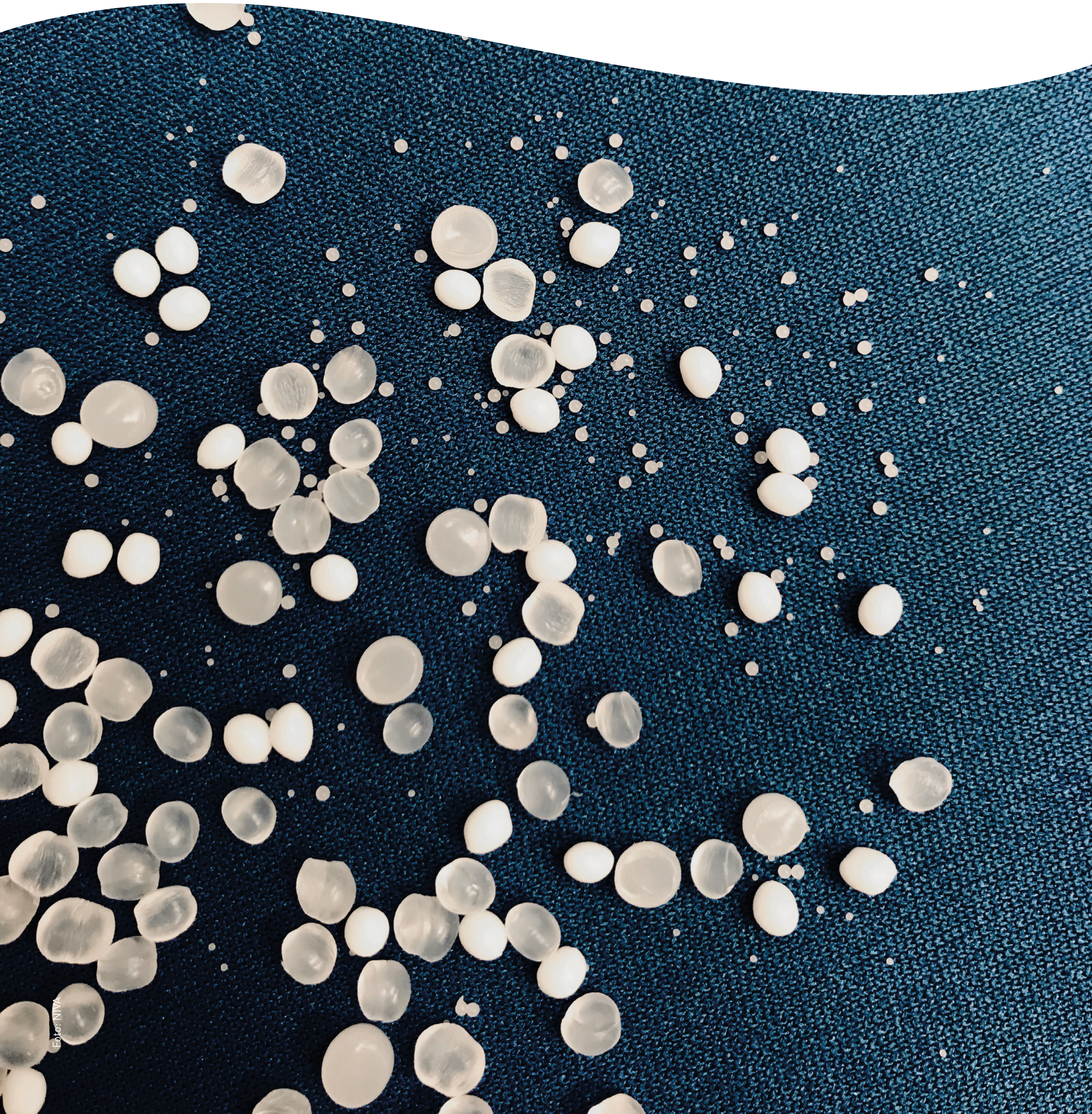
Mapping microplastics in sludge



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REPORT

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Summary

The main objective of this project was to characterize microplastics in sewage sludge from Norwegian domestic wastewater treatment plants applying different wastewater and sludge treatment technologies. WWTPs were selected to cover the three-main domestic WWTP categories in Norway and the main applied sludge treatment processes. Fenton's reagent was used to remove organic matter and density separations were employed to extract microplastics from sludge samples. Plastics were found in all ten sludge samples investigated from eight WWTPs. The overall average plastic abundance was 6 077 particles kg^{-1} (d.w.) (1701 – 19 837) or 1 176 889 particles m^{-3} (470 270 – 3 394 274). Based on the average microplastic abundance and the present application of sewage sludge in Norway, it was estimated that over 500 billion microplastics are released into the environment via sewage sludge application each year, to agricultural soils, green areas and soil producers. This likely represents a significant source of microplastics to terrestrial and marine systems.

Four keywords	Fire emneord
1. Microplastics	1. Mikroplast
2. Sludge	2. Avløpsslam
3. Wastewater	3. Avløpsvann
4. Soil improver	4. Jordforbedring

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Mapping microplastics in sludge

Preface

This report presents the results from the project Mapping microplastics in sludge (Kartlegging av mikroplast i avløpsslam). The project has been run in agreement between Miljødirektoratet as client and NIVA as project manager. The client contact has been Hannah Hildonen. Project leader at NIVA has been Marianne Olsen. Sampling was carried out by staff at wastewater treatment plants and coordinated by Christian Vogelsang. Method development, analysis of sludge samples and statistical analysis was carried out by Rachel Hurley and Amy Lusher. Additional dry weight and organic content analysis of sludge samples carried out by Eurofins. The report was written by Amy Lusher and Rachel Hurley with contributions from Christian Vogelsang and Marianne Olsen. Chris Harman carried out QA of the report. NIVA appreciates the opportunity to participate in this project and acknowledges everyone involved for good cooperation

Oslo, 11.12.2017

Marianne Olsen
Project manager

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Summary

The main objective of this project was to characterize microplastics in sewage sludge from Norwegian domestic wastewater treatment plants (WWTPs) which use different wastewater and sludge treatment technologies.

WWTPs are considered to be important receptors and processors for microplastics from households, industrial effluents and urban drainage. As the major fraction of the sludge produced in WWTPs is applied as a soil conditioner on farmland, it may represent a significant source of microplastics to the environment. It is therefore important to establish the content of microplastic particles in treated sludge. To map and compare the content of microplastics in sewage sludge from different types of wastewater treatment plants in Norway, it was necessary to identify the most appropriate and cost-effective method for analysing microplastics. Once methods were established, a comparison between different WWTPs could be made.

WWTPs were selected to cover the main categories of wastewater treatment processes and sludge treatment processes applied in Norway. WWTPs that participate in the five-yearly sludge screening campaign organised by Norsk Vann and the Norwegian Environment Agency and/or in the quarterly sampling to the Environmental Specimen Bank were prioritised. Samples were collected from each plant over several days and homogenised prior to the compositing of triplicate samples for microplastic analysis.

Four protocols for the removal of organic material from sludge samples were tested. Eight different polymers were treated and test sludge samples were used. The two alkaline treatments, Protocol 3 and 4 did not remove a sufficient proportion of the organic material present in the test sludge samples and there was evidence of degradation of some of the treated polymers. The peroxide-based treatments (Protocol 1a, 1b & 2) were significantly more effective in removing organic material from the test sludge samples; however, Protocols 1a and 1b demonstrated degradation of some of the polymers. The Fenton's reagent treatment (Protocol 2) showed no signs of degradation for any of the tested polymers. The treatment was also successful in reducing a large proportion of the organic content of the sludge samples. This protocol requires the shortest reaction times and the least laboratory infrastructure (e.g. incubators). Fenton's reagent was chosen as a cost- and time-effective method for extracting microplastics from sludge.

Sludge samples were thoroughly mixed before sub-sampling and triplicate sub-samples were taken to account for within-sample variability. Samples (10 g w.w.) were placed into clean, pre-washed containers. 20 ml of 30% H_2O_2 and 10 ml of ferrous sulfate catalyst solution was added to the sludge samples. The reaction took approximately two hours to complete at room temperature. This process removed most of the organic material. Following organic matter removal, microplastics were extracted through a density separation procedure (low density: 1 g cm^{-3} and high density: 1.8 g cm^{-3}).

Microplastic analysis was initially performed using a visual identification step. Suspected microplastic particles were identified using a rigorous visual protocol and further verification using the hot needle test. Manual identification of microplastic particles was supported by chemical characterisation using an infrared scanner: Fourier Transform Infrared Spectroscopy (FT-IR). Each microplastic particle was further characterised based on its morphological properties: size, shape, and colour.

Microplastics were found in all ten sludge samples investigated from eight WWTPs. The overall average plastic abundance was 6 077 particles kg^{-1} (d.w.) (1701 – 19 837) or 1 176 889 particles m^{-3} (470 270 –

3 394 274). Particles from sludge consisted of beads (37.6 %), fragments (31.8 %) fibres (28.9 %) and glitter (1.7 %). Most of the particles were clear in colour (41 %). Ten percent of the overall particles extracted were tested using FT-IR (n= 60). All particles tested were confirmed to be plastic. Polyethylene particles were the most common (30.5 %), followed by polyethylene terephthalate (26.7 %) and polypropylene (20.3 %). 62 % of plastics were extracted during the low density (1 g cm^{-3}) separation steps and 38 % were extracted at high density (1.8 g cm^{-3}).

No significant correlations were observed between microplastic abundances and the size of the WWTP (population equivalent), the influent volume or sludge production, and there were no significant differences between treatment processes or weather conditions, which indicates a complexity in the variables influencing microplastic contamination in the final sludge. The potential sources of microplastics to WWTPs may include domestic and industrial effluents as well as stormwater runoff. Based on the average microplastic abundance and the present application of sewage sludge in Norway, it was estimated that over 500 billion microplastics are released into the environment via sewage sludge application each year to agricultural soils, green areas and soil producers. This likely represents a significant source of microplastics to terrestrial systems.

Sammendrag

Tittel: Kartlegging av mikroplast i avløpsslam
År: 2017
Forfatter: Amy L. Lusher, Rachel Hurley, Christian Vogelsang, Luca Nizzetto og Marianne Olsen
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Hovedformålet med dette prosjektet var å karakterisere mikroplast i kloakkslam fra norske avløpsrenseanlegg med ulike rense- og slambehandlingsteknologier.

Avløpsrenseanlegg mottar mikroplast via avløpsvann fra husholdninger og industri, og urban overflateavrenning. Ettersom en stor andel av mikroplasten ender i det endelige slammet fra renseprosessene, og siden hovedmengden av dette slammet benyttes innenfor landbruket, kan slammet være en betydelig spredningsvei for mikroplast til miljøet. Det er derfor viktig å fastslå innholdet av mikroplast-partikler i behandlet slam. For å kartlegge og sammenligne innholdet av mikroplast i slam fra ulike typer avløpsrenseanlegg i Norge, var det nødvendig å identifisere de mest hensiktsmessige og kostnadseffektive metodene for analyse av mikroplast i slam. Når metodene var etablert, kunne det gjøres en sammenligning mellom forskjellige renseanlegg.

Ulike avløpsrenseanlegg ble valgt for å dekke de viktigste avløpsvann- og slambehandlingsprosessene i Norge. Anlegg som deltar i det fem-årige slamprogrammet organisert av Norsk Vann og Miljødirektoratet og / eller kvartalsvis prøvetaking til Miljøprøvebanken ble prioritert. Prøver ble samlet fra hvert anlegg over flere dager og homogenisert før det ble tatt ut tre parallelle prøver for mikroplast-analyse.

Fire protokoller for fjerning av organisk materiale fra slamprøver ble testet. Åtte forskjellige polymere ble testet. To alkaliske behandlinger, protokoll 3 og 4 fjernet ikke en tilstrekkelig andel av det organiske materiale som var tilstede i test-slamprøvene, og det var tegn på nedbrytning av noen av de behandlede polymerene. De peroksidbaserte behandlingene (protokoll 1a, 1b og 2) var signifikant mer effektive ved fjerning av organisk materiale fra test-slamprøvene. Fentons reagensbehandling (protokoll 2) viste ingen tegn på nedbrytning for noen av de testede polymerene. Behandlingen var også vellykket for å redusere andelen organisk materiale assosiert med slamprøvene. Denne protokollen er mest tidseffektiv og krever minst laboratorie-infrastruktur (for eksempel inkubatorer). Fentons reagens ble valgt som en kostnadseffektiv og tidsbesparende metode for ekstraksjon av mikroplast fra slam.

Slamprøver ble grundig blandet før uttak av prøver. Disse ble tatt i triplikat for å ta hensyn til variabiliteten i blandprøvematerialet. Prøver (10 g våtvekt) ble plassert i rene, forhåndsvaskede beholdere. 20 ml 30 % H_20_2 og 10 ml jernsulfatkatalysatoroppløsning ble tilsatt slamprøvene. Reaksjonen tok omtrent to timer ved romtemperatur. Denne prosessen fjernet det meste av det organiske materialet. Deretter ble mikroplast ekstrahert gjennom en tetthetsseparasjonsprosedyre (lav tetthet, 1 g cm^{-3} og høy tetthet 1.8 g cm^{-3}).

Mikroplast-analyse ble innledningsvis utført ved bruk av et visuelt identifikasjonstrinn. Potensielle mikroplast-partikler ble identifisert ved hjelp av en streng visuell protokoll og ytterligere testing ved bruk av en varm nål. Manuell identifisering av mikroplast-partikler ble støttet av kjemisk karakterisering ved hjelp av en infrarød skanner: Fourier Transform Infrared Spectroscopy (FT-IR). Hver mikroplast-partikkel ble videre karakterisert basert på deres morfologiske egenskaper: størrelse, form og farge.

Plast ble funnet i alle ti slamprøvene undersøkt fra åtte renseanlegg. Gjennomsnittlig antall mikroplastpartikler var 6 077 partikler kg^{-1} (d.w.) (1 701 – 19 837) eller 1 176 889 partikler m^{-3} (470 270 – 3 394 274). Partikler fra slam besto av perler (37.6 %), fragmenter (31.8 %) fibre (28.9 %) og glitter (1.7 %). De fleste partiklene var klare i farge (41 %). Ti prosent av de samlede partiklene som ble ekstrahert ble testet ved bruk av FT-IR (n = 60). Alle testede partikler ble bekreftet å være plast. Polyetylenpartikler var de vanligste (30.5 %) etterfulgt av polyetylentereftalat (26.7 %) og polypropylen (20.3 %). 62 % av plastpartiklene ble ekstrahert ut under separasjonstrinn med lav tetthet (1 g cm^{-3}) og 38% ble ekstrahert ut ved høy tetthet (1.8 g cm^{-3}).

Ingen signifikante korrelasjoner ble observert mellom forekomst av mikroplast og størrelsen på anleggene (Personekvivalent), behandlet mengde avløpsvann eller slamproduksjon, og det var ingen signifikante forskjeller mellom behandlingsprosesser eller værforhold (nedbør). Dette indikerer en kompleksitet i de variablene som påvirker forekomsten av mikroplast i slammet. Potensielle kilder til mikroplast som tilføres renseanlegg kan omfatte husholdnings- og industriavløp samt avrenning fra veier og andre tette flater i byene under nedbørshendelser. Basert på det gjennomsnittlige antallet mikroplast funnet i slamprøvene fra disse åtte avløpsrenseanleggene og den nåværende bruken av kloakkslam i Norge, ble det anslått at over 500 milliarder mikroplast-partikler slippes ut i miljøet hvert år via avløpsslam til landbruksjord, grønne områder og jordprodusenter. Dette representerer sannsynligvis en betydelig kilde til mikroplast til terrestriske systemer.

1 Introduction

The main objective of this project was to characterise microplastics in sewage sludge from Norwegian domestic wastewater treatment plants which apply different wastewater and sludge treatment technologies.

Widespread contamination of the environment by anthropogenic debris has become a major concern. Early work concentrated on large plastic items, termed *macroplastics*. However, the focus has now shifted onto smaller plastic particles known as *microplastics* and *nanoplastics*. These are produced from the breakdown of larger plastics, but also include specifically-engineered particles such as microbeads used in personal care products. Microplastics can be derived from a number of sources including the breakdown of textiles during washing, the abrasion of car tyres, and the fragmentation of plastic litter. Irrespective of their sources, microplastics pose a threat to ecosystems and biota. This can occur through a range of mechanisms including physical accumulation in digestive tracts and chemical transfer of sorbed contamination or plastics additives [1], though there are still knowledge gaps that need to be filled.

Microplastic particles have been detected worldwide. They have now been observed on every continent and in a range of environments including the oceans [2, 3], freshwater systems [4, 5], and terrestrial soils [6]. Following initial baseline assessments that revealed high levels of contamination, research now focuses on establishing potential sources and routes transport for microplastics to the environment. Wastewater treatment plants (WWTPs) are considered to be important receptors and processors for microplastics generated through urban drainage, and from municipal and industrial effluents [7, 8]. Microbeads from personal care products and synthetic fibres from the washing of clothes and textiles are released to wastewater from households. Plastic pellets or shavings may be released through industrial processes. Surface water drains may receive inputs from tire wear, road paint fragments, and degraded plastic litter in combined sewer systems [9]. WWTPs have been shown to be effective at trapping up to 99 % of microplastics larger than 20 – 45 µm [10, 11]. Although, often only larger size classes (typically >250 – 300 µm) are analysed. Murphy *et al*. [7] found that approximately 45 % of the microplastics by nu mber were captured together with the grit and grease removal at the entrance of the WWTP they studied. An additional 34 % was removed in the subsequent primary settler. These results are interesting for two reasons; (1) The large fraction of the microplastics that are collected together with the grit and grease may not end up in the final sludge, as at least the grit is typically sent away for destruction. However, due to the typically low specific density of the microplastics and short residence time in the grit chamber, it is likely that the majority of the collected microplastics are found in the floating grease fraction; (2) Relatively simple treatment is needed to remove the majority of microplastics >20 µm.

Limited biodegradation of the microplastics can be expected [12, 13], hence the majority of microplastic particles removed from wastewater will be found in the solid sludge phase. Sludge has to go through consecutive thickening, dewatering, stabilisation and hygienisation before it can be used for *e.g.,* soil conditioning. This may represent a significant source of microplastics to the environment. In Europe, up to 50 % of sewage sludge is used in this way [5, 14]. Once added to soil, microplastics may become incorporated into the soil matrix (e.g. through soil aggregation; [15]) or eroded to aquatic environments by wind or water [14]. Preliminary work suggests that biosolid treatment (outlined in **Figure 1)** does not have a large influence on reducing microplastic concentrations [16]; however, this needs to be assessed further. It is important to establish the content of microplastic particles in treated sludge.

Thus far, a number of different methods have been used to analyse microplastics in sewage sludge; however, they have not yet been properly tested or optimised. Analysis of sludge samples is challenging because samples usually consist of large, complex, highly organic material interspersed with other benign particles of different origin [17]. In addition, several different combinations of treatment methods are used to achieve a final sludge that is stabilised and hygienic (see **Figure 1**). The lack of standardized methods for sampling, extraction, purification and identification of microplastics complicates the comparison between the relatively few sludge microplastic studies conducted thus far. To map and compare the content of microplastic in waste sludge from different types of wastewater treatment plants in Norway, it is necessary to identify the most appropriate and cost-effective methods for analysing microplastics. This project had two aims:

1. To identify and test suitable methods for processing sludge samples
2. To analyse sludge and make a comparison between different WWTPs around Norway

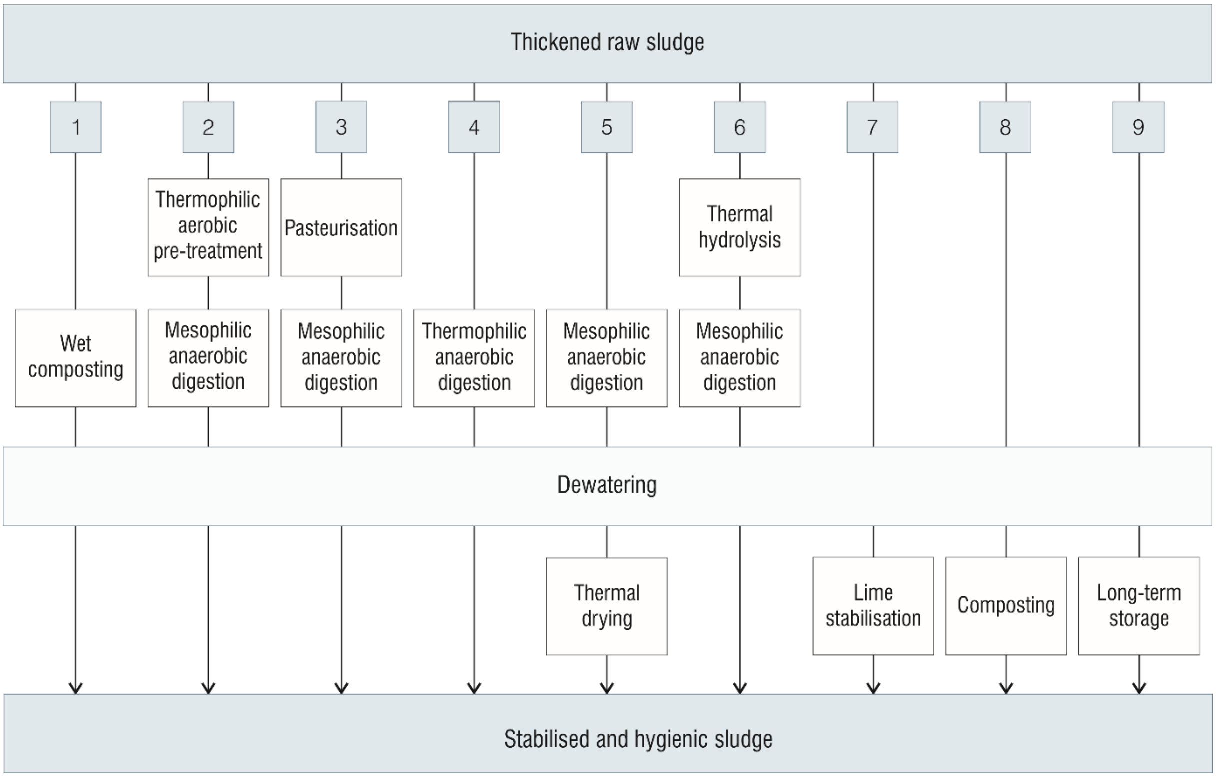

Figure 1. Overview of different sludge treatment processes. Adapted from Ødegaard et al. [18]

2 Materials and Methods

2.1 Wastewater treatment plant selection and sampling

Wastewater treatment plants were selected in agreement with the client. Eight sites were chosen, two of which were investigated twice.

2.1.1 Selection of wastewater treatment plants

WWTPs included in this study were selected to cover the three-main domestic WWTP categories in Norway (see **Box 1**) and the main applied sludge treatment processes (see **Figure 1**) except composting. An additional aspect was the influence from microplastics in road runoff, which was covered by sampling sludge under both dry and wet weather conditions at WWTPs expected to receive heavily polluted road runoff and by selecting an additional WWTP that was expected to receive limited road runoff[1]. WWTPs that participate in the five-yearly sludge screening campaign organised by Norsk Vann and the Norwegian Environment Agency and/or in the quarterly sampling to the Environmental Specimen Bank[2] were prioritised. **Table 1** summarises the treatment principles applied at the selected WWTPs, while **Table 2** summarises annual and dry weather influent loads and annual sludge productions. **Figure 2** shows the location of the selected WWTPs.

Box 1.
Main domestic WWTP categories in Norway.

Primary treatment: Approximately 20 % of the Norwegian population is connected to a WWTP that is only applying mechanical treatment to remove particulate matter.

Secondary treatment: A similar fraction of the population (38 %) is connected to a WWTP that apply secondary treatment, which in Norway typically imply chemical precipitation to ensure proper phosphorous removal and no biological process step.

Tertiary treatment: The largest WWTPs in Norway, receiving wastewater from approximately 37 % of all inhabitants (data for 2015 from KOSTRA[3]), apply advanced tertiary treatment (including both biological and chemical treatment steps).

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[1] The stormwater pipeline network in Lier is more or less completely disconnected from the domestic sewage pipeline network that leads to Linnes WWTP.

[2] https://miljoprovebanken.no/english/

[3] https://www.ssb.no/offentlig-sektor/kostra

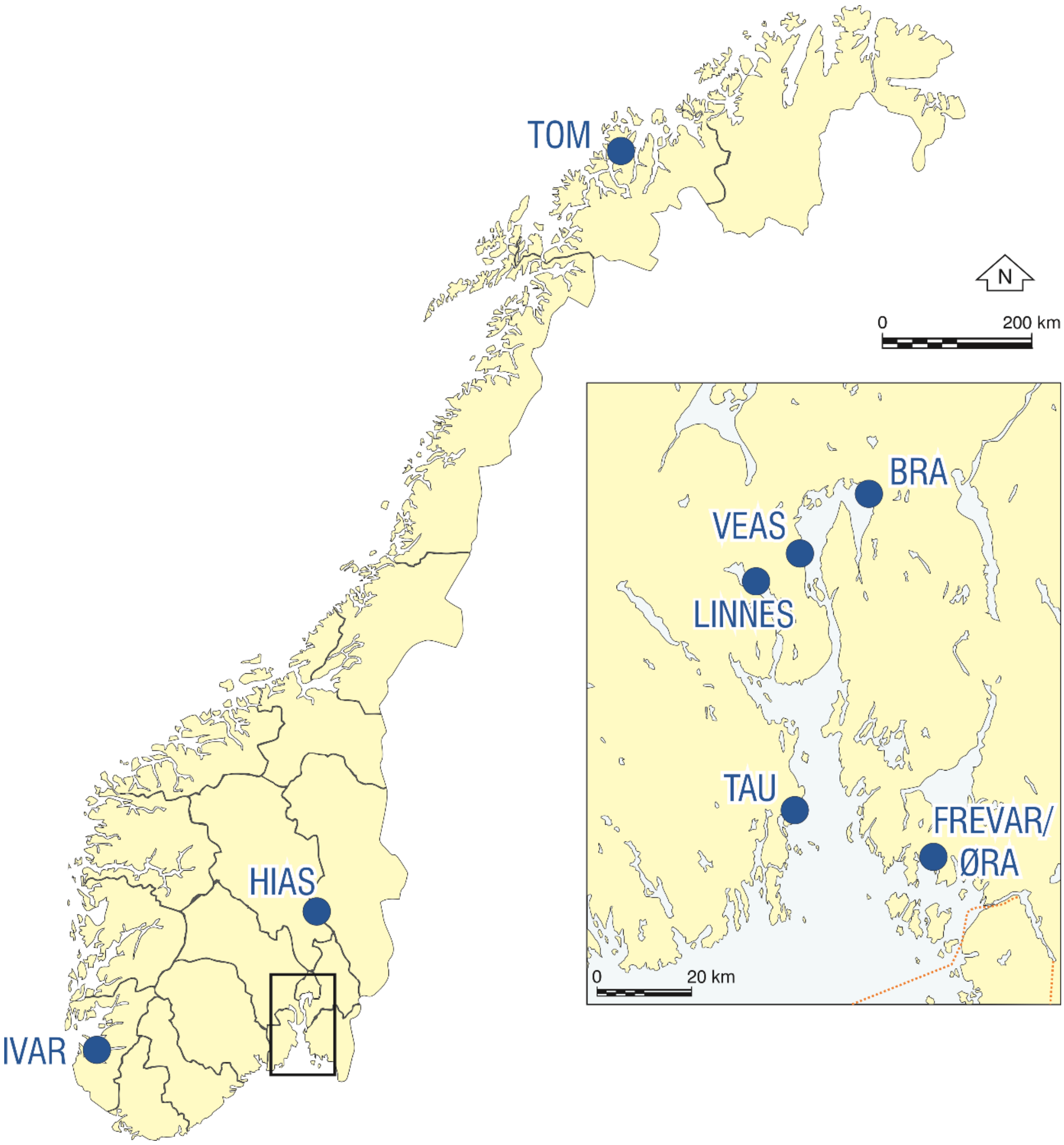

Figure 2. Site locations of wastewater treatment plants (WWTPs) sampled for sludge in 2017. Labels on the figure correspond to the site codes in **Table 1**

Table 1. Details on WWTPs that were investigated in this study including information on water and sludge treatment type. Water treatment is colour coded by treatment type: tertiary treatment (green), secondary treatment (yellow) and primary treatment (blue).

WWTP (Location)	Code	Number of samples	Water treatment	Sludge type	Sludge treatment	Sludge application
Bekkelaget WWTP (Oslo)	BRA	2[a]	Tertiary treatment; simultaneous precipitation and biological N-removal (activated sludge process).	Stabilised and dewatered sludge	Thermophilic anaerobic stabilisation over 14 days	Agriculture; primarily grain
Vestfjorden avløpsselskap (Slemmestad)	VEAS	2[a]	Tertiary treatment; chemical precipitation followed by biological N-removal (biofilm process). Separate excess stormwater treatment (mechanical/chemical)	Stabilised and dewatered sludge	Thermophilic anaerobic digestion and lime stabilisation.	Agriculture; primarily grain
HIAS (Hamar)	HIAS	1	Secondary treatment; chemical precipitation and biological treatment without N-removal (activated sludge process).	Stabilised and dewatered sludge	Thermal hydrolysis (Cambi process; 160°C) and mesophilic anaerobic stabilisation over 24 days	Agriculture; primarily grain
IVAR Sentralrenseanlegg Nord-Jæren (Stavanger)	IVAR	1	Secondary treatment; chemical precipitation.	Stabilised and dried sludge	Mesophilic (38-40°C) anaerobic stabilisation over 35 days, thermal drying and pelletisation	Compost, some for grain production
Tomasfjord (Tromsø)	TOM	1	Primary treatment; fine screen (350 µm Salsnesfilter and Masko Zoll on the sludge reject water).	Dewatered raw sludge	Dewatered sludge is sent to Stormoen in Balsfjord municipality for composting	-
FREVAR/ØRA (Frederikstand)	FREVAR	1	Secondary treatment; chemical precipitation.	Stabilised and dewatered sludge	Pasteurisation and thermal (60°C) anaerobic stabilisation over 12-15 days	-
Tønsberg renseanlegg (Tønsberg)	TAU	1	Secondary treatment; chemical precipitation combined with biological purification	Dewatered sludge (before lime stabilisation)	Lime stabilisation (slaked) after dewatering (the Orsa method)	-
Linnes renseanlegg (Lier)	LINNES	1	Primary treatment; fine screen	Dewatered raw sludge	Sends sewage sludge to Lindum Resource & Recycling for composting	-

[a] Sampling planned to be conducted both during dry weather and in connection with a major rainfall event.

Table 2. Annual and dry weather influent loads and annual sludge productions at the WWTPs included in the study. PE = population equivalent.

WWTP	Size	Dry weather flow	Sludge production
BRA	2016: 330 000 PE Ca. 49 mill. m^3	100 000 m^3/d	2016: 21 900 tonnes (28.3% TS)
VEAS	2016: 615 000 PE Ca. 97 mill. m^3	176 000 m^3/d	2016: 38 300 tonnes (45.8% TS)
HIAS	2016: 60 000 PE 7.0 mill. m^3	15 000 m^3/d	2016: 5 710 tonnes (36.5% TS)
IVAR	2016: 330 000 PE 27 mill. m^3	78 000 m^3/d	2016: 3 810 tonnes biopellets and 400 tonnes Minorga fertiliser (85% TS)
TOM	18 500 PE 3.6 mill. m^3	9 070 m^3/d	2016: 1 266 tonnes (20-25% TS)
FREVAR	2016: 80 000 PE 13 mill. m^3	22 500 m^3/d	2015: ca. 9000 tonnes
TAU	2015: 82 500 PE 14 mill. m^3	21 250 m^3/d	2015: 14 240 tonnes (39% TS)
LINNES	2016: 18 150 PE 2.86 mill. m^3	8 400 m^3/d	6 600 tonnes (30-32% TS)

2.1.2 Sampling waste water treatment plants

Samples were collected from each WWTP by staff from the individual plants. Sampling was conducted with a metal or wooden spoon and samples were placed into pre-annealed glass jars and frozen immediately. 5 – 10 grab samples of approximately 100 g each were collected on consecutive days, where possible, over a period from three days to two weeks, depending on the plant characteristics. **Table 3** provides more details on sample collection. From the plants that included sludge stabilisation (BRA, VEAS, HIAS, IVAR, FREVAR), three samples per day were collected, while from the plants without any sludge stabilisation (LINNES, TAU, TOM), only one sample per day was collected. At the end of the sampling period all of the 9 – 10 collected samples at each plant were placed in a cooling box and sent by overnight mail to NIVA for preparation of a composite sample under controlled conditions. The samples were homogenised prior to the compositing of triplicate samples for microplastic analysis.

Due to extended sludge treatment time at the plants applying sludge stabilisation (see **Table 3**), the influent conditions representative to the time of sampling were adjusted according to the sludge age at the respective plant. Sampling was sought to be collected during periods with limited rainfall to prevent inclusion of accumulated sewer sediments in the influent. As VEAS and Bekkelaget (BRA) WWTPs receive stormwater that is expected to contain large amounts of heavily polluted road runoff, two sets of samples were collected from these plants: one that would reflect typical dry weather conditions and another that would reflect a high stormwater inflow event.

Table 3. Details of conditions during sampling at each WWTP.

WWTP	Sampling period	# grab samples	Sludge production in period		Sludge age (d)	Adjusted influent period	Influent (m^3/d)
			m^3/d	tonnes TS/d			
BRA	19.09-21.09	9	55	18.0	14	5.9-7.9	145 000
	23.10-25.10	9	74	22	11-14	11.10-13.10	171 000
VEAS	10.10-12.10	9	105	48	23	17.9-19.9	249 000
	26.10-30.10	9	105	48	23	3.10-7.10	281 000
HIAS	16.10-20.10	9	58	20.5	27	19.9-23.9	23 700
IVAR	06.10-16.10	9	2	21.2	35	1.9-11.9	135 000
TOM	11.10-24.10	10	4.1	1.04	-	11.10-24.10	9 500
FREVAR	09.10-11.10	9	286	86	15	24.9-26.9	45 600
TAU	09.10-18.10	10	61	17.4	-	9.10-18.10	39 600
LINNES	30.10-03.11	10	8.5	2.4	-	30.10-3.11	7 900

2.1.3 Dry weight and organic content of sludge samples

Each sample was characterized in terms of dry weight (loss of water; heating at 105 °C until constant weight) and organic content (loss-on-ignition; heating at 550 °C until constant weight). These measurements were carried out by Eurofins.

2.2 Microplastic analysis

2.2.1 Methods development

Microplastic analysis generally describes the quantification and characterisation of microplastic particles from a sample. However, when this sample is derived from a complex environmental matrix, initial sample processing is required. Methods that are currently used to analyse sewage sludge involve very little processing and often rely on dilution, filtering or sieving [10, 19]. Sludge is largely composed of organic material as well as fine clay particles. Using these methods, most of this material will also be retained during the filtering/sieving stage, severely inhibiting the ability to accurately analyse for microplastic contamination. To overcome this issue, some studies have begun to incorporate a density separation step to isolate less dense particles from sediments using salt solutions [5, 16, 20]. This reduces the volume retained during filtering but is not effective at limiting particulate organic material which has a similar density (approximately 1.4 g cm^{-3}) to the target microplastics (approximately 0.9 – 1.8 g cm^{-3}). Hence, a further organic matter removal step is required. Thus far, this has been applied to sludge in two studies which utilize an alkaline treatment [21] or peroxide digestion [17]. This helps to drastically reduce the non-plastic particulate matter retained during filtering, and significantly reduces the time required to produce accurate microplastic data. However, thus far, the methods to remove organic material have not yet been tested or optimized for application to sludge samples. In this study, NIVA aimed to establish the optimum protocol for reducing sample volume whilst preserving polymeric particles.

It is important to note that some more advanced techniques to analyse microplastics in complex, organic-rich environmental samples have been proposed. These include thermal decomposition [22, 23] and pressurized liquid extraction [6]. However, they require specialized equipment and are more cost- and time-intensive to operate. Hence, they are not yet appropriate for use in environmental monitoring programmes. Furthermore, these methods involve the destruction of microplastic particles into a liquid or gas extract which eliminates important particle information related to the number of particles, their shape (fibre, microbead, fragment *etc.*) and their size, which can inform about sources and behaviour/fate in the environment. Enzymatic digestion of organic material has also been tested for complex environmental samples [e.g. 21]. However, due to the composition of organic material found within sewage sludge, the number of treatments that would be required will be high. Enzyme treatments are expensive, and each require a long incubation period. Hence, the cost of analysing samples is significantly increased. This method, therefore, is currently unsuitable for use in monitoring programmes. For these reasons, the above methods were not included in this study.

Method testing

The methods testing procedure was a two-step process:

1. Firstly, the effect of each protocol on virgin plastic pellets was tested to check for any degradation during digestion. This was important to ensure that the selected protocol would not have any effect on microplastics in real environmental samples.

2. Secondly, each protocol was applied to test sludge samples to assess the efficacy of the organic matter removal step.

Four protocols for the removal of organic material from sludge samples were tested. These have been adapted from existing studies which attempt organic matter reduction from sludge, soil, wastewater, and biota. The protocols are detailed in **Table 4**. Protocol 1 is a peroxide digestion, which aims to oxidise organic material. This was originally proposed for use with sewage sludge by Sujathan *et al.* [17]; however, the suggested temperature of 70 °C is higher than the continuous operating temperatures for some polymers. For this reason, this protocol was tested at two temperatures: 70 °C (Protocol 1a) and 60 °C (Protocol 1b). Tagg *et al.* [24] suggested a modification of peroxide digestion for use with wastewater samples. This utilises an iron catalyst and is referred to as Fenton's reagent. Protocol 3 and 4 are both adapted from studies which digest biotic material for microplastic analysis. They are both alkaline treatments. Two concentrations are commonly reported in the literature for NaOH (Protocol 3a and 3b): 1 M and 10 M. Both concentrations were tested to assess potential effects to microplastic particles and the amount of organic material that was removed.

Eight different polymers were tested during the methods testing procedure. Together these polymers account for >70 % of global plastic demand [25]. The tested polymers are listed in **Table 5** with a brief description of their properties and applications. They comprise polymers that are likely to be found in WWTP sludge including polyethylene (PE), which accounts for 93 % of microbeads from personal care products, and polyethylene terephthalate (PET), nylon (PA-66), and polymethyl methacrylate (PMMA), which make up a large proportion of synthetic fibres used in garments and textiles. Large (1 – 5 mm) virgin microplastic beads and pellets were used to permit accurate mass and size testing. The mass, size (a and b axis), FT-IR spectra and visual appearance of the polymers were recorded before and after treatment. Three beads/pellets were analysed per replicate and three replicates were run per treatment. Virgin microplastics were obtained from the JPI-Oceans BASEMAN project.

The two alkaline treatments: NaOH (Protocol 3) and KOH (Protocol 4) did not remove a sufficient proportion of the organic material present in the test sludge samples. Furthermore, there was evidence of degradation of some of the treated polymers including the breakdown of polyethylene terephthalate (PET) and polycarbonate (PC) (NaOH) and changes to the mass of polystyrene (PS) and PC (KOH). For these reasons, KOH and NaOH (Protocols 3 & 4) were disregarded as potential reagents for the extraction of microplastic from sludge samples.

The peroxide-based treatments (Protocol 1a, 1b & 2) were significantly more effective in removing organic material from the test sludge samples. Both polypropylene (PP) and PA-66 showed signs of degradation when heated to 70 °C. Additionally, PS exhibited visual changes indicative of minor degradation in both heated peroxide treatments (60 °C and 70 °C (Protocol 1a & 1b)). The Fenton's reagent treatment (Protocol 2) showed no signs of degradation for any of the tested polymers. The treatment was also successful in reducing the proportion of organic material associated with the sludge samples. This protocol requires the shortest reaction times and the least laboratory infrastructure (*e.g.,* incubators). Hence, it represents a cost- and time-effective method for extracting microplastics from sludge.

Table 4. Protocols tested for organic matter removal in test sludge samples.

Protocol		**Solution**	**Temperature**	**Exposure time**	**Adapted from**
Protocol 1	H_2O_2	30 % H_2O_2	70 °C (1a) 60 °C (1b)	6 h 6 h	[17]
Protocol 2	Fenton's reagent	30 % H_2O_2 with Fe^{2+}	Ambient	2 h	[24]
Protocol 3	NaOH	1 M NaOH (3a) 10 M NaOH (3b)	60 °C	24 h	[21, 26]
Protocol 4	KOH	10 % KOH	60 °C	24 h	[27, 28]

Table 5. Eight polymers used in the method testing procedure including details about their characteristics and usage.

Polymer		% Global plastic demand[a]	Density (g/cm^3)	Applications
Polypropylene	PP	23 %	0.82 - 0.90	Packaging, food containers, textiles (carpet, rope), reusable containers
Polyethylene (low and high density)	LDPE	17 %	0.92 - 0.93	Plastic bags, plastic bottles, packaging, plastic film, microbeads in personal care products
	HDPE	15 %	0.94 - 0.97	
Polystyrene	PS	7 %	1.05 - 1.06	Food containers, packaging
Polyethylene terephthalate	PET	7 %	1.31 - 1.43	Plastic bottles, plastic containers, synthetic fibres (polyester)
Polyamide	PA-66	1 %	1.13 - 1.15	Synthetic fibres (nylon), plastic film
Polycarbonate	PC	1 %	1.20 - 1.22	Plastic bottles, synthetic glass
Poly(methyl) methacrylate	PMMA	<1%	1.16 - 1.20	Synthetic glass

[a] PlasticsEurope [25]

2.2.2 Microplastic extraction

Microplastics were extracted from sludge from eight WWTPs around Norway. Sludge samples were thoroughly mixed before sub-sampling and triplicate sub-samples were taken to account for within-sample variability. 10 g of sludge (w.w.) was used for each sub-sample. Samples were placed into clean, pre-washed containers. 20 ml of 30 % (v/v) H_2O_2 and 10 ml of ferrous sulfate catalyst solution was added to the sludge samples. The reaction took approximately two hours to complete at room temperature. This process removed most of the organic material.

Following organic matter removal, microplastics were extracted through a density separation procedure. Microplastics were extracted using two density solutions: 1 g cm^{-3} and high density 1.8 g cm^{-3}. Firstly, microplastics were extracted at 1 g cm^{-3} to identify the proportion of microplastics that float in freshwater systems, hereafter referred to as low density. This was performed by filling the sample containers to the brim with filtered RO water and agitating the sample to bring low density particles into suspension. Once the solids had settled out, the overlying water was decanted, and vacuum filtered through Whatman GF/D filter papers. This process was then repeated with a high-density saturated NaI salt solution (density: 1.8 g cm^{-3}), to extract microplastics that sink in freshwater environments, hereafter referred to as high density. Filter papers were oven dried at 40 °C and analysed for microplastic particles. Particle density is determined by polymer density; however, microplastics may become biofouled which can alter particle buoyancy. By using two density extracts, it is possible to estimate the potential fate of microplastics in the environment.

2.2.3 Visual identification, characterisation and classification

Microplastic analysis was initially performed using a visual identification step. Filters papers were traversed at 30 x resolution using a stereomicroscope. Suspected microplastic particles were identified using a rigorous visual protocol and further testing using a hot needle point. The identification was performed by experienced researchers at NIVA and followed the European Guidelines for Monitoring Microplastics [29]. 25 % of samples were tested by another researcher in a dual-ID process to ensure consistency. The organic matter removal process significantly reduces the occurrence of non-synthetic particles in the final samples. However, all particles were still checked for absence of cellular or organic structures as well as homogeneous thickness, colour and shine. A lower size limit of 50 µm was imposed; no upper size limit was used.

The manual identification of microplastic particles was supported by further chemical characterisation using an infrared scanner; Fourier Transform Infrared Spectroscopy (FT-IR). This technique analyses the chemical composition of microplastic particles and reports the associated polymer type. Each scan produces a ‘fingerprint’, which was compared to a reference database available at NIVA. This database has been built through the JPI-Oceans BASEMAN project.

FT-IR analysis was performed for a subsample of 10 % of the identified particles to verify the visual identification step and to provide some information about the different polymers found in sewage sludge. This subsample size is recommended by the EU.

Each microplastic particle was further characterised based on its morphological properties: size, shape, and colour. Size is defined as the longest dimension of each particle. Shape is subdivided into three main categories: fragments (irregular shaped particles or films), fibres (microfibres or threads), beads (microbeads or spherical grains). In addition, glitter was identified as a hexagonal shaped plastic disc where, in some cases, remanence of foil and often iridescence was observed. All glitter particles were confirmed as plastic using FT-IR.

2.2.4 Quantification and reporting

Microplastic abundance in sewage sludge has been quantified in relation to sludge mass. This was performed for wet and dry weight. Results are primarily reported as particles kg^{-1}. Additionally, results are reported volumetrically as particles m^{-3} of sludge, to be consistent with volumetric reports provided by WWTPs. It was not possible, within the confines of this study, to establish true microplastics concentrations (mg kg^{-1}), due to the very low weight of microplastic particles. Current approaches to estimate mass based on density are not appropriate for use with fragments or fibres due to their irregular shape. The results have been converted to correspond with the population size associated which each treatment facility. Finally, the microplastic abundances have been further characterised by different size fractions and density (whether particles float or sink in freshwater).

2.3 Contamination control

Laboratory environments can be a source of microplastic particles which can overestimate analytical results. Several steps were taken to reduce and quantify this contamination to produce accurate outputs:

1. All sample containers were pre-washed with filtered distilled water before use.

2. Samples were kept covered as much as possible using aluminium foil or glass lids.

3. All equipment used in the processing and analysis stages were rinsed and checked under a microscope for any microplastic particles adhering to them. The vacuum filtering apparatus was rinsed with filtered water between each sample.

4. All reagents were vacuum filtered through Whatman GF/D filter papers immediately prior to use.

5. Sample processing was performed in a sterile cabinet.

6. Several procedural blanks were performed as negative control samples through the sample processing and analytical stages in order to test for laboratory contamination.

3 Results

Microplastics were found in all ten sludge samples investigated from eight WWTPs. The overall average plastic abundance was 6 077 particles kg^{-1} (d.w.) (1701 – 19 837) or 1 176 889 particles m^{-3} (470 270 – 3 394 274). Particles from sludge consisted of beads (37.6 %), fragments (31.8 %) fibres (28.9 %) and glitter (1.7 %). Most of the particles were clear in colour (41 %). Ten percent of the overall particles extracted were tested using FT-IR (n=60). All particles tested were confirmed to be plastic. Polyethylene particles were the most common (30.5 %) followed by polyethylene terephthalate (26.7 %) and polypropylene (20.3 %). 62 % of plastics were extracted during the low density (1 g cm^{-3}) separation steps and 38 % were extracted at high density (1.8 g cm^{-3}).

3.1 WWTP conditions during sampling

The daily average influent flow rates to the WWTPs that correspond to the respective sludge sampling periods, is shown in **Table 6.** It was sought to collect samples primarily during dry weather conditions, but as is evident from the ratio of dry weather influent, precipitation appeared to significantly influence the influent flow rates during most of the collection periods. Only the influent to Linnes and Tomasfjord (TOM) appeared to receive limited influence from stormwater runoff. Almost no precipitation was registered during sampling at Linnes, although this WWTP is not connected to a combined sewer system. The antecedent conditions in Tromsø were very dry (see **Appendix A**); however, a heavy storm event occurred during the sampling period at Tomasfjord (TOM). Due to the unusually dry conditions, it is possible that a large proportion of the rainwater may have been absorbed by soil and vegetation in the catchment area.

3.1.1 Sludge composition

Dry solids in the 10 sludge samples ranged from 25.5 % (TOM) to 87.3 % (IVAR) (**Table 7**). Tomasfjord (TOM) do not treat sludge onsite and so this value represents the moisture content of raw sewage sludge. In contrast, sludge at IVAR is dewatered and lime stabilised, producing a much drier final sludge. Organic content ranged from 38.8 – 38.9 % at VEAS up to 86.6 % for the raw sludge sampled at TOM. Mean organic content across the eight WWTPs (10 samples) was 56 %.

The volume of sludge produced per day ranged from 4.08 m^3 (TOM) to 286 m^3 (FREVAR). In regard to the volume of dry solids that this corresponds with, 1.04 m^3 d^{-1} was produced at TOM and 85.9 m^3 d^{-1} was produced at FREVAR. On average, across all the WWTPs, 26.9 m^{-3} of sludge (dry solids) was produced per day.

Table 6. Influent and rainfall during the sampling periods. More precipitation data are available in Appendix A.

WWTP	Daily average influent in period ($m^3 d^{-1}$)	Precipitation during sampling (mm)	Ratio of dry weather influent (%)	PE-specific Influent (l/PE*d)
BRA I	144 833	38.4	145	439
BRA II	170 817	23.8	171	518
FREVAR	45 552	1.0	203	569
HIAS	23 746	7.7	158	396
IVAR	134 640	78.3	173	408
LINNES	7 907	0.3	94	436
TAU	39 598	35.9	186	480
TOM	9 519	45.7	105	515
VEAS I	249 466	2.4	142	406
VEAS II	281 030	16.9	160	457

Table 7. Sludge characteristics and production at the WWTPs.

Site	Dry solids (%)	Organic content (%)	Sludge production	
			m^3 (w.w.) d^{-1}	m^3 (dry solids) d^{-1}
BRA I	33.0	49.3	54.7	18.0
BRA II	29.2	54.8	74.0	21.6
FREVAR	30.0	51.0	286	85.9
HIAS	35.2	59.3	58.2	20.5
IVAR	87.3	66.6	6.9	6.0
LINNES	28.3	69.5	8.5	2.4
TAU	28.7	49.4	60.6	17.4
TOM	25.5	86.6	4.08	1.04
VEAS I	44.8	38.9	105	48.1
VEAS II	40.7	38.8	105	48.1

3.2 Microplastic particle counts in sludge samples

Results of plastics in sludge samples are divided into sections. First quantitative results of plastics in samples are presented followed by classification by shape, size, buoyancy, colour and polymer identification.

3.2.1 Correction of microplastic counts

Airborne contamination was considered minimal due to routine monitoring in the laboratory. Procedural blanks were carried out during the digestion and extraction phase to monitor for contamination in the sampling procedure. These procedural blanks were processed in the sample way as sludge samples. Procedural contamination was accounted for based on particle shape, size and colour to reduce the bias. Only one set of blanks during the entire process contained suspected contamination therefore the corresponding sample results were corrected accordingly.

3.2.2 Abundance of microplastic in sludge

Plastics were found in all 10 sludge samples investigated from eight WWTPs, representing an average number of microplastic particles of 1 946 particles kg^{-1} (w.w.) (range: 464 – 5 792) or 1 176 889 particles m^{-3} (range: 470 270 – 3 394 274). The counts were standardised for differences in moisture content across the 10 samples, where the overall average was 6 077 particles kg^{-1} (d.w.) (range: 1 701 – 19 837). Abundances reported by weight are shown in **Figure 3** and details on microplastics particles by volume are given in **Table 8**.

The number of plastic particles within sludge samples vary across the different WWTPs (**Figure 3**). This may be related to the period of sampling (temporal resolution), sludge composition, or the differences in water and sludge treatment. The second sampling at BRA (BRA II) had the highest particle count within the sludge. High values (d.w.) were also observed for FREVAR, LINNES, and TOM. The difference between wet and dry weight results indicate the importance of standardising different moisture contents in the final sludge to produce comparable results.

Using details provided by the WWTPs, the abundance of microplastic particles in sewage sludge has been used to estimate the number of microplastics that are captured and added to sludge in a 24-hour period (**Table 8**). On average, 181 679 012 microplastic particles (50 – 5 000 µm) are captured and incorporated into sewage sludge per day. The highest value is associated with the second sampling period at BRA where over 750 000 000 microplastic particles are estimated to have been captured per 24 hours. The population equivalent can be used to assess the average number of microplastics per person that are released into and captured within sewage sludge in Norway. On average, 1 316 particles are associated with each individual per 24 hours. When extrapolated up to the national population, approximately 6.8 billion microplastics are captured and incorporated into sewage sludge per day.

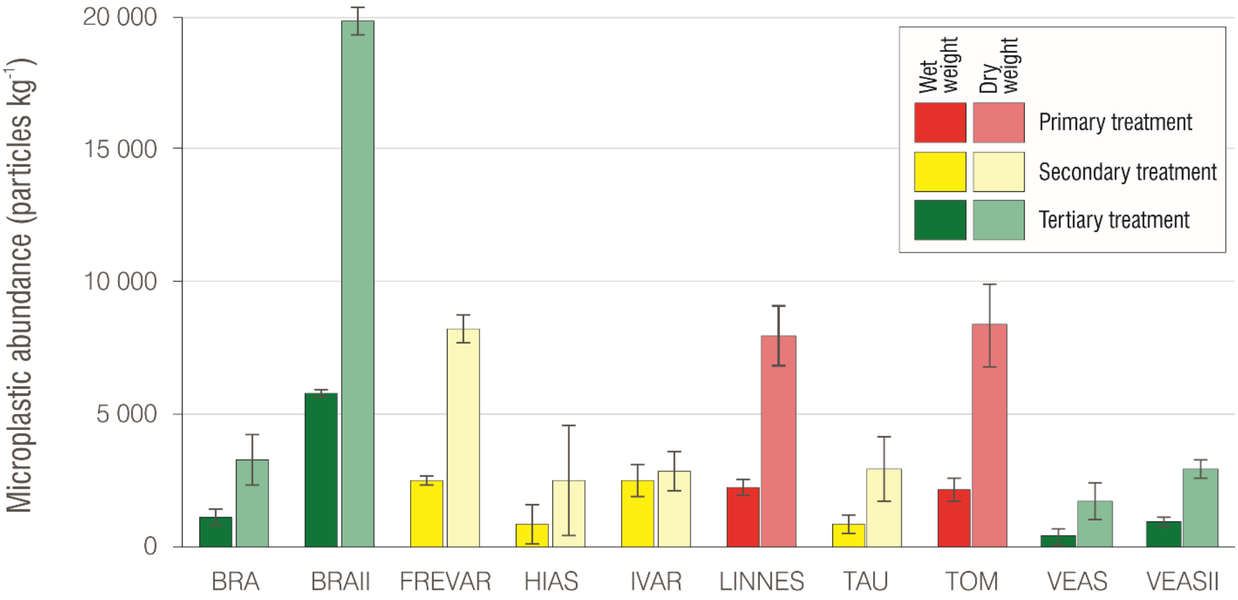

Figure 3. Microplastic abundances extracted from the ten sludge samples across eight sample locations. Abundances reported as particles per kg: wet weight (w.w) and dry weight (d.w).

Table 8. Microplastic abundances (particles m^{-3}), estimations for the number of microplastics captured in the sludge phase for each WWTP per day and the approximate number of microplastic per person that are incorporated into the finished sludge**.**

	Microplastic abundance (m^{-3})	**MPs 24h^{-1}**	**MPs 24h^{-1} person^{-1}**
BRA I	553 478	90 770 316	275
BRA II	3 394 274	753 528 895	2 283
FREVAR	2 173 985	622 484 318	7 781
HIAS	470 270	9 123 243	152
IVAR	762 246	152 449 184	462
LINNES	1 239 654	22 313 772	1 229
TAU	643 730	39 267 524	476
TOM	1 376 620	5 616 609	304
VEAS I	492 150	51 675 720	84
VEAS II	662 481	69 560 538	113

3.2.3 Influence of treatment type, size of plant and weather conditions on microplastic abundance

The highest microplastic count was associated with BRA WWTP which has tertiary treatment of wastewater. LINNES and TOM are primary treatment plants and both exhibit relatively high abundances of microplastic particles within the sludge; however, they are not significantly different from the count observed in sludge from FREVAR, a secondary treatment facility. Additionally, other secondary treatment facilities (HIAS, IVAR and TAU) are similar to BRA I and VEAS I, the two tertiary treatment plants investigated in this study.

Further, treatment of sludge does not appear to be the dominant control on microplastic abundance in sludge. LINNES and TOM WWTPs do not treat sludge onsite, where the samples tested here represent raw sewage sludge. Counts are higher than observed for other WWTPs; however, the microplastic abundance identified for BRA II was significantly higher. There is no significant difference between the sludge samples treated by anaerobic digestion (BRA, VEAS, HIAS, IVAR and FREVAR) compared to that treated by lime stabilisation (TAU). Similarly, there is no significant difference between thermophilic (BRA, IVAR) and mesophilic (HIAS, IVAR) anaerobic digestion. FREVAR, which stabilises sludge using thermal anaerobic digestion does exhibit a significantly higher microplastic count within the sludge; however, it is not possible to draw a statistically significant result from this single site.

There is no significant correlation between the population equivalent of the WWTP (**Table 2**) and the abundance of microplastics in the sludge (**Figure 3**) (Spearman's; $p = 0.302$). There is also no significant correlation between the abundance of microplastics and the dry weather flow ($p = 0.481$), influent volume ($p = 0.579$) or sludge production ($p = 0.589$) during the associated sampling period.

There is no correlation between microplastic abundance and precipitation (Spearman's; $p = 0.987$) or the ratio of dry weather flow ($p = 0.987$) across the 10 samples. Sampling was conducted twice at BRA and VEAS with the intention of capturing sludge produced under wet and dry conditions. However, this was not achieved (**Figure 3**). Despite this, the two sampling periods demonstrate significantly different particle counts (**Figure 3**). In particular, BRA II samples had a very high microplastic abundance.

3.3 Microplastic particle characteristics

3.3.1 Characterisation by shape

Microplastics were characterised by shape during the visual identification procedure. Beads were the most common form of plastics identified (37.6 %) followed by fragments (31.8 %), fibres (28.9 %) and glitter (1.7 %). Examples of some of the particles extracted are presented in **Figure 4**.

Figure 5 demonstrates the high variability between WWTPs based on microplastic abundance and shape characteristics. Microplastics within the sludge from TOM, IVAR, VEAS I and BRA II were dominated by microbeads. TAU and LINNES presented a high proportion of microplastic fibres, whilst fragments represented the majority component of microplastic contamination in FREVAR and BRA I sludge samples. All sites were contaminated by fibres and fragments, whereas microbeads were only absent in sludge samples from LINNES. Glitter – which is currently being considered in the ban on microplastics in personal care and cosmetic products in some countries – was identified in five of the samples: VEAS I, BRA II, FREVAR, HIAS, and IVAR (*e.g.,* **Figure 4d**). Notably, only two particles that resembled car tyre wear were identified in the sludge samples: one particle in BRA II and one particle in FREVAR.

The composition of microplastic shapes in the dual sampling that was conducted at BRA and VEAS shows a significant difference (**Figure 5**). At BRA, the number of microbeads was notably reduced, and the proportion of fibres and fragments increased in the second sampling period (BRA II). At VEAS, there was an increase in microbead contamination and a significant reduction in the number of fragments observed in the second sludge sample.

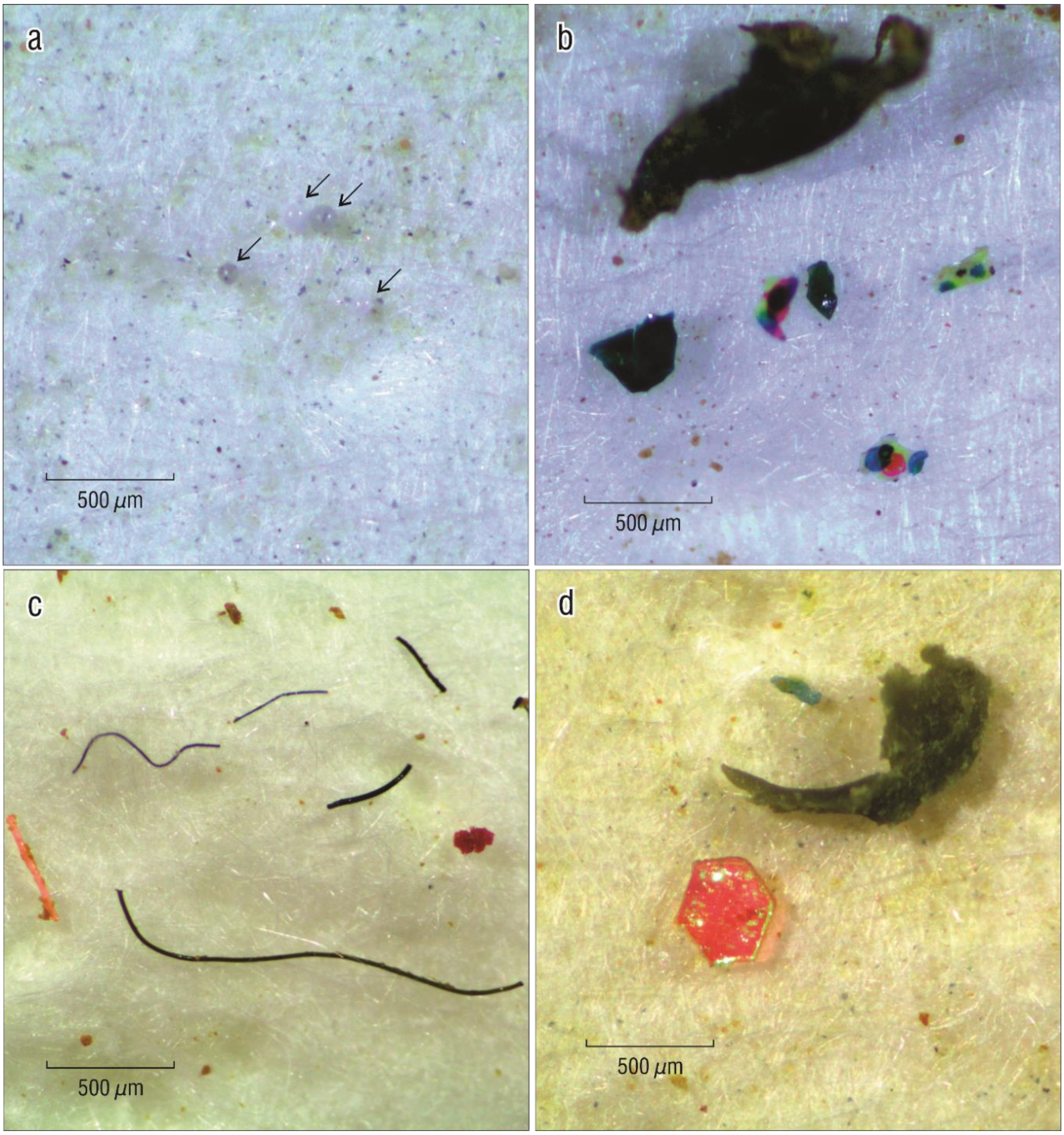

Figure 4. Examples of plastic particles extracted from sludge samples: microbeads (a), fragments (b), fibres (c) and glitter (d).

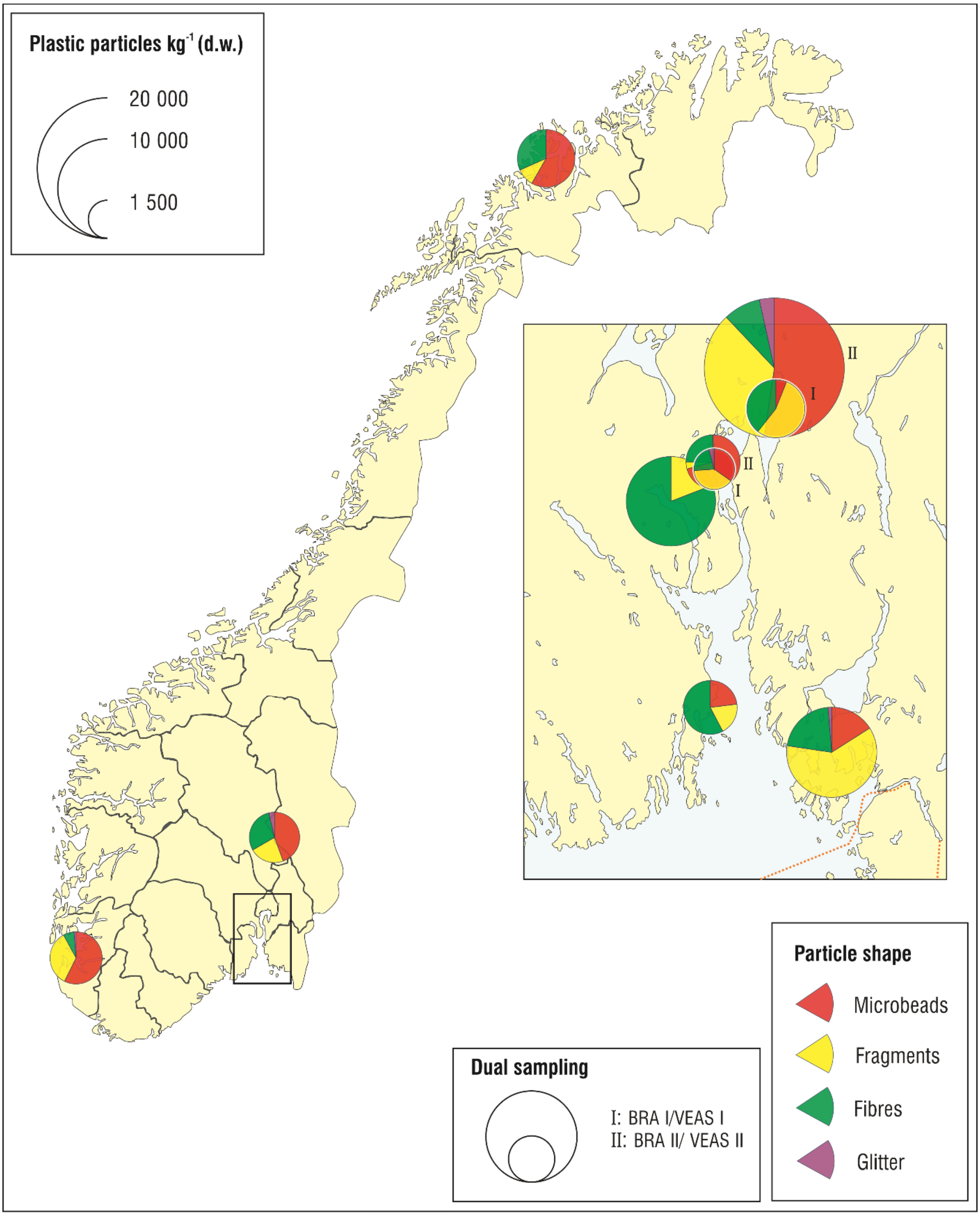

Figure 5. Map of microplastic abundance (particles kg^{-1} d.w.) in sludge samples from eight WWTP across Norway including breakdown by *particle shape*. Counts are represented as area-proportional circles. Dual sampling was performed at BRA and VEAS and is represented by overlain circles.

3.3.2 Characterisation by size

Sizes of plastics ranged from 54 µm (50 µm detection limit) to 4 987 µm with an average size of 644 µm. Based on the current convention for the classification of plastics, 81 % were small microplastics (<1 mm) and 19 % were large microplastics (1 – 5 mm) (**Figure 6**). 34 % of plastic particles were identified in the smallest size class analysed: 50 – 125 µm. It is important to note that due to size limitations in FT-IR analysis, none of these particles were chemically characterised or confirmed to be plastic. However, 97 % of these small particles were identified as microbeads, 98 % of which were extracted at 1 g cm^{-3}. The spherical shape and very low density of these particles points towards potential plastic composition.

Table 9 provides a breakdown of average particle size by shape and by sample. BRA II had the lowest mean particle size (399 µm) which is related to the large proportion of small microbeads identified. TAU presented the highest overall mean particle size (1 283 µm), linked to the higher percentage of fibres. Average microbead diameter ranged from 78.5 µm (IVAR) to 172 µm (HIAS). Mean fragment size was between 310 and 1 625 µm. Related to the long axis measurement, fibres were typically longer with mean lengths ranging from 790 µm to 2 025 µm. Glitter sizes were less variable, falling in the range 261 to 509 µm.

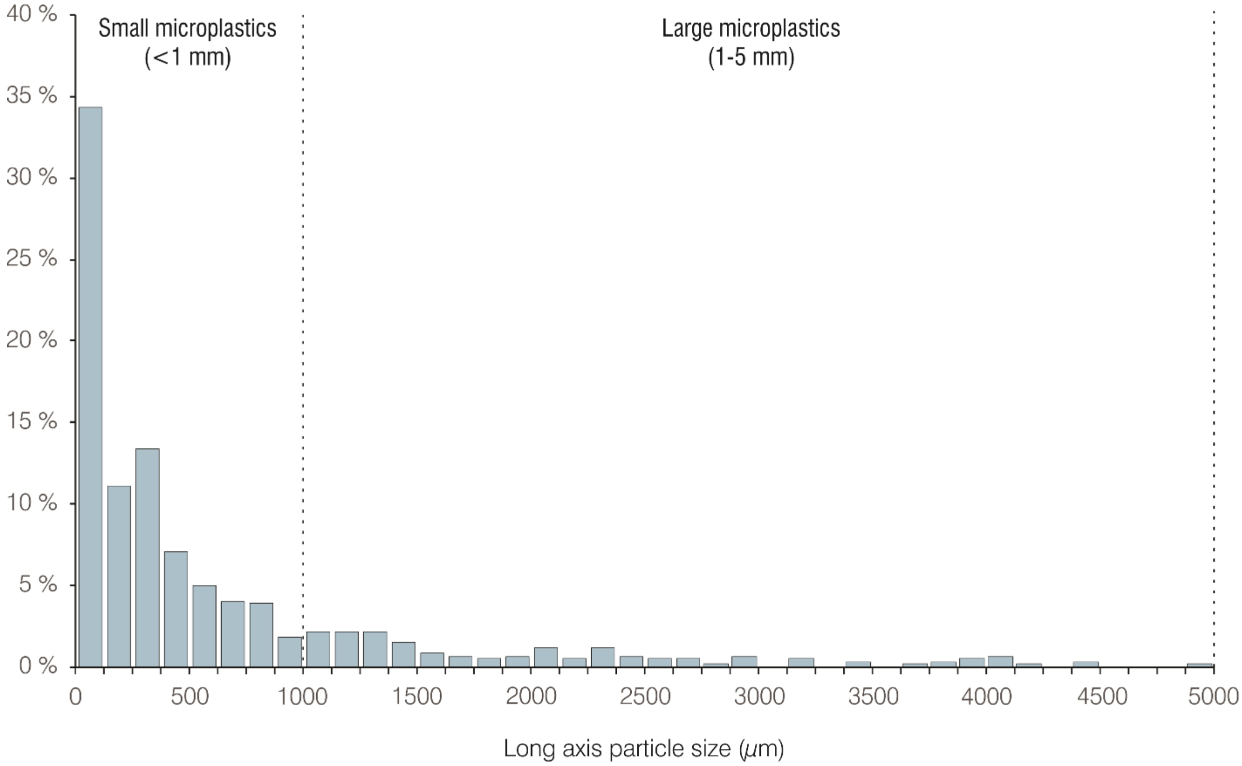

Figure 6. Size distribution of microplastic particles extracted from eight WWTPs across Norway.

Table 9. Size characterisation of particles extracted from sludge samples. Numbers in brackets represent the number of particles (*n)*. Values are reported in µm and refer to the longest axis.

	Average particle size				
	Beads	**Fragments**	**Fibres**	**Glitter**	**TOTAL**
BRA I	129 *(2)*	405 *(18)*	2025 *(13)*	*(0)*	1026 *(33)*
BRA II	94.1 *(91)*	422 *(62)*	1621 *(22)*	295 *(6)*	399 *(181)*
FREVAR/ØRA	78.5 *(12)*	922 *(46)*	1099 *(16)*	261 *(1)*	816 *(75)*
HIAS	172 *(13)*	777 *(6)*	2024 *(8)*	286 *(1)*	835 *(28)*
IVAR	82.4 *(43)*	1625 *(16)*	1025 *(7)*	313 *(1)*	708 *(77)*
LINNES	*(0)*	491 *(13)*	937 *(59)*	*(0)*	857 *(72)*
TAU	92.7 *(6)*	714 *(5)*	1950 *(15)*	*(0)*	1283 *(26)*
TOM	113 *(35)*	311 *(6)*	1560 *(23)*	*(0)*	652 *(64)*
VEAS I	132 *(8)*	806 *(5)*	790 *(5)*	509 *(1)*	502 *(19)*
VEAS II	105 *(17)*	310 *(5)*	1393 *(6)*	*(0)*	418 *(28)*

3.3.3 Characterisation by buoyancy

Across the WWTPs, 62 % of microplastic particles were extracted using the low-density solution, where 38 % were separated out using the high-density solution. Overall, 98 % of microbeads were associated with freshwater density extraction, and 74 % of fibres were extracted using the high-density solution. Fragments were evenly split across the density extracts, whilst all glitter pieces were associated with the high-density separation.

Figure 7 shows the different density profiles of extracted microplastics across the eight WWTPs. The majority of microplastics particles identified within TOM, IVAR, HIAS, BRA (wet) and both VEAS samples were <1 g cm^{-3}. Conversely, the majority of particles at FREVAR, TAU, LINNES and BRA (dry) were extracted at high density.

Only a small shift in the density profiles is observed between the two VEAS samples. In contrast, the two BRA samples show strong disparity. The low-density extract increases from 6 % to 82 % between the two sampling periods.

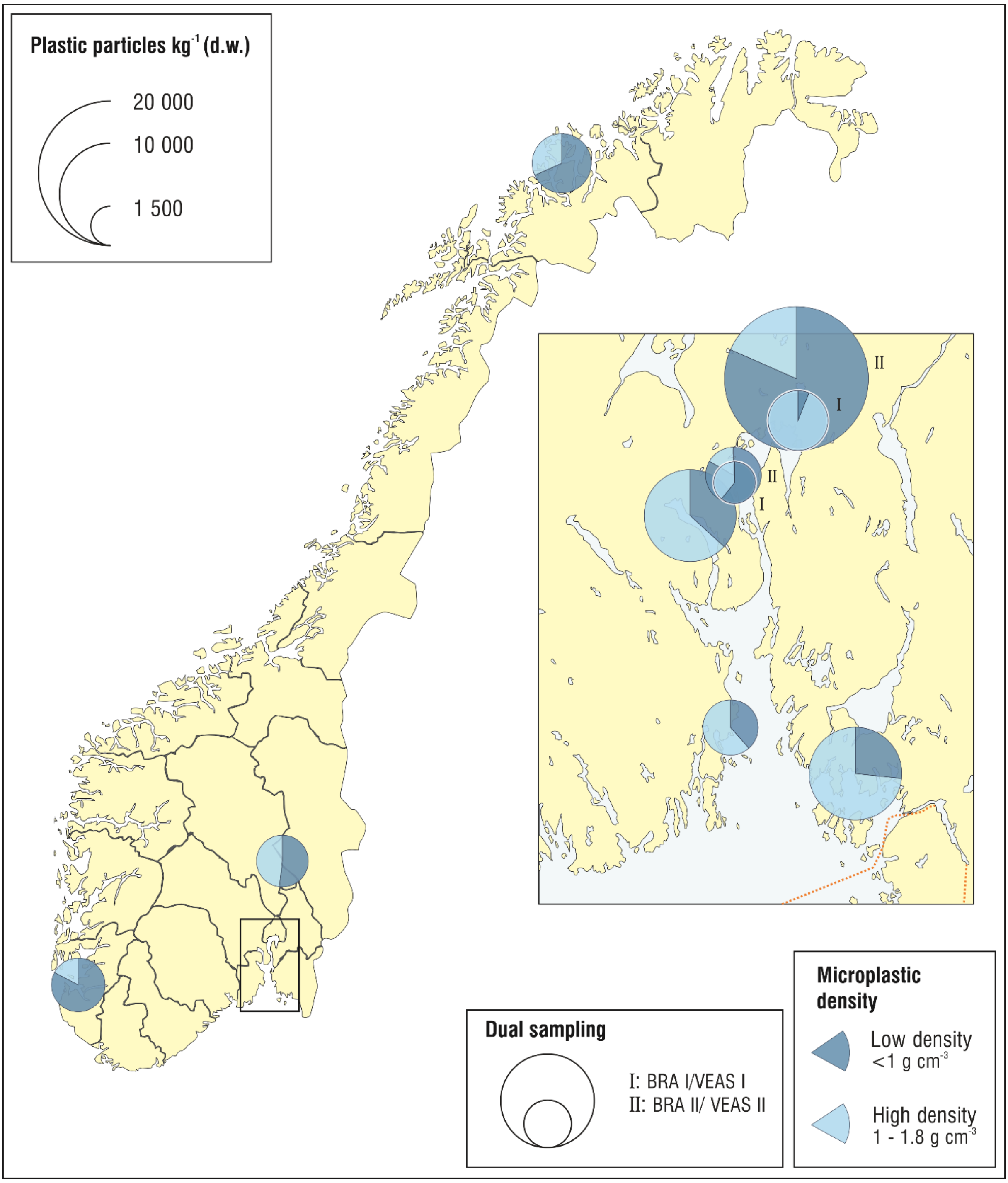

Figure 7. Map of microplastic abundance (particles kg^{-1} d.w.) in sludge samples from 8 WWTP across Norway including breakdown by *extraction density*. Values are represented as area-proportional circles. Dual sampling was performed at BRA and VEAS and is represented by overlain circles.

3.3.4 Characterisation by colour

The breakdown of particle colours is shown in **Table 10**. Clear particles were the most prevalent in colour (41 %). This large percentage was related to a high quantity of microbeads in the sludge samples: 84 % of microbeads were transparent in colour. As colour is a subjective identification parameter, results of particle colour are provided to give an impression of the variability in microplastic particles. There is no clear trend associated with colour across any of the sludge samples, with each sample composed of a variety of microplastic colours.

Table 10. Colours of plastic particles extracted sludge samples from eight waste water treatment plants around Norway. The distribution of colours is also broken down by microplastic shape.

	Overall %	**Bead**	**Fragment**	**Fibre**	**Glitter**
Clear	41 %	77 %	16 %	5 %	2 %
Black	13 %	0 %	15 %	85 %	0 %
Blue	13 %	1 %	41 %	58 %	0 %
Pink	6 %	0 %	53 %	39 %	8 %
White	6 %	92 %	8 %	0 %	0 %
Green	5 %	0 %	78 %	16 %	6 %
Grey	4 %	0 %	73 %	27 %	0 %
Turquoise	4 %	0 %	73 %	27 %	0 %
Multi-coloured	3 %	0 %	100 %	0 %	0 %
Red	1 %	0 %	33 %	67 %	0 %
Orange	1 %	0 %	43 %	57 %	0 %
Yellow	1 %	0 %	14 %	71 %	14 %

3.3.5 Characterisation by polymer

A subsample of representative particles extracted from all sites were subjected to chemical identification by FT-IR analysis. FT-IR was used to confirm the identity of plastic polymers by matching their IR spectra to a polymer library. A total of 60 particles were subjected to FT-IR analysis. This represents 10 % of the total particles that were identified visually. Many of the microbeads and fibres were too small for analysis using standard FT-IR. Hence, the results are obtained from larger particle sizes (>150 µm). All particles had a >80 % match to reference spectra. All tested particles were confirmed to be plastic. Details of the particle analysed by FT-IR is provided in Appendix B.

Many microbeads were too small to be analysed using standard FTIR. However, 98 % were extracted at freshwater density. This indicates that these particles are likely to be polyethylene or polypropylene, which both have a density <1 g cm^{-3}. All microbeads that were large enough to be tested for polymer composition were found to be polyethylene.

A range of polymers were identified (**Figure 8**). Polyethylene was the most common (30.5 %) closely followed by PET (26.7 %) and PP (20.3 %). A small number of particles (n = 2) were found to be composed of styrene butadiene rubber, which is an important polymer used in car tyre manufacture. No significant trend related to polymer composition was observed across the eight WWTPs, where a variety of polymer types were detected in each of the sludge samples.

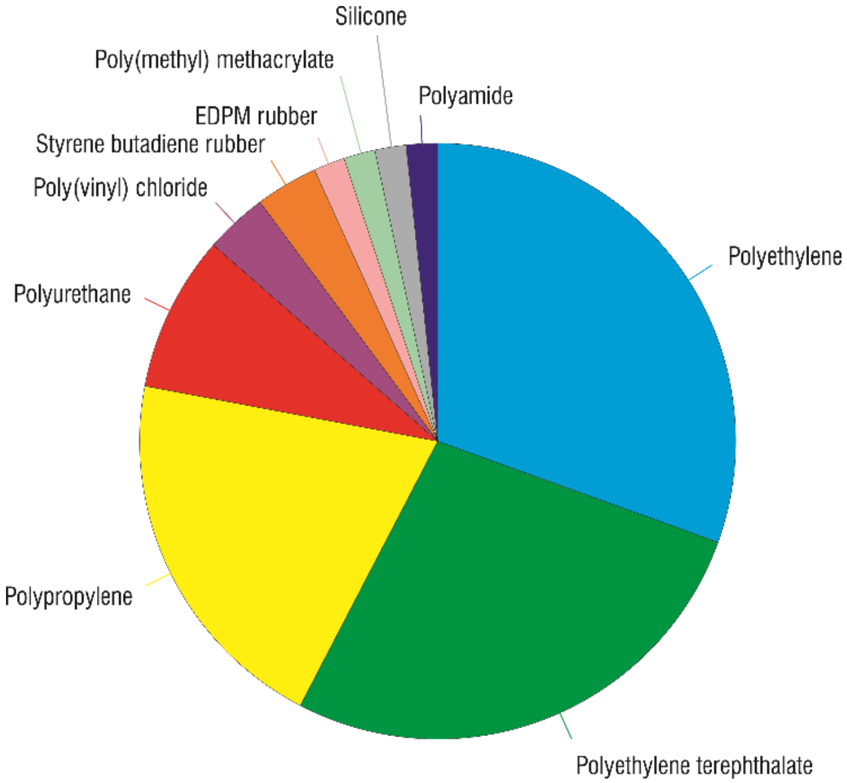

Figure 8. Polymer composition of microplastic particles tested using FT-IR analysis. A subsample of 10% (n=60) of the particles were chemically characterised by FT-IR.

4 Discussion

4.1 Uncertainties related to sampling

4.1.1 Heterogeneity of microplastic abundance in sludge samples

Several measures were taken to increase the representativeness of sludge samples analysed for microplastics. Sampling was conducted over consecutive days to account for short-term fluctuations. Sludge samples were thoroughly mixed, and composites were produced to account for potential variability. Furthermore, three replicates were analysed for each sample (10 samples from eight WWTPs). Hence, the resulting particle counts are considered to be representative of the sludge produced during the sampling period.

In general, the variability observed across the replicates analysed for each sample was relatively low. The coefficients of variance (CVs) associated with the replicates were <1 for all samples. Additionally, CVs were below 50 % for all samples except HIAS (CV = 82 %). This indicates reasonably low variability across the replicates analysed. However, as replicates were composed of composited and homogenised samples, it is not possible to establish the degree of heterogeneity in microplastic abundance in the sludge within the sample period. Instead, the sample processing was effective in accounting for potential variability.

Further work is required to establish the temporal variability of samples across various timescales. Furthermore, the influence of the sampling strategy, including the mode of extracting samples from the sludge phase, should be tested.

4.1.2 Temporal variability of microplastic abundance in sewage sludge

Sludge was extracted during two sampling periods at VEAS and Bekkelaget (BRA) WWTPs. It was intended that this would encompass dry and wet weather conditions; however, as shown in **Table 6**, stormwater inflow provided a significant influence during both periods at both WWTPs. It is notable that despite similar weather conditions, the two sampling periods both reveal significant variations at both WWTPs. The overall change is much smaller at VEAS, which may relate to the steady sludge production that is maintained at this plant (**Table 7**). Higher microplastic abundances are associated with the VEAS II sampling, which corresponded with a rainfall event. This may have increased the number of microplastics entering the plant through stormwater inflow. However, the observed change is driven by an increase in microbeads which are not typically associated with stormwater sources. Hence, the increase at VEAS II may simply reflect small temporal variability in microplastic abundance.

In contrast, there are large shifts in the breakdown of microplastics by shape and density at Bekkelaget (BRA) between sampling periods. There is a significant increase in the abundance of microbeads, which is coupled with an increase in the proportion of microplastics extracted at low density (**Figure 7**). This may reflect an influx of polyethylene microbeads during the second sampling period (BRA II). As the level of precipitation associated with both sampling periods was similar, it is possible that the changes observed reflected changing source dynamics and that this variable is important for determining sludge microplastic abundance. This may also explain the lack of significant correlations between other variables such as the size of the plant or the volume of influent. This may point towards source dynamics being the dominant control on sludge microplastic abundance and composition (shape, density *etc*.). Further work is required to characterise these dynamics and examine variability in the microplastic counts related to influent and the corresponding sludge samples.

Samples were composited to account for potential variability across small temporal scales (3 – 10 days). However, it is not possible to conclude from this study how the abundance of microplastic in sewage sludge varies over longer timescales. It has been suggested that potential sources of microplastics to WWTPs may exhibit seasonal fluctuations. For example, washing machine use has been shown to be 700 % greater during the winter than in the summer months [30]. Hence, it is likely that fibre contamination may significantly increase during cold weather periods. Furthermore, the influence of increased precipitation and resulting influent volumes may have a flushing or diluting effect on particle counts in sewage sludge by altering the volume of sludge produced. Further work is required to examine seasonal variability in microplastic abundance in sewage sludge.

4.2 Evaluation of methodologies selected to extract microplastics

4.2.1 Organic matter removal technique

One of the key barriers to establishing microplastic abundances in sewage sludge is the lack of a standardised or validated analytical technique. Several methodologies that have been put forward do not go far enough to isolate microplastic particles from solid samples. Sludge has a high organic matter content which is varied and complex in its composition. Fine clays and other mineral material may also be captured and added to the sludge in WWTPs. Due to the volume and nature of the solid substrate, simple techniques such as dilution and filtering are not sufficiently effective in reducing the amount of non-plastic material extracted alongside microplastic particles. Furthermore, a proportion of this material may have a similar density to the target microplastics, which reduces the efficacy of a density separation-based approach. When there is a large volume of non-plastic material left on filter papers, it is difficult to sift through the material and produce a reliable count of extracted plastic particles. Hence, organic matter removal techniques were tested to establish the optimum procedure for the reduction of non-plastic material in sludge samples. This was followed by a density separation procedure to isolate plastics from the higher density mineral component. As part of this testing, this study investigated the influence of different reagents on reference particles, for a variety of polymers, to ensure no degradation occurred as a result of the treatment.

The tested method proved effective at significantly reducing the volume of organic material without degrading any of the tested polymers. This included eight common polymer types which account for >70 % of global plastic demand. The extract left on filter papers was sufficiently clear to easily identify suspected microplastic particles. Furthermore, the particles themselves were cleared of potential biofouling so as to facilitate effective FT-IR analysis. The selected technique – Fenton's reagent – also represented the quickest organic matter removal technique that was tested. The chemicals required to produce the reagent are inexpensive and readily available. Hence, this process is time- and cost-effective for use within a monitoring framework.

Despite this, further testing of the technique is required to continue to validate and optimise its use. Namely, the effect of the organic matter removal stage on semi-synthetic fibres was not tested. This includes materials such as rayon and viscose which are produced from cellulosic material but treated with a polymerisation process. Whilst the significance of these particles has not yet been fully established, such as the extent of environmental contamination and their ecotoxicological implications, it is likely that WWTPs receive a high volume of these fibres from laundry effluents. Preliminary work points towards the potential biodegradability of semi-synthetic fibres such as rayon in wastewater treatment processes and sewage sludge [31]. However, the degree of polymerisation is important, and biodegradation has not been analysed under a range of treatment conditions. Many of the sludge samples had high fibre counts, but it was only possible to chemically characterise a small number of fibres using FT-IR analysis based on size limitations in that technique and the very small diameter of many fibre particles (ca. >20 µm). All of the fibres that could be tested using FT-IR were confirmed to be plastic – *i.e.,* polymers derived from petrochemicals such as polyethylene terephthalate (polyester) and polyamide (nylon). It is not possible, therefore, to establish whether the methodology used here was effective in extracting semi-synthetic fibres. Further methods testing and additional study to fully examine the threat posed by semi-synthetic fibres, their behaviour in WWTPs and their resistance to oxidative treatment is important.

Finally, alternative techniques such as thermal decomposition and pressurised fluid extraction yield more precise and accurate results. It is possible that these analytical approaches will be favoured in the future to provide more rapid and accurate analysis of microplastic contamination in sludge. However, for the purposes of this study as the first baseline assessment, it was considered of great importance to establish the *characteristics* of microplastics in sludge. Namely, the above-mentioned techniques are destructive and only produce a concentration for each polymer type. It is not possible to establish the quantity of microplastics nor their composition: size, shape or density. These characteristics are highly valuable for establishing the behaviour, fate and ecotoxicological significance of environmental microplastic particles. Hence, the method employed in this study was favoured to produce a wider range of information related to sludge microplastic contamination.

4.2.2 Using sequential density extraction

Following the organic matter removal step, a density separation process was conducted to isolate microplastics from the remaining solid material. This was performed using two density solutions in sequence to establish a density profile for the extracted microplastics: low density (<1 g cm^{-3}) and high density ($1 – 1.8$ g cm^{-3}). This can be used to estimate the proportion of microplastics that are buoyant in freshwater upon release into the environment [32].

Microplastic buoyancy is related to the density of the polymer it is composed of, as well as shape and any further additives [33]. However, once a particle has been exposed to the environment, it is possible that it will become biofouled by colonisation of microrganisms on the particle surface. This coating can influence the overall density of the particle and affect buoyancy in aquatic environments [34]. However, as organic matter removal was performed prior to density separation in this study, it is likely that a large proportion of this biofouling will have been cleared from microplastic particles. Biofouling may increase the density of a particle, reducing its buoyancy and causing it to sink in the various wastewater treatment settling steps. Hence, the microplastics extracted at freshwater density may represent biofouled particles that in fact sank during the WWTP process and sludge capture [11]. It is therefore not possible to draw conclusions about the stages at which these particles were captured in the treatment process based on the density at which they were extracted following organic matter removal. However, as the particles have been cleaned of any biofilms, the density extraction procedure used here may indicate potential polymer composition where polyethylene, polypropylene and foamed polystyrene will float at freshwater density. This is confirmed by the FT-IR results where, of the 60 particles that were chemically characterised, 88 % of the low-density extract particles were polyethylene, polypropylene or SBR (Appendix B). Just 8 % of the particles extracted at higher density were composed of these low-density polymer types. This may relate to the incorporation of some microplastics in composite particles that were not completely broken down by the organic matter removal process.

Furthermore, the high organic content and potential for microbial activity within WWTPs may increase the likelihood of a particle becoming biofouled during treatment or following incorporation into the sludge phase [11]. However, it was not possible to examine the extent of this process based on the potential cleaning of particles during organic matter removal.

The inability to establish biofouling on the extracted particles also limited interpretations of the fate of microplastics upon release into the environment. The density of microplastic particles will determine their behaviour and fate, such as potential for erosion or transport processes in aquatic systems [35]. Whilst biofouling is very important in this process, it is also a seasonal and successional phenomenon [36, 37]. Namely, the microbial assemblages may fluctuate in coverage and possibly completely removed over time. Based on the methods employed and results obtained from this study, it is not possible to establish the influence of wastewater treatment and the capture of microplastics in solid sludge on biofilm development or removal. Hence, we cannot make conclusions regarding the specific behaviour of microplastic if they are added to soils or released into the environment. However, it is notable that the majority (62 %) of identified microplastic particles were low density and would likely be buoyant in freshwater if they are free from biofouling. This gives some indication of the potential for microplastics to be eroded from soils and transferred to other environments such as freshwater systems. Furthermore, it points towards their potential behaviour if they are ultimately transported to the marine environment.

Further work to unpick biofilm dynamics related to wastewater treatment processes and the extent of biofouling on microplastic particles added to soils is required. This includes the efficacy of performing density separation prior to organic matter removal to include the influence of biofouling on particle density.

4.2.3 Size limitations

Due to analytical limitations, it was not possible to investigate the entire size spectrum of microplastic contamination in sludge samples. The lower limit for visual identification was set at 50 µm. Hence, microplastic particles below this threshold were not quantified. This limit was imposed based on the constraints of visual identification – small particles cannot be reliably visually identified as plastic particles. In fact, many studies utilise size limits higher than that imposed here (e.g. 150 µm). However, the majority (96 %) of particles smaller than 150 µm observed in this study were microbeads. Due to their sphericity and shine, it was easier to recognise these particles and qualify suspect plastic composition compared to fibres and fragments. It is not possible to state with confidence that particles below this size threshold are suspected microplastics due to difficulties in observing key characteristics or testing using a hot needle.

An additional size limitation was introduced by the FT-IR analysis that was used in this study. Standard FT-IR is only capable of testing larger microplastic particles. There is no prescribed lower limit for this technique; however, the spectra produced from particles that do not cover enough of the sample window do not yield acceptable matches with the reference library. This effect is enhanced for fibrous particles, which exhibit a very small diameter despite potentially long lengths. Hence, many of the very small (<150 – 200 µm) microplastics and a large proportion of the fibres could not be tested using FT-IR. Additional techniques such as the use of a hot needle test was used to provide greater confidence in the likely plastic composition of suspected microplastic particles. It is important to note that all of the tested particles, representing 10 % of total identified particles, were confirmed to be plastic. This points towards a rigorous visual identification procedure and may imply a high proportion of untested microplastic are also likely to be plastic.

Given the high number of microplastics concentrated in the lowest size fraction (**Figure 6**) it is indeed possible that there are a large number of microplastics below this size threshold within the sludge samples. It was not possible to test this component of microplastic contamination within the confines of this study; however, further research using more advanced techniques is recommended to establish the nature of this contamination. Furthermore, it is not yet possible to analyse *nanoplastics* in environmental samples. Indeed, thus far, only one study has been effective in reporting potential nanoplastic concentrations under environmental conditions [38]. The current study was not able to characterise the nanoplastic component of sewage sludge. It is important to note the increased risk posed by these smaller plastic particles [39]. Hence, the extent of nanoplastic contamination of sewage sludge should be tested in the future.

4.2.4 Reporting units

Correcting for moisture content across the 10 sludge samples, as shown in **Figure 3**, demonstrated the need to standardise microplastic counts for sample dry weight. This prevents reporting relationships within the data that are merely a component of substrate characteristics or sludge treatment process. In addition, it is important to report counts in a manner that can be compared with other studies. Based on the findings of this project, it is recommended that future counts are reported as particles kg^{-1} (d.w.) or volumetrically as particles m^{-3}.

4.2.5 Potential for laboratory contamination

Routine testing of the laboratory environment was performed during the processing and analysis of the sludge samples. Potential sources of microplastics in the laboratory were controlled to reduce the potential for contamination. The risk for contamination from the laboratory environment was considered to be low based on this monitoring.

The use of Fenton’s reagent, as an organic matter removal step, was effective in accelerating the reaction time required. During non-catalysed peroxide oxidation, samples must be kept only partially covered to permit vapours to escape. The shorter reaction time reduced the time during which samples were partially exposed to potential laboratory contamination. The samples were processed in a clean cabinet during step to further reduce the potential for airborne contamination during this stage.

Furthermore, it was possible to keep the samples covered for the majority of the sample processing procedure. This was highly effective in limiting contamination, as demonstrated by the very low number of plastic particles observed within the blanks. Only one set of blanks were found to contain microplastics and the corresponding batch of samples has been corrected for this potential contamination.

4.3 Importance of influence characteristics and treatment process

4.3.1 Influence of WWTP characteristics and treatment processes

Based on the small number of WWTPs investigated in this study and the variety in treatment process, it was not possible to derive statistically significant findings related to the influence of water and sludge treatments or the size of the plant. Further study is recommended to establish the effect of different treatments (*e.g.,* primary, secondary, tertiary) and sludge finishing (*e.g.*, anaerobic digestion, lime stabilisation, thermal drying) on microplastic abundance. This could be enacted through the WWTP process to establish the stages during which microplastics are captured and added to the solid sludge phase and by testing sludge samples before and after stabilisation and hygienisation.

Additionally, there are a number of steps in the wastewater treatment process where microplastics may be removed. This includes grit traps and grease skimming stages. The fate of the solid material captured during these steps is not known for the WWTPs examined here. In many cases, this component is removed from the WWTP and is not added to the sludge [11]. It is possible that a significant proportion of the microplastic load associated with wastewater influent is captured during these steps and may subsequently be sent to landfill or incinerated. In theory, many of the low-density plastics such polyethylene and polypropylene may float and be captured as part of the skimming process, for example. These represent a significant proportion of microplastic contamination in the environment [34]. Murphy *et al.* [7] only identified microbeads, which are typically composed of polyethylene, in this grease skimming stage. It is possible that the low-density plastics identified in this study represent biofouled particles that were no longer positively buoyant due to increased particle density. The proportion of microplastic removed during these steps represents a removal of microplastics that are not added to agricultural soils or released into the environment.

4.3.2 Influence of weather conditions

Wet and dry weather sampling at VEAS and BRA did not successfully capture contrasting weather conditions. Sludge produced in the VEAS II sampling corresponded with a low magnitude rainfall event. The abundance of microplastic particles in the sludge was significantly different (**Figure 3**); however, the change was not dramatic. BRA II is associated with a slightly lower intensity rainfall event; however, both sampling periods were characterised by precipitation. The notable change between BRA I and BRA II samples may reflect one or a number of variables, many of which were not tested in this study, for example: influent microplastic concentrations, accumulation of microplastic in the environment between rainfall events and seasonal changes in microplastic release.

No statistically significant correlation was observed between microplastic abundance across all samples and the associated level of precipitation. It is likely that the influence of weather conditions on microplastic concentrations in influent and resulting sludge is highly complex and possibly related to shifting source dynamics, seasonal trends, and antecedent conditions. This study represents an initial baseline assessment of microplastic abundance in sludge across Norway. Further testing should incorporate systematic sampling across different temporal scales. Moreover, additional plants should be included to separate the influence of microplastic inflow to the plant from the effect of the wastewater treatment process.

4.4 Potential sources of microplastic to sludge

Attributing potential sources to microplastic contamination is complex [40]. Based on the very wide range of sources and the ubiquitous nature of plastic particles, it is not currently possible to deliver reliable estimates for different microplastic sources to WWTPs. However, it is feasible to establish some broad potential sources for the observed microplastics based on particle shape and polymer composition.

Microbeads are specifically engineered spherical plastic particles. Hence, the range of sources is generally smaller than fragmented microplastics. All the microbeads analysed in this study were composed of polyethylene, although a large proportion were too small to be tested using FT-IR. 93 % of microbeads in personal care and cosmetic products (PCCPs) are composed of polyethylene [41]. This indicates that the microbead contamination observed in sludge samples in this study may be derived from the use of PCCPs and the incorporation of these particles into municipal effluent. However, the mean particle size observed here is lower than typically found in such products. Hence, it is not possible to conclusive link this contamination to PCCPs. It is also possible that these microbeads could instead be derived from industrial effluents, where microbeads are sometimes used as 'airblasting' media [42]. However, the particle size range used in these applications has not yet been assessed.

Plastic fibres are likely to be derived from synthetic textiles. Studies have shown that >1 900 particles are released from a single synthetic garment per wash [30]. As washing machines discharge into the wastewater system, there is a direct link to WWTPs. Many fibres are high density – *e.g.*, polyester: 1.24 – 2.3 g cm^{-3} [34] – and so are likely to settle out in the sludge capture stages. However, it is not possible to derive domestic vs. industrial contributions of textile fibres, where the effluent from industrial laundry facilities and textile production plants may be very high.

It is not possible to establish the source of microplastic fragments. It is possible that small fragments could be found in the influent and be derived from urban sources such as road dust or urban runoff. Alternatively, they may have fragmented from larger plastic debris within the WWTP as a result of the treatment process. Exposure to warm temperatures, mechanical process and biological activity may increase the fragmentation of plastic particles. It is important to establish this effect in the future to examine the extent to which WWTP actively *produce* small microplastic particles.

Several studies point towards tyre debris as a major source of microplastics to the environment [43–45] and the typical density of tyre wear debris (1.2-1.3 g cm^{-3} [46]) indicates that such particles are likely to be captured in the solid sludge phase, as opposed to the grease skimming steps for example. However, potential tyre wear particles were not observed in the visual identification step in this study. It is possible that the tyre wear particles may be captured and removed prior to the sludge phase. Alternatively, they may be smaller than the size limit imposed in this study. This is supported by the findings of Kreider *et al.* [47] who established 5 – 25 µm as the key size class for tyre wear particles. However, the above study also found many particles up to 350 µm in size, which were not observed in this analysis. Further, only a small number of black fragments identified in the sludge samples were composed of SBR. It has been noted that standard FT-IR analysis may not be ideal for analysing car tyre particles based on absorption of the IR beam by the carbon black component [46]. Regardless, a successful characterisation of suspected particles as SBR was achieved through FT-IR analysis used here.

From the results of this study, it is not possible to state the likely pathways or accumulation zones for car tyre-derived microplastic particles. Instead, the findings simply point towards a lack of such particles reaching the solid sludge phase of WWTPs. Further work is required to track microplastics derived from car tyre wear to establish their fate within WWTPs.

4.5 Significance of microplastic abundance in Norwegian sludge samples

Thus far, seven studies have examined microplastic abundances in sewage sludge. This section reviews the key findings from those studies in order to contextualise the results from Norwegian WWTPs.

In a study of wastewater treatment in Sweden, 16 7000 microplastic particles kg^{-1} (d.w.) were identified in the sludge phase from a single WWTP in Lysekil [10]. The majority of this contamination was composed of fibres, with a smaller contribution from plastic fragments and paint flakes. Only microplastics >300 µm were analysed and the WWTP had a low population equivalent (12 000). Based on these details, it seems likely that the relative microplastic contamination was greater than those observed for Norwegian WWTPs.

Mintenig *et al.* [21] identified microplastic abundances ranging from 1 000 to 24 000 particles kg^{-1} (d.w.) in dewatered sludge from six WWTPs in NW Germany. However, they did not quantify fibres as they had not controlled for potential contamination from the laboratory environment. The authors found that all microplastics were <500 µm, where 66 % of microplastics are within that size class in this study.

Mahon *et al.* [16] observed abundances of 4 196 – 15 385 particles kg^{-1} (d.w.) from seven WWTPs in Ireland. The authors observed a significant difference in the number of microplastic particles in sludge treated with anaerobic digestion and thermal drying. Sludge samples treated by lime stabilisation presented the highest microplastic counts; however, it was noted that this may have been a factor of a different methodological approach. One WWTP in this study utilises lime stabilisation (TAU) and did not present similarly elevated particle counts. A higher proportion of fibres (75.8 %) were observed than in Norwegian WWTPs (28.9 %) and a far lower contribution from microbeads was identified (0.3 % compared to 37.6 % in this study). However, this may be linked to the lower size limit of 250 µm that was used, where the majority of beads identified in Norwegian WWTPs were below this size.

Return activated sludge from a WWTP in Seelze, Germany, was analysed by Sujathan *et al.* [17]. Very high counts of 495 000 particles kg^{-1} (d.w.) were reported. Microplastics were analysed using confocal Raman microscopy which permitted a lower size threshold of 0.48 µm, where the majority of particles were between 20 and 100 µm. This indicates that abundance of microplastics in sludge may indeed by much higher, with a large number of plastics contained within the smallest size classes. Sujathan *et al.* [17] found a similar proportion of microbeads (36 % compared to 37.6 % in this study), where the mean diameter was approximately 30 µm.

Three studies report microplastics as particle kg^{-1} wet weight or l^{-1}, which complicates comparisons with this project. Murphy *et al.* [7] identified fewer than 2 000 particles kg^{-1} (w.w.) in the sludge cake from a WWTP in Glasgow, UK. The highest abundances were observed in samples obtained from the grease skimming step (7 868 particle kg^{-1} (w.w)). They found no short-term temporal variations in microplastic concentrations within the sludge cake sample (24 hours). Microbeads were only observed in the grease sample. Return activated sludge was sampled from a WWTP in California by Carr *et al.* [11]. Only one particle in 20 ml of sludge was identified in the sampling. Instead, microplastics counts of approximately 5 000 particles kg^{-1} were observed in the solid skimming from the primary settling tanks sampled from three WWTPs. This component is not added to the final sludge at these sites and so represents a removal of microplastics. However, it is not stated whether this value relates to wet or dry weight. Finally, Leslie *et al.* [5] analysed microplastics in sludge from three WWTPs in the Netherlands. Counts ranged from 370 to 950 particles kg^{-1} (w.w.), where the dry weight is reported to be <1 % for all samples.

These reported abundances compare to those observed in WWTPs across Norway: 1 701 to 19 837 particles kg^{-1} (d.w.). However, based on the differences in microplastic extraction and the size classes analysed, it is difficult to draw meaningful comparisons between the studies. Despite this, it is unlikely that microplastic abundance in sludge from Norway is significantly higher or lower than from sludge produced elsewhere. All results fall within the same order of magnitude, with the exception of Sujathan et al. [17]. That study, based on a German WWTP, investigated smaller microplastic particles. This points towards the potential for significantly higher microplastic particle counts in Norwegian sludge that exist below the size threshold examined in this study.

4.5.1 Implications related to the use of sludge as a soil improver

Up to 99 % of microplastics (>150 – 250 µm) that enter WWTPs are captured [10, 11]. A large proportion of this is incorporated into the solid sludge phase [7, 11, 16]. The results presented here demonstrate a high abundance of microplastic particles in sewage sludge compared to other environmental substrates such as river or lake sediments [e.g. 48–50]. A common practice involves the reuse of this sludge as a soil improver in agricultural systems. Nizzetto *et al.* [14] highlighted this as, theoretically, the key pathway for microplastic release in to the environment. Current estimates suggest that approximately 63 000 to 430 000 tons of microplastic are added to European farmlands each year as a result of this practice. This has been of concern as microplastic abundance in sludge that is applied to fields had not yet been ascertained for Norway.

Based on the abundance of microplastics in sludge revealed in this study, and details on the application of sewage sludge in Norway [51], it can be estimated that approximately 446 billion microplastic particles are spread on agricultural soils, 27 billion microplastic particles are added to green areas and 112 billion microplastic particles are sent to soil producers per year. This equates to over 584 billion microplastics that are released into the environment via sewage sludge application each year in Norway.

Once applied to the fields, microplastic may become incorporated into soils [15]. Erosion by wind and water can lead to the transfer of microplastics to other environments, such as rivers, lakes and oceans. This has the effect of propagating contamination across a wider area and potentially contaminating systems otherwise free from microplastics [35]. Additionally, soil biota such as earthworms (*Lumbricus terrestris* [52–55], *Eisenia Andrei Bouché* [56]) and Collembola (F*olsomia candida, Proisotoma minuta* [57]) may interact with microplastic particles. Existing studies have noted effects on soil organism health and behaviour, including histological damage in the gut tract [52–54]. Ingestion of microplastics by these organisms also presents an opportunity for trophic transfer, which has been demonstrated for terrestrial birds in China [58]. Thus, the application of microplastic contaminated sewage sludge may have ecosystem impacts.

Contaminants may sorb onto particles prior to, during, or following wastewater treatment and these can be released upon ingestion and cause toxic effects. These may include typical wastewater contaminants such as phenanthrene, tonalide, and benzophenone [59]. Hydrophobic contaminants may preferentially sorb onto microplastic particles leading to an enrichment of microplastics with organic pollutants and metals [60, 61]. The sorption capacity of typical wastewater microplastics, such as microbeads, has been confirmed [41].

Fibres have been identified as having the greatest ecotoxicological impact based on their increased surface area [62]. High fibre counts were observed in the sludge from some of the WWTPs, such as LINNES. It is possible that this increases the ecological significance of sludge spread from those plants. On the other hand, small particle size, as observed for microbeads, may increases the chances of particle ingestion by biota. Browne *et al.* [63] demonstrated that contaminants may be transferred to organisms upon ingestion, hindering healthy functioning and reducing biodiversity. Once ingested, sorbed contaminants are bioavailable [64], particularly when exposed to gut conditions [65]. Hence, microplastics added to soils from sludge may represent a transport pathway of contaminants to soil ecosystems. The concentrations of contaminants sorbed onto microplastics from WWTPs must be tested in more detail, including the influence of water and sludge treatment on enhancing sorption/desorption of these potentially harmful chemicals.

Finally, the small microplastic (<50 µm) and nanoplastic component was not investigated in this study. It is not yet understood how these particles behave once added to soil systems; however, their small size increases the risk posed by environmental contamination. For example, nano-sized particles may be taken up by plants and, potentially, crops [66]. This may contaminate food products produced for human consumption with particle derived from sewage sludge. However, this has not yet been demonstrated under environmentally-relevant conditions.

5 Recommendations for future work

This study presents a baseline assessment of microplastic abundance in sludge from WWTPs across Norway. The results presented here provide useful insights into the level of microplastic contamination and estimates the release into the environment through the application of sludge as a soil improver. However, this report also raises a number of interesting questions related to sludge microplastic dynamics. Future work should focus on capturing more potential variables (such as temporal changes and the influence of treatment processes) and the optimisation of analytical methodologies (including size limitations). Some specific recommendations are detailed below:

- Examine the influence of the sampling strategy, including the mode of extracting samples from the solid sludge phase.
- Establish the temporal variability of microplastic abundance in sludge samples across various timescales including seasonal changes and different weather conditions.
- Perform systematic sampling of a range of wastewater treatment (primary, secondary, tertiary) and sludge finishing (*e.g.,* anaerobic digestion, lime stabilisation, thermal drying) processes to establish their influence on microplastic abundance in the final sludge.
- Investigate the significance of semi-synthetic fibres in WWTPs including their behaviour and fate, their environmental significance, and the ability of the methodological technique to effectively extract semi-synthetic particles.
- Investigate biofilm dynamics of microplastic particles within the wastewater treatment process and the extent of biofouling on particles that are added to soils including the potential for performing density separation prior to organic matter removal to establish the influence on particle density.
- Examine the small microplastic (<50 µm) and nanoplastic component of plastic contamination in sludge.
- Identify the transfer pathways and fate of microplastics derived from car tyre wear in sewer systems and WWTPs.

6 Conclusions

This study presents the first baseline assessment of microplastic abundance in sludge produced in WWTPs across Norway. The results highlight variability between the sludge samples including temporal variability at the Bekkelaget (BRA) and VEAS WWTPs. The composition of microplastic contamination was also variable, including varying dominance of different microplastic shapes and densities across the sludge samples. In general, extracted microplastics were small, where 81 % were below 1 mm and 34 % were concentrated into the lowest size class analysed (50 – 125 µm). These findings may help contribute to developing potential solutions to microplastic contamination in sludge in the future.

The values obtained here correspond with other WWTPs from across Europe and North America; however, methodological disparities complicate efforts to compare results. This highlights the need for a standardised analytical procedure. Within this study, several potential extraction protocols were tested and a time- and cost-effective method was identified and optimised. However, additional analytical parameters such as the size classes analysed, and the units used to report data must also be addressed to improve comparability between different studies.

It is notable that there was no significant correlation observed between microplastic counts and many of the potential influencing variables including: the size of the plant (population equivalent), the treatment processes employed, influent volumes, or weather conditions. This may be a factor of the number of samples taken, where a larger-scale, systematic study is recommended in the future. However, this finding may also point towards complexity in the nature of different variables. Further work must also characterise the effect these have upon microplastic abundance in the final sludge to provide recommendations for reducing microplastic contamination and release into the environment.

The potential sources of microplastic to WWTP are likely to be numerous and source dynamics are expected to be complex. It was not possible, within the confines of this study, to confidently state the sources of different microplastic shapes; although, municipal and industrial effluents as well as storm water runoff all may play a potential role. Notably, particle counts did not include a large proportion of microplastics that resembled car tyre debris, nor were confirmed to be composed of typical car tyre polymers (e.g. SBR) in the FT-IR analysis. This is contrary to reports highlighting car tyre wear as one of the largest potential sources of microplastics. The fate of tyre debris particles in sewer systems and WWTPs has not yet been identified. The absence of tyre wear particle identified in this study indicates this component of microplastic contamination could have a different fate within WWTPs.

Estimates for the scale of microplastic release via sewage sludge to agriculture, green areas, and soil producers highlight this reuse practice as a potentially major source of microplastics to the Norwegian environment with a potential transfer to other environments. The risk posed by this environmental contamination has not yet been fully assess.

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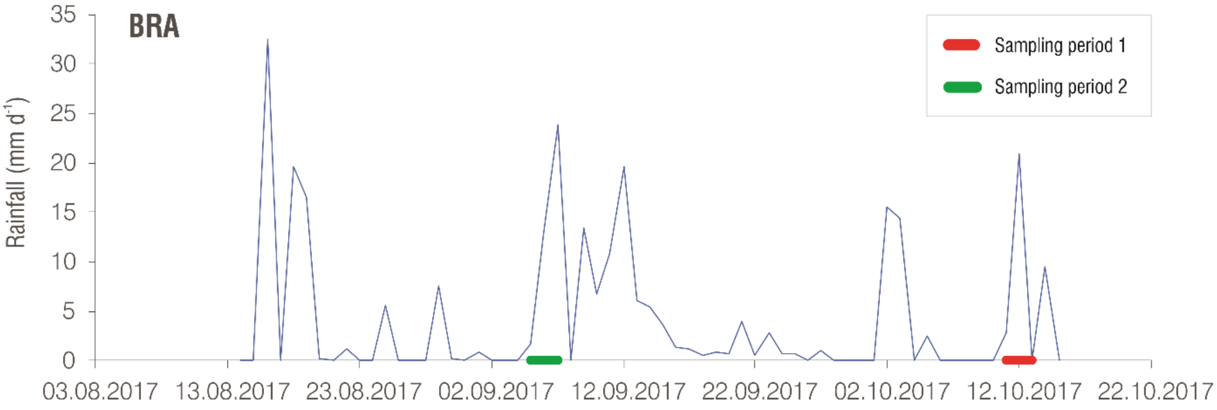
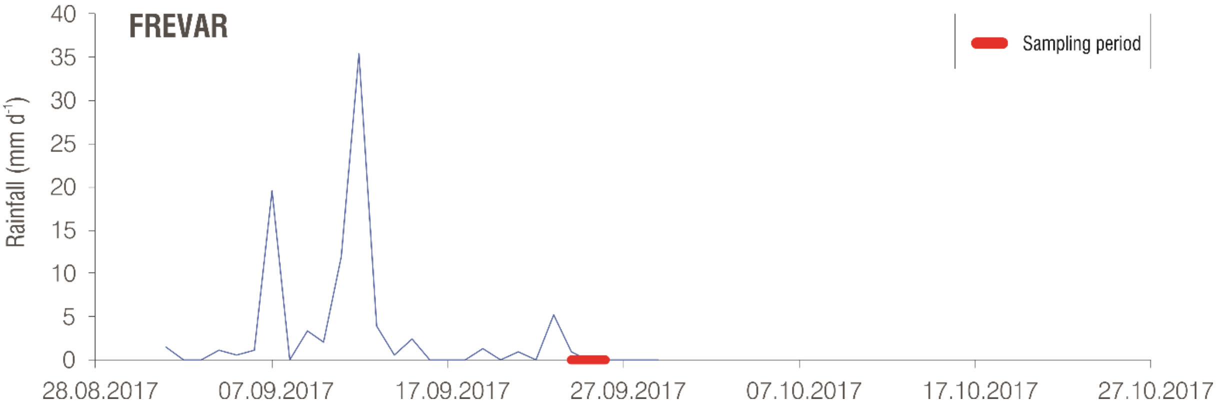
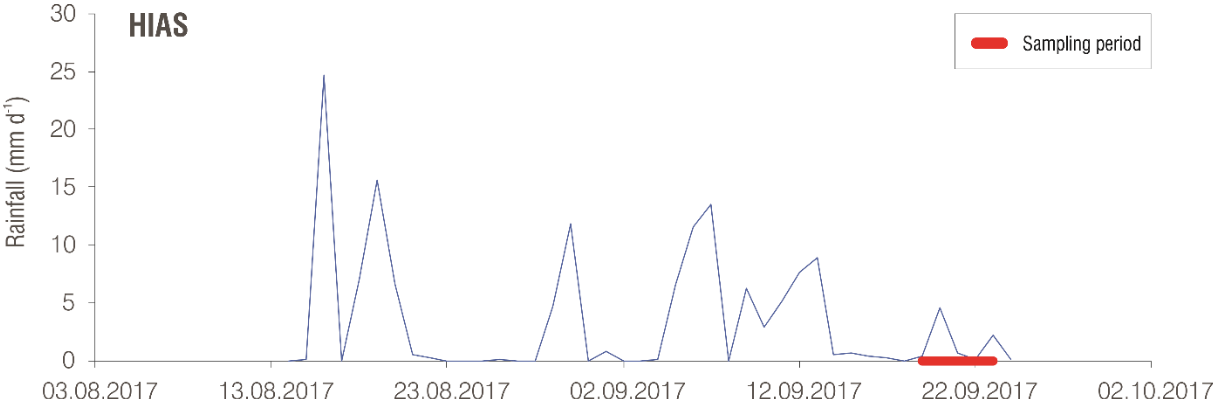
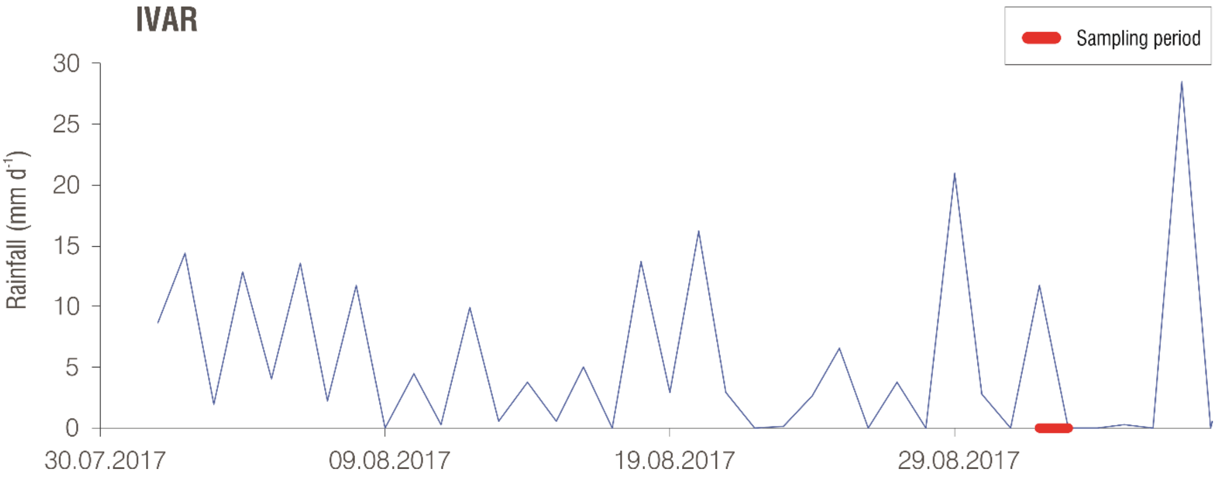
Appendix A. Rainfall events during sampling

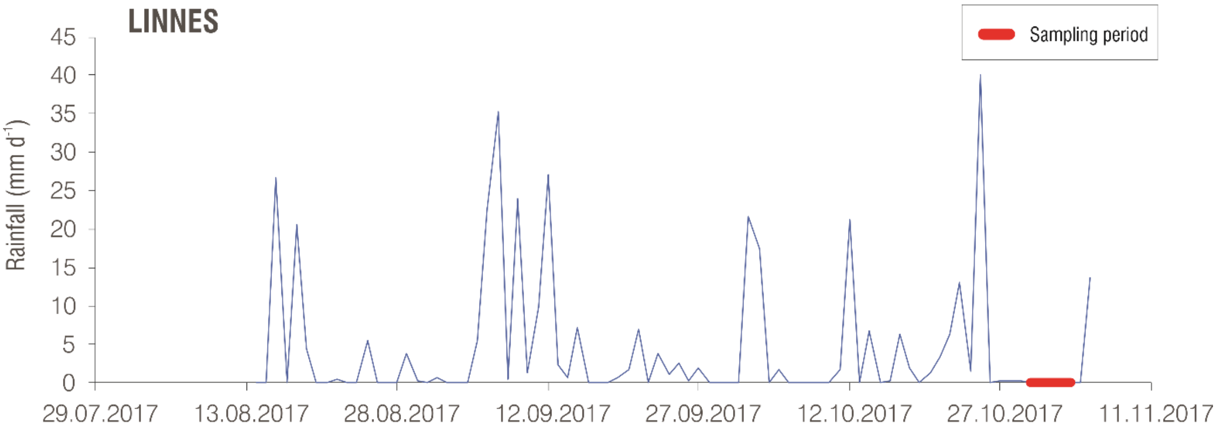
The below figures show rainfall events in the period prior to and during the sampling campaigns at the nearest available Met.no monitoring station to the respective WWTP. Sampling periods are marked with green/red bars.

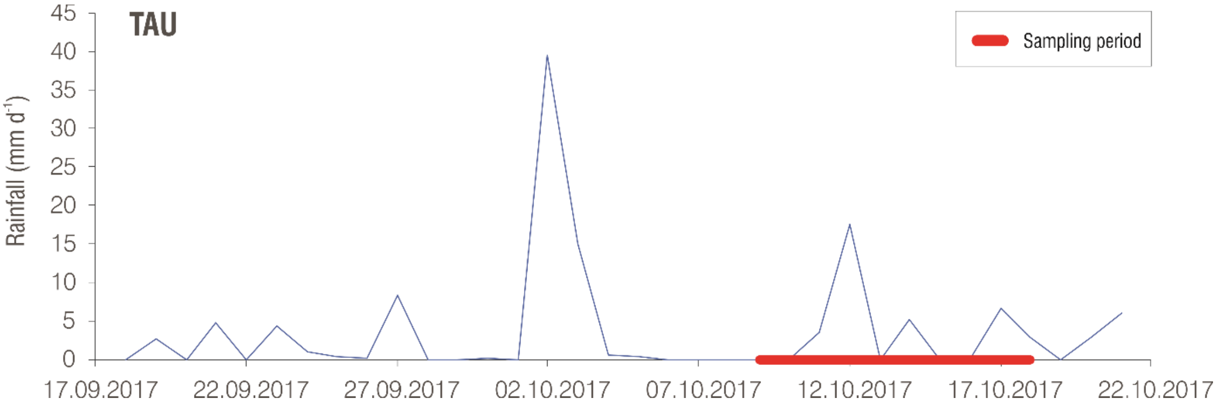


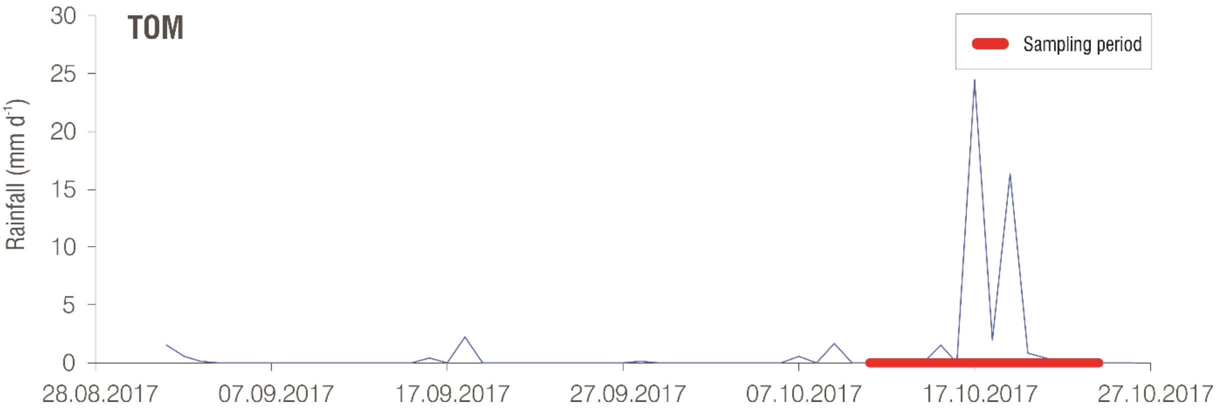


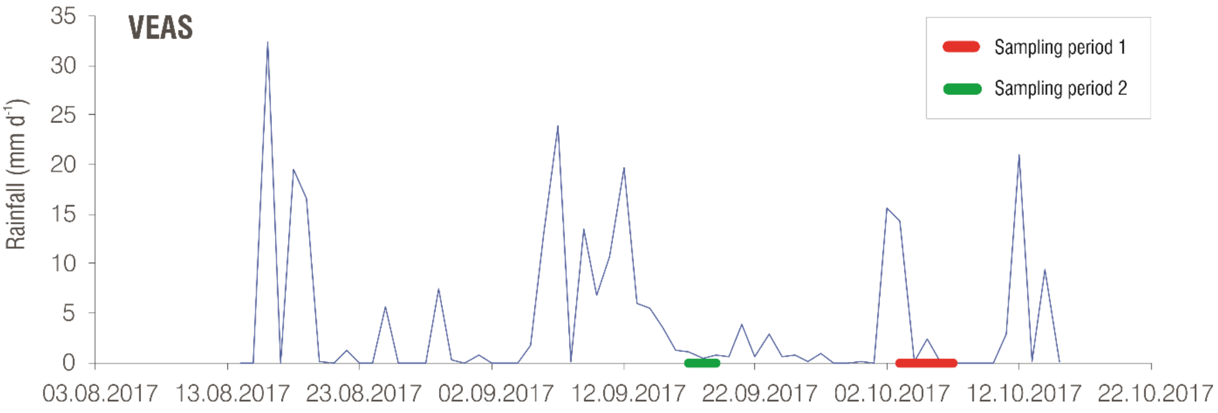



IVAR
Sampling period
Rainfall (mm d⁻¹)
0
5
10
15
20
25
30
30.07.2017
09.08.2017
19.08.2017
29.08.2017


LINNES
Sampling period
Rainfall (mm d⁻¹)
0
5
10
15
20
25
30
35
40
45
29.07.2017
13.08.2017
28.08.2017
12.09.2017
27.09.2017
12.10.2017
27.10.2017
11.11.2017


TAU
Sampling period
Rainfall (mm d⁻¹)
0
5
10
15
20
25
30
35
40
45
17.09.2017
22.09.2017
27.09.2017
02.10.2017
07.10.2017
12.10.2017
17.10.2017
22.10.2017


TOM
Sampling period
Rainfall (mm d⁻¹)
30
25
20
15
10
5
0
28.08.2017
07.09.2017
17.09.2017
27.09.2017
07.10.2017
17.10.2017
27.10.2017


VEAS
Sampling period 1
Sampling period 2
Rainfall (mm d⁻¹)
35
30
25
20
15
10
5
0
03.08.2017
13.08.2017
23.08.2017
02.09.2017
12.09.2017
22.09.2017
02.10.2017
12.10.2017
22.10.2017

Appendix B. Breakdown of FT-IR analysis

Site	Density	Shape	Colour	Size	Polymer type	FTIR Match (%)
BRA	High	Fragment	Grey	1213.21	PMMA	91
BRA	High	Fibre	Blue	3246.85	PET	87
BRAII	Low	Fibre	Black	1853.7	PP	80
BRAII	Low	Fragment	Blue	357.68	PP	80
BRAII	Low	Fragment	Turquoise	342.02	PE	92
BRAII	Low	Fragment	Pink	336.3	PP	80
BRAII	Low	Fragment	Clear	265.08	PP	89
BRAII	Low	Fragment	Clear	209.95	PE	91
BRAII	Low	Fibre	Grey	469.6	PET	95
BRAII	Low	Fibre	Red	1578.02	PP	89
BRAII	Low	Fragment	Clear	1093.12	PET	89
BRAII	Low	Fragment	Blue	684.76	PP	84
BRAII	Low	Fragment	Black	1342.11	PP	84
BRAII	Low	Fragment	Turquoise	396.87	PE	84
BRAII	Low	Fragment	Black	798.18	SBR	86
BRAII	High	Fragment	Grey	711.67	PP	86
BRAII	High	Glitter	Green	322.48	PET	91
BRAII	High	Fragment	Blue	563.55	PET	83
BRAII	High	Glitter	Pink	442.88	PET	84
BRAII	High	Glitter	Pink	339.36	PET	83
BRAII	High	Fibre	Pink	4400.82	PES	84
FREVAR	High	Fragment	White	2348.19	PVC	91
FREVAR	High	Fragment	Black	2370.52	PET	91
FREVAR	High	Fragment	Black	3486.32	SBR	81
FREVAR	High	Fragment	Multi	518.05	PUR	81
FREVAR	High	Fragment	Black	3998.26	PE	91
FREVAR	High	Fragment	Multi	321.78	PUR	84
FREVAR	High	Fragment	Green	796.32	PET	83
FREVAR	High	Glitter	Yellow	261.27	PET	87
FREVAR	High	Fragment	Multi	509.1	PUR	82
FREVAR	High	Fragment	Multi	463.42	PUR	88
FREVAR	High	Fragment	White	4409.64	PET	85
HIAS	Low	Bead	Clear	382.96	PE	93
HIAS	Low	Bead	Blue	651.87	PE	90
HIAS	Low	Fragment	Clear	2077.93	PE	94
HIAS	Low	Bead	Clear	156.23	PE	96

HIAS	High	Glitter	Clear	285.66	PET	82
IVAR	Low	Fragment	Blue	3219.89	PP	82
IVAR	Low	Fragment	Green	1023.51	PP	81
IVAR	Low	Fragment	Clear	1577.86	PE	88
IVAR	Low	Fragment	Clear	619.19	PE	86
IVAR	Low	Fragment	Black	850.38	PE	87
IVAR	Low	Fragment	Orange	4712.8	PVC	90
IVAR	Low	Fragment	Clear	651.85	PE	85
IVAR	Low	Fragment	Turquoise	715.78	PE	91
IVAR	Low	Fragment	Green	3402.34	PE	87
IVAR	Low	Fragment	Black	596.41	PP	85
IVAR	High	Glitter	Clear	313.29	PET	81
IVAR	High	Fragment	Multi	2597.26	PUR	82
LINNES	Low	Fragment	Blue	1454.04	PE	90
LINNES	High	Fibre	Orange	2992.79	PET	89
LINNES	High	Fragment	Pink	1255.08	Silicone	97
TAU	Low	Fragment	Green	2552.07	PP	88
TOM	Low	Fibre	Black	2919.88	PA	84
TOM	Low	Bead	Clear	306.95	PE	93
VEAS	Low	Bead	Clear	171.06	PE	91
VEAS	High	Fragment	Black	2710.42	EDPM	83
VEAS	High	Glitter	Clear	508.52	PET	84
VEASII	Low	Bead	Clear	171.66	PE	92

NIVA: Norges ledende kompetansesenter på vannmiljø

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