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Method Validation in microplastic research in the Arctic. A study of microlitter in arctic sediments in Adventdalen, Svalbard

Master's thesis in Geography

Supervisor: Chantel Nixon

Co-supervisor: Mark Furze

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Abstract

Field and lab methods in microplastics research have been widely inconsistent since microplastics were first acknowledged as an environmental pollutant. So far, the most common methods applied to the study of microplastics in the environment have been borrowed from fields such as micropaleontology, sedimentology, and marine biology; however, the adaptability of such methods for all environments and geographic locations is questionable. For example, how much sediment sampled from the seabed of a high Arctic fjord is enough to characterize the concentration of microplastics in that location? And are techniques such as filtering or sieving to concentrate a specific size fraction, an efficient way to isolate microplastics from sediments? Finally, are the methods equally appropriate in all environments, from the tropics to the poles? This thesis presents successes and failures in processing glaciomarine and glaciofluvial sediments from Adventfjorden, Svalbard, for microplastics (5mm to 100 μm).

A key finding has been that density flotation and filtration separation techniques have limited efficacy with the silty-clay sediments of Adventfjorden due to the similarities in density and (in some cases) morphology between the sediments and the microplastics. A pre-step of sieving the samples at 63 μm , improved their extraction potential by ~40%. Testing of particle chemistry using Raman Spectroscopy revealed that a small number of 3% of the extracted particles were paint flakes, rather than microplastics, indicating that reliance on visual identification of microplastics, observations of colour, size, and morphology, is insufficient and will not produce reliable, replicable results. This thesis argues that the utilization of analytical instruments like FTIR or Raman Spectroscopy should therefore be required for reliable data production on the presence and abundance of microplastics in all study areas. Finally, the glaciomarine and glaciofluvial sediments in Adventfjorden showed no similarities with respect to their microplastic content, potentially indicating that the source of microplastics in Adventfjorden is not Longyearbyen itself, although many more samples are required to confirm this. With thorough sediment grain size analysis and microplastic abundance data, sample size can be determined across different environments.

Sammendrag

Metoder for felt og laboratoriearbeid i mikroplastforskning har vært svært inkonsekvente siden mikroplast ble anerkjent som miljøforurensing. De vanligste metodene brukt i mikroplastforskning har blitt tilpasset fra metoder i geologi og biologi, men tilpasningsdyktigheten til slike metoder for mikroplastforskning i ulike miljøer er lite forstått. For eksempel, hvor mange sediment prøver fra bunnen av en arktisk fjord er nok for å karakterisere mengde mikroplast over et gitt område? Er sedimentologiske og mikrofossile ekstraksjonsmetoder, f.eks. oksidasjon av organisk materiale, filtrering og siling for å konsentrere en spesifikk fraksjon, tilstrekkelige og brukbare for å isolere mikroplast i sediment prøver i alle miljøer, fra tropene til polene?

Denne studien setter søkelys på mikroplast (<5mm til 100µm) i glasimarine og glasifluviale sedimenter fra et arktisk fjordmiljø (Adventfjorden, Svalbard). Den presenterer suksess og feil i prosesseringen av disse sedimentene for mikroplastanalyse. Et viktig funn har vært relatert til flyte- og filtreringsmetoders begrensede effektivitet med silt/leire sedimenter fra Adventfjorden på grunn av deres likheter i tetthet og til dels morfologi som mikroplast. Siling av prøvene ved 63µm, forbedret ekstraksjons potensialet av partikler. Testing av partiklene i Raman Spektroskop avdekket at et lite antall partikler var malingspartikler og ikke mikroplast. Funnene indikerer at visuell identifikasjon av mikroplast basert kun på observasjoner av farge, størrelse og morfologi ikke er tilstrekkelig og vil ikke produsere tillitsverdige, gjenbrukbare resultater.

Utnyttelse av analyseinstrumenter som FTIR og Raman spektroskop bør bli standardisert for tillitsverdige dataproduksjon vedrørende konsentrasjon av mikroplast i alle studieområder.

Glasimarine og glasifluviale sedimenter viser ingen likhet i forekomst av partikler. En mikroplast partikkel ble funnet i glasifluviale sediment prøve. Med nøyaktig analyse av størrelsesforhold av sediment partikler og forekomst av mikroplast, kan mengde prøver bli bestemt for ulike miljøer.

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1.0 Introduction

To combat the monumental challenge of plastic litter in marine and terrestrial environments it is vital to increase our understanding of the behaviour of microplastics as they are transported through different environmental compartments (e.g., river, ocean, water column, seabed). One key starting point for tackling such a challenge should be to evaluate of the efficacy of different methodologies for sampling and processing of microplastics from different environmental compartments in different geographic locations (Singh et al., 2020). Microplastic research is a relatively new field of study, and methodologies have for the most part been adapted from geology, biology, and other disciplines (Torres & De-la-Torre, 2021; Cutroneo et al., 2020; Solomon & Palanisami, 2016) As the environmental behaviour of microplastics is assumed to be comparable to that of minerogenic and organic particulates of similar size and density (Harris, 2020), it seems natural to simply apply extraction and other processing methods from other disciplines.

Examples for bulk sampling microplastics include using plankton nets floating on the ocean surface (Cutroneo et al., 2020), which allows for large areas to be covered in a short period of time. Problems with this method include clogging of nets when mesh sizes are too small, and the inability to capture the smallest size fractions. On the other hand, geological sediment sampling techniques such as piston, gravity and box coring are also frequently applied in microplastics research (Torres & De-la-Torre, 2021). Extraction of microplastics from sediments have been accomplished using density separation. This method is mostly used in industry for separation of plastic waste (Gent, Menendez, Toraño, & Diego, 2009). This methodology of separating plastic from solid matrices provides excellent capabilities of separating microplastic from inorganic sediments. The methodology of density separation display issues in terms of isolating microplastics, as most material with same or lower density than the dense liquid added to the sample, will make the material float as well as potential microplastics. Furthermore, methods for digesting of organics have been adapted from both geology and biology in most microplastic studies to limit the influence the organics have on the results (e.g., trapping microplastics, cause issues in filtration) (Munno, Helm, Jackson, Rochman, & Sims, 2018). Organic digestion does need adaptation to not influence the microplastic material, which may at too high temperatures as

a result of the exothermic reaction in the digestion process cause deformations of the microplastic particle.

Against this backdrop and following the unanswered questions of Singh et al. in the MIREs report on microplastics in Svalbard (2020), this thesis attempts to determine whether selected field and laboratory methods are appropriate and effective for surveys of marine and terrestrial environments in the high Arctic via testing some of the most common sampling, chemical treatment, extraction, and quantification techniques. The results from the high Arctic specifically inform whether additional research is required, which will serve to strengthen microplastic sampling and processing methodologies for this fragile environment.

The overarching aim of this thesis is to determine to what extent previously established methodologies for extracting and quantifying microplastic concentration in sediments can be adapted in a high Arctic environment, specifically a glaciated catchment adjacent to Adventfjorden, Svalbard (Fig.1).

1.1 Structure of thesis

This paper will first look into study area, in order to provide a detailed environmental location backdrop for the discussion. Secondly, the paper will provide information on the different transport mechanisms which disperse microplastic in the environment and identify areas in which the settling of microplastic particles is likely to occur. Third, a review of selected methodologies is presented to give an overview of commonly used methodologies today and the advantages and disadvantages with said methodologies. Fourth, the thesis will introduce the methodologies utilized for this project specifically, before delving into a discussion on the challenges and solutions identified for such methodologies throughout the fieldwork and laboratory work. Fifth, the results of the project will be presented. This includes amount of potential microplastic per sample and the results from material composition testing with Raman spectroscope. Sixth, the methodologies are discussed. This includes their efficiency and the alterations done to cope with the methodological challenges that occurred during the process of researching this thesis. The results are also discussed in this final part of the paper. Finally, the main findings of the paper will be presented in the conclusion.

2.0 Study Area – Adventfjorden, Svalbard

The aim of section 2.0, Study Area, is to provide a backdrop for succeeding discussions of theory, methodology, results, and discussion, by laying out some of the most relevant features of this high Arctic environment. Key focuses include potential sources of pollution and sediment sources, transportation, and deposition between the delta and the fjord.

2.1 People and pollution in Adventfjorden

Adventfjorden is a small (8 km long, 4-12 km wide), NW-SE oriented fjord arm of Isfjorden, Spitsbergen, Svalbard (Fig.2). Longyearbyen, the largest community on Svalbard, is located on the south side of Adventfjorden (Fig.2) has a population of 2869 (SSB, 2021). People have lived in Longyearbyen since the early 1900s when it was established as a coal mining town. Coal mining in Longyearbyen experienced a peak in the mid-1900 before WWII with multiple coal mines in operations. Today, only Mine 7 is still in operation. There are several cabin settlements outside of Longyearbyen, along Adventfjorden, used by local community members. Some of these are only accessible snowmobile (winter) or by foot in summer due to environmental laws prohibiting the use of all-terrain vehicles to conserve fauna. Other cabins are accessible by road. The traffic in Longyearbyen and the surrounding road is comprised of cars owned by residents, snowmobiles owned by residents and used by tourists as well as trucks transporting coal from Mine 7 in the far end of the valley and the powerplant (Fig.4). The spread of snowmobile trails in the area surrounding Longyearbyen is extensive, as semi-permanent trails are not set. There is a network of roads in the town of Longyearbyen, and one road stretching along the western side of the fjord (Fig.4).

In peak tourist season (March – October), Adventfjorden and Longyearbyen experiences high traffic from cruise ships and shipping (Kugiejko, 2021; Glomsrød, Duhaime, & Aslaksen, 2020), with thousands of tourists coming ashore to go on guided snowmobile trips, hiking tours, sled-dog tours, local boat trips and a variety of different activities. Longyearbyen is also accessible by plane, with an airport located on the western side of the mouth of Adventfjorden (Fig.4).

Approximately 7-9 flights land in Longyearbyen per week.

2.2 Sources of pollution

Pollution in Adventfjorden comes from the surrounding settlements; untreated, raw sewage from Longyearbyen is directly disposed of into the Adventfjorden at a depth of 50 m (Fig.2) (Evenset Anita, 2009). Infrastructure supporting the disposal of waste is located close to shore (Figure 4). This area may be a major source of plastic pollution to the environment in Adventfjorden, notably in seasons with high tourist traffic. In winter, recreational trips with snowmobile is common. Microplastic from snowmobile belt and plastic skis can enter the environment in around town and easily mobilized for further transport into the fjord during summer or winter in times of strong winds (Singh et al., 2020).

2.3 Physical geography

Adventfjorden is influenced by several glacially fed rivers and streams, some of which flow through the centre of Longyearbyen. These include Longyearelva, which drains Larsbreen, Longyearbreen and Platåbreen glaciers; and Adventelva, a braided river system that flows east of Longyearbyen, transporting glacial meltwater and sediment from the glaciers and valleys of Adventdalen itself (Brooks, Ffolliott, & Magner, 2012; Zajączkowski & Włodarska-Kowalczyk, 2007) (Fig.5). Local glacial activity delivers eroded sediment particles to the glaciofluvial system and deposition on the tidal flat and storage in the glaciofluvial system is governed by seasonal freeze-thaw cycles, slope processes and channel migration. Sediment flux to Adventfjorden is mainly supplied by Adventelva and a large delta has developed at the head of Adventfjorden (Weślowski, 2011). During melt season, Longyearelva experiences large flow of water. Significant modifications have been done to the river path to account of large flow of water and to spare infrastructure from overflow and erosion. Further in the Adventdalen, Adventelva has eroded a channel which induce slope processes like slides and fall of rocks and sediments.

Paraglacial sediments are seasonally transported from the steep slopes and valleys to Adventfjorden mainly via slope processes and glaciofluvial erosion and transport, although aeolian transport and coastal erosion are also important (Rydberg et al., 2016; Ta, Wang, & Jia, 2015) The seasonal transport dynamics are clearly visible in Adventfjorden as a sediment plume of variable size, which is deflected towards the north. The steering mechanisms of sediment plumes in arctic glacial fjords are likely governed by the Coriolis effect, this has been

documented in numerous studies. (Pawłowska et al., 2017; Nilsen, Cottier, Skogseth, & Mattsson, 2008; Syvitski, 1989; Hamblin & Carmack, 1978), it is also influenced by local currents.

On land, during the freeze-up in the autumn, but before significant snow cover, dust is readily mobilized from the subaerially exposed parts of the riverbed in Adventdalen. Much of the river sediments in Adventelva is derived from the surrounding glaciers, and this includes abundant silt and clay-sized particles (Gilbert, 1983). This leads to frequent dust storms that are clearly visible from town. The town of Longyearbyen and Adventdalen thus experience significant, fine-grained aeolian deposition in early winter.

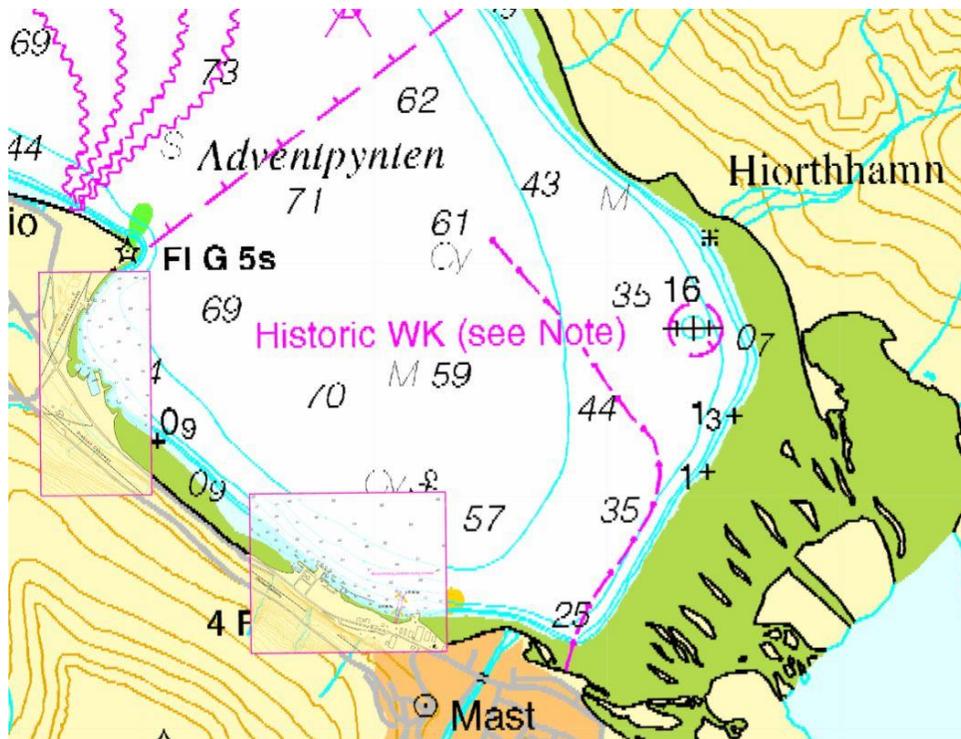


Figure 1 Nautical map of Adventfjorden displaying extent of sewage pipe (NorwegianPolarInstitute, 2021).

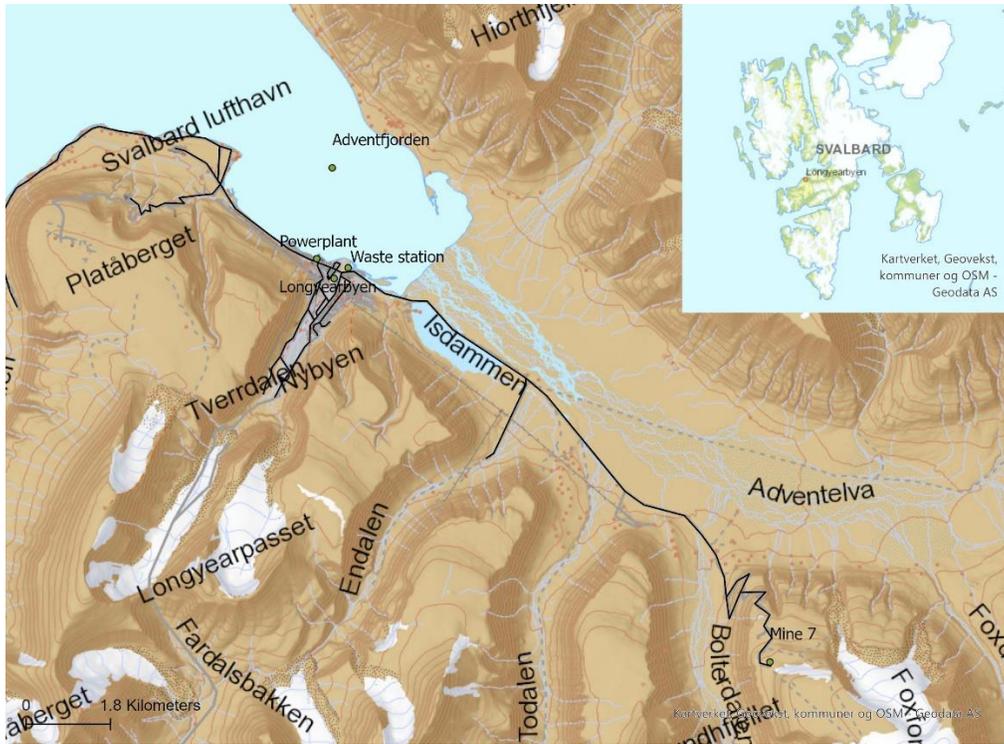


Figure 2 Overview map of Adventfjorden and surrounding network of roads

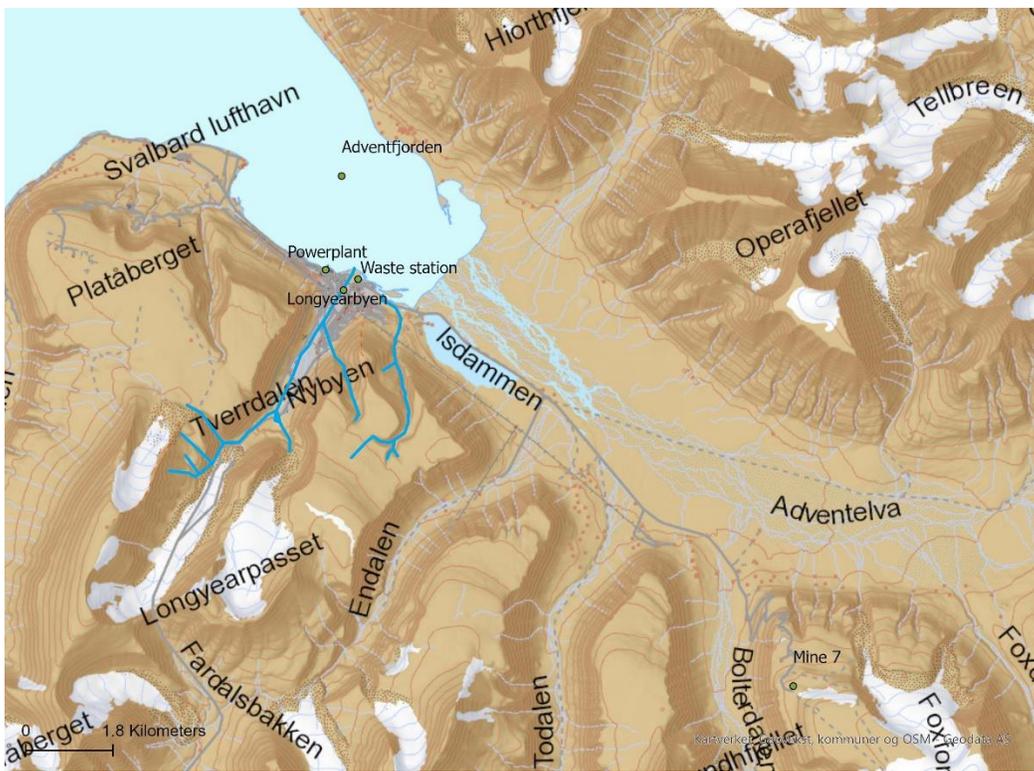


Figure 3 Larsbreen, Longyearbreen, Platåbreen and their respective meltwater flow path

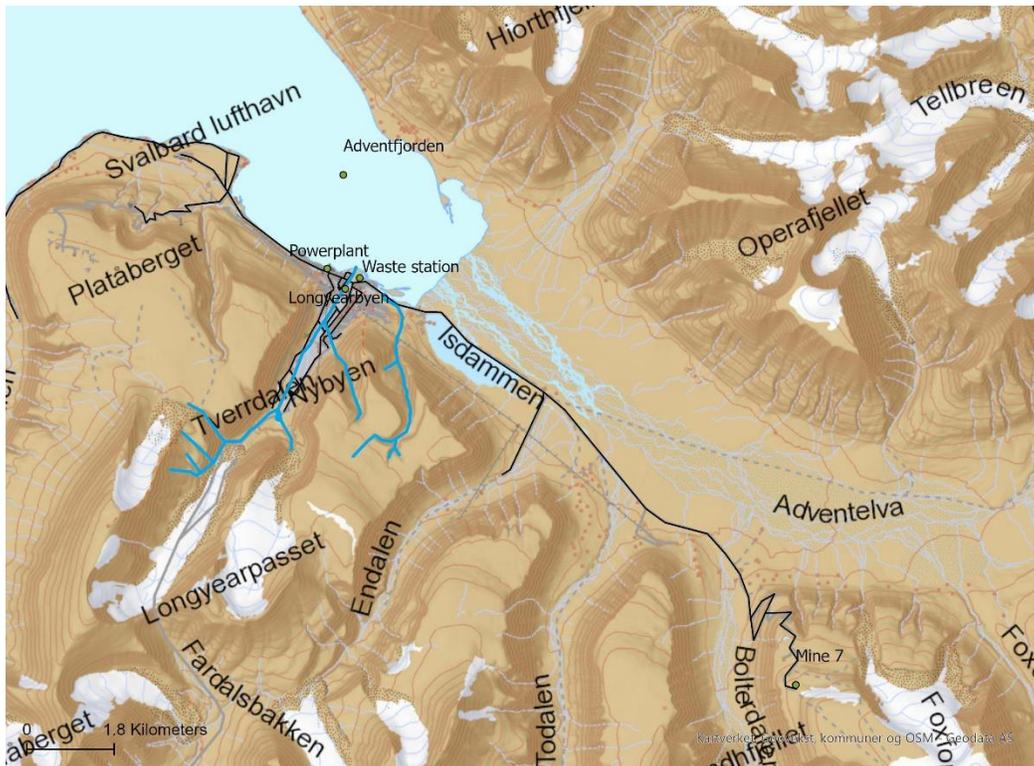


Figure 4 Overview of the roads in around Adventfjorden and Adventdalen. Mine 7 located in the right lower corner and the powerplant located in the centre of town

2.4 Ocean currents and bathymetry

The currents surrounding Svalbard include the East Spitsbergen and West Spitsbergen currents and the coastal currents (Fig.5). The East Spitsbergen and West Spitsbergen currents meet along the western coast of Svalbard, which forces the West Spitsbergen current northwards (Fig.5). Arctic water has also been detected flowing into Isfjorden, which comes from the West Spitsbergen Current (Skogseth et al., 2020). Arctic water is warmer and saltier than the cold fjord water. The mixing of the two water masses increases current activity in the fjords (Skogseth et al., 2020). Below the surface, Adventfjorden has high occurrence of turbidity currents, which weaken with distance from river mouths (Zajączkowski & Włodarska-Kowalczuk, 2007).

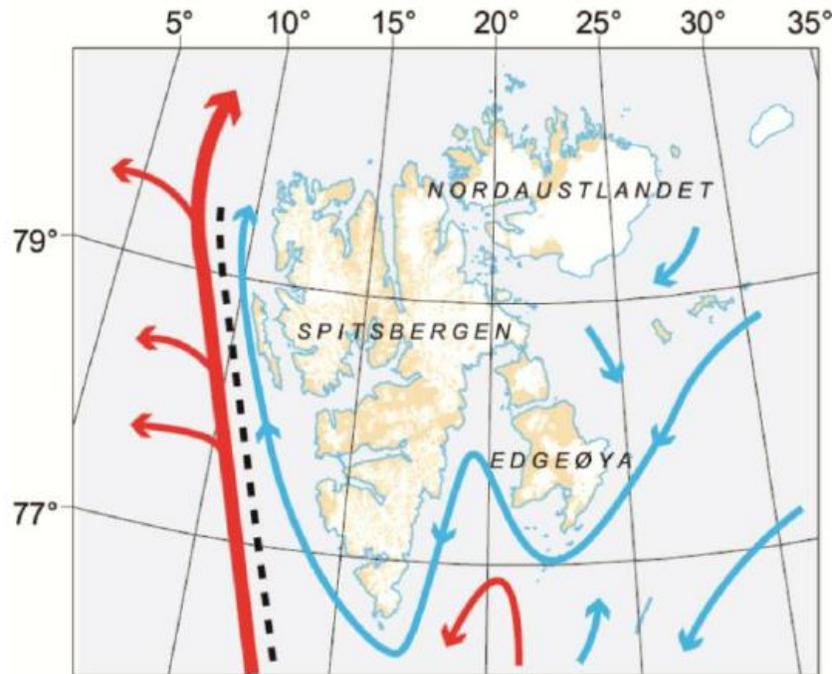


Figure 5 Map displaying main currents; the East Spitsbergen Current (Blue) and West Spitsbergen Current (Red). The dashed line indicates the frontal area of the two currents. Map from Svendsen et al. (2002)

Adventfjorden has a depth that varies from 50 to 80 meters and at the mouth reached 100 meters (Zajączkowski, Nygård, Hegseth, & Berge, 2010). Adventfjorden is underlain by steep, unstable slopes, as the glaciomarine sediments in the fjord are easily reworked (Weślowski, 2011). The deposition of sediments in the fjord flow in as glaciofluvial sediments and are deposited on the tidal mudflats or enters deep into the fjord and settle. Due to high hydrodynamic forces in the centre of the fjord, the sediments are easily reworked and suspended in the water column, they are easily visible as sediment plumes (Weślowski, 2011; Zajączkowski et al., 2010; Zajączkowski & Włodarska-Kowalczyk, 2007). Due to the Coriolis effect, the sedimentation may be occurring more on the right-hand side of the fjord (north) (Pawłowska et al., 2017; Hamblin & Carmack, 1978).

2.5 Climate and climate change

The average annual temperature of Adventdalen is -2.7 degrees C, measured as mean temperature the past 5 years (SeKlima, 2021). Wind directions in Adventdalen tend to be from inland out towards the fjord (Svendsen et al., 2002). At the broadest level, Svalbard is influenced by occasional storms, seasonal changes in light conditions and ocean currents and sea ice extent

(Svendsen et al., 2002). In winter, low pressure systems from Iceland and the high-pressure system from Greenland tend to pushing humid air from the North Atlantic Ocean over Svalbard. Temperatures are becoming increasingly warmer on Svalbard and the occurrence of sea-ice in winter is decreasing (SeKlima, 2021; Cryosmap, 2021; Hanssen-Bauer et al., 2019). For Adventfjorden, fast sea ice has not been observed in the fjord since 2005 (Zajączkowski et al., 2010).

3.0 Microplastic theory and literature review

The study of microplastic debris as a specific class of environmental litter has increased in scope and popularity, but relatively little is still known about its behaviour in the environment. In this section, microplastic as an environmental litter is presented, along with the mechanisms that transport and distributes microplastic from source to sink. The mechanisms that influence the behaviour of microplastics in the environment are highlighted, providing a foundation for understanding what affects the successes and failure of different methodologies for sampling and extraction of microplastics in sediments.

3.1.1 Plastic production

Raw plastic production was measured to be close to 370 million tons in 2019 and is continuously reaching new heights(PlasticsEurope, 2020). Plastic has gained a reputation in the past years for being a persistent and ubiquitous source of pollution in the environment, facilitated by its importance as a material for the global economy and a key foundation for numerous businesses and jobs. Due to the COVID-19 pandemic, the European production of plastics experienced a decline in 2020, but forecasts show that a re-stabilization in production in the future is likely (PlasticsEurope, 2020). At the same time, there is also a big push for limiting production and distribution of single-use plastics (EuropeanCommission, 2019). It is estimated that about 8 million tons of plastic pollution is entering the environment every year globally, and increased production and limited changes to behavioural patterns regarding waste management will tend to

increase the number of plastics entering the environment (PlasticsEurope, 2020; Hallanger, Gabrielsen, & Norsk, 2018; Lebreton et al., 2017; Galgani, Pham, & Reisser, 2017)

3.1.2 Pollution

The term pollution encompasses all human-produced material that is deliberately discarded or lost into the environment through inadequate waste management, and that which is not naturally removed or repurposed into nutrients (Muralikrishna & Manickam, 2017). Pollution can also mean the presence of an unnatural substance that is not human produced, but human induced. Plastic litter is an umbrella term for a large group of different polymers, which have gained much attention over the past decades as they are clearly visible in their macro form and ubiquitous from the tropics to the poles (Hallanger et al., 2018; Galgani et al., 2017). Microplastics which are between 1 micron and 5 mm in size (Frias & Nash, 2019), may be invisible to poorly visible, but are potentially even more harmful to the environment compared to macroplastics, given their small size (Goss, Jaskiel, & Rotjan, 2018; Fang et al., 2018; Critchell & Hoogenboom, 2018)

Microplastics are either industrially produced or byproducts of plastics and are deliberately or unintentionally discarded in the environment across the world (Galgani et al., 2017). Once released into the environment, microplastics are regarded as pollution as the environment lacks the ability to remove the substance naturally. Microplastics can be grouped based on two parameters, size fraction and origin. Defining the microplastics based on size fraction puts them in groups of large (1mm to 5mm) and small (<1mm). The second grouping of microplastics relates to their origin, either primary (that originally produced in the microplastic size range) or secondary (macroplastic litter that has subsequently degraded to microplastic) (Collard et al., 2021; Frias & Nash, 2019; Hallanger et al., 2018; Trevail, Kühn, Gabrielsen, & Polarinstitut, 2015) To better understand the pathways through which microplastics travel to get to the environment, it is important to address the differences between the groups of microplastic pollution

Primary microplastics originate from industry as intentionally produced particles that serve a purpose in industry and consumer products (Wang et al., 2019). The most commonly produced microplastics are used in what is referred to as Personal Care and Cosmetic Products (PCCP)

(Cheung & Fok, 2017). Plastics are cheap and easy to produce, which makes them ideal for mass production for consumer products. Cosmetic exfoliants, for example, have been identified as major sources of primary microplastic pollution (Napper, Bakir, Rowland, & Thompson, 2015). Due to their small size, such particles easily bypass sewage treatment plants and, in most cases, end up in the aquatic environment (Klein, Worch, & Knepper, 2015)

Secondary microplastics are a byproduct of degraded macroplastics (Cole, Lindeque, Halsband, & Galloway, 2011). Degradation of macroplastics occurs due to different stresses acting together, such as UV radiation and mechanical weathering. Ultraviolet radiation works by breaking down the polymeric structure of plastics (Hale, Seeley, La Guardia, Mai, & Zeng, 2020; Cole et al., 2011). This means that the chemical compound of the polymer is degraded, and the plastic material becomes brittle. Over time, this leads to fracturing and division of the plastic debris into smaller pieces and eventually microplastics (Barnes, Galgani, Thompson, & Barlaz, 2009). It is not only the UV degradation of macroplastic litter that creates secondary microplastics, although is a major driver of this process. Physical or mechanical abrasion through wave action, wind erosion and other mechanisms also contribute to the production of microplastics.

3.2 Transportation and deposition of microplastics

There are several means of transportation of microlitter that move the pollutant from source to the sinks, with sinks being in most cases, the marine environments. Atmospheric transportation moves the smallest and lightest of the particles across the globe (Chen, Feng, & Wang, 2020), while rivers are responsible for all sizes of macro and microplastic transport (van Emmerik & Schwarz, 2020). Between the terrestrial source and the marine environment, some plastic is lost in terrestrial sinks along the way (lakes, flood/overbank deposits next to rivers and streams, riverbeds, beaches, etc; (He et al., 2021)) There are also marine sources of macro and microplastic, including tourism, fishing, and shipping industries (Andrady, 2011). Each of these transport and depositional steps in different environmental compartments for microplastics are explored in further detail, below.

3.2.1 Transportation of and deposition of microplastics in fluvial and marine environments

Given their densities (typically from 0,9 to 1.5 g/cm³ (Campanale, Savino, Pojar, Massarelli, & Uricchio, 2020; Kooi & Koelmans, 2019)), the many positively buoyant microplastics float in water, and are most comparable to suspended sediment loads in rivers and streams in the clay size fraction of less than 2µm (Helmke, Koons, Schomberg, & Iskandar, 1977) or to wood fragments or leaves. Transportation of microplastics in fluvial systems is closely governed by density differences between the particle and the liquid in which the particle is situated in (Brooks et al., 2012). In general, if a particle has a lower settling velocity than the surrounding eddies' buoyancy velocity, the particle will be transported together with the sediments in suspended load of the river. Changing energy conditions, for example spring melt vs. winter freeze-up may result in deposition of the particles on the riverbed, in bars or as overbank deposits.

Particles that have a higher settling velocity than the natural buoyancy velocity of the surrounding water will naturally sink and become part of the bed load. The bed load defines the location in the fluvial river column where solids that have negatively buoyant properties are not in suspended load, but rather move along the riverbed or in series of long jumps which is referred to as saltation (Bagnold, 1973). Burial can also happen here if energy conditions suddenly decrease causing bedload transportation to stop and/or suspended load to come out of suspension.

Studies of sinks of microplastics in fluvial sediments show that deposition of microplastics depends on the density of the particle. Typically, microplastics with low-density are retained with the suspended load of the river and are transported over longer distances (e.g., the ocean). For the high-density microplastic particles, these tend to be deposited on the riverbed close to their source, depending on the flow velocity of the river (He et al., 2021).

In Arctic rivers, the influx of meltwater from the surrounding catchment can be very high. Rough riverbeds and uneven topography due underlying glacial and paraglacial sediments, as well as active slope processes during summer when the active layer has thawed, can create high levels turbulence. Such conditions decrease the likelihood of the deposition of microplastic on the riverbed and the possibility of riverbeds being sinks of for microplastics (Lebreton et al., 2017), although strong seasonal variations in energy levels (frozen winter vs melting summer) means

that finer-grained sediments and low density microplastics can get locked up in over bank deposits. That said, if there is a long-enough period between freeze-up and significant snow fall, the microplastic in the fine-grained over bank deposits may be further transported by aeolian processes (McKenna Neuman, 1993)

With high-density microplastics, these can sink and move down stream via saltation. However, some studies have shown that the saltation of microplastics increases mechanical abrasion, which alters the texture of the particle, decreases its density, and consequently moves the particle from bedload to suspended load (K. Zhang et al., 2016). On the other hand, the density of microplastics can also be altered via the process of biofilm accumulation on the polymer (Horton & Dixon, 2018). These processes confirm that microplastics do not have a constant density level throughout their lifetimes and therefore move differently in their environment over long timescales.

3.2.2 Microplastics in marine environments

Marine microplastics have the past decades been recorded in growing volume in the world's oceans (Clark et al., 2016). As mentioned in chapter 3.2 about pollution, microplastic pollution enters the marine environment due to inadequate waste management or deliberately discarded into the environment. Microplastics are buoyant and are readily transported and dispersed in the marine environment by ocean currents. Sinks form in the ocean, which for plastic pollution and microplastics usually refers to floating garbage islands (Ramos, Purba, Faizal, Mulyani, & Syamsuddin, 2018). Although microplastics are caught in floating garbage patches or float with the ocean currents around the world's ocean, large numbers of microplastic particles sink in the water column due to alterations to the buoyancy velocity of the microplastic particle and eventually end up on the seafloor (Barnes et al., 2009). More on the alterations of density and transport in the water column will be highlighted below in chapter 3.2.3.

Microplastics in Svalbard seabed has been found to vary from 560 particles/kg dry sediments to 230 ± 180 particles/kg dry sediments (Jensen & Bellec, 2019). Previous surveys on microplastic concentrations around Svalbard revealed a considerably higher amount in the Hausgarten project (53 to 810 particles/kg to 320 ± 330 particles/kg dry sediments). (Bergmann et al., 2017). This indicates variations in amounts, although the surveys focused on different fractions. The types of

microplastics that were found in the study by NGU with Jensen & Bellec (2019) and Hausgarten project with Bergmann et al. (2017) were typically PE (Polyethylene), Phenoxy resin and rubber resins together with other microplastics. This indicates that microplastics are present in the seabed in around Svalbard, though sizes vary but mostly found in fraction ranging from 10µm to 1mm.

3.2.3 Biofouling

Biofouling has been widely acknowledged as a process through which low-density microplastic particles become more dense and sticky, thereby facilitating flocculation (which can also increase density) and increasing the deposition potential of the particle (Hale et al., 2020; Goss et al., 2018; Kaiser, Kowalski, & Waniek, 2017). Biofouling is defined as the process in which a submerged material is colonized by organic material (bacteria or algae, for example) and hence its buoyancy is augmented (Kooi, Nes, Scheffer, & Koelmans, 2017; Kaiser et al., 2017).

Biofilm created by algal growth on the material will when the material experiences a negative buoyancy, settle in the water column (Ye & Andrady, 1991). In seawater, the difference in density of seawater varies in the water column, which influences the settling patterns of particles due to biofouling. There are therefore reasons to expect that microplastics in seawater are kept in a suspended state for a considerably long period of time (Kooi et al., 2017; Kaiser et al., 2017).

There are also studies showing that microplastics experience circular movements in the marine environment, namely a sink/resurface process. Ye and Andrady (1991) presented findings that show a fouling-defouling cycle of particles causing a negative buoyancy -positive buoyancy-sinking-resurfacing process (Fazey & Ryan, 2016). In terms of timescale, the fouling process is a function of the environment the microplastic particle is situated in and the type of polymer that the fouling process is working on (Kaiser et al., 2017). The residency time of a body of water and the polymer type therefore determine the potential for a specific microplastic particle to have for settling in a specific area.

As biofouling alters the buoyancy properties of a microplastic particle via colonisation of organic material as a biofilm, adhesion of sediment particles may also be beneficial for the transportation of microplastics in the water column (Collard et al., 2021; Li et al., 2020). Biofouling of microplastics floating in the water column may cause organic and inorganic particles (sediments) to be trapped by the sticky film, thereby altering the density and the vertical transport in the water column may increase (Wu et al., 2020).

3.2.4 Aeolian transportation of microplastics

Atmospheric and aeolian transportation of microplastic is poorly understood. Generally, atmospheric transportation of microplastic is regarded as an effective way to spread the smallest microplastics over greater distances from sources to remote sinks (e.g., alpine lakes, ice-caps etc.) (Wright, Ulke, Font, Chan, & Kelly, 2020; Y. Zhang, Gao, Kang, & Sillanpää, 2019). Some studies have demonstrated that wind erosion has the ability to pick and deposit microplastics in areas of wind eroded sediment catchments (Rezaei, Riksen, Sirjani, Sameni, & Geissen, 2019). Furthermore, when dry environments, the generally light density of microplastics and some morphologies (e.g., flake and fibre forms), allows for easy mobilization and transport during periods of strong winds, and microplastic particles may travel long distances before deposition when wind intensity declines (Bullard, Ockelford, O'Brien, & McKenna Neuman, 2021).

3.2.5 Sea-ice as temporal microplastic sink and means of transportation

Microplastics have been detected in sea-ice in the Arctic Ocean (Kanhai, Gardfeldt, Krumpen, Thompson, & O'Connor, 2020; Peeken et al., 2018; Obbard et al., 2014). Floating microplastic particles are picked up during sea-ice formation and are temporarily locked up in the ice masses (Obbard et al., 2014). Due to the major surface currents of the Arctic Ocean (in general water flows from the Pacific through the Bering Strait to the North Atlantic Ocean), the incorporation of microplastics in the sea-ice in the shallow coastal waters of Siberia, Alaska and NW Canada, allows for long distance transportation towards areas such as Svalbard and the Canadian Arctic Archipelago (Peeken et al., 2018; Obbard et al., 2014). Sea-ice has therefore been considered as a possible vehicle for microplastics in the high Arctic (Bergmann et al., 2017). During summer

melt, the sea-ice locked microplastic particles are released to the water column once more, and are consequently deposited into the environment (von Friesen et al., 2020). Finally, sea-ice deposited onshore in Svalbard may deliver microplastic particles picked up North of Svalbard and deposit the microplastic particles on land.

3.2.6 Sedimentation of microplastics

Behavioural patterns of microplastics and other microlitter are closely linked to the behaviour of organic matter in fluvial systems (Harris, 2020). Microplastic show similar characteristics to Particulate Organic Matter (POM) and may have the tendency to infiltrate sediments through pore spaces between sediment particles, and can also be re-suspended if disturbed and reworked in the sediment column due to bioturbation (Harris, 2020; Willis, Eriksen, Wilcox, & Hardesty, 2017). As such, it is reasonable to state that in case of deposition of microplastics in the benthic sediments, this does not necessarily mean a permanently settled state of a microplastic particle. Ongoing sedimentation can bury microplastics, incorporating them into the stratigraphic record, but as stated, microplastics can move further via pore-spaced migration (highly depended on the shape and size of the sediments and the microplastics), bioturbation, or disturbance, such as submarine landslides and current erosion (Harris, 2020). On the seabed, Benthic feeders can ingest and transport microplastics deeper in the sediments and also be the direct cause of burial of microplastic particles (Setälä, Lehtiniemi, Coppock, & Cole, 2018).

4.0 Methodologies applied in this thesis

The target size fraction for this study was set to 100µm to 5mm. This fraction was set based on the reasoning that sizes smaller than 100µm would be difficult to locate with a traditional stereoscopic microscope, and the time that would be spent on this was not allowed due to the timescale of the project. Methods for extracting microplastics from marine sediments also most commonly consider the size fraction (e.g., (Cadiou et al., 2020)) Below is a presentation of the different methods used in this thesis. A review of selected methodologies is found in Appendix (Fig.25)

4.1 Field

The sampling stations were divided into three stations, two of which are marine, and one terrestrial. The coordinates for the stations are listed in Table 1 on a map (Figure 1).

Station number	Coordinates
1	N78° 13.857 E015°40.327
2	N78°15.704 E015°35.124
3	N78° 12.350 E015°48.447

Table 1 Coordinates for sampling stations. Coordinates in Degrees Minutes Seconds

4.1.1 Fjord samples

On 28.09.2020, two sampling stations in Adventfjorden were visited (Figure 1). Sediment samples from the seabed (35-80m depth) were collected using a small Van Veen grab (250 cm², 3.14 litre; Figure 7) deployed from a Polarcirkel 1050 boat (dimensions: 1050cm long and 350 cm wide). Four samples were taken from each drop of the Van Veen grab, with 5 drops per site. Sediments were extracted from the top of the grab through four doors using a metal spoon and transferred to 290ml glass sample jars with screw-on metal lids. The jars were filled approximately half full. One extra sample of similar size from each station was collected and placed in plastic Ziplock bag for later grain-size analysis.

Between each sampling from the grab, the spoon was rinsed with fjord water to avoid cross-contamination. The grab was cleaned between drops by lowering and raising it in the fjord. During sampling of the grab, a glass jar labelled “blank” was opened and kept nearby to collect any background contamination from either our clothing or the boat. Samples in the labelled glass jars were immediately transferred to a cold storage facility at UNIS and kept at ca. 4 degrees C until they were analysed in the lab.



Figure 6 Van Veen Grab in closed position connected to the crane on Polaris, the research boat used in this study

4.1.2 Terrestrial sampling

Station 3 located on a subaerially exposed channel bed, which is part of the braided river system of Adventelva (Fig.8). A sampling quadrat of 1 square meter was measured with a wooden measuring stick and sediments from the surface and down approximately 2 cm were collected with a metal tablespoon (Fig.9) Sediments were immediately transferred to 290 ml glass jars, which were covered with aluminium foil before the lid was screwed on top. An extra sample from the station was collected for grain size analysis. As for the boat sampling, a blank jar was also used to capture background contamination from clothing, etc., during sampling.



Figure 7 Sampling area for glaciofluvial sediments. Material seen in the photo is ice.



Figure 8 Sampling of glaciofluvial sediments

4.1.3 Contamination samples

During collecting and processing of samples, the risk of contamination from airborne microplastic particles is an ever-present challenge. To help quantify potential background contamination, blanks were created in a couple of different ways depending on the situation and stage of the project. For the sampling stage, a selection of sample jars were used as designated blanks. A blank jar was left open during sampling to gather potential airborne particles. During the drying of the sediments, the same blank jars were left in the drying oven for the same purpose. The idea behind using the same type of jar as a blank is that it has the same potential of trapping airborne particles as the sample jars used for the sediments.

The risk of contamination is also present during the laboratory stages of microplastic studies (see section 4.2 below), due to airborne microplastics inside the laboratory space (Scopetani et al., 2020). As such, numerous blank Petrislides were placed throughout the workspace to gather any airborne particles that are present in the vicinity of where the different analyses were conducted. For the indoor contamination samples, Petrislides with lids (Merck Millipore PetriSlide®) and glass-fibre filters (2.7µm GF/D Glass Microfiber) were left labelled and uncovered for the duration of the different lab analyses. During work under the fume hood, two Petrislides® were placed on either side of the fume hood and soaked in deionized water (DI- H₂O). The same was done for during the filtration step.

4.2 Laboratory

4.2.1 Sample drying

Sediment samples from Adventfjorden and Adventelva were dried in a drying oven at 40°C for a minimum of 48 hours (Campanale et al., 2020; Lares, Ncibi, Sillanpää, & Sillanpää, 2019). At the end of each drying period, the tops of the glass sample jars were covered with new piece of aluminium foil, pre-cleaned with DI- H₂O. The dried samples were then stored in a refrigerated room until organic matter digestion, density floatation, and filtration could be done.

Three metal bowls were filled with extra sediments collected for grain-size analysis from each station and dried together with the microplastic sediment samples for the same duration. These samples were then stored together with the microplastics sediments while awaiting further processing.

4.2.2 Rehydration and digestion

After the drying, sediments had consolidated in the sample jars and therefore required rehydration with DI- H₂O, in order to proceed to the next step of organic matter digestion (OMD). The process of rehydration relied on a plastic squirt bottle with the DI- H₂O liquid and refilling of the sample jar with approximately 3-4 dl of this liquid. The sample jar was left uncovered for 10-15 minutes for the water to saturate into the sample sediments before a spoon was used to stir the samples and loosen up the sample for further processing.

OMD relies on adding a chemical to the sample which will oxidize the organic matter present, thereby removing it from the non-organic part of the sample (minerogenic sediments and microplastic). For this project 30-32% proof Hydrogen Peroxide (H₂O₂) was used for this step (Gewert, Ogonowski, Barth, & MacLeod, 2017).

For the process of digestion, a 500 ml Erlenmeyer glass flask was rinsed three times with DI- H₂O, dried, and filled with approximately 200ml of H₂O₂. A glass cork was put on top to seal the flask when not in use. A small lime glass 230mm Pasteur pipette with a rubber bulb was used to transfer the H₂O₂ from the flask to the sample. The reactions were conducted inside the fume hood. One to two pipettes of H₂O₂ were added to the samples. When no more reaction with the sample could be observed (i.e., bubbling) following the addition of H₂O₂ the reaction was considered to be complete. Samples were then covered with DI-H₂O rinsed aluminium foil, lids screwed on top, and stored in the refrigerated room awaiting until further processing.

4.2.3 Flotation

Density separation or flotation as it is also referred to, relies on the morphological properties of a particle and its difference in density to the liquid it is in and to the other particles present. For density separation, a saturated sample of NaCl solution was created by heat-stirring H₂O with 292.21 (58,44 g/mol) g of normal table salt from Jozo™. The amount of salt to the solution was chosen based on the molar mass of the solution which was desirable for the project. A 5 molar solution was the desired saturated solution which gives it a density of approximately 1,29 g/cm³(1,292g /1litre H₂O) Different molar densities were experimented with at this stage and are discussed the chapter 5.1.6. All solutions that were made for flotation purposes were vacuum filtered through a 0.27µm glass fibre filter. The NaCl solution was added to the sample jars such that it reached up the brim of the glass sample jar and shaken for one minute and thereafter left covered for 24 hours for sediments to settle and the lighter particles to float to the surface.

4.2.4 Decanting and filtering

Samples were carefully decanted into a Buchner funnel and filtered through a 0.27µm glass fibre filter with the water tap vacuum device running. A 50ml syringe was filled with the same NaCl solution and used to clean down the sides of the Buchner funnel to gather any potential particles that stuck to the sides of the Buchner funnel. Once all the liquid was filtered from the sample, the filter was placed on a petri slide, covered, and labelled with sample number and molar density of the NaCl used for density separation.

4.2.5 Microscopy

Microscopy is a common way of localising, identifying and quantifying microplastics in microplastic studies. There are a wide variety of microscopes that can be utilized for such a process. For this thesis, a Leica S9E stereoscopic microscope was used for localising, identifying, and quantifying potential microplastic particles on sample filters following the process of isolation and extraction steps were complete. The microscope was also used underway to identify the efficiency of the extraction process during extraction of spiked samples (see section 5.1.6).

4.2.6 Separation into species

In terms of identifying potential microplastic particles in the samples, a system of labelling the morphology and colour was created for consistency. The initial separation by morphology, with beads, oblong particles, fibres, and films. Beads include all particles with rounded morphologies. Oblong particles are rounded, but clearly oblong in shape. A fibre category included: long, thin, and rounded particles. Films included flat, long particles and clusters of flat particulates. If the particle had a weathered and splintered look, that was noted down as well. Particles were then differentiated by colour.

Separation of potential microplastic particles into species and by colour and morphology is necessary to map out potential microplastics on the glass fibres for speed and efficiency during the spectroscopy step (see section 4.2.8), where polymer type is determined. Detailed descriptions of the appearance of the microplastic particles also gives a good overview of what kind of particles are found in the area and may provide insight into the origin of the particles.

4.2.7 Contamination identification

Contamination is an ongoing challenge in microplastic studies. Every study of microplastics will face this challenge and individual development of methods for limiting contamination is common. Such methods include limiting clothing which can contaminate and extensive cleaning of sampling equipment and containers (Cutroneo et al., 2020). In this thesis, all stages, from field sampling to laboratory work, were carefully planned with respect to potential contamination of the samples. In the field, blanks were made to account for any airborne particle contamination of the samples during transferring of sediments to the sample jars.

Prior to sampling, all sample jars were rinsed with DI- H₂O in a series of three times before covered with aluminium foil and a lid screwed on shut. The sample jars were not opened until they were used in the field and left closed until needed in the lab. Blank samples were taken at all stages, ranging from blanks to sample filters

Identifying what is contamination and what is not, is not an easy process and is not deemed unproblematic (Scopetani et al., 2020; Cadiou et al., 2020). Contamination can include a wide

variety of sizes, morphologies, and colours of microplastics. For this project, the blanks were analysed to determine whether there was any contamination present in the nearby surroundings of the sample and sample jar once opened. On the aforementioned filter papers or in these blanks identification could be done. For proven contamination of microplastic particles, a Raman spectroscope or FTIR step also must be included to identify the compound of the particle. To account for microplastic contamination, particles of similar characteristics need to be tested to differentiate between contamination and non-contamination. (e.g., blue particles in contamination and field samples)

4.2.8 Raman Spectroscopy

Raman Spectroscopy has been widely used in determining the chemical composition of different materials as well for identifying chemical bonds in various materials (Meng et al., 2015). Raman Spectroscopy relies on the principle of the Raman effect which defines the light-scattering effect on a material (Staveley, 2016). The principle defines the change in wavelength from reflected photon radiation. Most of the reflected light from the material that is the same as the emitted laser is referred to as Rayleigh scattering, but the differences in wavelength that are detected by the CCD (Charged Couple-Device) corresponds to a material's chemical bonds also referred to as the Raman scatter (Lewis & Edwards, 2001; Brame, 1978). The peak in the resulting graph corresponds to the wavelength of the vibration of a specific chemical bond or single chemical. This data can then be utilized in acquiring a probability of resemblance to the signature of known chemicals. In the Raman software, databases of known chemical and prior tested material are available.

For this project, a Wintec Confocal Raman Spectroscopy held in the Department of Material Science and Engineering at NTNU was utilized in the testing of microplastic samples from Adventfjorden. Before a test was conducted with the Raman, the microplastic sample was examined to determine the location of the particle of interest on the filter. This was then marked with scratching the filter paper or using a blue pen on the white filter. It was then placed on the working table under the lens of the Raman and the particle was localized on the connected computer screen where a camera connected to the Raman lens was showing. The process started on a 10X zoom lens and after focus was acquired, the lens was changed to 50X zoom, and the software was adjusted accordingly to match the hardware. Once the camera was focused in and a

spot on the particle was selected as suitable for a test, the camera was closed off on the Raman spectroscope, and the lens was changed to a dark setting for the laser not to be reflected into the camera, as the laser will destroy the camera.

The databases connected to the software located in the Laboratory Raman computer was ST_Japan, a commercial database of over 3000 spectra from many different materials and a polymer database created at the laboratory for tested material of known chemical composition.

4.2.9 Sedigraph

A combination of manual analysis where samples were weighed before and after drying process and a Sedigraph, were used to determine grain size distributions for each of the three sampling stations (two from Adventfjorden and one from Adventelva). The grain size distributions of the sediment samples are important to show the differences in the sedimentary regimes. The Sedigraph, housed in the Department of Arctic Geology at UNIS, uses a combination of settling sediments in a constant environment and an x-ray beam to determine relative mass and distribution of particle sizes in a sample (Micromeritics, 2020).

The principle that makes it possible for the sedigraph to determine fraction in a solution of sediments builds on Stoke's law and X-ray absorption (Beer-Lambert law) (Micromeritics, 2020). Stoke's law defines the process of determining particle size distribution in a body of sediments by the process of sedimentation (Banerjee, Bhatkar, & Jain, 2017; Clifton, McDonald, Plater, & Oldfield, 1999). The second variable that is utilized to determine the size distribution of sediment particles in a solution is the Beer-Lambert's law that defines the absorption of X-radiation. In more definitive terms, the law explains the length of a beam in an absorbance medium from its emitting location (Swinehart, 1962).

The principle of the sedigraphs is the function of time and velocity of settling particles in a solution and the transmittance of the X-radiation beam from the cell to the receiver. The beam is sent through the sample and the length of the beam is measured and the hindered transmittance is recorded and calculated according to Beer and Lambert's law (Singer et al., 1988) The resulting data represents particle diameter and cumulative mass (Micromeritics, 2020; Swinehart, 1962)

5.0 Results: Methodological challenges and solutions

This section describes in detail of the challenges that arose during the different sampling and processing steps for extracting microplastics from the sediment samples from Adventfjorden and Adventdalen and describes the modifications that were made along the way to work around the various hurdles.

Adventfjorden: Sites 1 and 2:

5.1 Grab sampling of marine sediments

The main challenge with Van Veen grab was the early release of the locking mechanism due to a faulty winch. The winch was operated by a lever that was very sensitive to changes in pressure being put upon it. At times, the winch stopped unspooling due to uneven pressure loading on the lever, and the immediate stop of the unspooling pulled on the wire making the mechanism on the grab release. This was only discovered when the grab was hoisted to the surface. The other challenge that proved to make drops of the grab unsuccessful were occurrences the grab did not release at all.

The next challenge was that the boat had to be repositioned following each drop due to drifting with currents and wind. Luckily at the time of the sampling, there was only a small breeze. The boat had to be repositioned to counteract the drift, but it was not possible to reposition it perfectly to match the initial drop site, which meant the position and depth of the drop changed between drops. It is also important to note that the location of where the grab touched down was not exactly the position reported from the GPS on the boat as currents move the grab as it was lowered to the seabed. The challenges that appeared during this stage likely had no implications on the final results and all planned 60 samples were obtained.

5.1.2 Organic Matter Digestion

The samples from sites 1 and 2 (Fig.1) contained significant amounts of organic matter (fig.5), which needed to be removed prior to the microplastics analysis. The reason for this is that organic material can disrupt the identification process by either trapping potential microplastics or by increasing the possibility of misidentifying organics as microplastics. Some samples had macro-organic particles (e.g., bivalve, crab; Fig.9), which could be easily picked out with tweezers prior to digestion of micro-organic matter. During the stage of acid digestion, H_2O_2 was transferred from the Erlenmeyer glass flask to the samples via pipette and stirred with a glass rod. For some samples, the reaction was violent, even with only one drop of H_2O_2 , causing some of the samples to rise over the brim of the jar. Once the reaction ended, more H_2O_2 was added to the sample and this process was meant to be repeated until there was no reaction. For the samples from Adventfjorden, reactions to the H_2O_2 were still occurring after approximately 2 weeks of digestion, but limited laboratory time meant that no additional time could be spent on organic matter digestion and samples had to move on to the flotation step. This means that there was still some organic matter left in the samples, which would very likely float along with the microplastics during the flotation step and can also be very similar in appearance to plastic, especially when dried. For the samples that reacted violently to even very small amounts of H_2O_2 and overflowed their sample jars, there may have been some loss of microplastics.

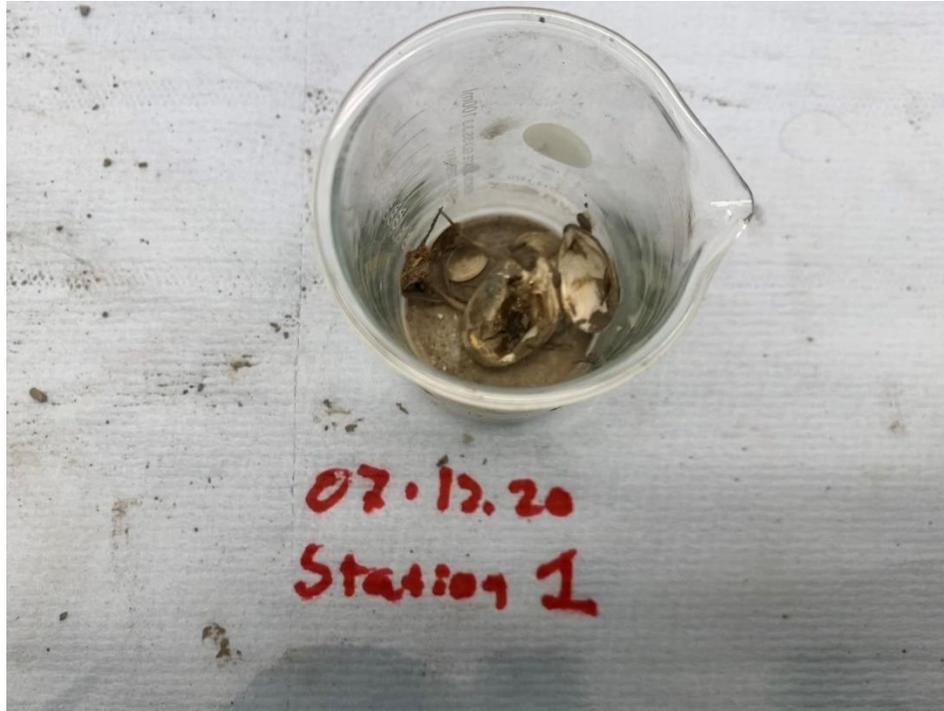


Figure 9 Macro organic material in glaciomarine sample 1. Picked out with tweezers.

Adventdalen: Site 3

5.1.3 Terrestrial sampling in Adventelva

Terrestrial sampling was easier than seabed sampling in terms of getting nicely grouped samples within a small area. Sampling in the early autumn along dried channels in riverbed of Adventdalen also proved to be successful, as the upper layer was soft, but the layers underneath had frozen, which made traversing the area and locating a good area to sample, relatively easy. Terrestrial sampling experiences share some of the same challenges as grab sampling from a boat, as the challenge of contamination is ever-present. At the time of the terrestrial sampling, a slight breeze was recorded which highlights the possibility of a potential contamination to have come from further in the valley.

5.1.4 Organic Matter Digestion

The samples from site 3 in Adventelva did not contain as much macro-organic matter as sites 1 and 2 in Adventfjorden (Fig.10), although some plant material was removed prior to flotation (Fig.11). H_2O_2 was added to the samples and digestion of organic matter progressed in the same

way as for samples from sites 1 and 2. The 20 samples from site 3 similarly continued to show reactions after two weeks, which led to the termination of the procedure in order to move on to the next step, which was flotation.



Figure 10 Organic material in glaciofluvial samples from site 3

5.1.5 Flocculation and deflocculants in samples from Adventelva

After agitation of the samples from site 3 in Adventelva during the stage of flotation step, visible change in the sample jar were clear. The samples had "grown" in size after the agitation of the sample jars. This means that the finer material that were settling slower than the coarser material were caught in a hindered settling state, they did not fully settle but remained in suspension. The stage in which the sediments were settling in can be referred to as the "hindered settled" state (Guo, Zhang, Song, & Wang, 2015). This state occurs during a settling stage of finer sediment particles, where the liquid in which the sediments are situated in is denser than the sediment

particles. Additionally, the container the sediments is situated in do does not settle and is continuously suspended in the solution.

During this stage, the hindered settlement of the sediments was initially thought to be flocculation of the sediments in the sample, due to the cavities that were created in the solution. More specifically, it was as if the clay particles formed a layer with air pockets. Clay particles adhere to each other by electrostatic forces which can be removed by deflocculation, allowing settling to occur. To combat this issue, a common treatment used to disperse clay particles within sediment solution was used. Sodium Hexametaphosphate (and can also be found in consumer cleaning agents such as Calgon) $(\text{NaPO}_3)_6$ was added to the samples and stirred in. This aqueous solution was created by adding 50g of $(\text{NaPO}_3)_6$ to 350ml of DI-H₂O and stirred on a heat-stirrer until dissolved. This solution had a density of 0,143 g/cm³. How this reacts with the NaCl that is already in the sample is uncertain but seems likely to not react or change the density of the solution. That said, in this case the addition of Sodium-Hexametaphosphate did not improve the settling of the particles, which suggests that it either increased the density to a value greater than that of flocculated clay particles, or it did not work in terms of dispersing them.

5.1.6 Flotation

Flotation relies on the utilization of a dense liquid to create a means for buoyant particles to rise to the surface of the solution and then be separated via a decanting of the heavy liquid from the heavier particles, which settles out after some time. Naturally, this dense liquid should be denser than the particles of interest, the microplastics, but not denser than the minerogenic sediment. The liquid utilized for this thesis step is recommended to be $>1.2 \text{ g/cm}^3$ for flotation to be successful (Cadiou et al., 2020; Dekiff, Remy, Klasmeier, & Fries, 2014). A flotation liquid of similar density (1.2 g/cm^3) was created and initially tested samples that were spiked with a known amount of microplastic particles. The microplastic particles added to the test samples were made by filing down red plastic push pin (most likely High-density Polyethylene) into shavings of $<500\mu\text{m}$. A known quantity of shavings (8-20 particles) was added to the test sample consisting of sediment from the glaciofluvial site, agitated vigorously in the heavy liquid for 1 minute and left for 24 hours to settle. The microplastics were not visible after 24 hours and it was therefore determined that the flotation liquid was not dense enough. To combat this challenge, a

strong dense liquid of NaCl was made. It was decided upon to make a 5-molar solution, which saturates the liquid with NaCl to the point in which no more can be dissolved at room temperature. The 5-molar solution required 292.21 g of NaCl (normal table salt) per 1 litre of distilled water. The same procedure of adding dense liquid, agitating the sample, and filtering was applied to the new test samples, using the 5-molar solution. This time, spikes could be seen floating on the surface of the dense liquid and they could therefore be retrieved from the test samples.

Following flotation, the spiked samples were filtered and microplastic particles counted to determine the percent loss of microplastic during the flotation and filtering steps. The challenge of utilizing a denser liquid for flotation is that many non-microplastic particles (e.g., organic matter, clay), which are not relevant to this study will be caught during the flotation step. This is an issue that was experienced to a high degree in this project and is discussed further in this section.

A post-organic matter digestion, pre-flotation step of sieving with a 63 μ m mesh was tested in an attempt to minimize the problem of accumulating too many non-plastic particles during flotation in the heavy liquid. Here, all particles larger than 63 μ m were kept while those less than 63 μ m were disposed of. This step greatly improved the flotation of microplastics and settlement of denser liquid. A drawback to this additional step was the increased potential to contaminate the sample as the sieving was done in an open area with the showerhead from a tap. This open area was the same lab as the previous stages, and blanks were placed near the sieving area and left out for the entire procedure. The possibility of microplastic contamination from the showerhead on the tap was considered, and a litre of water was sampled, filtered, and analysed. Analysing these filters turned up no particles in the targeted size range of 100 μ m – 5 mm.

5.1.7 Filtration

The procedure of filtration relies on the decanting of dense liquid with potential microplastic particles into a Buchner funnel onto glass fibre filter. Using a vacuum supplied by a water tap vacuum device, the heavy decanted liquid was pulled through the glass microfibre filters, sample by sample. The challenge that was encountered at this stage was that due to the amounts of

sediments that did not settle, decanting of the dense liquid occasionally included small to large amounts of clay. This covered the filter with sediments and made analysis of the filter in microscope later impossible at a later stage.

A post flotation procedure was developed to ensure that fewer non-microplastic particles were caught onto the filter papers. The developed procedure consisted of placing an open sample jar into a rinsed 2 litre glass beaker. A 50 ml syringe was filled with the same NaCl solution and was carefully emptied in the sample glass jar, until an overflowing of the sample jar was visible.

Some of the dense liquid in the sample glass jar was decanted straight into the 2-litre glass beaker carefully not to include micro-sediments. Following this step, the dense liquid in the glass beaker was decanted into the Buchner funnel and filtered with vacuum. Using the syringe, the glass beaker was rinsed into the filter to gather any potential leftover microplastic particles. Still sediments were caught in the filter paper, although notably less than in the previous filtration, which may be the result of leftover flocculated clay clusters in the sample that were not possible to remove during that sieving stage.

5.1.8 Raman Spectroscopy

Raman spectroscopy was decided upon as the preferred means of the analysis of the chemical compound of the suspected microplastics in the samples. This decision was primarily based on access to a database of tested polymers located in the software of the Raman. Locating particles of interest to test was done on a dissecting microscope at hand next to the Raman microscope. All samples which contained particles approved for testing were marked with a needle or a pen in the filter for easy detection in the Raman.

Setting up the Raman for analysis of a sample, the 10x EC Epiphlan lens with a working distance of 10mm was chosen for easier detection. Once the particle was detected and focused on, changing to 50x Epiphlan Neofluar dic (microscope lens) with a working distance of 9,1mm was done for finding the location on the particle for testing. The following settings were set for all sample testing with some changes done depending on sample test outcome. The integration time was set to 10 seconds, which states the time the spectrometer is measuring photons. The Accumulations were set to 6. This tells the spectrometer to do photon measurement for 10 seconds in 6 different accumulation periods, which sets the time for analysis to 60 seconds.

These 6 accumulations are combined to produce one spectrum. The laser intensity was set to a low 10 mW (Milliwatt) to not accidentally burn through the material before spectra could be recorded. Fluorescence is a challenging hurdle that may in some cases disrupt the reading of chemical signature in a material; to combat this issue the Raman has a function called Oscilloscope. The Oscilloscope function was used for most samples to alleviate fluorescence. Doing this for some particles, at high intensity (higher than 10mW) proved to burn through the material as the material did not have the capability to withstanding such intensities (See Fig. 23 in chapter 6).

5.1.9 Sedigraph complication

As will be stated in chapter 6.1.5, the first run of the sediment samples in the sedigraph proved to include some complications which rendered the data from the analysis unusable. Most of the sediments from the marine stations ran with no problems and data could be retrieved, however, for the terrestrial some complications arose as the sedigraph experienced problems related to clogging. The sedigraph has a maximum grain size of 300 μm , any over that and the machine may get clogged. Uncareful work with the sedigraph and through the adding of sediments that were not sieved appropriately to separate the particles larger than 300 μm clogged up the machine and a full system clean had to be done. Following this, a new protocol was produced for sediment grain size analysis.

The new protocol to alleviate the problems met in the first run of the sedigraph included a combination of sedigraph and manual analysis. The sediments were accordingly sieved with a 250 μm sieve to separate the two size classes of sediments: >250 μm (anything courser than fine sand) and <250 μm (fine sand, silt, and clay). Following the sieving, organic matter digestion was applied to the sediments (as for microplastic samples) and left for a week with an occasional top-up of H_2O_2 until reaction in the flask stopped. The process of rinsing the samples with DI- H_2O , centrifuging, decanting the liquid, and repeating this process four times was applied, although some of the sediment particles were so fine that they did not settle, even after centrifuging for 5 minutes at 5000RPM. Following the rinsing, the samples were left for 48 hours to properly settle. Once the sediments had been cleaned, some DI- H_2O was left in the samples they were followed up with the addition of the deflocculant Sodium hexametaphosphate ($(\text{NaPO}_3)_6$) The deflocculant was added to the samples and stirred vigorously for 1 minute and left for 24 hours

until samples could be used for sediment analysis. The larger particles were then weighed in a wet state and the beaker was subtracted from the total weight. They were then left in an oven for 24 hours at 45 degrees Celsius for the water to evaporate from the samples. The samples were weighed again, and the beaker weight was subtracted from the total weight.

For the second run of the sedigraph, the sediments had been treated and prepared according to the protocol. The machine was left running for at least two hours before analysis, and the samples were added to beakers marked according to sample and sieve size to avoid further clogging of the machine. The sample was then added to the sample cylinder on the sedigraph and loaded into the machine. The sedigraph pumps the sediments into a suspension in the sample window and directs them in front of the X-ray beam. The sediment solution was altered according to the K counts from the machine to be within 50 and 105 for optimal reading. Too low and the solution need more water, too high and the solution needs more sediments. The size fraction that was set as the baseline for the machine was 250 μ m to 0.18 μ m.

The results from the sedigraph presented here rely on the fraction set by the sedigraph and its proposed diameter on particles and cumulative percentage outside of the set fraction. For the two marine samples that were tested, the sediment liquid to sediment ratio was satisfactory for good reading. This was not present in the testing of the glaciofluvial sediments from site 3 as the solution, was too thin. This means that there were not enough sediments in the solution. For the glaciofluvial sediments, the percentage from 5 μ m and smaller showed a negative percentage. This is likely indicating that there is too much water content in the sample. All data from 0% and negative has been disregarded and values changed to NULL for the creation of the plot, thus renders the data for the glaciofluvial site unusable. The data is still shown in the graph figure 18 in the next chapter.

6.0 Results: Microplastics

In the previous two chapters, both the methods and detailed description of problems encountered throughout the collection and processing of samples are described in detail, including the necessary modifications made to the methods in order to successfully collect, extract, and quantify microplastics from the study areas. Although the modifications made to some of these

oft-used methods represent some of the most important results from this thesis, this section describes the characteristics and quantities of microplastics discovered in the samples from the study sites. Numbers of potential microplastic particles and Raman spectroscopy confirmed microplastics are present. Particle size analysis of sediments from three sample sites are also presented in section 6.1.5.

6.1 Particles observed (pre-Raman screening)

Adventdalen: Site 3

Nineteen out of 20 glaciofluvial sediments samples from site 3 were analysed in full and therefore form the largest part of the dataset presented in this thesis (Table.3). One sample (sample 5; table 3) was broken during the laboratory stage and was discarded, while another (sample;19, table 3) ended up with too much sediment on one of the two filter that did not allow for the counting of microplastics. The total number of particles suspected to be microplastics based on their appearance under the microscope was 202 (Table 3). The 202 potential microplastic particles were divided into 13 groups based their different morphologies and colours (Fig.11). In the bead group, the blue and yellow beads were the most common. Red, orange, and white beads were also found, but not more than 1-3 per group. The fibre group was dominated by blue and transparent fibres, which were found in almost all the samples. Black fibres were also observed in most samples, which ranging from 1-5 particles per sample. Red and yellow fibres were observed in smaller numbers, ca. 1-3 particles per sample. For the group of films, yellow films were the most common, one transparent film particle was also found observed.

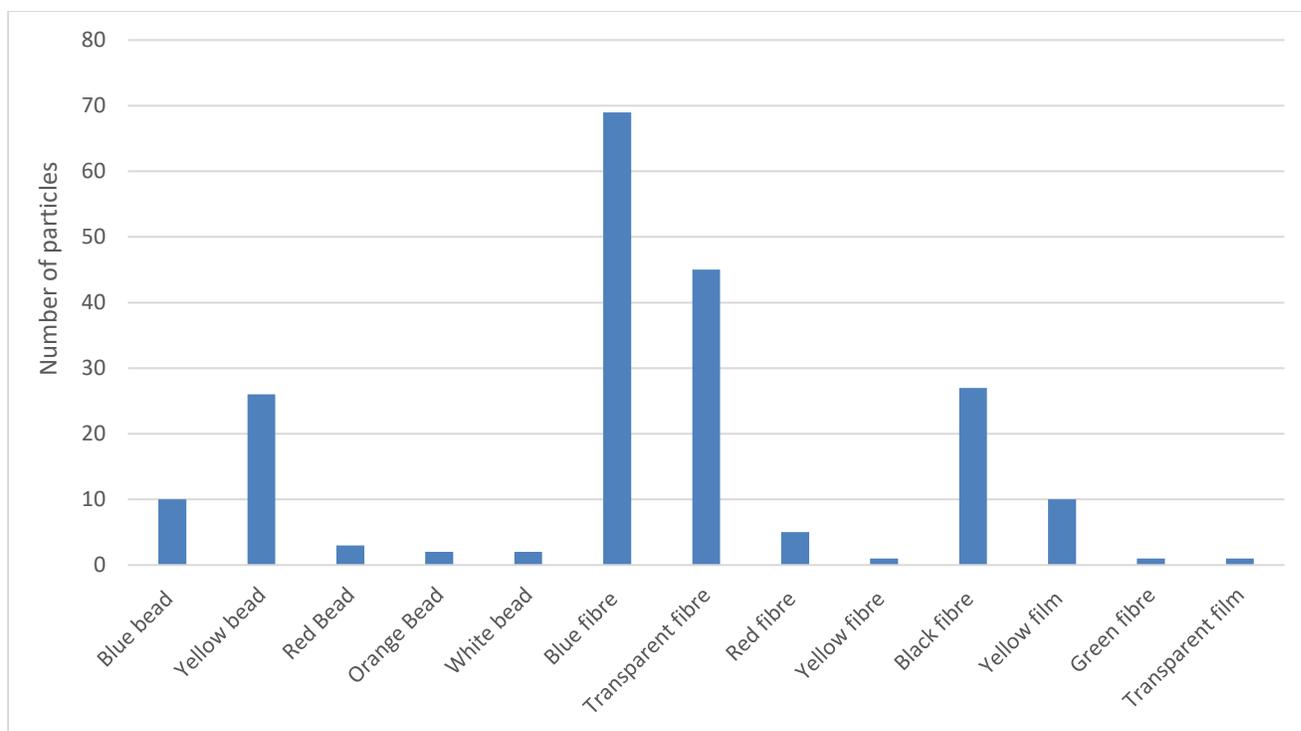


Figure 11 Amount of possible (i.e., pre-Raman confirmation) microplastic particles per group from site 3

Adventfjorden: sites 1 and 2

Six samples of distal glaciomarine sediments from Adventfjorden (Fig.2) were examined under the microscope for potential microplastics. Three of the samples are from site 1, and three are from site 2 (Table 3). Potential microplastic particle identification and counting of the filters under the microscope revealed 21 particles from site 1 and 27 particles from site 2 (Table 3). The most common type of particle observed in site 1 samples included black and blue fibres, the same was included in site 2. Blue fibres with 19 particles were detected and 16 black fibres were detected (Fig. 12). Yellow beads with similar characteristics as for the glaciofluvial sediments was located and counted. 4 yellow beads were located, and 1 red bead was found. As for the remaining particles, transparent, yellow, and red fibres were counted, totalling 48 particles for site 1 and 2, respectively. As for films, one yellow particle was observed.

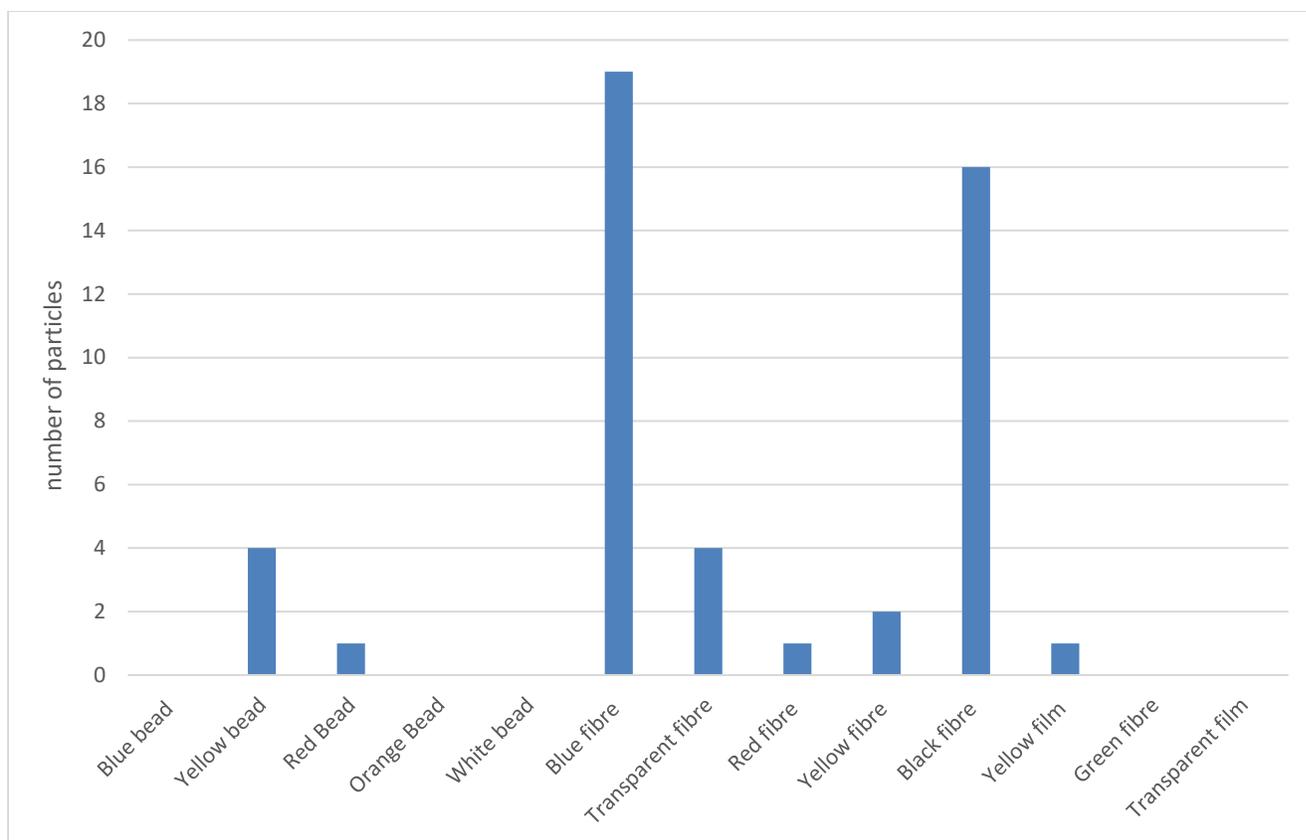


Figure 12 Amount of possible (i.e., pre-Raman confirmation) microplastic particles per group from site 1 and 2

6.1.2 Contamination samples

From the contamination samples placed in the laboratory during procedures, blanks from the marine and terrestrial sampling were tested. The jars labelled blank did not return any contamination particles. The Petrislides from the laboratory procedures were tested and mostly red and blue fibres were located. These fibres were tested in the Raman spectroscope, which will be highlighted in chapter 6.14.

6.1.3 Spiked samples

Four spiked samples from sites 3 and 1 were prepared in order to test the retrieval rate of particles following digestion of organic matter, density separation and filtration. Three of the samples that were tested with spikes came from the glaciofluvial site (from site 3), and one fjord

seabed sediment samples was spiked (from site 1). Particles retrieved from first filtration pre sieving were added to the number of particles retrieved post sieving. The samples with glaciofluvial sediments (site 3) had a retrieval rate varying from 40% to 87%. For the only spiked sample from site 1, 26% was retrieved post flotation and filtering.

Site	Sample no	Amount spiked	Retrieved	Retrieval%
3	2	8	7	87.5
	10	15	11	73
	11	20	8	40
1	39	39	10	26

Table 2 The table show the samples labelled by the number they were assigned. The number of particles that they were spiked with and how many were retrieved. The retrieval percentage is also presented in this table

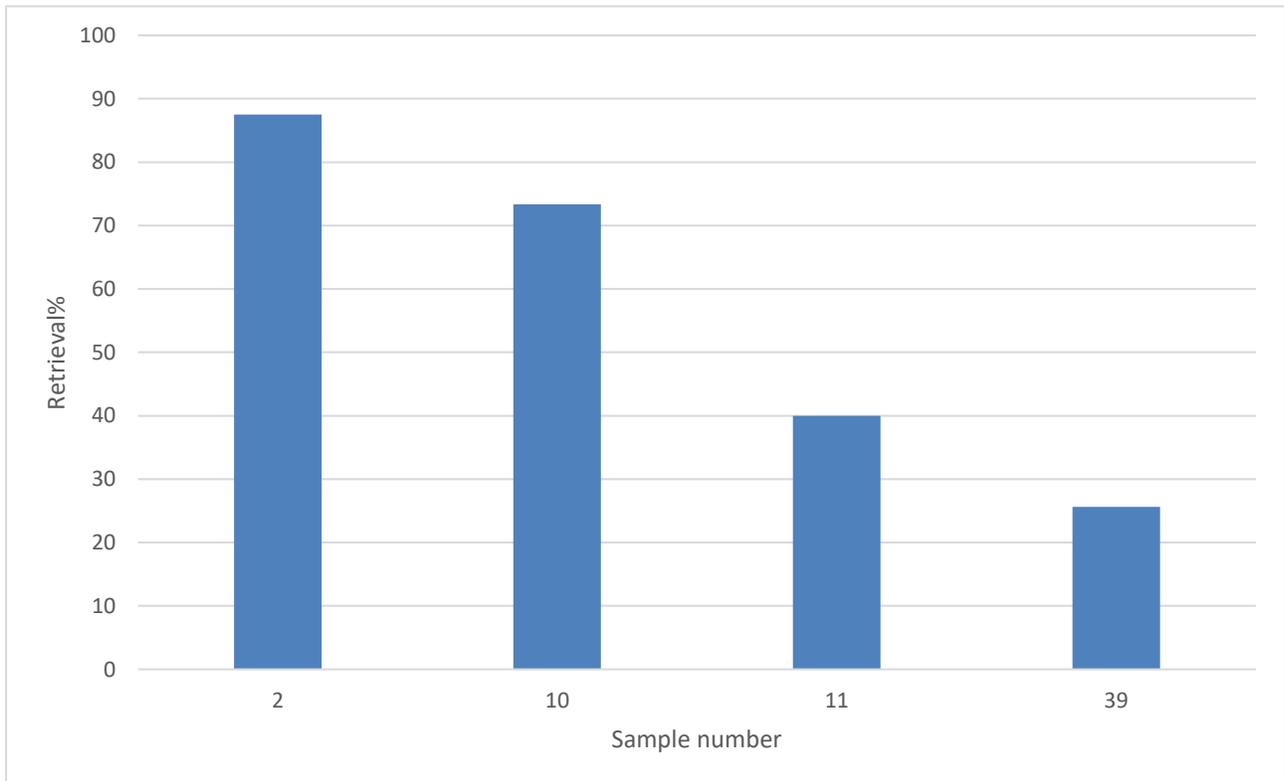


Figure 13 Retrieval in percentage of spiked samples. Data shown in table 2

6.1.4 Raman results

Potential representative microplastic particles from the different colour-morphology classes (Approximately 3 of each) from 8 samples from site 3 were analysed using NTNU's Raman spectroscope. Three blue fragments (Fig.14) were tested for chemical signatures and revealed a spectrum (Fig.15) which has a 27.86% similarity to Phthalocyanine Blue ($C_{32}H_{16}CuN_8$). Based on the ST_Japan database. Phthalocyanine Blue is mostly found in paint and plastics as an insoluble pigment. A second blue particle from sample 1 was tested (Fig.16) and revealed a close similarity to the first particle (Fig.17). Three blue fragments (beads) were tested in all, and all revealed between 25-31% similarities in chemical composition to Phthalocyanine Blue.

Of the blue fibres tested, sample number 3 (Fig. 20 and 21) from site 3 showed a 77.60% similarity to Zexlon. Two-three of the blue fibres from site 3 are likely organic matter as they had signatures (44-66% similarity) to Diamino Benzene ($C_6H_6Cl_2N_2$) and Rutin ($C_{27}H_{30}O_{16}$), both of which are organics.

One sample of yellow bead from sample number 18 (site 3), revealed a spectrum of 33.60% similarity to Ethyl trimethylammonium chloride ($C_5H_{14}ClN$) (Fig. X). One yellow fibre from sample 9 (site 3) showed a 31.36% similarity to Phenyl fluorene ($C_{19}H_{14}$).

Some yellow beads and all black fibres proved to be impossible to get a spectrum from with Raman spectroscopy. Either the laser burned through these particles at low intensity (Fig. 22 and 23) or was unable to get a reading from due to high fluorescence. Readings are not possible to retrieve in cases of burning of the material as the material is burned and destroyed before the accumulation of reflected signal is done.

From the contamination samples, the red and blue fibres were tested and revealed spectra with similarities to Algin, Gellan Gum, which are organic.

For the Raman spectroscope 14 samples were tested from site 1, 2 and 3, in addition to contamination samples from two labs. Of the 14 site samples; sample 1, 3, 9 and 18 showed similarities to chemicals most commonly used in production of plastic products. For the samples that contained particles that showed indications to be organics, sample 45, 47, the lab

contamination sample and sample 10 had similarities to organics. From the Raman testing 8 out of 14 samples showed results. The remaining 14 samples were either burned or did not reveal any readings.



Figure 14 Picture of blue bead containing Phthalocyanine Blue in sample 1 from site 3

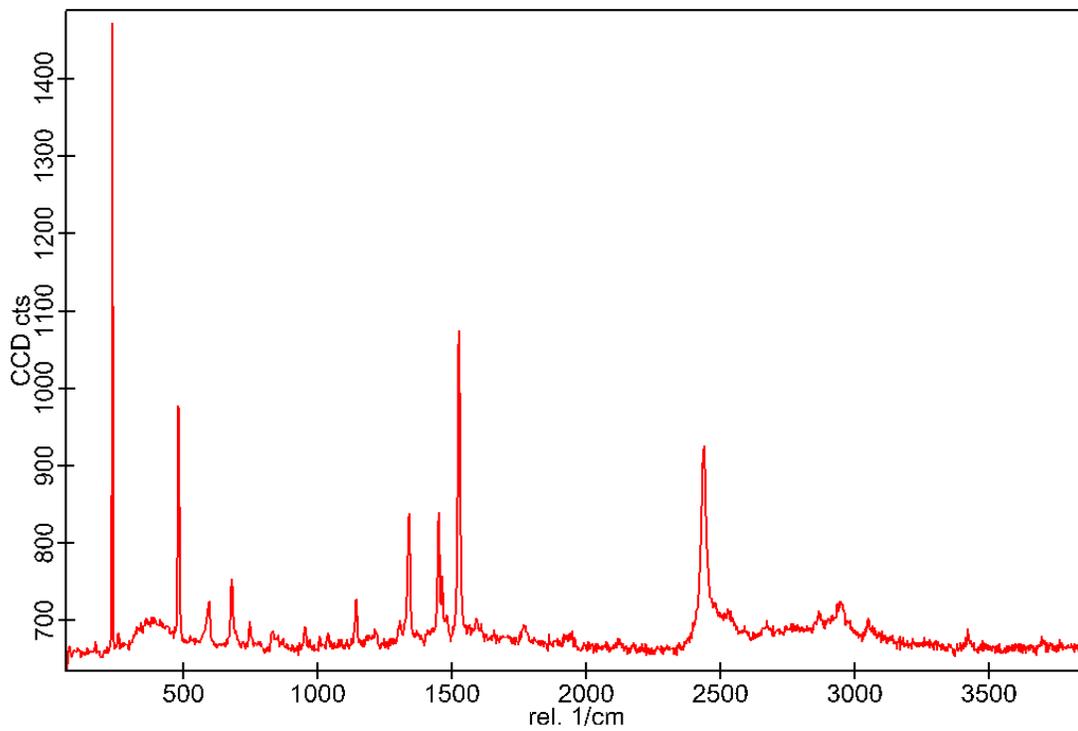


Figure 15 Raman spectra of blue bead in sample no.1. TR1_Particle1

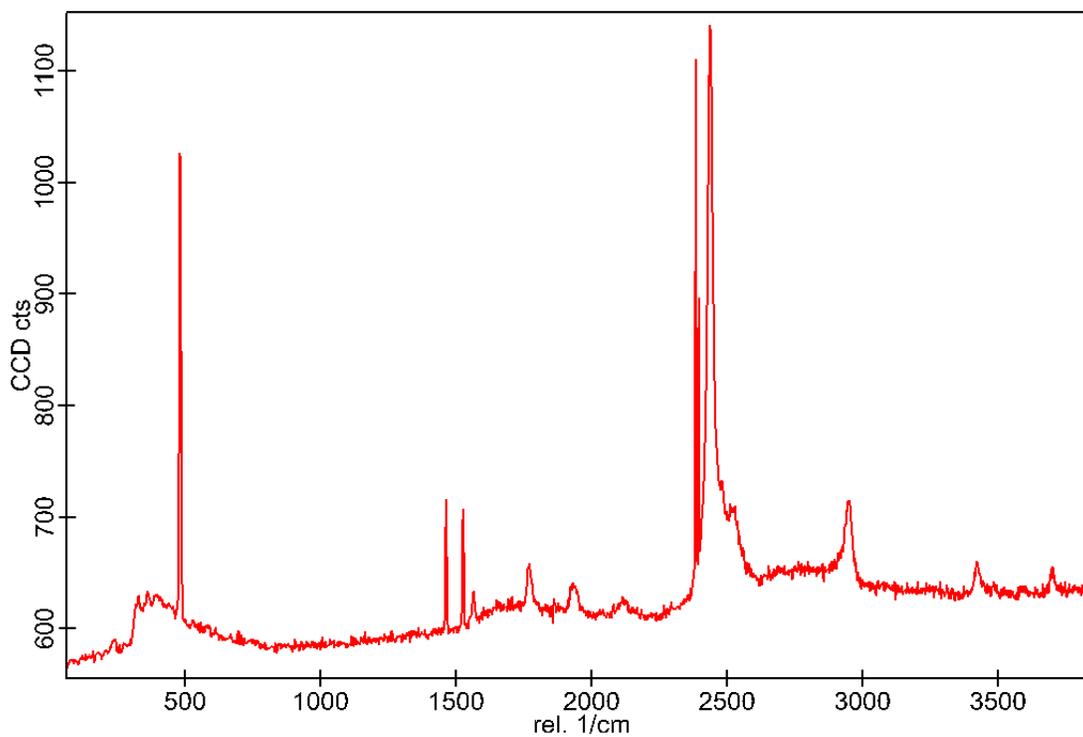


Figure 16 Raman Spectra of second blue bead in sample no.1 TR1_Particle2

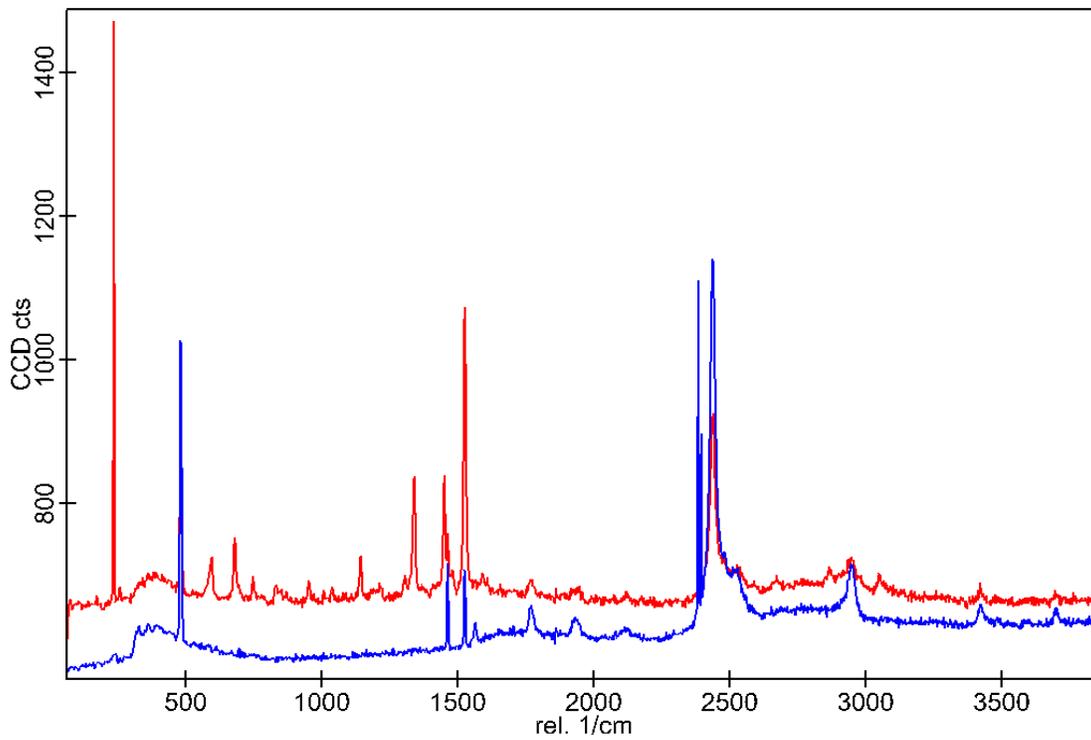


Figure 17 Comparison of spectra from TR1_Particle1 and TR1_Particle2

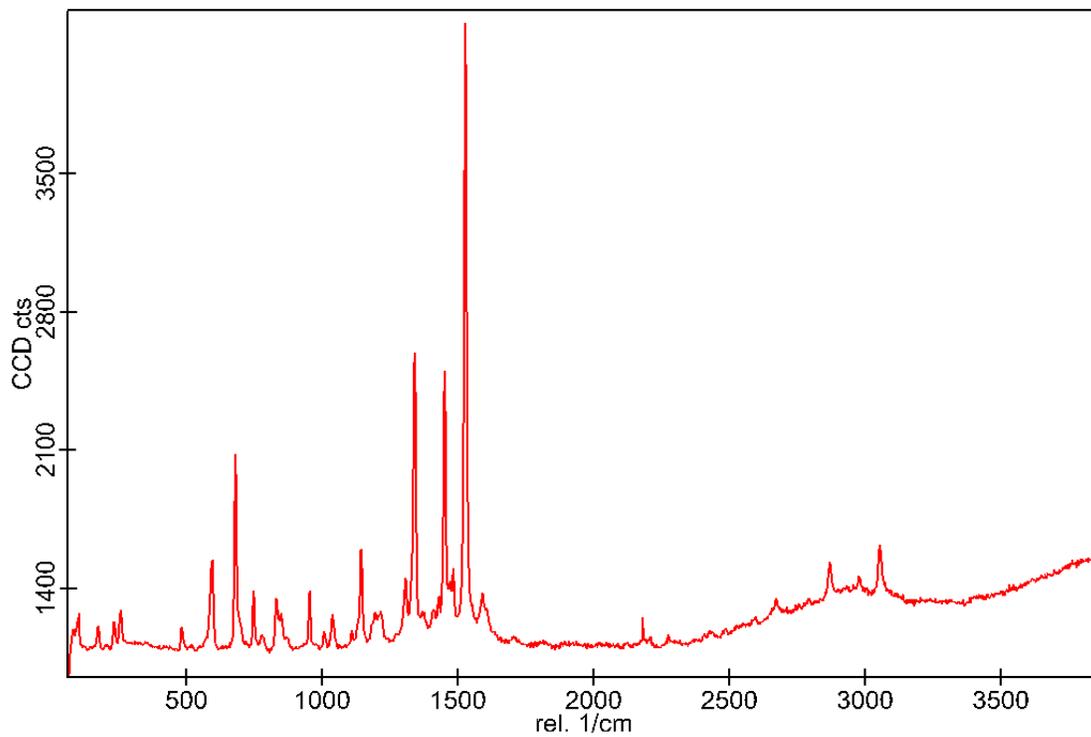


Figure 18 Raman spectra of blue bead in sample no15. TR15_Particle3

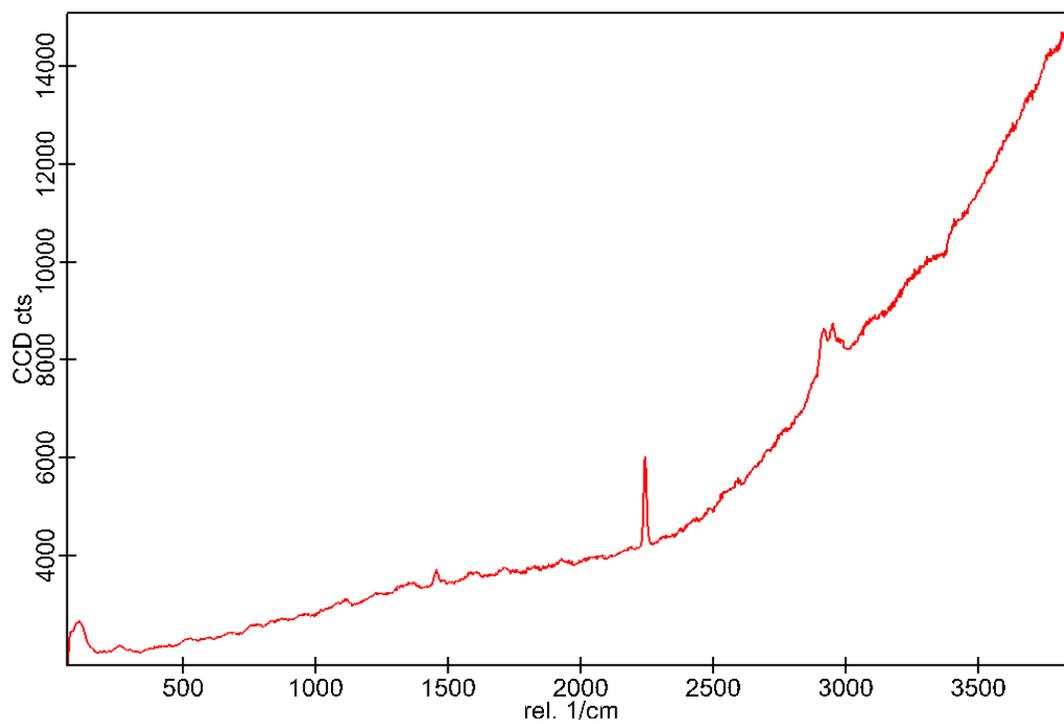


Figure 19 Raman spectra of blue fibre from sample no3. TR3_Particle1



Figure 20 Blue fibre from sample no 3. TR3_Particle1

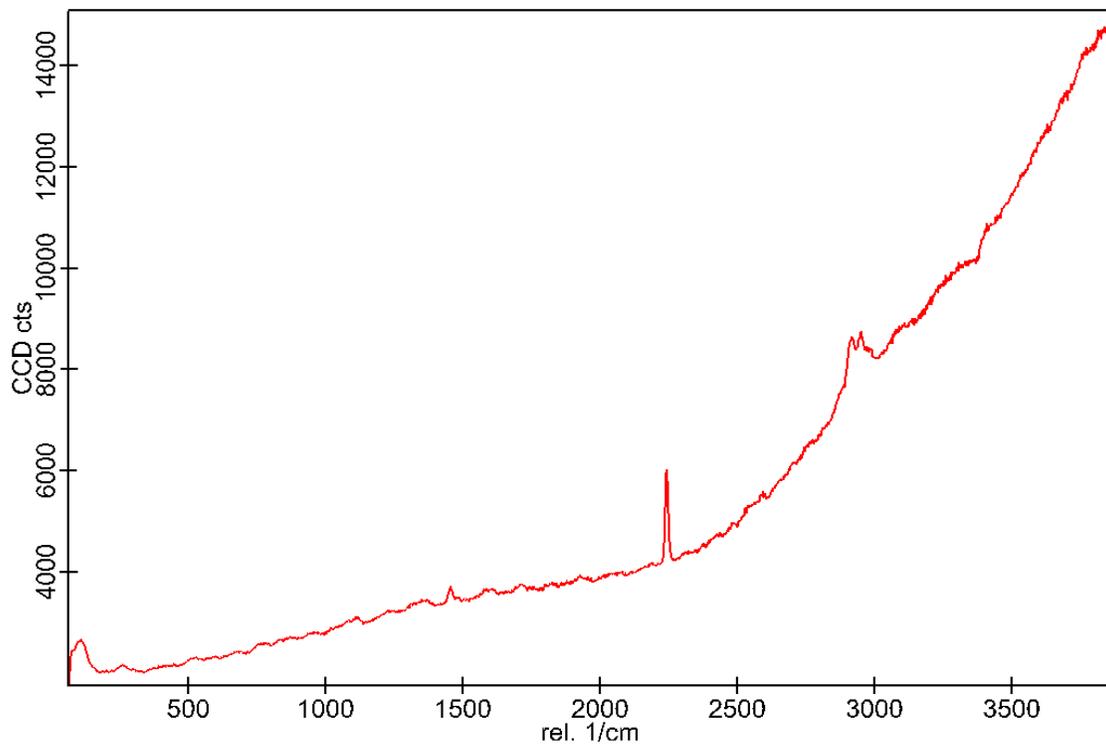


Figure 21 Raman Spectra of blue fibre in sample no.3. TR3_Particle1

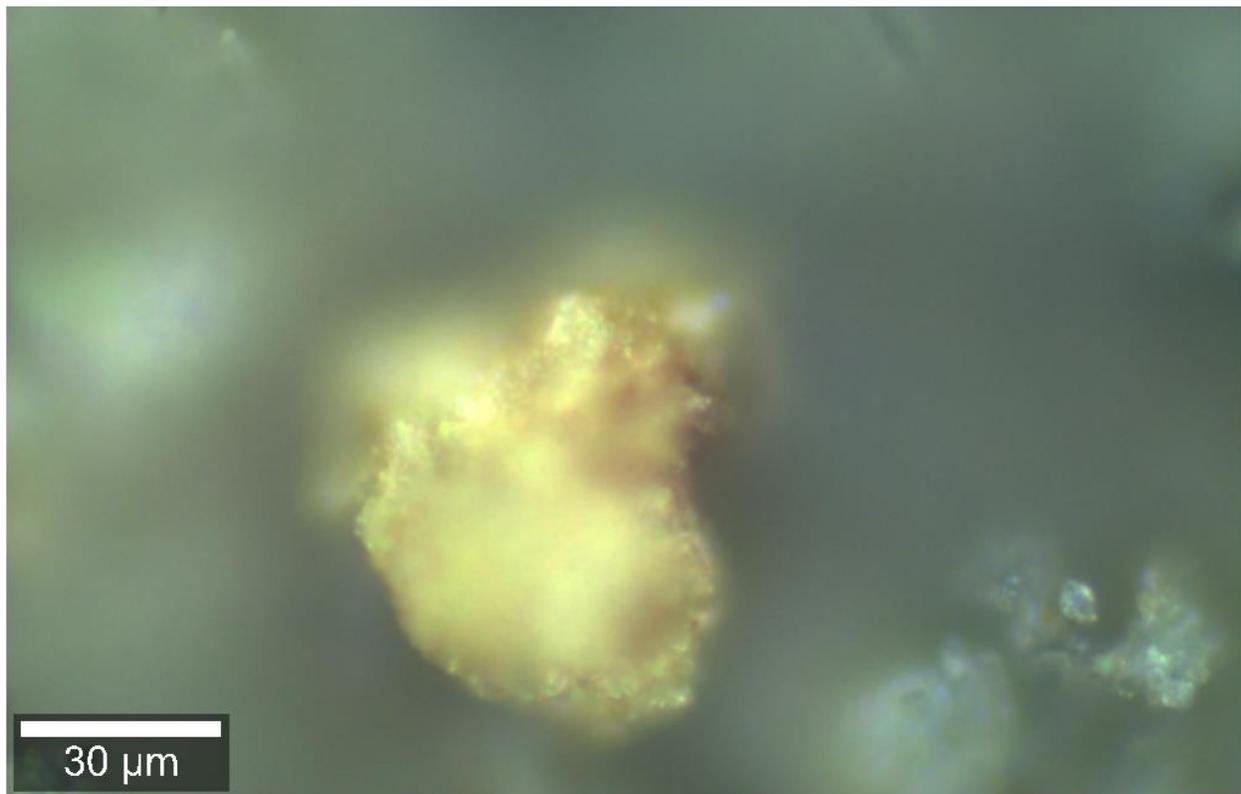


Figure 22 Yellow particle before Raman test

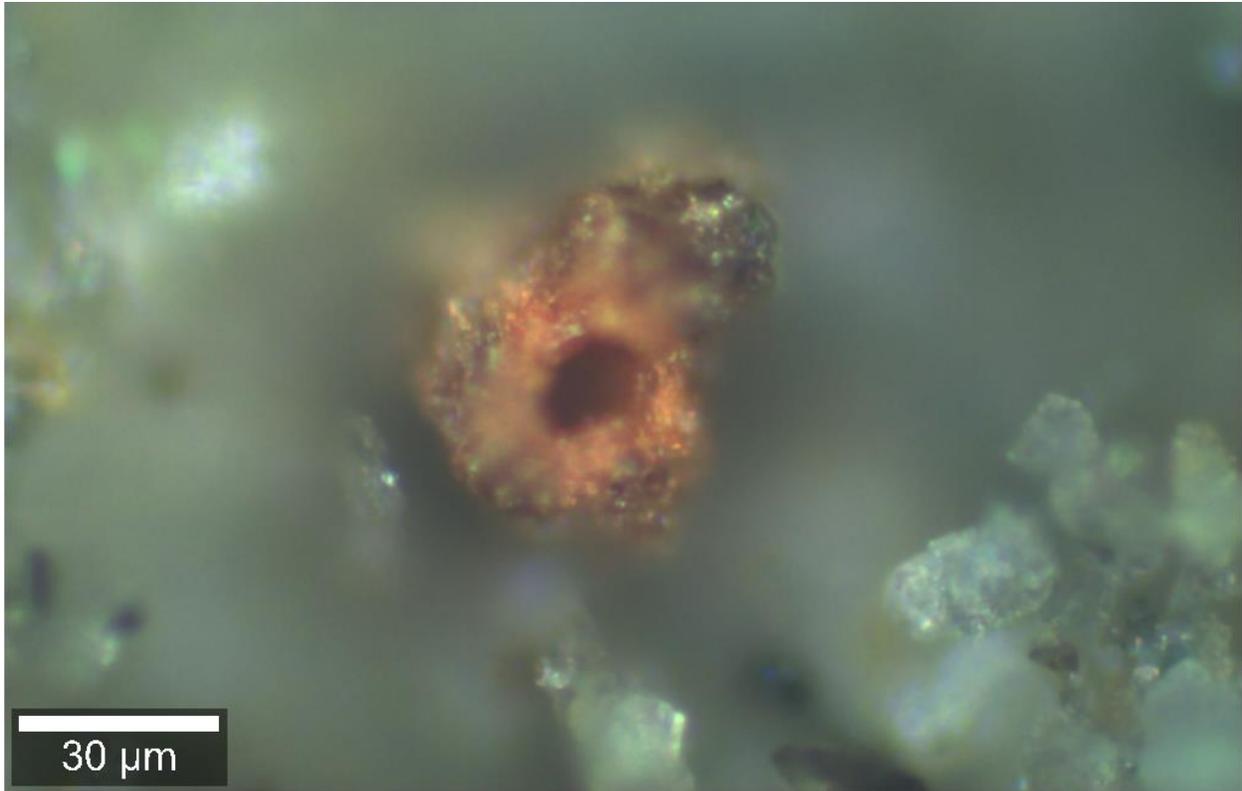


Figure 23 Yellow particle after Raman test. No reading possible due to burning of material.

6.1.5 Sediment grain size analysis

Grain size analysis for one representative sample from each of the three study sites is presented below (Fig. 24). The Sedigraph is not able to measure particles under $0,18\mu\text{m}$ but can calculate the probability of sizes. The results show the lowest and the highest size that was possible for the machine to calculate and cumulative finer mass percentage. The cumulative finer mass percent shows the percentage of sediment particles that are smaller than a given size.

Sediments from sites 1 and 2 consist of approximately 25% and 34% particle sizes that are $1\mu\text{m}$ and smaller (i.e., clay sized particles) (Fig. 24). All samples have a cumulative finer mass percent of 80-95% for sizes larger than $100\mu\text{m}$ (i.e., coarser than very fine sand). Sites 1 and 2 show a large distribution of finer particles than site 3, which consists of nothing finer than very fine silt.

The sediment fraction larger than $250\mu\text{m}$ (i.e., larger than fine sand) is not available in full due to loss of registered data. This data loss was due to a technical error on the computer the data was stored on with a forced restart incurring the data loss. The time schedule of the project did not

allow for redoing of the sediment grain analysis. Therefore, only the data for $<250\mu\text{m}$ is displayed in this paper.

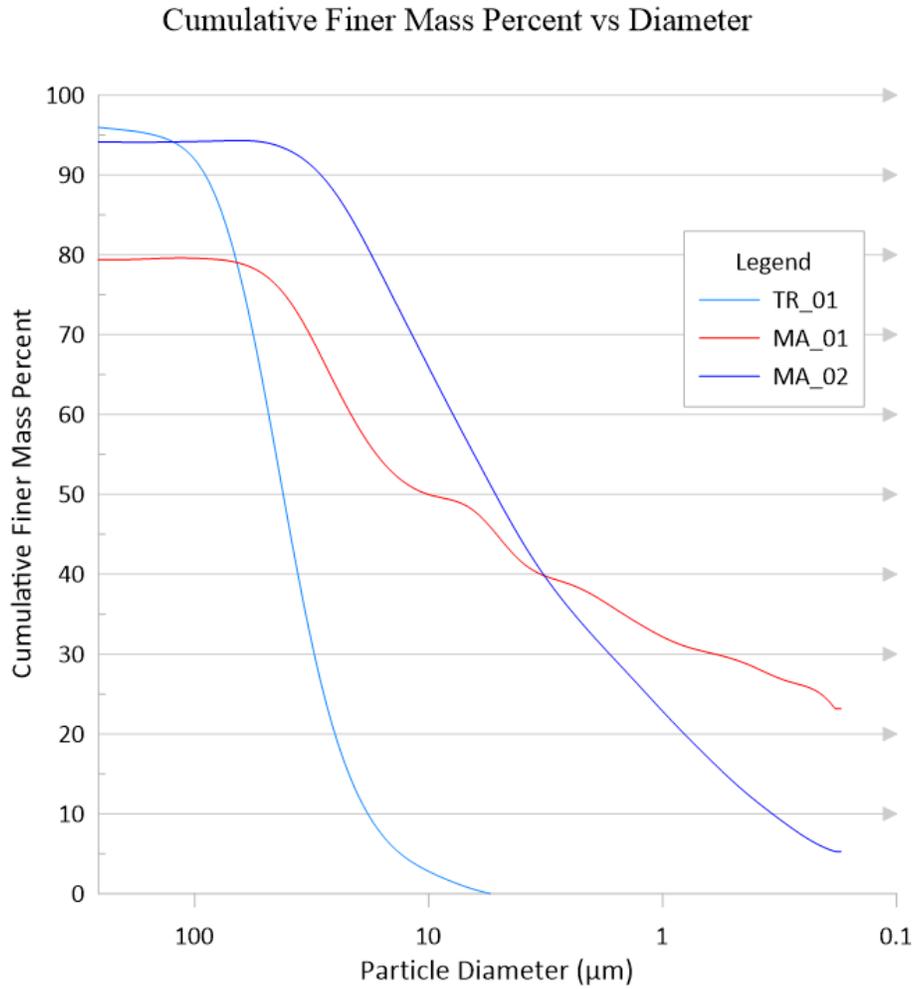


Figure 24 Sedigraph analysis showing data for grain size $<250\mu\text{m}$. Red (MA_01) is site 1, Dark blue (MA_2) is site 2 and Light blue (TR_01) is site 3. Legend = Sampling sites

7.0 Discussion

7.1 Discussion of challenges in methodology

Chapter 5 and 6 outlined how some processing and extraction methods for microplastics in high Arctic fjord and glaciofluvial sediments require specific modification to overcome various challenges. In this section these modifications are discussed further, including whether the sample size and methods used were adequate for defining concentrations of microplastics in these high Arctic environments. Furthermore, this section discussed what other methods could be experimented with in future studies that may contribute to more reliable, replicable, and representative results.

7.1.2 Digestion of organic matter

Digesting the organic matter in both fjord and glaciofluvial sediments samples prior to density separation and minerogenic sediments with H_2O_2 proved challenging and ultimately ineffective. Organics were still detected in the samples even after two weeks of adding H_2O_2 every 3-4 hours during the day, and due to the time allowance for this thesis, the procedure had to be terminated before digestion was fully complete. On closer inspection, the digestion step did not oxidize the largest pieces of organics, but rather bleached them. This is a common outcome that has been experienced in other studies as well (Hurley, Lusher, Olsen, & Nizzetto, 2018; Gewert et al., 2017; Nuelle, Dekiff, Remy, & Fries, 2014). Nonetheless, with organic matter bleached, and this more visible under the microscope, it was found that in some cases, this actually aided in differentiating organic matter from microplastics. On the other hand, the digestion process as followed was not an effective way to reduce organic material, and much of it was still left in the sample, ultimately leading to a 'crowded' filter, which took a long time to scan through during the search for potential microplastics. As a result, reducing organic matter from sediments analysed for microplastics and misidentification of organic matter for microplastics remains a challenge.

To combat the low efficiency of oxidation of organic matter using H₂O₂, Fenton's reagent can be added (Hurley et al., 2018; Tagg et al., 2017; Ravit et al., 2017). Fenton's reagent is a catalyst of iron sulphate that is mixed with H₂O₂ to increase digestion capabilities (Walling, 1975). Fenton's reagent as a catalyst has been used before in microplastic research previously and would likely have aided in organic matter digestion of the fjord and glaciofluvial sediment of this study. Of concern, however, was that Fenton's reagent would cause the exothermic reaction of oxidation to produce too high temperatures and deform potential microplastic particles (Al-Azzawi et al., 2020), thereby making identification even more difficult. This is seen as risky in such environment where it is possible that there are relatively few microplastics, which may also be highly degraded to begin with.

Faust et al (2019) point to that organic matter digestion of Arctic fjord sediments will always be difficult due to the large amounts of organics present in the sediments. This is based on the environmental setting and sedimentation rates in arctic fjords burying organics and essentially trapping them. The large amounts of organics may also have implications on the biofouling processes that induce sinking and burial of microplastics (Wu et al., 2020; Hoellein, Shogren, Tank, Risteca, & Kelly, 2019; Kooi et al., 2017; Kaiser et al., 2017; Fazey & Ryan, 2016). The article suggests that the organic matter composition in the fjord beds is mostly comprised of marine derived organics, not terrestrial organics. In this thesis, samples from glaciofluvial site (site 3) have been shown to include large amounts of organics, which negatively influenced the efficiency of the digesting procedure (Fig. 10). This may indicate that terrestrial organics inflow into Adventfjorden amounts to the high levels of organics in the fjord. This is backed up by the K. Kozirowska et al (2017) pointing to large terrestrial organics near glacier fronts. As Faust et al (2019) suggests, the environmental characteristics of the high Arctic, notably Svalbard, points to low amounts of organic matter in the glaciofluvial environment. The levels of organics found in the glaciofluvial sediments of this thesis, begs the question on the source of the large amounts, and that it may be due to thawing and deepening of permafrost. This release more organic matter to the fluvial sediments. This is an important question to be answered by future studies. Potential organics in different environmental compartments can easily be determined by Loss on Ignition (LOI).

7.1.3 Flotation

The flotation step to separate the heavier minerogenic sediments from the lighter microplastics also proved challenging and led to a slight change in direction of the study from its initial goal, which was to determine the number of samples needed to provide foundations for a good statistical count of microplastics in high Arctic sediments. Clay particles in the samples from sites 1 and 2 proved to be the most significant challenge as they do not appear to fully settle in the heavy liquid solution, and clog up the filter following decanting, making it difficult to later scan under the microscope for microplastics. Those that do settle may be holding onto microplastics, causing a loss of sample.

The dense liquid that was used for this project was a high saturated NaCl solution with a density of 1,29g/cm³. This should have allowed all particles denser than 1,29g/cm³ to sink and lighter microplastics to float. Microplastics commonly have densities ranging from 0,9 to 1,5g/cm³ (Campanale et al., 2020). The denser particles could have been included with a denser liquid such as NaI (sodium iodide), which was available for the project. Sodium iodide can be used to float particles lighter than 1,4 g/cm³, however, NaI is highly toxic for aquatic organisms, but mainly the time of discarding of the waste NaI proved to decrease efficiency in the long run to the level of the liquid being disregarded for further use. The density of the solutions may have been altered when added to the samples due to the sediments in the sample being saturated in fresh water.

Although using a liquid with higher density, such as NaI may have yielded more microplastics separated from the settled sediments, the challenge of high clay content was not overcome, causing problems during filtration. As the dense liquid kept the clay in suspension, particles of interest may have been trapped below the suspended clays or trapped with the settled clay. In terms of flotation of sludge-like sediments and sediments with a high level of clay particles, dense liquids can cause more problems than they solve. As such, for this project, the clay size fraction was removed by vigorously agitating the sample and then rinsing them through a sieve with a 63µm metal mesh before flotation. The success of this pre-step was clearly seen with the spiked sample pre- and post-sieving (section 6.1.3) where the retrieval rates were much improved after sieving. Although adding a laboratory step to processing of the sediment samples

for microplastics may increase the chance for contamination, blanks were used to account for this.

7.1.4 Decanting and filtration

Following flotation, but prior to filtration, the dense liquid floating the potential microplastic particles must be transferred from the sample jar and run over the filter via decanting. Decanting of the heavy liquid from the sample jar unfortunately reduced to many sediments to the filter paper due to the clay particles remaining in suspension even after 48 hours, partly due to increased turbulence during decanting procedure partly due to clay particles having similar to densities to microplastics as $1,09 \text{ g/cm}^3 - 1,706 \text{ g/cm}^3$. The process of sieving with $63\mu\text{m}$ metal sieve was introduced as mentioned above to counteract this problem (section 7.1.3). Decanting also resulted in sticking of particles to the sides of the sample jars, leading to potential loss of microplastics. Overflowing of the sample jars in a 2L glass beaker proved to reduce turbulence and resuspension of settled sediments during removal of heavy liquid, and therefore also the sticking of particles to the sides of the sample jar. Some turbulence was unavoidable, and some sediments were still caught on the filters. This technique proved to be successful in the spiked sample experiment and was therefore applied to the samples from Adventfjorden and Adventdalen. However, as this process relied on transferring from the sample jar to another beaker before filtering, the risk of contamination also increased.

No alterations to the vacuum filtration step were made as there were no substantial challenges identified during this process. Vacuum filtration is simple and straightforward, but there are some points at which contamination and cross-contamination may occur, such as in between each filtration and inadequate rinsing between each filtration. The vacuum flask is also the container for the filtered liquid and a 1-liter flask was not large enough to filter large numbers of samples as the flask filled up quickly. However, the efficiency for filtering one sample proved to be no problem.

7.1.5 Identification and species determination

Identifying microplastics based on microscopy is and will always be bound by the fact that the process relies on subjective analysis. Two different researchers may classify particles differently accepting and disregarding particles as microplastics based on subjective opinion. As organic

material sometimes has similar characteristics to microplastic particles, they can easily be mistaken for microplastics. This greatly impacts the eventual results of the survey and can in certain cases render a study unreliable and incomparable to other studies. Leaning on the side of too inclusive when deciding whether or not to count a particle as ‘potentially microplastic’ for using Raman or FTIR spectroscopy later confirmation is prudent.

Identifying and dividing the particles into groups or classes in the identification stage provide an efficient foundation for locating and assessing the particles during Raman analysis and also helps in quickly assessing overall abundance and similarities between sites. A good system of defining microplastic particles based on morphology, colour, and what state they are in, give a good foundation for future studies and can help provide greater insights on similarities and differences between sites with various environmental characteristics.

7.1.6 Raman analysis

Raman analysis was used to determine whether classes of identified particles were microplastics or not. As mentioned previously, Raman analysis relies on the emission of laser and the reading of the reflected photons in a sensor to determine the chemical bonds of the material. There are benefits with a Raman over an FTIR, which also is widely utilized as a means for chemical analysis. Firstly, the Raman does not require too much preparation of the sample beforehand as the Raman is a microscope with a camera. This makes it possible to identify and locate the material of interest before testing. With the FTIR however, the particles must be separated and prepared in such a way that the FTIR manages to read the signatures by isolating the potential microplastics from sediments and organics. This highlights the importance of the modifications to flotation procedure presented earlier in this chapter, where as much removal as possible of minerogenic material like clay, is critical. Removing as much organic matter as possible also increases efficiency when using FTIR to identify microplastics.

The camera and microscope on the Raman make the preparation work much less time consuming, however the localization of material that is too small for easy recognition can increase time spent searching in the Raman. There is also the problem of the burning of the potential microplastic particle. For example, a given particle can withstand a laser intensity of 10mW, but its resistance is not known until a burning has been done at a certain intensity. In

samples with low overall numbers of microplastics, this can be problematic. During the work with the Raman, some particles experienced a burning at low intensity, and some could withstand substantially higher intensity. Tests were undertaken on known samples of polystyrene to discover a typical intensity load that polymer could withstand. From the testing, it was found that during the use of oscilloscope function on the Raman, the intensity threshold was 40 mW before a burning of plastic was witnessed at higher intensity. This does not fully prove the intensity load a plastic particle can withstand, but it indicates that plastic has a higher threshold than organics as organic material burned at low intensities (e.g., 5-10mW)

7.2 Discussion of results

7.2.1 Amounts per sample

The samples from the glaciofluvial and the fjord sites show overall low numbers of particles that may or may not be microplastics. An overall average of 11 particles in glaciofluvial and an average of 8 particles per sample in fjord samples were registered. To what extent this is a direct result of the methodologies and protocols used is uncertain, but the results do point towards a low probability of there being high numbers of microplastic particles in the glaciofluvial sediments of Adventdalen and fjord sediments of Adventfjorden. Site 3 is a glaciofluvial environment with intermittent (often seasonal) periods of high intensity flow, for example during the melt season, and low to no flow (during freeze up) (Hoellein et al., 2019). As discussed earlier (chapter 3.2.1), if the buoyancy potential of a microplastic particle exceeds the settling potential of the water it is situated in, the particle will be delivered to suspended load and therefore be transported further downstream and possibly into the fjord, until the settling velocity exceeds the buoyancy potential. It is therefore important to study the flow intensity of the study area in different seasons to map out possible sinks where temporary trapping of microplastic particles may occur. Site 3, which clearly experienced high flow in the past (ripples can be seen in this dry channel: Fig. 7) but is currently experiencing no flow since the sediments are subaerially exposed, is an example of a good area to sample glaciofluvial sediments from. It is

possible that some microplastics may have been resuspended here already due to strong winds, but these may equally be lightest, smallest particles, which are not included in this study.

Based on settling velocity and buoyancy potential, the low numbers of microplastics may also be due to the potential denser particles in the sample which have negative buoyancy exceeding the density of 1.2g/cm^3 . In this case, the use of NaI (sodium iodide) as density separation liquid could have been more effective, but the additional time required for cleaning meant that this was not possible to achieve in the later stages of the project.

In the glaciofluvial site, the types of microplastics found are most commonly used in textile production. This polymer is referred to as PAN or Polyacrylonitrile and may have originated from clothing of passing hikers or during winter season of scooter traffic. The paint chips that were found contained the dye Phthalocyanine Blue. Possible source of this particle may be from ship traffic, where particles deposit on the beach and by aeolian transportation get transported further in land when dry. The particles could also have originated from heavy equipment from the mining industry.

Because of all the challenges during digestion of organic matter, density separation and decanting, the abundance of microplastics in Adventdalen and Adventfjorden registered in this thesis should be considered as a tentative first look, and provide only a starting point for future work, that utilizes more appropriate methodologies from the onset. Interpretations of microplastic concentrations from such clay-rich, organic-rich sediments, given the many challenges encountered during digestion or organic matter and separation of microplastics and minerogenic sediments, also stresses the importance of taking the local environmental and sedimentological characteristics into account and determining the most appropriate methodology accordingly.

7.2.2 Spiked samples

Exploring the potential of the extraction processes with more extensive spiking of samples should have been done to discover and eliminate problems in the process from the beginning. The results of the spiked samples show scattering in extraction and makes methodology standardisation next to impossible. Whether it is the particles that were used that influenced the results or not, it would be beneficial to utilize microplastic particles such as fibres or other

preproduced microplastics particles such as glitter or something similar. A successful extraction should have at least 80% retrieval rate (Budimir, Setälä, & Lehtiniemi, 2018). The range of 40-87% retrieval rates from site 3 and 26% retrieval from site 1 and 2 is an indication of gaps in methodology. Whether the sample was too wet and altered the density of the NaCl liquid due to dilution or that the overflowing/decanting process was inadequately executed is difficult to say. Further testing is necessary.

7.2.3 Raman results

The results of the Raman spectroscopy tests are inconclusive due to the low number of particles of each group that was tested. This limits the possibility of producing statistically sound results. The reasons for the low number of tests are a result of the low number of samples to test, as well as time limitations due to more time having been spent on improving pre-processing methods. From the tested particles, the blue fibres stand out as the most interesting due to the variable result on similar looking particles. In this case the particle that was deemed a microplastic particle from its signature close to Zexlon or Polyacrylonitrile, show interesting results as some of the other particles tested did not have similar spectra. The other particles had spectrums like organic material, such as Rutin and Algin. Similar looking particles show different spectrums and in a group of four particles, one might be a microplastic particle while the rest are organics, increasing the importance of spectroscopy for testing of material composition. Solely reporting abundance of microplastic particles based in visual inspection is not enough by far, and it is absolutely critical to perform extensive testing for as many of the potential microplastic particles as possible, rather than relying of visual similarities.

As mentioned, some particles were not possible to test due to their low threshold for withstanding laser intensities. Combining two spectroscopes, Raman spectroscopy and FTIR spectroscopy may increase the testing capability and produce results which will give the potential in defining ratios for microplastic abundance in samples. This is based on the flaws that the other spectroscopy can overcome. As an example of this, the thesis reported several particles to be either portraying too much fluorescence which rendered some data unusable. FTIR should have made it possible to overcome this problem and a possible result on material should be possible to acquire. This combination of spectroscopes and extensive testing may increase the

possibility of defining microplastics based on appearance and increase the value of visual inspection. Doing a dual spectroscopy testing was not possible for the project due to the cost of utilizing both spectroscopes simultaneously and the time allocated for the project did not allow for such extensive testing.

7.2.4 Sediment analysis with Sedigraph

Due to uncareful storage of notes and accidental deletion of data from the computer, data was lost for the manual analysis of the sediments from the stations. Therefore, this thesis only includes data from size 250 μm and down, however, visual assessments suggest that grain sizes larger than 250 μm did not exceed 1 mm and did not make up a large portion of the sample. For that smaller than 250 μm , sediments from sites 1 and 3 are mostly between 250 μm to 0.1 μm . The marine samples contain significant clay content (35%), while the glaciofluvial site is mostly silt and fine sand, however, there were some technical problems with the sedigraph, and given the similar problems with microplastic extraction from all sample sites, likely related to clay content, it is very likely that there is at least some clay content in the glaciofluvial sediments as well.

8.0 Conclusion

This thesis has explored well-established methodologies for microplastic research and their limitations when applied to high Arctic fjord and glaciofluvial sediments from Svalbard. Isolating and extracting microplastics from these sediments proved to be more challenging than initially thought, which was very likely due to the high clay contents of the samples. Clay minerals appear to readily trap microplastic particles either in the water column or in the settled sediments during density flotation. By taking the pre-step of agitating and sieving the samples through 63 μm metal mesh to remove as much clay as possible provided a work around for this issue, increasing the potential of retrieval of spiked particles and potential microplastic particles. The second challenge that was experienced during laboratory work was the limited efficiency of organic matter digestion without Fenton's reagent.

The initial count from visual identification showed 202 particles in the glaciofluvial sediment from site 3. The Raman-confirmed number of microplastic particles in the glaciofluvial sediments was only 1 particle, which may reflect the low, overall abundance of microplastics in the area. Notably, it was discovered during testing that most of the particles tested with similar

visual characteristics were actually quite different with respect to material. For example, the only microplastic fibre, had the same characteristics as other blue fibres which were identified as organic material. This indicates that determining microplastic abundance solely based on visual inspection with microscopy will not provide trustworthy results. Vigorous testing of particles in Raman spectroscopy and FTIR is important to reduce the subjectivity of many microplastic studies (Käppler et al., 2016; Lenz, Enders, Stedmon, Mackenzie, & Nielsen, 2015). The subjectivity of microplastic studies is its weakest link, as the people undertaking microplastic studies might define microplastics differently. To fully standardise methodologies in microplastic research, the differentiation between microplastic and non-microplastic material should be of highest priority.

One microplastic particle was confirmed found in the sediment samples through Raman analysis, although only particles from 6 samples were examined on the Raman due to extensive problems during processing (organic digestion and flotation). 4 particles with chemical signatures matching different dyes were found in the glaciofluvial environment, which are also classed as pollution (Collard et al., 2021).

This thesis has demonstrated the importance of characterising local environments with respect to water energy, organic matter, grain sizes, prior to study of microplastics in sediments in order to choose the most appropriate and necessary sampling and processing steps to isolate microplastics. The importance of confirming a particle as plastic using Raman and/or FTIR rather than relying on the assumption that all particles of the same colour and morphology will be formed from the same material, has also been confirmed in this study. Altogether, these results and lack of results provide valuable information for future microplastic studies.

9.0 Appendix

Sample no	Code	Filter 1*	Filter 2*	Total count MP
1	TR	6	8	14
2	TR	2	5	7
3	TR	0	31	31
4	TR	0	8	8
**5	TR	0	0	0
6	TR	4	8	12
7	TR	3	14	17
8	TR	3	5	8
9	TR	0	10	10
10	TR	2	17	19
11	TR	1	4	5
12	TR	0	11	11
13	TR	2	12	14
14	TR	0	3	3
15	TR	2	12	14
16	TR	0	2	2
17	TR	0	5	5
18	TR	2	7	9
***19	TR	0	9	9
20	TR	0	4	4
Total TR				202
Average MP per Jar				11
39	MA1	9		9
43	MA1	3		3
47	MA1	9		9
48	MA2	6		6
45	MA2	10		10
46	MA2	11		11
Total MA				48
Average MP per Jar				8

Table 3 The table is separated into sample nr which refers to the number on the sample jar. The code TR stands for "Terrestrial", MA is for "Marine". As the terrestrial samples was filtered twice due to alterations during filtration process, the labels show: Area, Jar number, Filter number and amount per filter as well as total amount.

* Two filtration steps, 1. before sieve and 2. after sieve with 63µm ** Broken or no sample to see *** 1 filter rendered unusable due to too many sediments on the filter.

Dry weight incl.jar (grams DW)	Dry weight excl. Jar (grams DW)	Average weight jar grams
397.55	181.17	216.38
410.53	194.15	216.38
404.72	188.34	216.38
448.58	232.20	216.38
477.04	260.66	216.38
422.81	206.43	216.38
429.57	213.19	216.38
420.90	204.52	216.38
453.89	237.51	216.38
429.18	212.80	216.38
441.30	224.92	216.38
422.08	205.70	216.38
438.75	222.37	216.38
409.42	193.04	216.38
442.37	225.99	216.38
403.74	187.36	216.38
446.76	230.38	216.38
394.03	177.65	216.38
440.80	224.42	216.38
425.12	208.74	216.38
Average DW	211.58 grams DW	

Table 4 Dry weight sediments from terrestrial site (site 3). Average dry weight in grams.

Sample no	Type particle	Amount
1	Blue bead	2
	Blue fibres	3
	orange bead	2
	Transparent fibre	3
	Black fibre	2
	Yellow film	1
	Yellow bead	1
	2	Transparent fibre
Black fibre		2
White bead		1
Blue fibres		1

3	Blue bead	1
	Blue fibres	25
	Red fibre	1
	Red bead	1
	yellow film	2
	Yellow bead	2
4	Nothing	
5	Broken	
6	Blue fibres	4
	Black fibre	1
	Blue bead	1
	Transparent fibre	2
	Yellow bead	4
	7	Yellow bead
Yellow film		2
Transparent fibre		4
Blue fibres		5
Black fibre		3
White bead		1
8		Yellow bead
	Blue fibres	1
	Transparent fibre	2
	Black fibre	2
9	Yellow fibre	1
	Transparent fibre	5
	Black fibre	1
	Blue bead	1
	Red fibre	2
10	Red fibre	1
	Blue bead	2
	Blue fibres	7
	Black fibre	2
	Yellow bead	2

11	Black fibre	2
	Transparent fibre	1
	Blue fibres	1
	Yellow bead	1
12	Transparent fibre	3
	Transparent film	1
	Black fibre	3
	Blue fibres	1
	Yellow bead	2
	Yellow film	1
13	Transparent fibre	9
	Yellow film	1
	Red fibre	1
	Black fibre	1
	Blue fibres	2
	Yellow bead	1
14	Transparent fibre	2
	Black fibre	1
15	Transparent fibre	8
	Yellow film	1
	Yellow bead	3
	Blue fibres	3
16	Yellow film	2
17	Transparent fibre	2
	Black fibre	1
	Yellow fibre	1
	Green fibre	1
18	Black fibre	3
	Yellow bead	5
	Blue fibres	1

19	Blue fibre	3
	Transparent fibre	2
	Blue bead	1
	Red film	1
	Black fibre	1
	Yellow bead	1
20	Black fibre	2
	Blue fibre	1
	Blue bead	1
Site 1 & 2		
39	Black fibre	3
	Blue fibre	3
	Yellow film	1
	Yellow bead	2
43	yellow bead	1
	Black fibre	1
	Transparent fibre	1
47	Red fibre	1
	Black fibre	5
	Blue fibre	3
48	Blue fibre	2
	Black fibre	4
45	Yellow bead	1
	Blue fibre	2
	Transparent fibre	3
	Black fibre	2
	Yellow fibre	2
46	Black fibre	1
	Blue fibre	9
	Red bead	1

Table 5 Amount of particles per type per sample from site 3

Methodology	Procedure	Pros	Cons
Sampling			
Grab hooked to a wire dropped to the bottom of lake/sea, when mechanism is initiated locks the grab	Large amounts of sediments in one grab. Subsamples can be taken. Easy and effective	Relies on machinery for larger sizes to pull up. No control of the location grab lands. Mechanism can fail. Disturbs the benthic top layer	
Van Veen Grab sampling	Same mechanism as Van Veen grab, preserves the benthic layer	Relies on machinery for larger sizes to pull up. No control of the location grab lands.	
Box Core sampling	Tube penetrates the sediment layers and acquires chronological data of sediment layers	Chronology of the sediment layers	
Core sampling	Water is sampled by filling water sample bottles.	Suspended microplastic can be collected.	Large amounts of water needed for good statistical count. For column sampling large sampling rig is needed. Not efficient
Water sampling	Floating device with a net and sampling container, usually connected to the back of a boat	Catches surface debris, large area can be covered in short amount of time	Need for boat, can be contaminated by air pollution and boat
Trawler net sampling	Water is sampled by filling water sample bottles.	Suspended microplastic can be collected.	Need large volumes for good statistical count.
Fluvial water sampling	Sediments are sampled with trowl and transferred to sample jars	Potential settled microplastics can be sampled.	Locations may have no particles due to high intensity flow and microplastics cannot settle.
Fluvial sediment sampling			
Laboratory			
Organic Matter Digestion	Chemical oxidation of organic material with Hydrogen Peroxide.	Easy processing. Cheap.	Bleaching of organic material is common. Needs catalyst to work efficiently. Exothermic so can deform microplastic particles
Density Separation	Dense liquid of NaCl or NaI	Easy processing. Cheap.	NaI is expensive and toxic to life in water, demands waste disposal. Mineralogic particles and organics may also be separated together with sediments.
Filtration	Vacuum filtering of liquids	Constrains the particles to filter paper which makes easier for identification	In case of too much sediment, the filter gets clogged. Contamination is always a risk with this.
Microscopy	Identification and quantification with visual means	Easy and cheap. Efficient	Misinterpretations are possible. Subjective identification is possible
Raman Spectroscopy	Laser and spectroscope measuring reflected photons to determine chemical bonds	Easy process and need little preparation, database readily available with chemical signatures	Is highly affected by fluorescence, expensive
FTIR	infrared spectrum obtained by absorption or emission of a material	Easy to use, not affected by fluorescence	Expensive, need thorough preparations before testing.

Figure 25 Review of selected methodologies for field sampling and laboratory extraction/analysis

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