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Microplastic occurrence in selected sediments samples in Rogaland – a pilot study

by

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List of commonly used abbreviations

UiS – University of Stavanger

SO – Sola beach

SO-1 – Sample identification label for surface sample from Sola beach

SO-2 – Sample identification label for subsurface sample from Sola beach

BO – Bore beach

BO-1 – Sample identification label for surface sample from Bore beach

BO-2 – Sample identification label for subsurface sample from Bore beach

SS – Steinerskolen i Stavanger

SS-1 – Sample identification label for surface sample from Steinerskolen i Stavanger

SS-2 – Sample identification label for subsurface sample from Steinerskolen i Stavanger

XRD – X-Ray Diffraction

PSD – Particle size and distribution

SPT – Sodium polytungstate

Units

μm – micrometer

m^3 – cubic meter

cm – centimeter

mm – millimeter

g/cm^3 – grams per cubic centimeter

C – celcius

μ – micron

nm – nanometer

cm^{-1} – reciprocal wavelength

g – gram

km – kilometers

km^2 – square kilometer

Abstract

Microplastic pollution is of growing concern and is today one of the most important problems of the Earth. When microplastic is released into the environment it can have a major impact on both humans and other living beings. Microplastics have been found in all natural environments, including oceans and the atmosphere (Ferrero, et al. 2022). This project research aims to present the abundance of microplastic at two different beaches and one public school and comment on the types of plastic within the samples. However, the main objective is to present a technique for sampling, separation, and identification via Raman.

In this study, selective and bulk sampling was used for the sampling and density separation for the separation process. The methodology was optimized for the purpose of this study and different density fluids were used to develop the best possible method to be used. With comparison to an experienced Raman laboratory, where we measured all standards and samples, we could define the ideal analytical set up for plastic research with Raman at UiS: 633 nm laser, 1 s of exposure time, 30 of accumulations fixed at 10 %.

Sampling has been carried out at two popular beaches in Rogaland, Sola (sample set SO), Bore (sample set BO) and in a sandbox of a public school (sample set SS). Results showed similar mineralogical composition in all the samples. However, the SS samples contained a significant higher abundance of chlorite and mica and was poorly sorted. In total the SO sample, the best sorted, has the higher abundance of microplastic, while the SS samples have the highest abundance of microplastic particles smaller than 1000 μm . The BO sample also had a lot of microplastic nurdles at its surface while no nurdles were observed at SO or SS.

This study only concentrated on material larger than 32 μm as this already had been sufficient material for this thesis project. Samples at the same location from 30 cm depth show much less or were even barren in regard of microplastic $> 32 \mu\text{m}$. Significant drainage could for that size of plastic not be observed. Maybe in smaller fraction such an effect is possible. The plastic identified were mostly larger than 1000 μm in both, SO and BO samples but only on the surfaces. Still, contamination of deeper parts of the sediments took still not place for large plastic particles. Generally, SS contained as well smaller samples but was less contaminated by plastic than the beach samples. The main occurring plastic types in all three samples has been polypropylene, polyethylene and poly (ethylene-vinyl acetate).

Suggestion for further study is to investigate in sediments the grain size smaller than 10 μm to target plastic fibres between 5-10 μm . Nevertheless, the defines methodological set up can also be used for that fraction at UiS.

1 Objective

The objective of this study is to compare different public exposure in jæren in regard of the contamination by microplastic in sediments. The study will also compare surface sediment samples with other collected at the same location in a different depth. The research likes to present if plastic is abundant and if so, what types of plastic exists. This will be determined using Raman spectroscopy. However, main objective is to present a sound sampling technique and an analytical recipe for the identification via Raman application. The reason is that such a study has not been yet carried out at UiS. Quantification and the study of nanoplastic is out of the scope of this research project. The microplastic will first be identified at UiS and results and methodology compared with studies using Raman at an experienced laboratory in Italia to compare and validate the results. This activity also implies the necessary knowledge transfer for future studies on microplastic at UiS.

2 Introduction

Plastic pollution is of growing concern and is today one of the biggest global challenges we are facing. As the plastic is released into the environment it can have major impact on both humans and other living beings. The main concern regarding wildlife is the ingestion as then these gameplayers are potential part of the food chain, including the human food chain (Ritchie and Roser 2018). Currently there is very little evidence of the impact that microplastics can have on humans. Although, it is widely acknowledged that the full extent of the impacts of plastic on the ecosystems is not yet fully understood. However, it is obvious that plastic as a substance which does not decay easily will increase in its abundance and will cause, or already does, and enormous effect on animal, plant and human tissue and life in general. “The term microplastic (MP) generally refers to any piece of plastic smaller than 5 mm to 1 μm in size along its longest dimension” (Crawford and Quinn 2016). Microplastic can further be classified into two categories. Primary microplastic that is plastic produced intentionally (Bergmann, Gutow and Klages 2015), and secondary microplastic which are smaller particles of irregular plastic that will be formed in the environment from the fragmentation of larger plastic material into ever-smaller particles (Cole et al, 2011).

Different approaches within earlier studies, makes it difficult to compare studies; yet no systematic methodology or data bases are present. As there is a lack of standardised ways to carry out research, the aim of this study will be to develop a methodology for sampling, separation, and identification of microplastic that can be used later for other studies in the future. This is the first-time that studies regarding microplastic at the University of Stavanger (UiS) have been carried out on sediments in Rogaland. Therefore, this study will be a pilot study to select areas of contamination in the public sector.

The samples were collected using a combination of selective and bulk sampling. Two of the most popular beaches in Rogaland, Sola beach (Fig 1 a) and Bore beach as seen in (Fig 1 b) were chosen. Two samples were taken at each outcrop. One surface sample (Fig 2 a) and one at a depth of no more than 30 cm (Fig 2 b), indicated by the black photo lens cover.



Figure 1 Sampling area for the beach samples a) Sola beach - the sampling area; b) Bore beach - the sampling area.



Figure 2 Pictures of the sampling process. a) Surface sample at Sola beach collected in a glass jar. b) Picture showing the sampling of SO-2.

Both beaches are located in jåren on a large moraine that continues out to the sea. (Humberset 2013). The facies for both beaches are clastic shallow marine coastal environment in the wash-over facies close to the dune area. “The area was formed during the last ice age that ended about 10 000 years ago and rests partly on solid rock (mainly gneiss and granite) and partly on a seabed from earlier geological periods that has been transformed into shale and partly sandstone. The moraine layer is so thick that only a few hilltops are visible before we reach a few kilometres in from the coast” (Humberset 2013).

Two beach samples sets were combined with one sample set collected from the sandbox at a public school (Steinerskolen i Stavanger), sampling area (Fig 3), to compare and see if there are any differences in the abundance of plastic at the beaches and artificial sand complex, which has been emplaced for playing for children of all ages at the school. This was a major criteria to try to monitor plastic contamination in an area frequently used by children.

The samples were processed at UiS for Raman studies at UiS and the Università Milano Bicocca (Italy), which is a major gameplayer within Raman analytical science in geology. The study will demonstrate the main sizes of plastic contamination and determine the type of plastic particles. Focus, however, within this BSc is on the methodology and will provide a sound analytical recipe for plastic identification via Raman application.



Figure 3 Sampling area for the public school, playground at Steinerskolen i Stavanger.

3 Sampling

Several sampling methods for microplastic are described in literature. However, there are no universally accepted methods and the method used should be depending on several factors. The environment which is being sampled and the size limitations of the microplastic to be collected are factors that need to be considered (Crawford and Quinn 2016). Also, the method to be used largely depend on the research question (Lushe, et al. 2017) as well as the “economic proportionality of the methods and also the study compartment” (Razeghi, et al. 2021). It is important to have this in mind as the studies should easily be replicated and reproducible.

For this study, bulk and selective sampling were used. Bulk sampling is a sampling technique where the entire sample is taken without reducing its volume during the sampling process. The benefit of bulk sample is that all the microplastic in the sample should be collected regardless of their size and visibility. Processing the whole sample also prevent any microplastic from being lost or overlooked during the sampling process. Selective sampling is a process where all the recognizable items is collected by hand. This method has a higher change of microplastic to be overlooked. Therefore, for this study the bulk sampling is done

first and then if there are any visible particles left, they have been collected by hand and added to the sample. Each outcrop had a surface area of about 0.25 m² as seen in Fig 4 a). Closer look at the surface area before sampling are seen in Fig 4 b). The first sample were collected in the morning 09:30 o'clock at Sola beach (sample identification label: SO), the 29.09.2022 (coordinates: 48.88614° N, 5.60228° W; Fig. 4 a). The weather was fair with no wind and clear skies. After the area of sampling was selected, the surface was scratched with a metal spade about 5 cm before the samples were collected with the spade and introduced carefully into a glass jar. All the visible plastic left at the surface was then collected by hand and added to the glass jar. After sampling, the jar was closed and labeled; Fig. 2 a). Two samples were taken at each outcrop. One surface sample and one at a depth of no more than 30 cm as seen in Fig 2 b), indicated by the black photo lens cover.

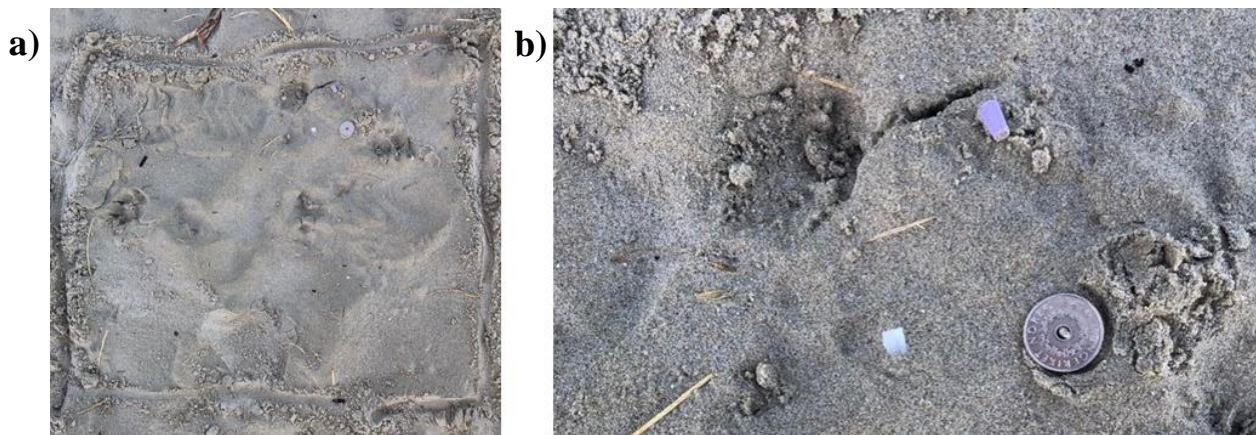


Figure 4 Pictures of the sampling area at Sola beach. a) The surface area before sampling; b) Detail of the surface area before sampling.

For the subsurface sample, a hole has been dug of about 30 cm depth with a metal spade as seen in Fig 5 a). Sample area after sediments were collected are seen in Fig 5 b). The sample was then collected by using the spade to dismember carefully sample material of about 1-3 cm into the vertical sand wall. The sediments were then filled carefully into a new glass jar before any visible plastic particle left were added to the sample jar. The same procedure was used in all the three outcrops.

The second outcrop at Bore beach (sample identification label: BO) was collected the same day the 29.09.2022 at 10:40 o'clock (Coordinates: 58,73805° N, 5,51351° W; Fig 1 b). The weather was still fair with no wind and clear skies.

The third sample set was collected the 06.10.2022 at the time 09:15 o'clock (coordinates: 58.93914 N, 5.73477 W; Fig 3) at the playground of Steinerskolen i Stavanger (sample identification label: SS). The weather has been windy with some rain.

After the collecting process, the samples were transported to the laboratory for further treatment. The samples did not get in contact with plastic tools and the sample jar were devoid of any plastic to avoid unnecessary contamination.

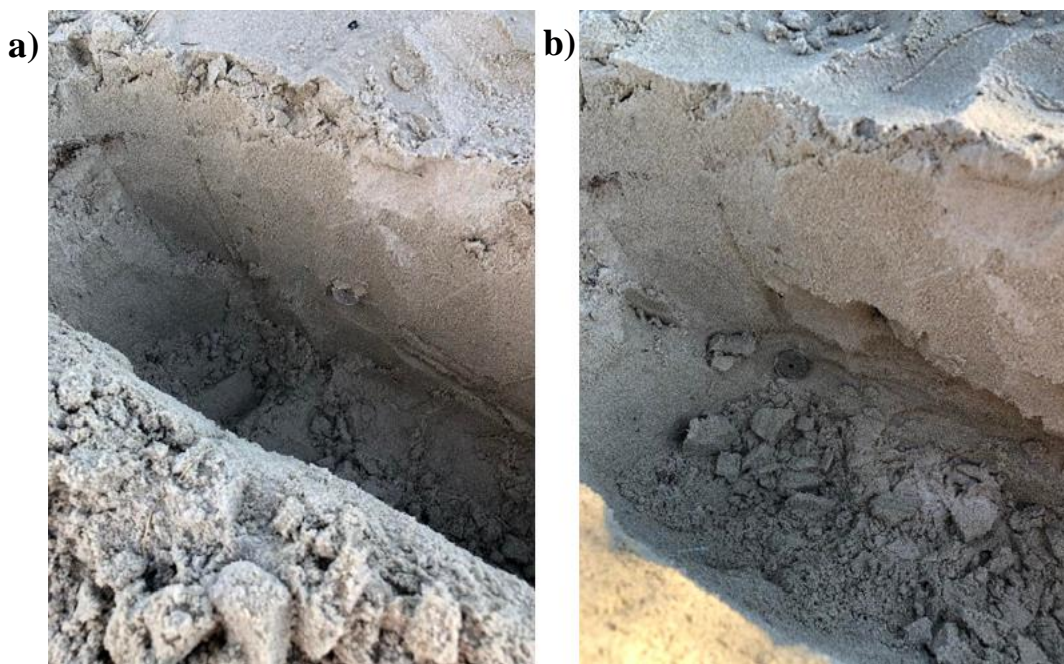


Figure 5 Pictures of the sampling area for SO-2 at Sola beach. a) Before sampling; b) After sampling.

4 Methodology

The methodology of this study was based on the method described in ‘Microplastic Pollutants’ (Crawford and Quinn 2016). Selective and bulk sampling was used for the sampling, density separation for the separation process and Raman spectroscopy for the identification. The methodology was optimized for the purpose of this study, which was to contribute to knowledge about plastic pollution in Stavanger and to develop a methodology for sampling, separation, and identification of microplastic in sediment samples that was easily reproducible and repeatable. In addition, the methodology focused on precision, accuracy, and minimal contamination. As Crawford and Quinn (2017) states contamination of an environmental sample either in liquid or in solid with microplastic that were not part of the original sample is one of the major challenges in these types of studies as microplastic is so

ubiquitous of nature, existing almost everywhere. To avoid contamination from plastic sources, the use of plastic equipment was avoided as much as possible and replaced by either glass, aluminum, or Teflon in all steps from sampling to analysis. The samples were also at all times stored in closed or covered equipment, either in glass jars, Teflon beakers or covered by aluminum foil.

4.1 Sample preparation

4.1.1 Splitting the samples

Working with several samples at the same time increases the risk of contamination between samples. Particles left in the sample splitter, on the equipment used to handle the samples or laboratory bench are examples of how easily contamination can occur. It is therefore important to carefully wash the equipment well between each sample and always keep the laboratory environment clean. The splitting process is an important step in the sampling preparation as by splitting the sample there will be two representative parts, where one part can be kept in reserve either to compare the results with or to reproduce the study. It is important not to have too much material in the sieve, as it can clog the mesh in the strainer and prevent finer material from passing through to the next sieve. Therefore, it is important to divide the sample during the preparation process.

Before the splitting took place the sample splitter was blown with pressure air and closely inspected for any material stuck in the splitter. This was done before each sample was split. Each sample was first split one time (Fig 6). In this split, the first part yielded one half of the original sample and was stored for later use. The other half was split again into two quarters of the original sample. One part was sieved while the other part was split again into two sixth of the original sample to be used for XRD and PSD analysis.



Figure 6 Pictures of the splitting process with the sample splitter to generate two similar samples for further processing.

4.1.2 Dry sieve analysis

The method involves separating material of different sizes by passing the sample through a series of sieves with a decreasing mesh size. The sieve analysis is done to separate the sample into fractions based on their particle size and to get a more even sample to analyze.

Before the sieving process all the sieves were cleaned and carefully blown with pressured air. As for the splitting there is always the risk of contamination between samples when working with several samples. For the sieving process there might be particles left in the sieve or on the other equipment. Therefore, the sieves were cleaned well between each sample to avoid contamination. All other equipment to be used as metal spoon and needle was also cleaned before use. When all the sieves were cleaned and blown with pressured air a microscope was used to look for any smaller particles left in the sieves. If so, a needle was used to remove this, without damaging the sieve. The sieves were then placed in consecutive order with the coarsest sieve (2 mm) on top and the finest (32 μm) at the base. In total seven sieves were used with the mesh sizes 2 mm, 1mm, 500 μm , 250 μm , 125 μm , 63 μm and, 32 μm . The sample was weighted and poured carefully into the coarsest sieve. The sieve stack was placed onto the sieve shaker. The machine was set to a vibration intensity of 6/10, intervals of 3 seconds and a total time of 25 minutes as seen in Fig. 7a). The shaking of the sieve stack causes the material to pass through until the mesh size is too small, thereby resulting in separation of the material based upon size.

The material was carefully recovered from each sieve one by one starting at the top. The sediments left in the coarsest sieve (> 2 mm) for sample BO-1 can be seen in Fig 7 b). The sediments were then carefully transferred to a clean paper sheet. A metal spoon and needle were used to get all the sediments left out of the sieve and on to the paper sheet. The sediment collected from the sieve was then carefully transferred to a glass bowl to be weighted as seen in Fig 7 c) before the sample was taken back to the paper sheet. The paper sheet was rolled as a cone and the sediments were put in a new Teflon beaker labeled with sample name and the fraction size.

-The results of the sieving analyses for each sample can be found in appendix A.

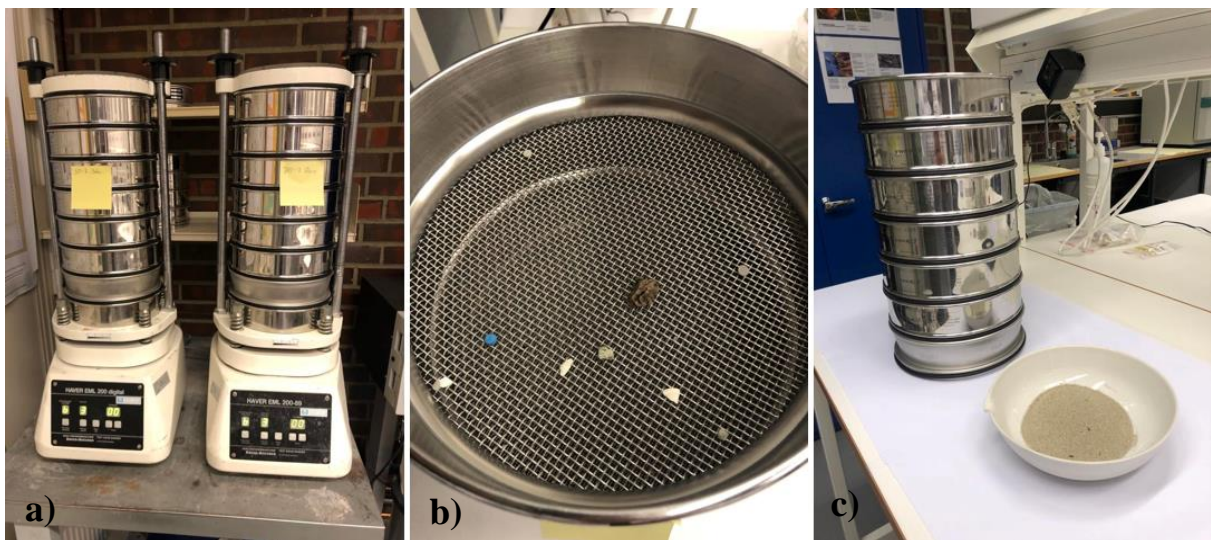


Figure 7 Pictures of the sieving process. a) The sieve stack placed in the sieve shaker; b) Closer look at BO-1 > 2 mm; c) BO-1_500-250 μ m in glass bowl after weighting.

4.1.3 Optical microscope before separation

Pictures were taken with an optical microscope to document the process and to get an overview of the samples. This is valuable information that can be used to compare if there are any differences in sediments where the microplastic is found. The optical microscope works by using light to enlarge the object in focus. Light from a light source will pass through a condenser that focuses the light onto the object to be examined. The light that hits the object is then reflected and passes through the object which then will magnify the image. All equipment to be used was cleaned properly before use and between each sample and fraction. A metal spoon was used to carefully place one spoon of each sample onto a petri dish one sample at a time for inspection. The petri dish was then placed under the microscope. Optical pictures taken before the separation can be seen in Fig 8.

-Additional optical pictures before separation in appendix B.

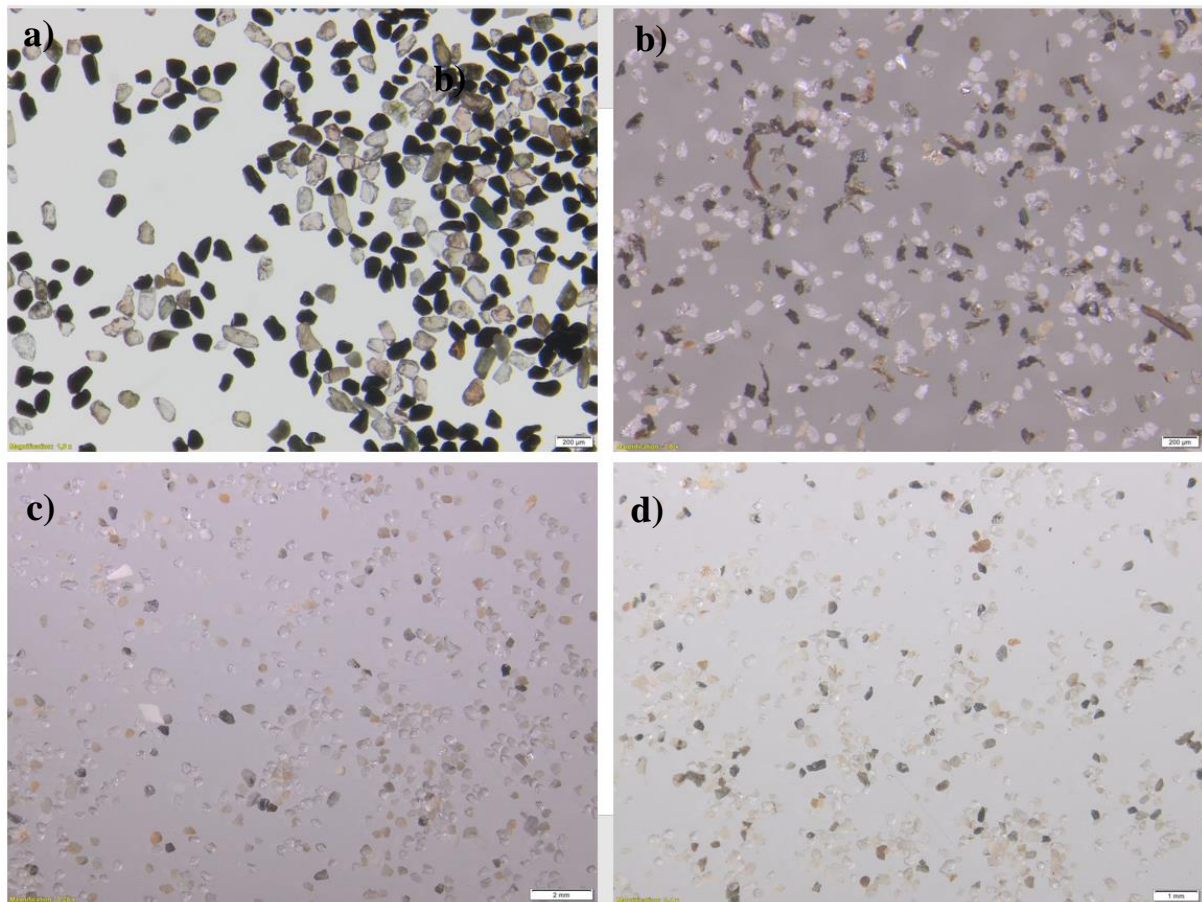


Figure 8 Optical pictures before the separation. a) BO-1_125-63 μm overview of the sample; b) BO-2_63-32 μm ; c) SO-2_500-250 μm overview of the sample; d) SS-1_250-125 μm overview of the sample.

4.1.4 Density separation

The method used to separate microplastic from other sediments depends on several factors as described in the introduction. Grain size and shape, the material, and the amount of material to be separated are factors that need to be considered. Although there is a lack of standardised ways to separate microplastic from other sediments density separation is a widely used method. “Density separation is today the most reliable and commonly used method for the separation of microplastics from sediment, or sand, because the density of the liquid (typically a sodium chlorite or other salt solution) can be adjusted to allow certain plastic materials to float on the surface” (Crawford and Quinn 2016).

As microplastic and other sediments have different densities with microplastic ranging from 0.808 to 1.238 g/cm^3 and other sediments typically 2.65 g/cm^3 (Crawford and Quinn 2016) by using a liquid with intermediate density such as distilled water with a density 1.00 g/cm^3 it is possible to separate all the microplastic with a density lower than 1.00 g/cm^3 . Any particles

with a density lighter than 1.00 g/cm^3 will float in the liquid and thereby separate it from the other sediments which will sink to the bottom.

For this study the liquid used for separating microplastic from the other sediments was sodium polytungstate (SPT). As there was no previous experience with separation of microplastics in the laboratory used for this study, experimental tests with different fluids and different densities were performed. The first separation tests were performed using distilled water with a density of 1.00 g/cm^3 , later sodium polytungstate (SPT) with a density of 1.4 g/cm^3 was used for all samples.

To start the separation, process all equipment to be used was thoroughly cleaned both before and between each sample and fraction. In the microscope it was easy to see that particles were stuck onto other sediments. Therefore ultrasound bath was used to try to loosen the particles that was stuck onto other sediments. The sample was transferred to a beaker before it was filled with distilled water as seen in Fig 9 a). The beaker with distilled water and the sample was placed in an ultrasound bath for 3 minutes as seen in Fig 9 b).

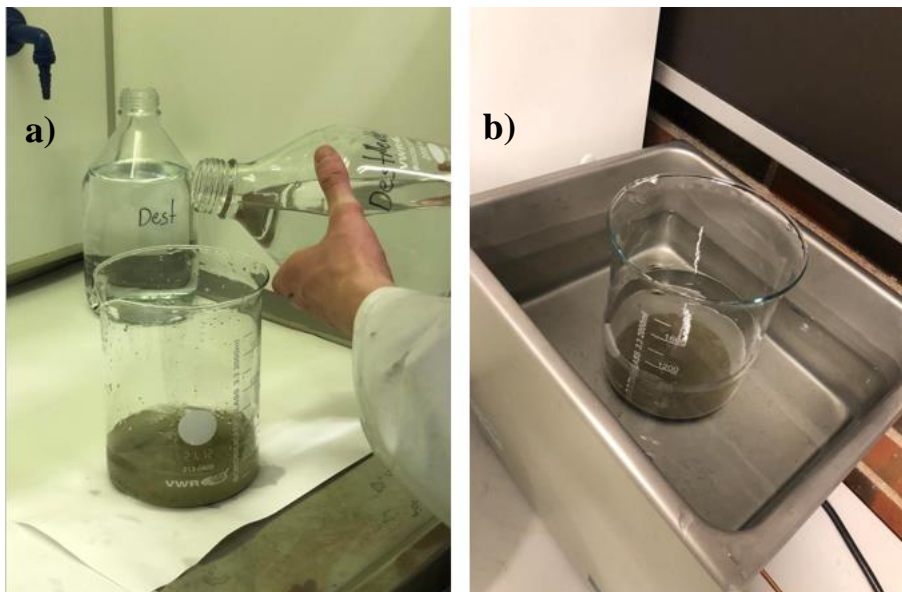


Figure 9 Sample preparation. a) Sample preparation before ultrasound bath; b) Beaker with sample and distilled water placed in ultrasound bath.

After the ultrasound bath the beaker was placed in a fume hood. A metal funnel was placed at the top of the cylinder and distilled water was then poured into the cylinder by holding a finger to the spout to avoid any liquid or sample to get out (Fig 10). The cylinder was filled about half full of distilled water. The beaker with the sample was decanted carefully into the funnel. To get all the sediments out of the beaker a squirt bottle was used (Fig 10). After all

the sediments was out of the beaker the funnel was flushed to any sediments left in the funnel added to the cylinder. The funnel was carefully taken out of the cylinder to avoid it from getting in contact with the fluid and to avoid contamination. The cap was then placed on top of the cylinder. The cap was held in place and the spout was still covered while the cylinder was turned up and down 10 times as seen in Fig 11, to mix the sample properly with the distilled water. The cylinder was placed under ventilation with the beaker placed under the spout. The time was set to 60 minutes, allowing the sample to settle (Fig 12 a).



Figure 10 The process of decanting the sample into the separation cylinder.

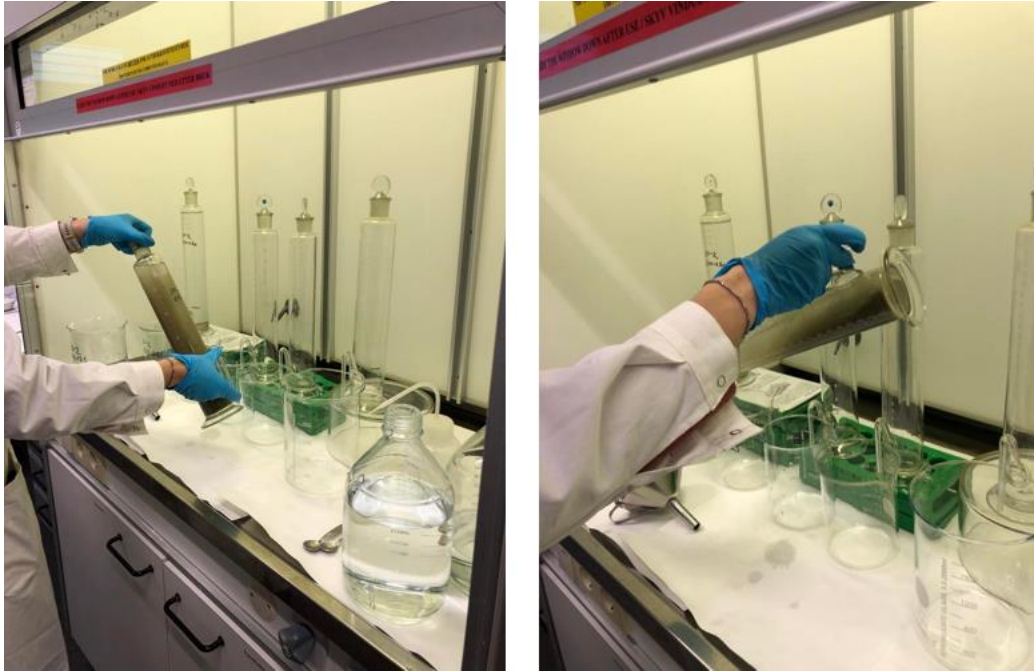


Figure 11 Showing the separation cylinder turn up and down to mix the sample with the distilled water.

After 60 minutes the cap was removed, and a squirt bottle was used to avoid getting any sediments left at the walls of the cylinder as the material floating in the liquid was decanted into the marked beaker placed under the cylinder as seen in (Fig 12 b).

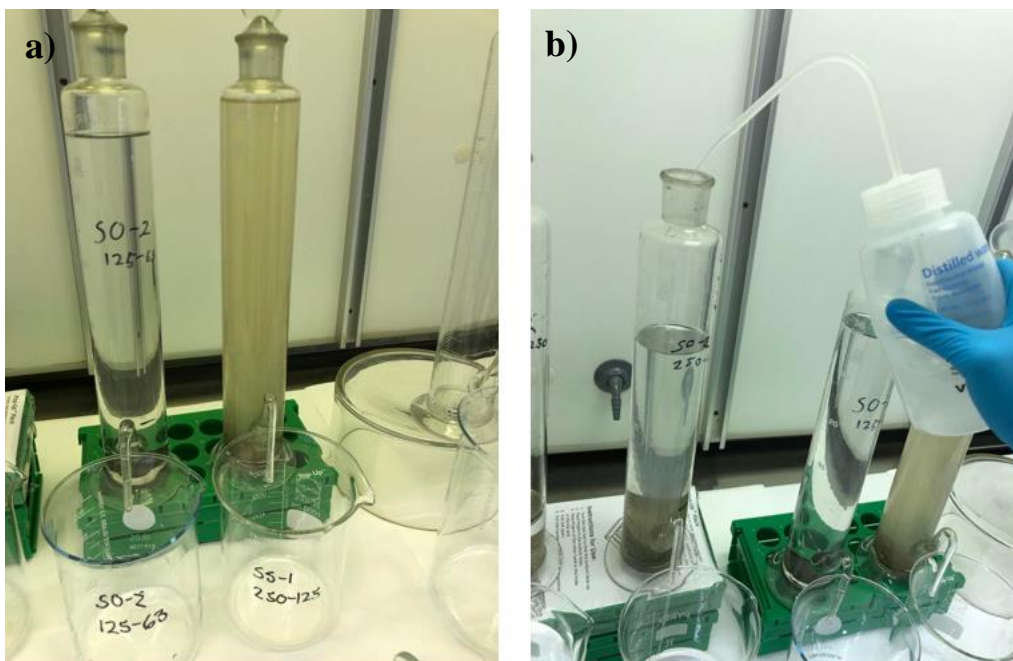


Figure 12 Density separation. a) Separation cylinders filled with distilled water and sample; b) Squirt bottle used to flush the cylinder and avoid sediments left at the wall.

The beaker with the sediments and distilled water was filtrated through a 25 μm metal filter. The filter was cut with scissor in proper size of about 15 x 15 cm. Every filter was weighted

before use. The filter was placed on a bottle and attached with hemp as seen in Fig 13. The beaker with the distilled water and the material was decanted carefully over the filter to filtrate out the material collected. The filter was carefully loosened from the bottle, closed, and placed in plastic beakers that was covered by aluminum foil as seen in Fig 14 a). The beaker was labeled with name and fraction and stored in a warming cabinet. The maximum temperature used for drying should not be higher than 60° C (Campanale, et al. 2020). The temperature used in this study was 40° C. The sample was left to dry over the night as seen in Fig 14 b).

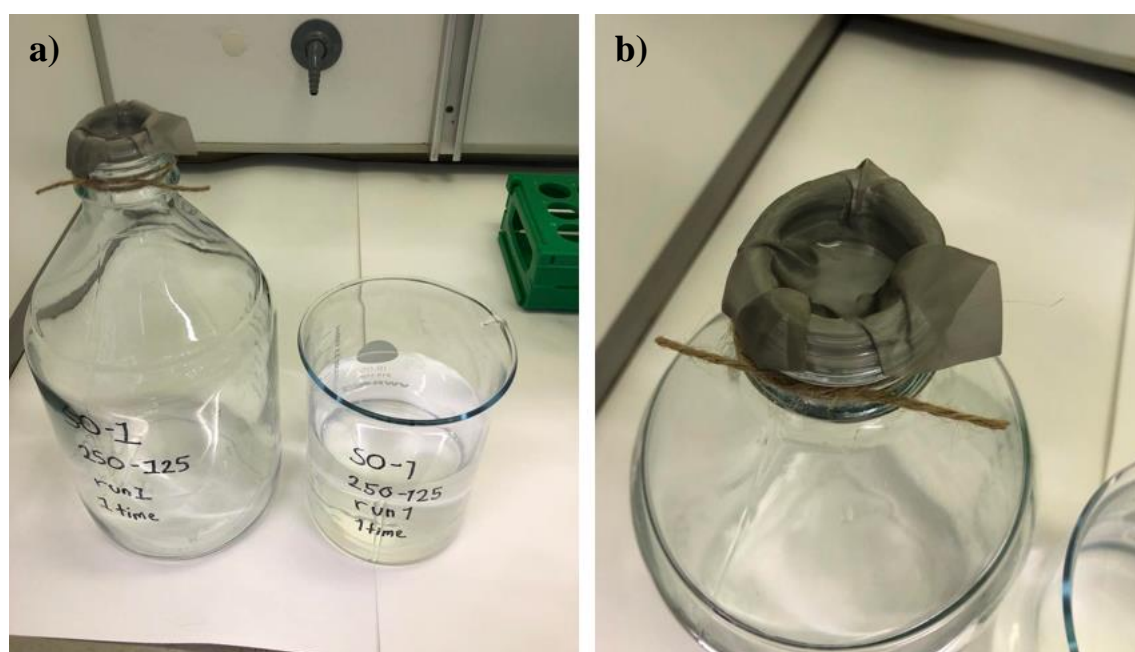


Figure 13 The filtration process for sample SO-1.

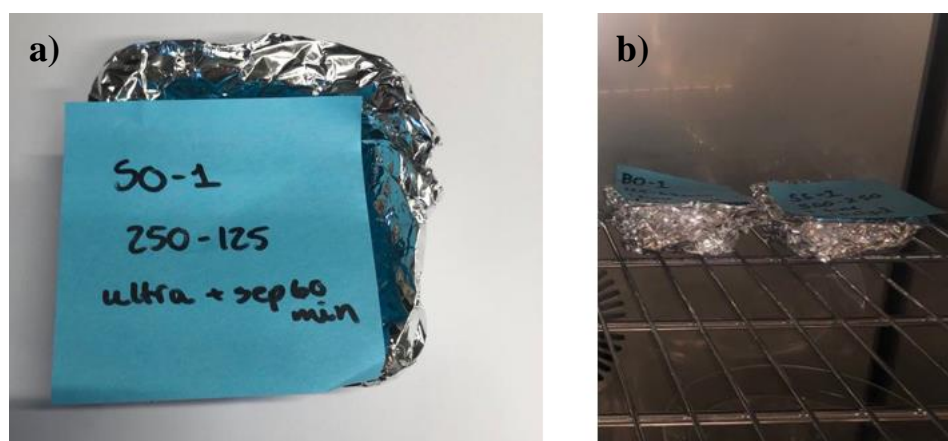


Figure 14 Storing of the samples after separation. a) Beaker covered by aluminum foil; b) Samples stored in the warming cabinet.

The bottom residue in the cylinder was taken out by turning the cylinder up down over the beaker that was used for the decantation. To recover all the sediments left in the cylinder a squirt bottle was used. When all the sediments were collected the material and the distilled water in the beaker was filtrated in paper filters as shown in Fig 15.



Figure 15 Filtration of the bottom residue.

After the filtration, the filters were closed and placed in the warming cabinet. All samples were defined as larger than 25 μm in the sieving process, hence the residue in the beaker already has particle sizes less than 25 μm . Therefore, there was no need to save the residue in the beakers at this stage. After one day in the warming cabinet the filters with the sample were taken out and weighted again to see how much material was collected in the filters, (appendix C). Every sample was inspected again under optical microscope as seen in Fig 16 after the density separation to control the yield. Additional optical pictures before separation in appendix D.



Figure 16 Optical pictures after separation with distilled water. a) BO-1_250-125 μm overview of the collected sample where a blue fiber can be seen; b) SO-1_500-250 μm overview of the collected sample where fibers can be seen.

After a few samples were separated with distilled water the same samples were again separated with SPT. The density of the SPT had to be 1.400-1.403 g/cm^3 to be accepted. The procedure of density separation with SPT are similar as for the process with distilled water only now the distilled water was changed with SPT. After the separation with SPT the material that was collected in the filters was properly cleaned to avoid any solvent of SPT to be left in the sample before the samples were stored in the warming cabinet overnight. SPT crystallizes quickly which can lead to false interpretation results. After one night in the warming cabinet the samples were inspected under optical microscope as seen in Fig 17.



Figure 17 Optical pictures after separation with SPT. a) BO-1_250-125 μm several fibers in multiple colors; b) SO-1_500-250 μm several fibers pushed together into a ball.

4.1.5 Samples larger than 2000 μm

After sieving the samples all visible microplastic was collected by hand or tweezers. Double-sided tape was cut in proper size with scissors and glued onto glass slides. One slide was used for each sample. The collected microplastic was glued onto the glass slide using the tape as seen in Fig 18. The slides were covered by a thin glass cover to protect the samples for damage and contamination.



Figure 18 Microplastic particles from samples larger than 2000 μm .

4.2 In-house library

In-house library was made using known plastics collected at home. The plastic was cut off with a metal knife circa 1 x 1.5 cm small pieces. The pieces were glued with double-sided tape onto glass slides as shown in Fig 19. The slides were covered by a thin protection glass to protect the samples for damage and contamination.

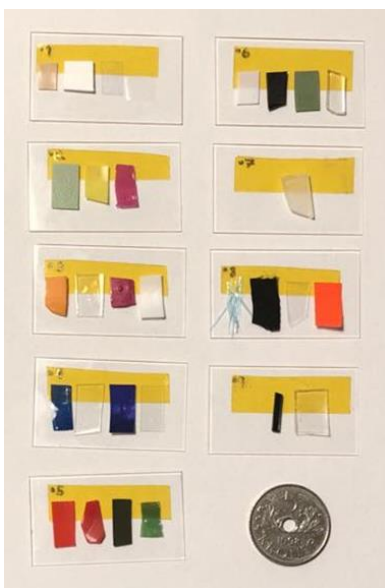


Figure 19 Self-prepared plastic particles collected at home and prepared at the laboratory.

4.3 XRD

X-Ray Diffraction (XRD)-analysis are carried out to determine the chemical compositions in the analysed samples. An XRD analysis is performed by exposing the sample to be examined with x-rays that will hit the sample. The sample then will spread out in a specific pathway that depends on the crystal structure of the sample. The samples to be analysed were first grounded, which is necessary to obtain a uniform and homogeneous powder. It is important that the powder to be analysed has a larger surface area for it to interact with the X-rays as it will be easier for the x-rays to hit the crystals in the sample and measure the spread of the x-rays.

Experimental setup:

Diffractometer: Bruker D8 Advance Eco diffractometer equipped with a Lynxeye detector (Cu-K α radiation, 40 kV voltage, 25 mA current).

Measuring conditions: 5-90 degrees (2theta), 0.6 mm divergence slit, measurement time 0.6 seconds pr step, increment 0.01. Rotation on (rotating the sample continuously while measuring). Total measurement time 85 minutes pr sample.

4.4 Particle size and distribution

It is important to measure the size and distribution of particles in samples because this can have a major impact on the properties and functionality of samples and assist when interpreting results. By measuring the particle size and distribution (PSD), we can better understand the properties of samples and optimize it for different applications. In a study on microplastics, the microplastic particles can be so small that they cannot be seen with the naked eye meaning that they can be difficult to distinguish from natural particles in a sample. Master sizer 3000 was used for particle and size distribution analyses on dry samples. Every sample was first split two times and then sieved manually with a 2 mm mesh. Every sample was weighted before samples were fed into the machine which uses laser diffraction to measure the particle size and distribution. Every sample was tested four times and non-spherical particles were assumed. The machine gives robust and rapid particle size measurements over a range of 0.01-3500 μ . The results are displayed in nearly real time and the software Malvern Panalytical calculates immediately the range of grains (span) and the variation of grains (uniformity), and the range of the most abundant grain sizes D(10), D(50)

and D(90), where D(10) means that 10 % of the grains have a size of the given value as a lower value, D(50) means 50 % have the given values and D(90) means that 10 % of the largest grains have a size of the given value.

4.5 Raman spectroscopy

Raman spectroscopy was used to identify the possible microplastics. “The analysis of plastic materials using Raman spectroscopy was first published by Signer and Weiler in 1932, in which they obtained a Raman spectrum for polystyrene” (Crawford and Quinn 2016). Since the arrival of the laser Raman spectroscopy, a lot of literature has been published for use of Raman techniques regarding microplastics. Raman spectroscopy is a non-destructive chemical analysis application which provides detailed information about the chemical structure of the exposed molecule/material. The method uses a combination of light microscope and a Raman spectrometer allowing high magnification visualization of a sample and Raman analysis with a microscopic laser spot (Nandi 2021).

Raman spectra are obtained by exposing a sample to a high-intensity laser beam. The sample will then scatter the incident light where most of the scattered light will have the same wavelength as the laser source and therefore does not provide any useful information. This is called the Rayleigh scattering. However, a small amount of the light (usually 0.0000001%) is scattered at different wavelengths. This is called the Raman Scattering (Nandi 2021). The energy difference between the incident light and the scattered light is called the Raman shift (Nandi 2021).

Raman spectra were obtained by using a combination of in Via Renishaw, a Leica stereomicroscope, and a x-y stage. Laser sources of 532 nm and 633 nm were used. Calibration was made using an integrated internal standard of Silicon before each session. Before every session two calibrations were performed. One quick calibration and one template calibration. The calibration is important to check the center, width, and height of the peak. The center should be at about 520.5 cm^{-1} and the height depends on the used laser.

The Raman spectra were measured by centering the spectra range on spectrum ranging from $100\text{-}3200. \text{ cm}^{-1}$ and using a combination of the 532 nm and the 633 nm laser. The magnification for all the samples was regulated between 5x and 50x depending on the fibers or particle size. Laser power was controlled to avoid heating effects. A quick 10 sec test, with 1 accumulation and the intensity of the laser fixed at 50% was carried out at each

microparticle. The standard for each sample was set to 10 sec with 5 accumulations and the laser fixed at 50 % and then adjusted as appropriate for each sample. When the signal was collected, the baseline was extracted to remove the background noise, and then cosmic rays were removed if any. A total of 37 Raman spectra were collected. The collected spectra were classified comparing the unknown spectrum with two different libraries (University of Milano-Bicocca, Italy n.d.) and (PublicSpectra 2019). Matching percentages were calculated automatically by the spectral search function at (PublicSpectra 2019).

5 Theoretical background

The recent years rapid increase in both production and consumption of plastics has serious impact on both the environment and humans (Heidbreder, et al. 2019). Billions of tonnes of plastic are already released into the environment and most of the plastic are still present in one way or another. Plastic is highly resistant to degradation, and it may take thousands of years to completely degrade. “In 1950 the world produced 2 million tons of plastic annually and since then annual production has increased to 460 million tons in 2019. The production is further expected to double by 2050” (Ritchie and Roser 2018). Although plastic has contributed to a lot of achievement within technological innovation it may ultimately lead to significant environmental problems (Crawford and Quinn 2016).

5.1 What is plastics

Plastic is a synthetic material that is made through a process known as polymerization, illustrated in Fig 20. Plastic substances are composed of large chain-like molecules, termed macromolecules (Crawford and Quinn 2016). Each individual molecule in a polymer chain is called a monomer where each of the monomer are a single unit. These single monomers have the ability to bound together and form long chains through the process called polymerization. Substances with this molecular arrangement are called polymer (Crawford and Quinn 2016). When chains are formed, they can be moulded and shapes to form new solid objects. “The vast majority of polymers in today’s world are the synthetic plastics created by humans. However, there are also many natural polymers in existence. Thus, it is important to note that although all plastics are polymers, not all polymers are plastics” (Crawford and Quinn 2016).

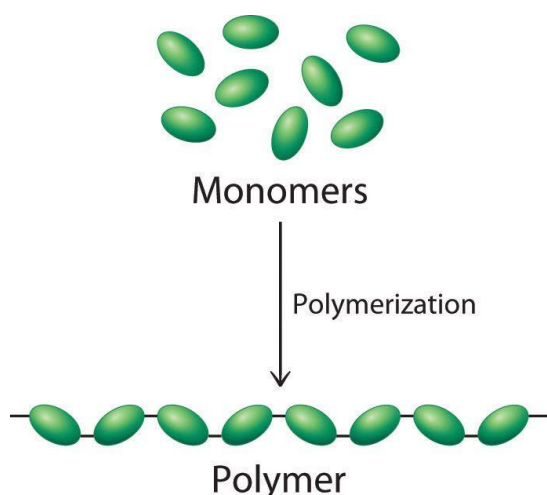


Figure 20 Illustrating how polymers are formed from single units of monomers through polymerization. (Bradford, What Is a Polymer? 2017)

5.2 Classification

Crawford and Quinn (2016) define a piece of plastic that is equal to or larger than 25 mm in size to be classified as microplastic. Pieces of plastic that is smaller than 25 mm are further classified based on their size where the size refers to the size along its longest dimension as shown in Table 1.

Table 1: Size categories for pieces of plastic compiled from (Crawford and Quinn 2016)

Category	Size
Macroplastic	≥ 25 mm
Mesoplastic	<25 mm-5 mm
Plasticle	<5 mm
Microplastic	<5 mm-1 mm
Minimicroplastic	<1 mm-1 μ m
Nanoplastic	<1 μ m

5.3 History

The term plastic was first used in the 1630s and the term was used to describe a substance that could be molded and shaped. The term has its origin from the Ancient Greek term plastic that refers to something that is suitable for or moulding, and the Latin term plasticus which pertain to moulding or shaping (Crawford and Quinn 2016). In 1863 an advertisement offered a price of \$10 000 to anyone who could find a substance to replace ivory. An American named John Wesley Hyatt took up the challenge and in 1868 he invented a new material called celluloid. Celluloid is a material that is easy to mould and shape and celluloid are known as the first plastic. However, celluloid could not replace ivory because the material wasn't heavy enough.

Celluloid is also highly flammable which makes the production risky. In 1907 a Belgian chemist named Leo Hendrick Baekeland combined phenol-a waste product of coal tar-and formaldehyde, creating a hardy new polymer. Baekeland named the new substance after himself and it was called bakelite. Bakelite was much less flammable than celluloid and the raw materials used to make it were more readily available. Bakelite transformed the world at the time and by the end of the 1930s more than 200.000 tonnes of Bakelite was produced. (Crawford and Quinn 2016). In the 1920s, researchers first commercially developed polystyrene, a spongy plastic used in insulation. Soon after came polyvinyl chloride which was a substance that are both flexible and hard. Acrylics created transparent, shatter-proof panels that mimicked glass, and in the 1930s nylon took centre stage, a polymer designed to mimic silk, but with many times its strength. Starting in 1930, polyethylene became one of the most versatile plastics, still used today to make everything from grocery bags to shampoo bottles.

5.4 Plastics and general classification

Plastic can be derived into two main categories, thermoplastics, and thermosetting plastics. Thermoplastics can be reshaped if it is heated while thermosetting plastic will have its finale shape when the plastic is hardened. Most common and abundant plastic types are shown in Table 2 and 3.

Table 2: Most common plastics compiled from (Crawford and Quinn 2016)

Name	Abbreviation	Classification	Chemical formula
Polyethylene terephthalate	PET or PETE	Thermoplastic	$(C_{10}H_8O_4)_n$
Polyethylene	PE, LDPE, LLDPE, HDPE	Thermoplastic	$(C_2H_4)_n$
Polyvinyl chloride	PVC	Thermoplastic	$(C_2H_3Cl)_n$
Polypropylene	PP	Thermoplastic	$(C_3H_6)_n$
Polystyrene	PS	Thermoplastic	$(C_8H_8)_n$
Polycarbonate	PC	Thermoplastic	$(C_{16}H_{18}O_5)_n$
Polyamide (nylon6)	PA	Thermoplastic	$(C_6H_{11}NO)_n$
Polyamide (nylon6.6)	PA	Thermoplastic	$(C_{12}H_{22}N_2O_2)_n$

Table 3: Most abundant plastics compiled from (Canals, Haan and Sàncnez-Vidal 2019)

Name	Percentage (%)
Polyethylene	54.5 %
Polypropylene	16.5 %
Polyester	9.7 %

6 Results

6.1 Grain size distribution

The sand samples have been studied in terms of the grain size distribution. Particle size distribution (PSD) was performed with Malvern Master Sizer 3000 on dry samples. The generated cumulative percentage curves for each sample are shown in Fig 21-26. The steeper the curve the better sorted is the material. As seen in Fig 21-26 the curve is steeper for SO than for both BO and SS, indicating that SO has the best sorting. SO has span of grainsizes by 0.704 for SO-1 and 0.686 for SO-2. The grain distribution curve for BO is less steep than SO but steeper than for the samples of SS. The grain size span for BO is 0.913 for BO-1 and 0.829 for BO-2. SS is the least steep curve and has even a span of 1.938 for SS-1 and 2.099 for SS-2 making the SS the poorest sorted sample. The span varies and for BO and SO it is less wide for the deeper samples but not for SS where the deeper sample is even less sorted. SO is the most uniform sample with a variation of grains sizes ranging from 229-245 μm for SO-1 and 232-247 μm for SO-2 (fine sand) and are more uniform than BO. BO is more uniform than SS with a variation of grain sizes ranging from 328-366 μm for BO-1 and 302-331 μm (medium sand) for BO-2. SS is poorly sampled with a variation of grain sizes of 552-948 μm for SS-1 and 443-812 μm (medium to coarse sand) for SS-2. The distribution of grain sizes in the samples are shown in detail in Table 4. The span shows the range of grains, (if perfectly sorted then span = 0), uniformity the variation of grains. The D values [3,2] and D [4,3] is the range of the most abundant grain sizes. D(10) means that 10% of the grains have a size of the given value as a the lower value, D(50) means 50% of the grains have the given value, and D(90) means that 10% of the largest grains have a size of the given value.

Table 4 The distribution of grain sizes in the samples.

Sample	Span	Uniformity	D [3,2] (µm)	D [4,3] (µm)	Dv (10) (µm)	Dv (50) (µm)	Dv (90) (µm)
SO-1	0.704	0.217	229	245	168	237	335
SO-2	0.686	0.211	232	247	171	239	335
BO-1	0.913	0.282	328	366	223	346	538
BO-2	0.829	0.256	302	331	212	316	474
SS-1	1.938	0.596	552	948	279	802	1830
SS-2	2.099	0.655	443	812	221	663	1610

SO

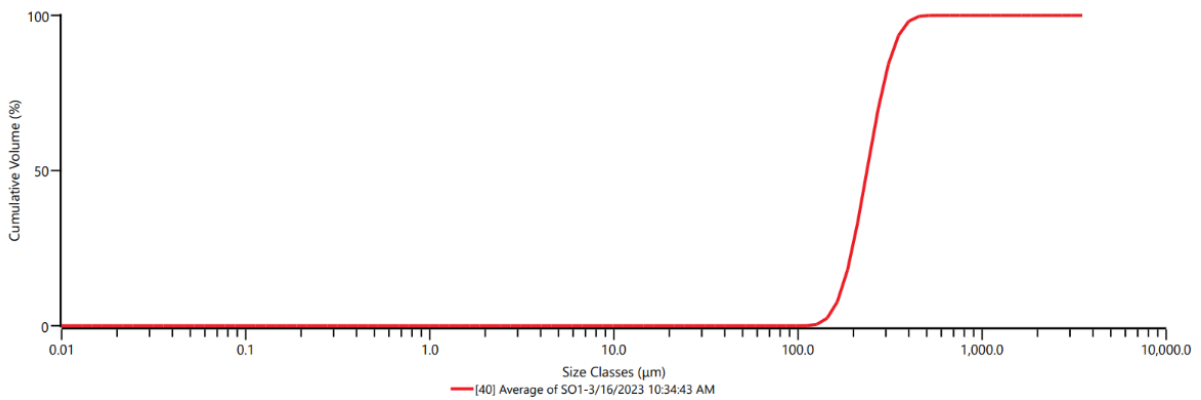


Figure 21 Cumulative percentage curve for SO-1.

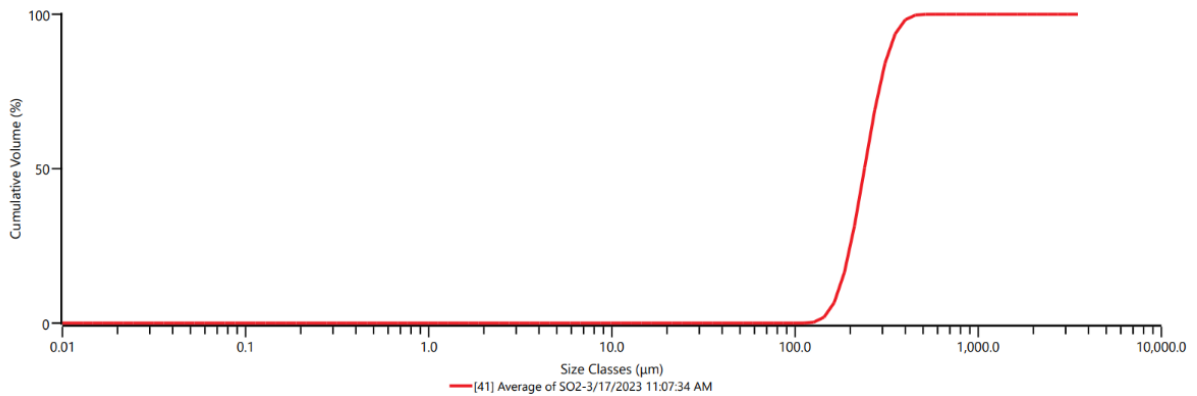


Figure 22 Cumulative percentage curve for SO-2.

BO

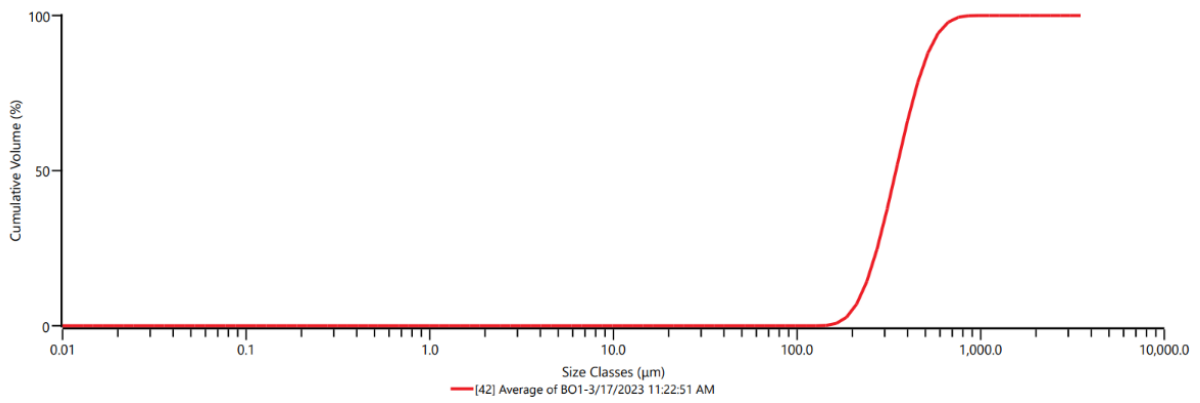


Figure 23 Cumulative percentage curve for BO-1.

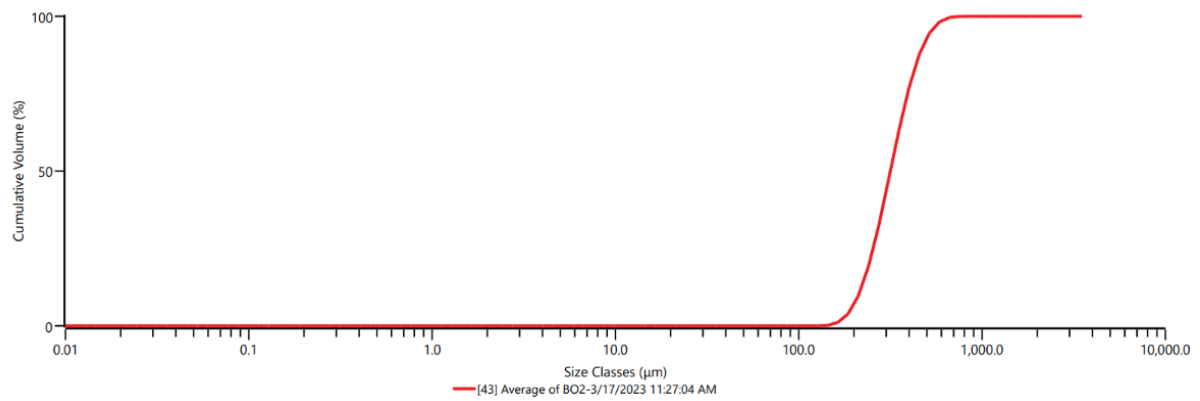


Figure 24 Cumulative percentage curve for BO-2.

SS

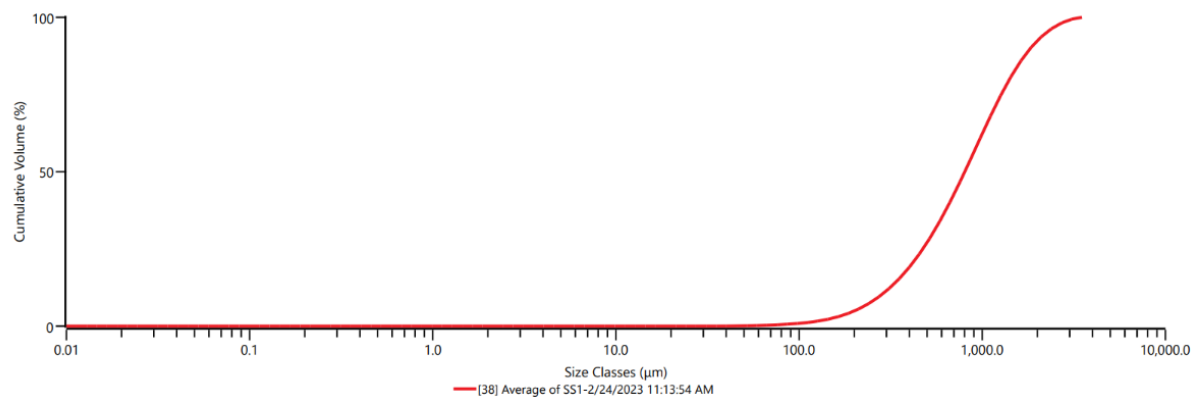


Figure 25 Cumulative percentage curve for SS-1.

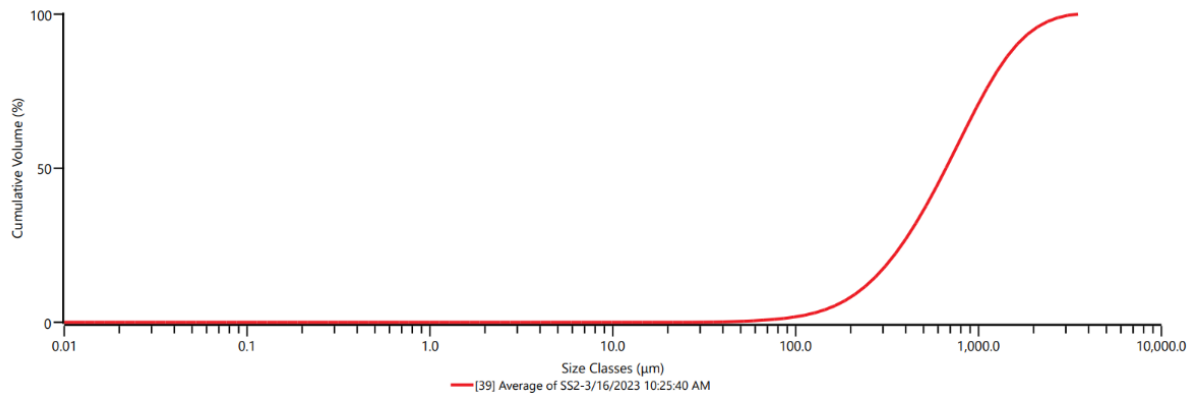


Figure 26 Cumulative percentage curve for SS-2.

-Full PSD analysis results in appendix E

6.2 Mineralogical composition of the samples

Results from XRD analysis show that the main mineral groups in the samples are quartz, feldspar, micas, chlorite, and amphibole (Fig 27-32). All samples have a similar composition with the difference to SS sample which has considerable higher amounts of chlorite and micas than SO and BO.

SO

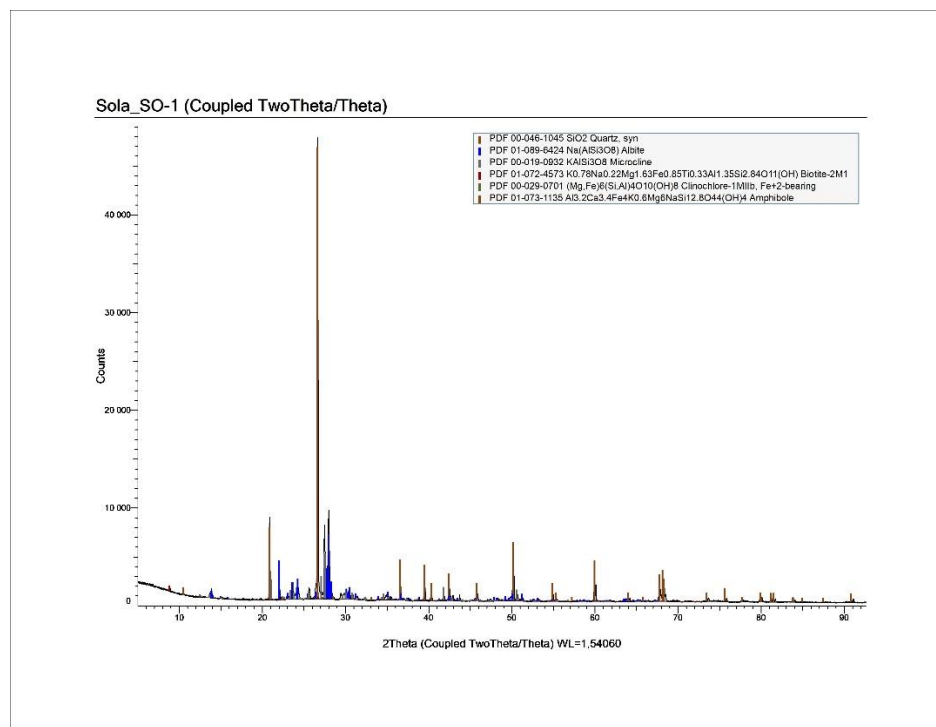


Figure 27 XRD patterns of SO-1.

Sola_SO-2 (Coupled TwoTheta/Theta)

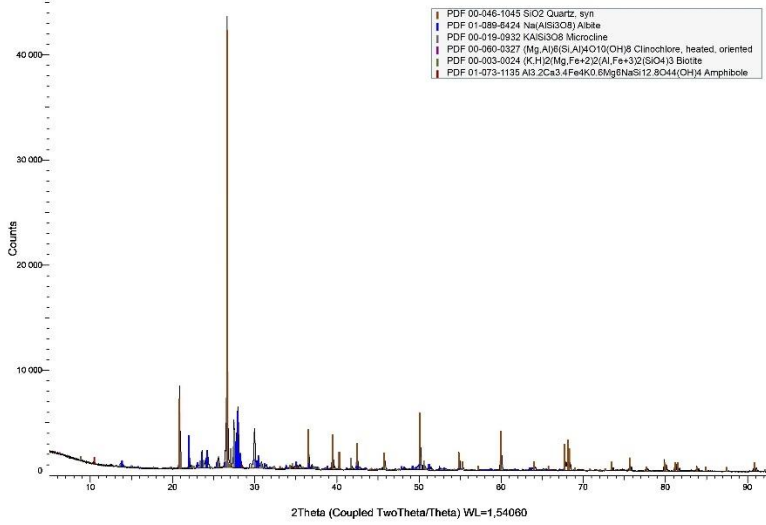


Figure 28 XRD patterns of SO-2.

BO

Bore_BO-1 (Coupled TwoTheta/Theta)

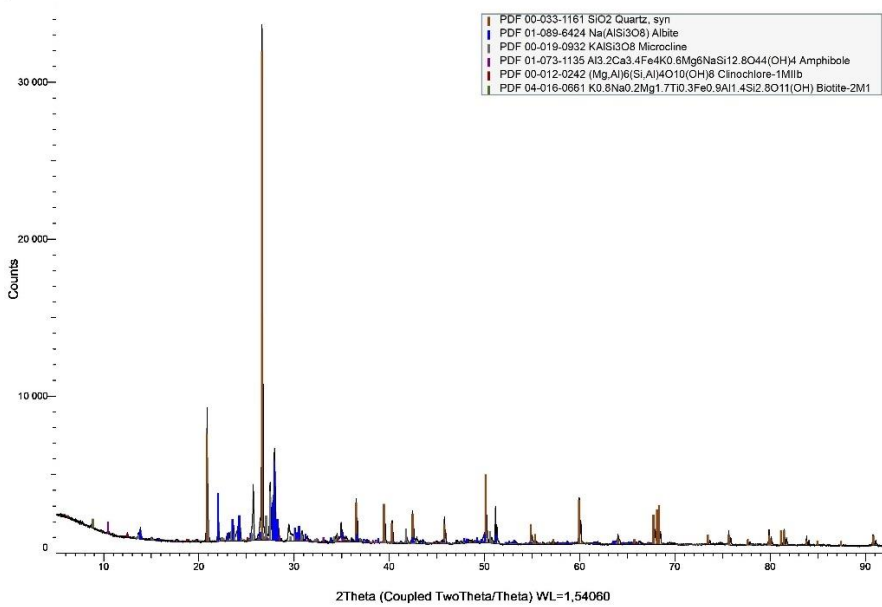


Figure 29 XRD patterns of BO-1.

Bore_BO-2 (Coupled TwoTheta/Theta)

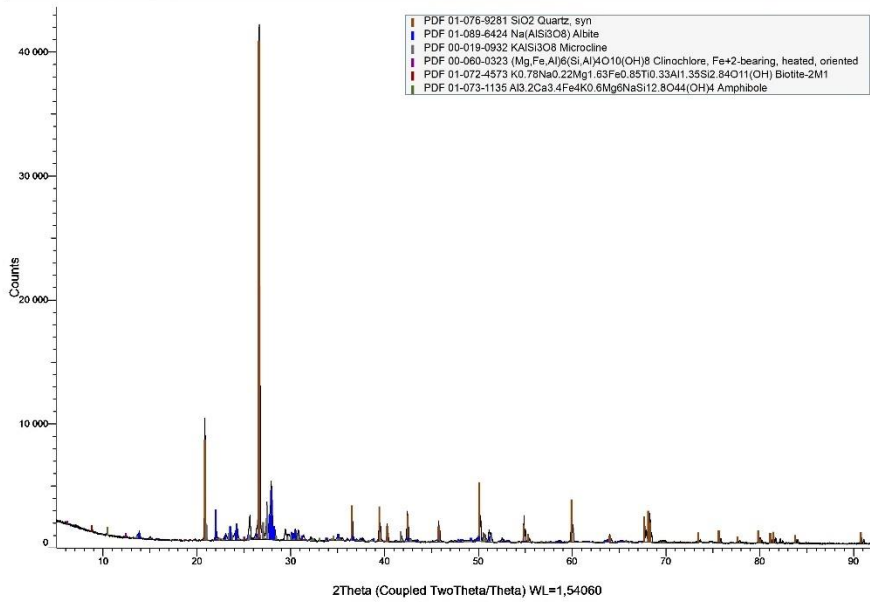


Figure 30 XRD patterns of BO-2.

SS

SS-1 (Coupled TwoTheta/Theta)

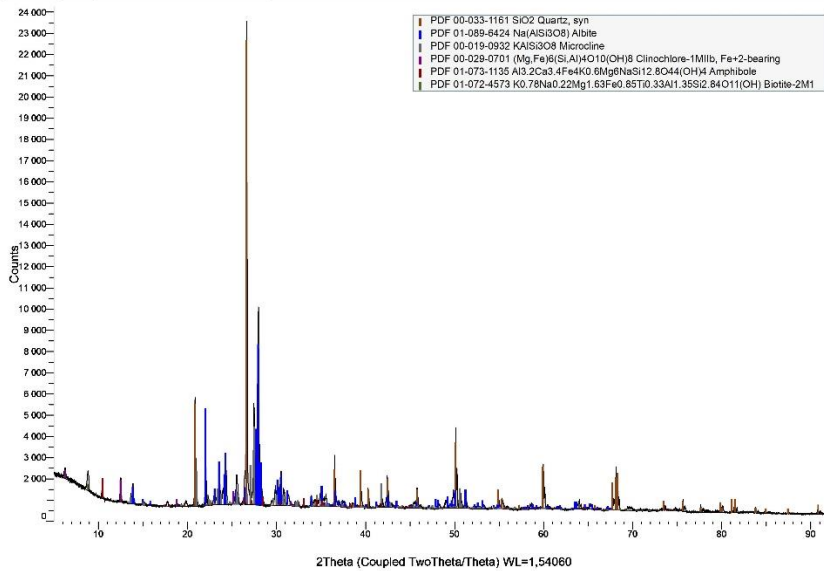


Figure 31 XRD patterns of SS-1.

SS-2 (Coupled TwoTheta/Theta)

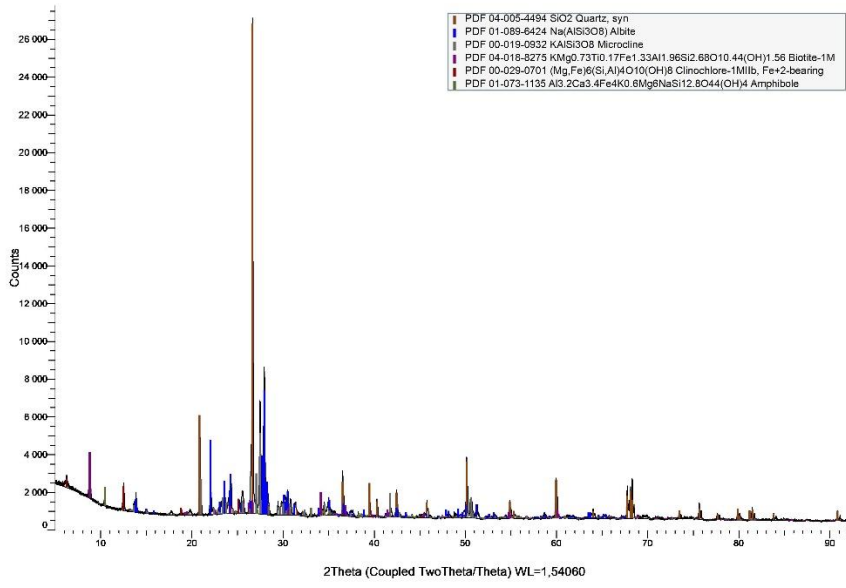


Figure 32 XRD patterns of SS-2.

6.3 Optical microscopy

Several samples had particles of fibres that were easy to identify using an optical microscope (Fig 33-34).

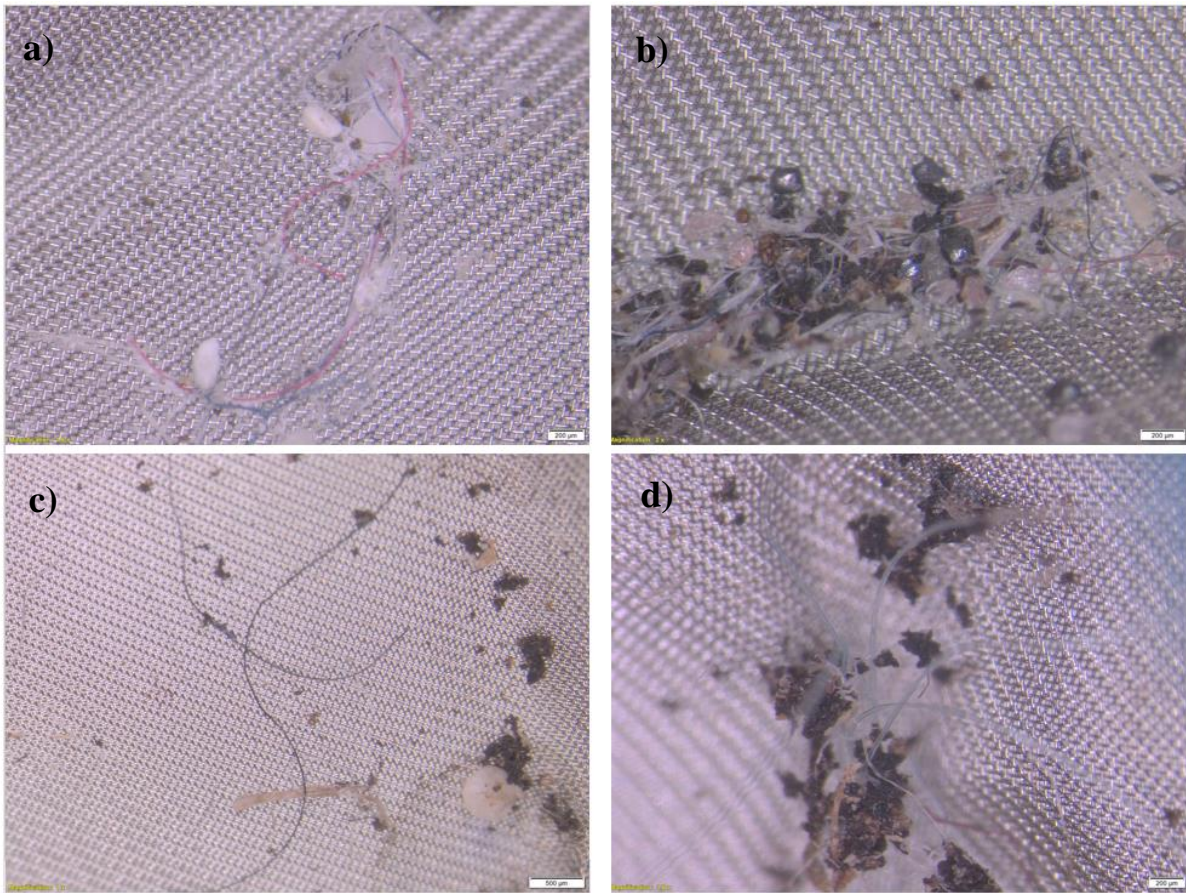


Figure 33 Optical microscope images of the collected samples after sieving and separation a) BO-1_250-125 µm overview of the collected sample showing several fibers; b) BO-1_125-63 µm overview of the collected sample showing several fibers; c) BO-2_500-250 µm two black fibers; d) BO-2_125-63 µm several transparent fibers.

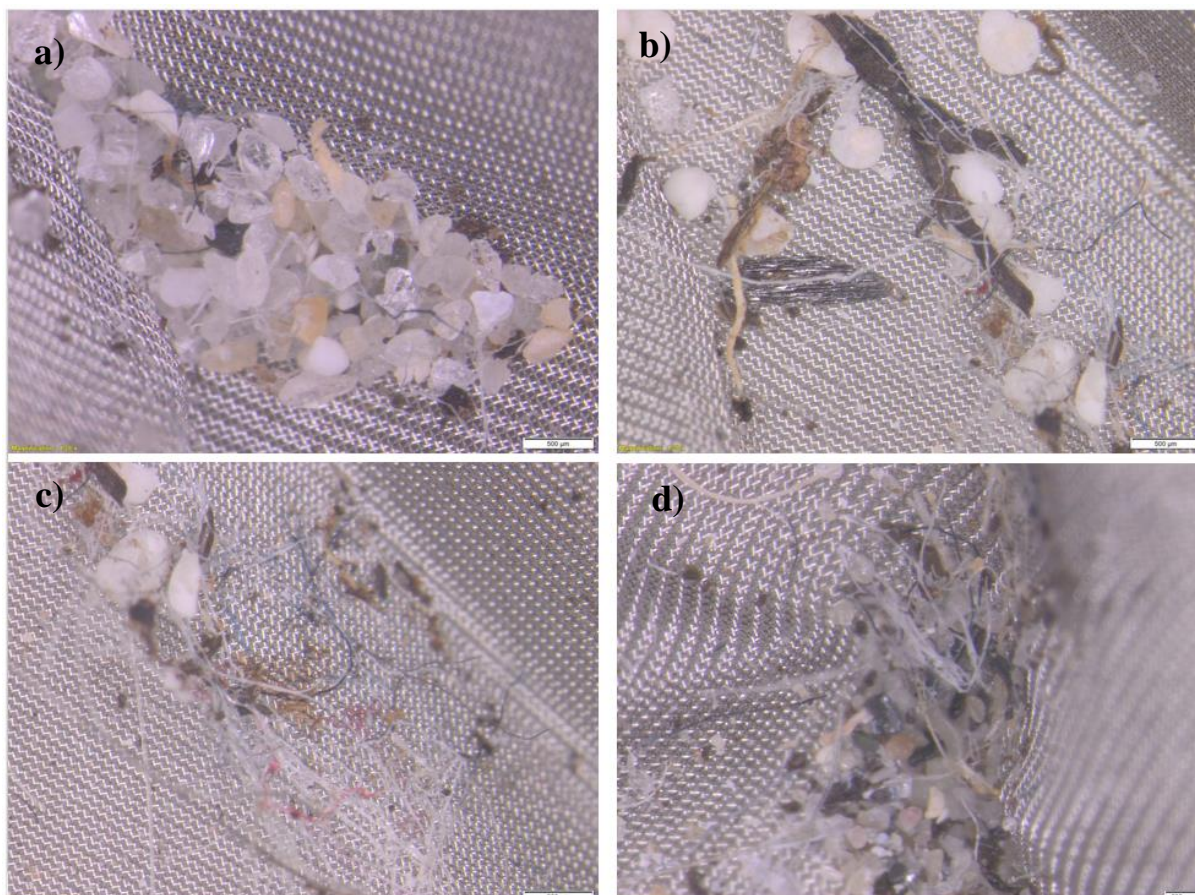


Figure 34 Optical microscope images of the collected samples after sieving and separation a) SO-1_250-125 μm overview of the sample; b) SO-2_500-250 μm overview of the sample showing several fibers; c) SO-2_500-250 μm overview of the sample showing several fibers; d) SO-2_125-63 μm overview of the sample showing several fibers.

-Compiled workflow diagram for the methodology can be seen in appendix F.

6.4 Raman spectroscopy

6.4.1 In house (UiS) library

The aim was to determine a Raman spectrum of samples which have been known as they are labelled as specific plastic types and collected in the household. The spectra were then compared to those from literature. This allowed to control if labelling is correct and if variation within composition in the selected sample was observed. From each sample a small block had been cut off for analysis. The microscope glass, a paper block and the Teflon beaker were also tested. As seen if Fig 35-45 the obtained spectra are highly comparable to those spectrum published in literature (PublicSpectra 2019). A matching percentage had been calculated according to the described method in chapter 4.5.

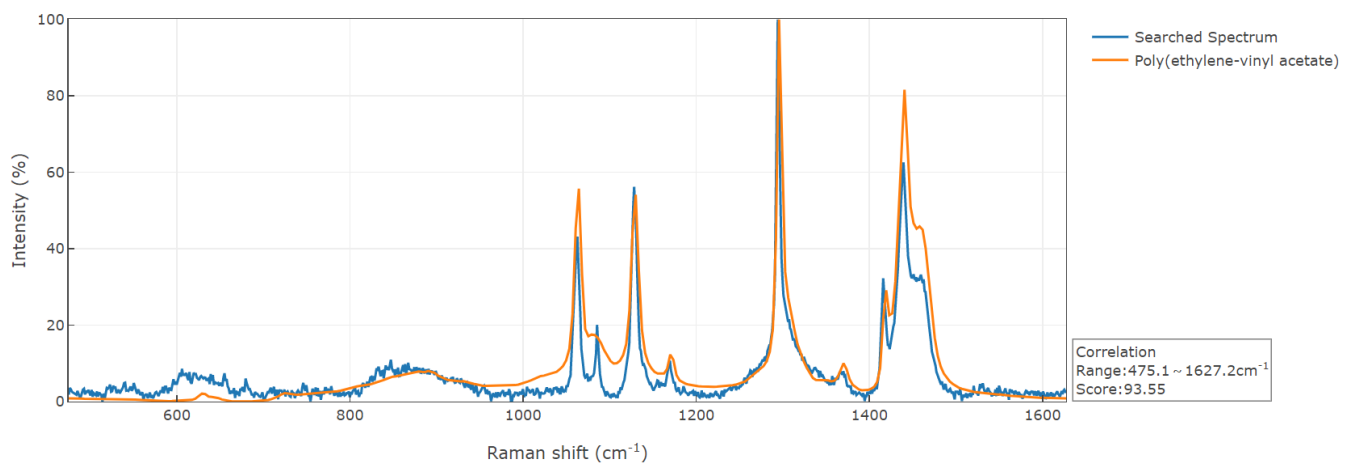


Figure 35 Obtained spectra for plastic grocery bag (searched spectra) labeled PE-LD with 93.55 % match with ethylene-vinyl acetate (PE-LD). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

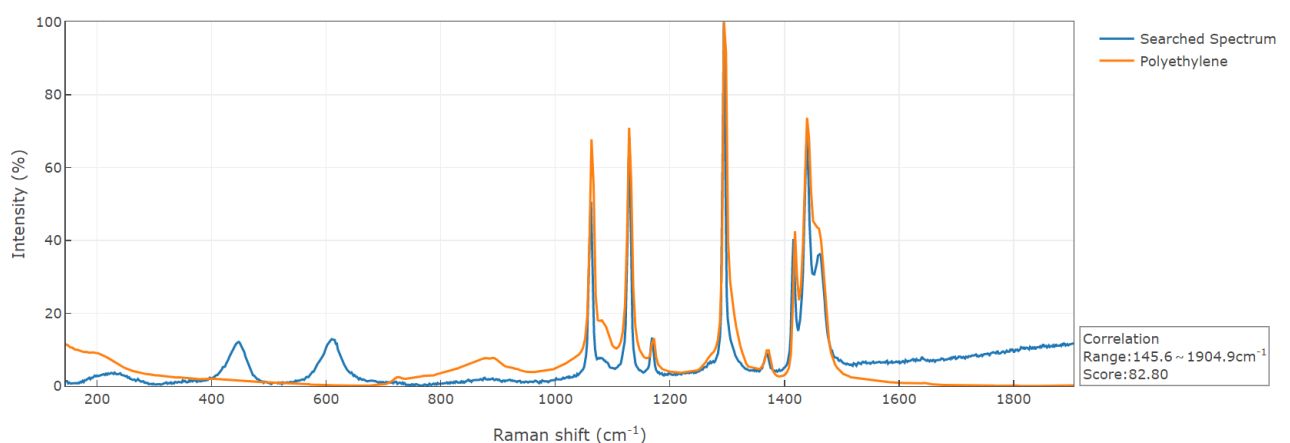


Figure 36 Obtained spectra for a shampoo bottle (searched spectra), labeled HD-PE with 82.80 % match with Polyethylene (PE). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

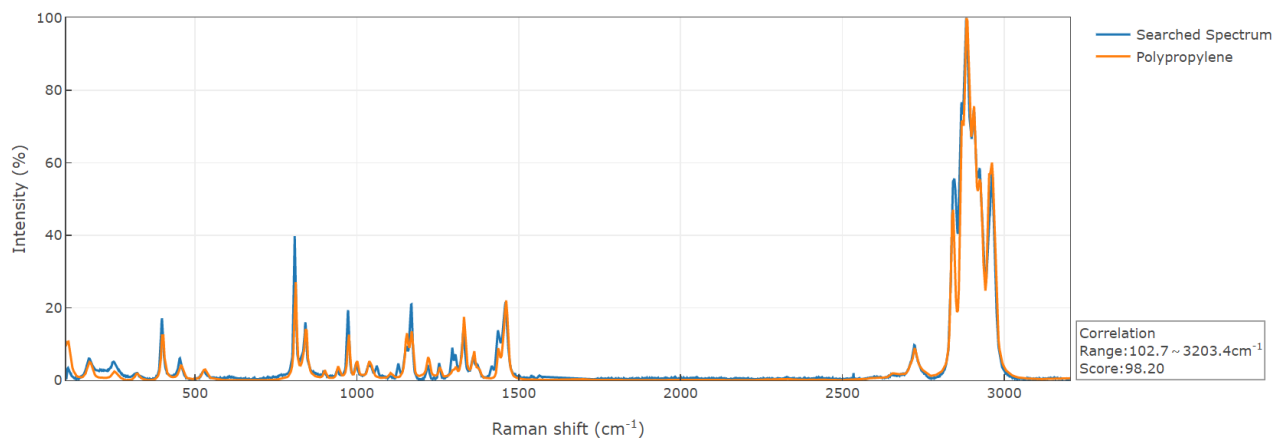


Figure 37 Obtained spectra for kinder maxi surprise egg (searched spectra), labeled PP with 98.20 % match with polypropylene (PP). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

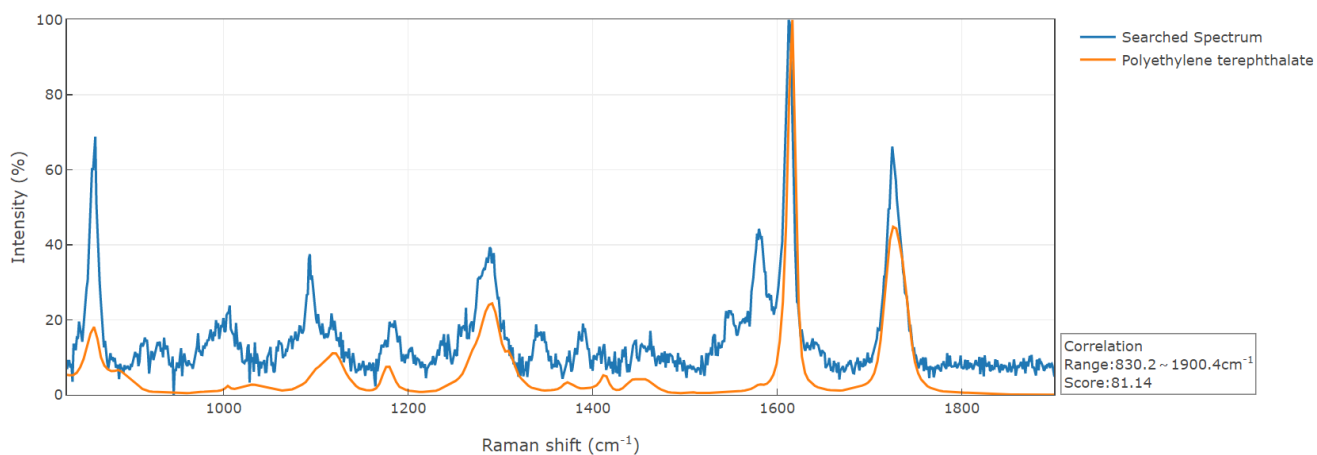


Figure 38 Obtained spectra for jacket fabric (searched spectra), labeled polyester with 81.14 % match for Polyethylene terephthalate (polyester). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

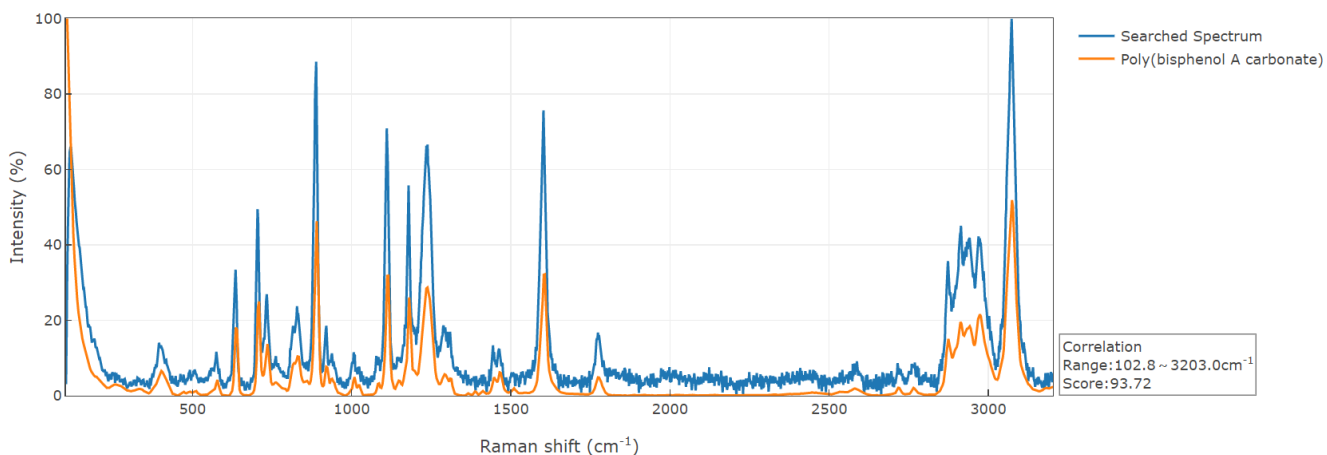


Figure 39 Obtained spectra for a piece of light bulb (searched spectra), labeled PC with 93.72 % match for poly (bisphenol A carbonate) (PS). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

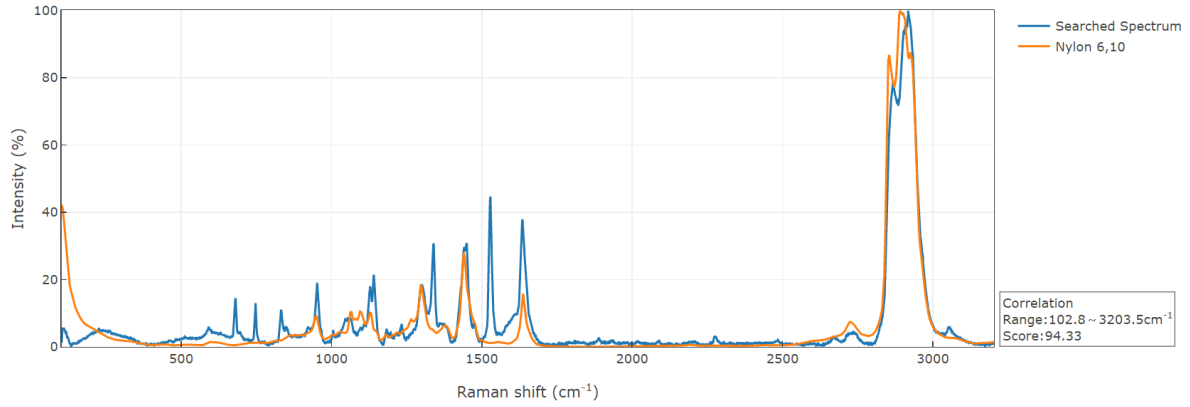


Figure 40 Obtained spectra for a black nylon strip (searched spectra), labeled (nylon) with 94.33 match for Nylon 6.10. Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

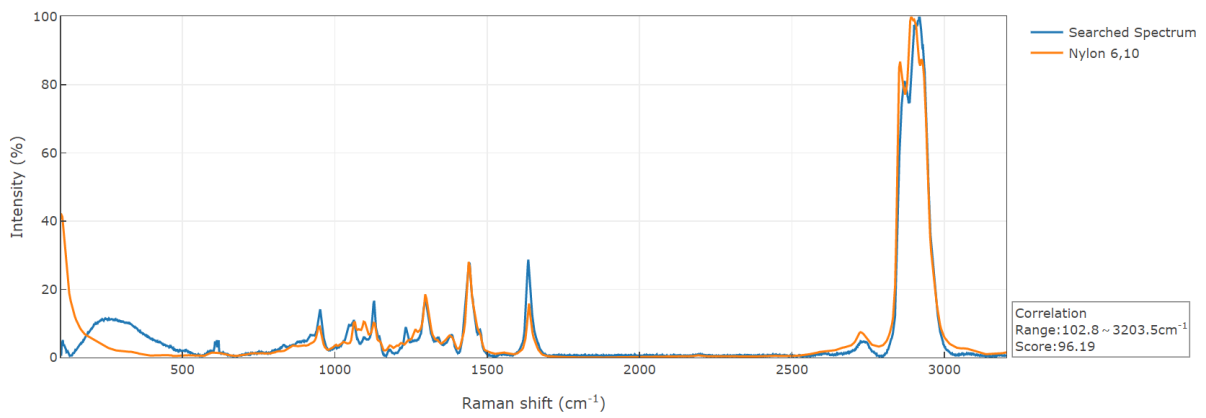


Figure 41 Obtained spectra for a white nylon strip (searched spectra), labeled (nylon) with 96.19 % match for Nylon 6.10. Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

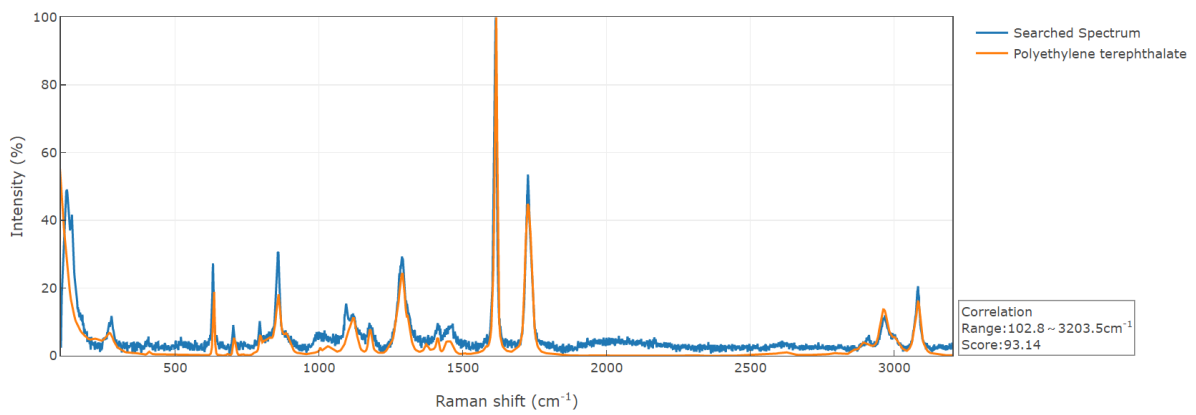


Figure 42 Obtained spectra for polyester rope (searched spectra), labeled (polyester) with 93.14 % match for Polyethylene terephthalate (polyester). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

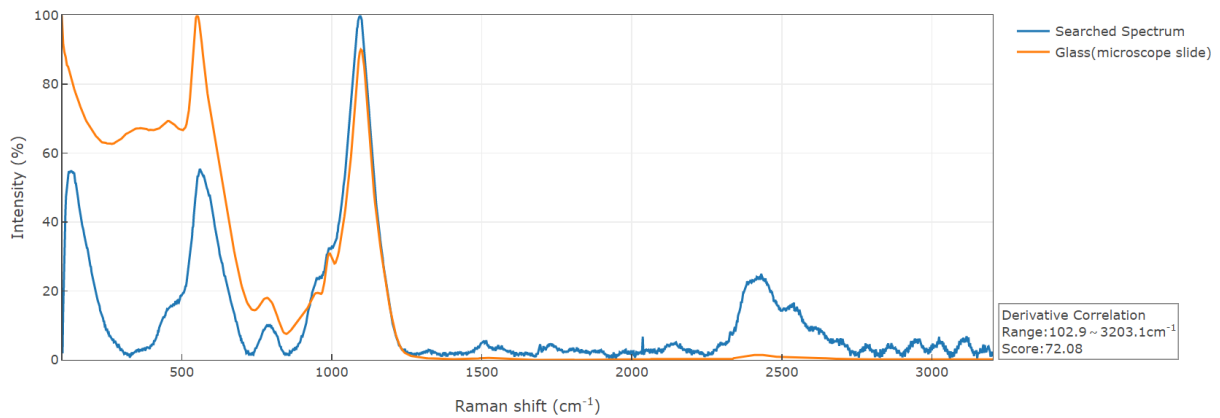


Figure 43 Obtained spectra for petri bowl (searched spectra), with 72.08 % match for glass (microscope slide). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

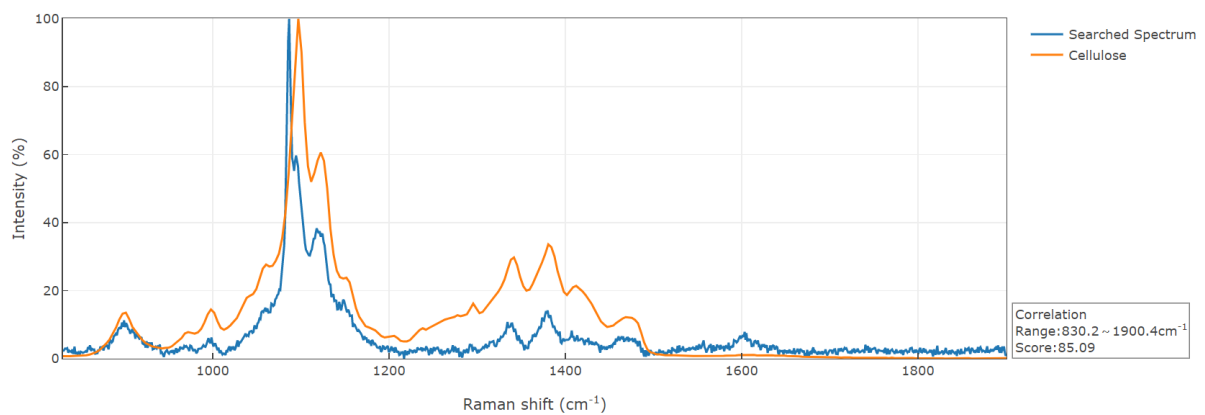


Figure 44 Obtained spectra for a paper block (searched spectra), with 85.09 % match with cellulose (natural paper). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

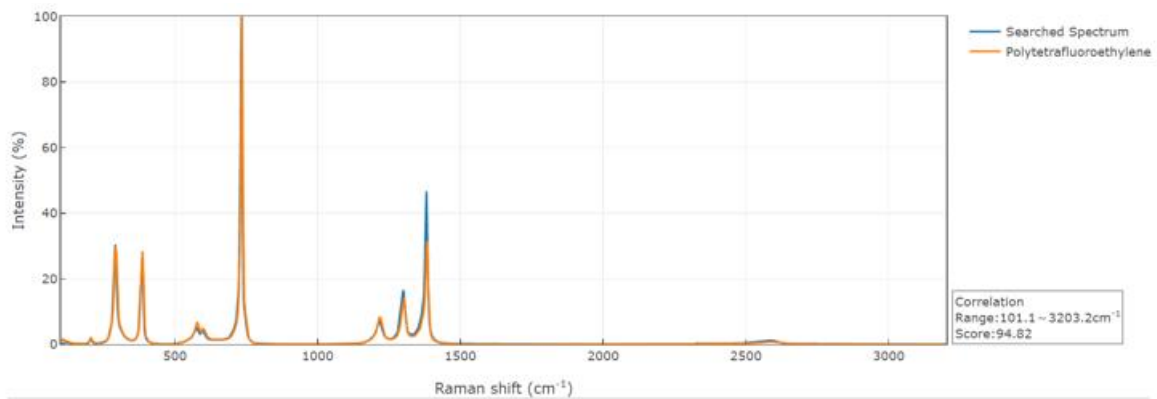


Figure 45 Obtained spectra for Teflon beaker (searched spectra), with 94.82 % match for polytetrafluoroethylene (Teflon). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

6.4.2 Obtained spectra for microplastics from collected samples

Fractions larger than 2000 μm and not smaller than 32 μm were also studied and separated from the collected samples. In total 20 microplastic particles were identified using the Raman spectroscopy. The obtained spectrum for each particle was compared

with the open Raman spectral database (PublicSpectra 2019). A match over 80 % were accepted as identified microplastic according to (PublicSpectra 2019).

The most abundant microplastic identified in this study was polyethylene with 14 spectra matching and polypropylene with 5 spectra matching. Nylon was also identified in one sample. In total 16 microplastic particles larger than 2000 μm were identified and 4 microplastic particles smaller than 2000 μm . The results are shown in Fig 46-55. The obtained spectrum labeled (a), comparison spectra (b), and both spectra added together (c). Obtained spectra are compared with spectra from the open Raman spectral database (PublicSpectra 2019). Other spectra that were identified are cellulose and silicon and are shown in Fig N a) and l) in appendix G.

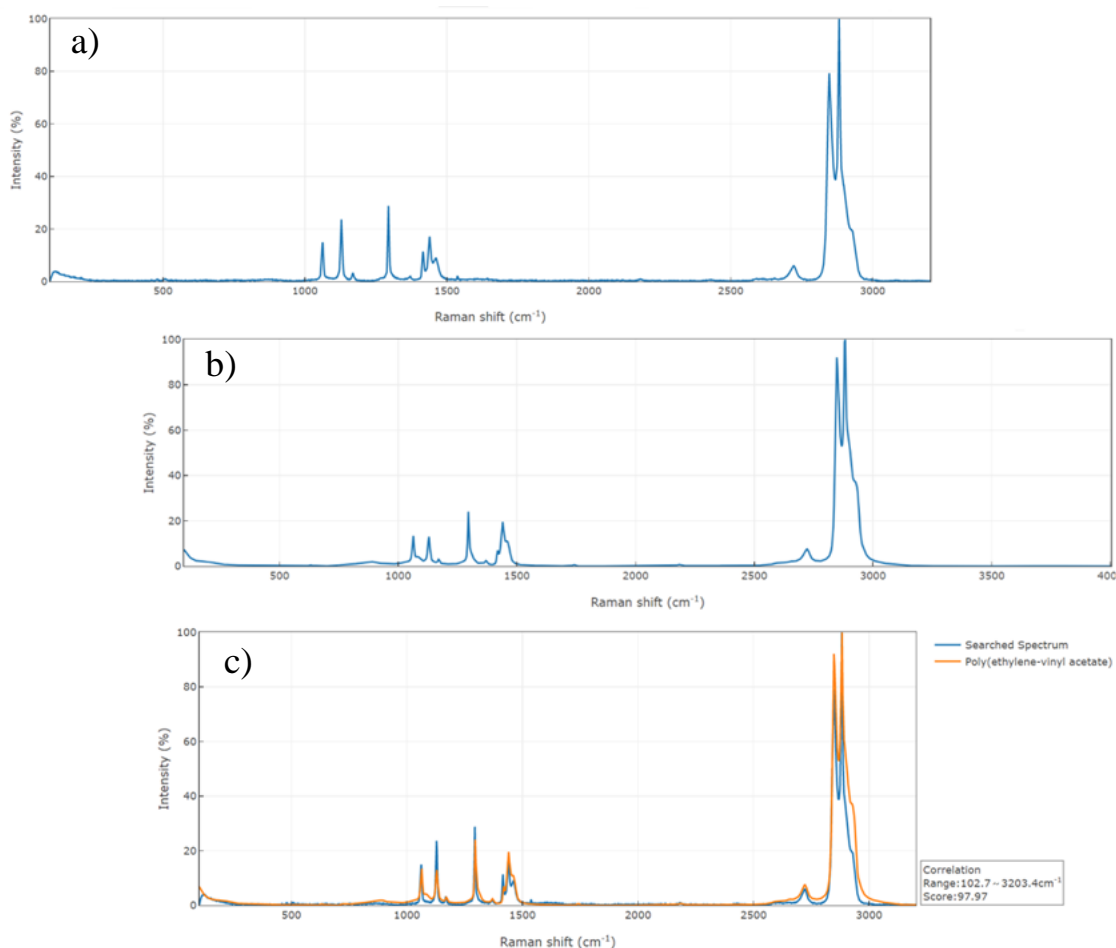


Figure 46 Three large samples (specs) from SO-1_1, all classified as poly (ethylene-vinyl acetate).

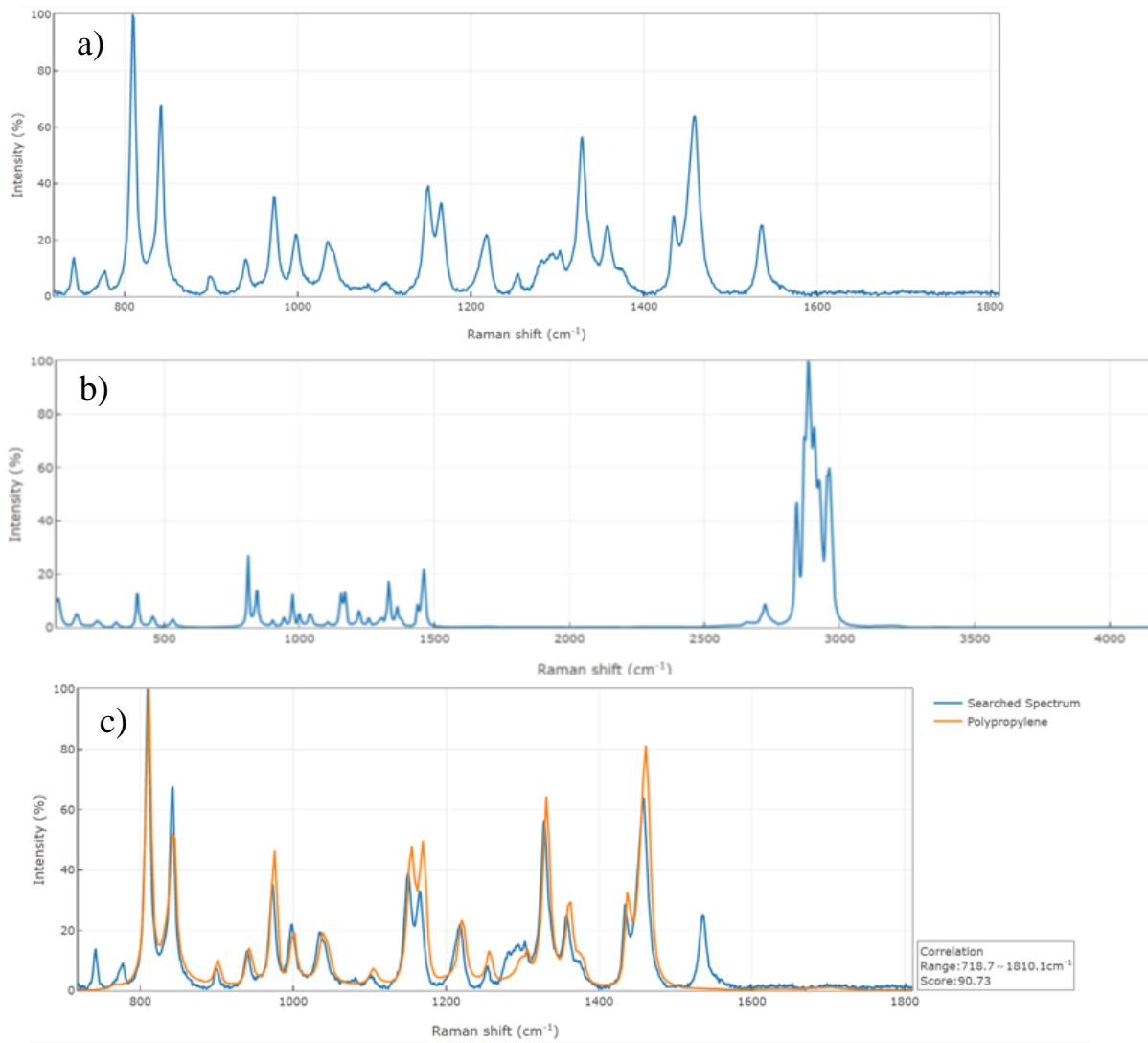


Figure 47 Three large samples (specs) from SO-1_Leave_1, all classified as polypropylene.

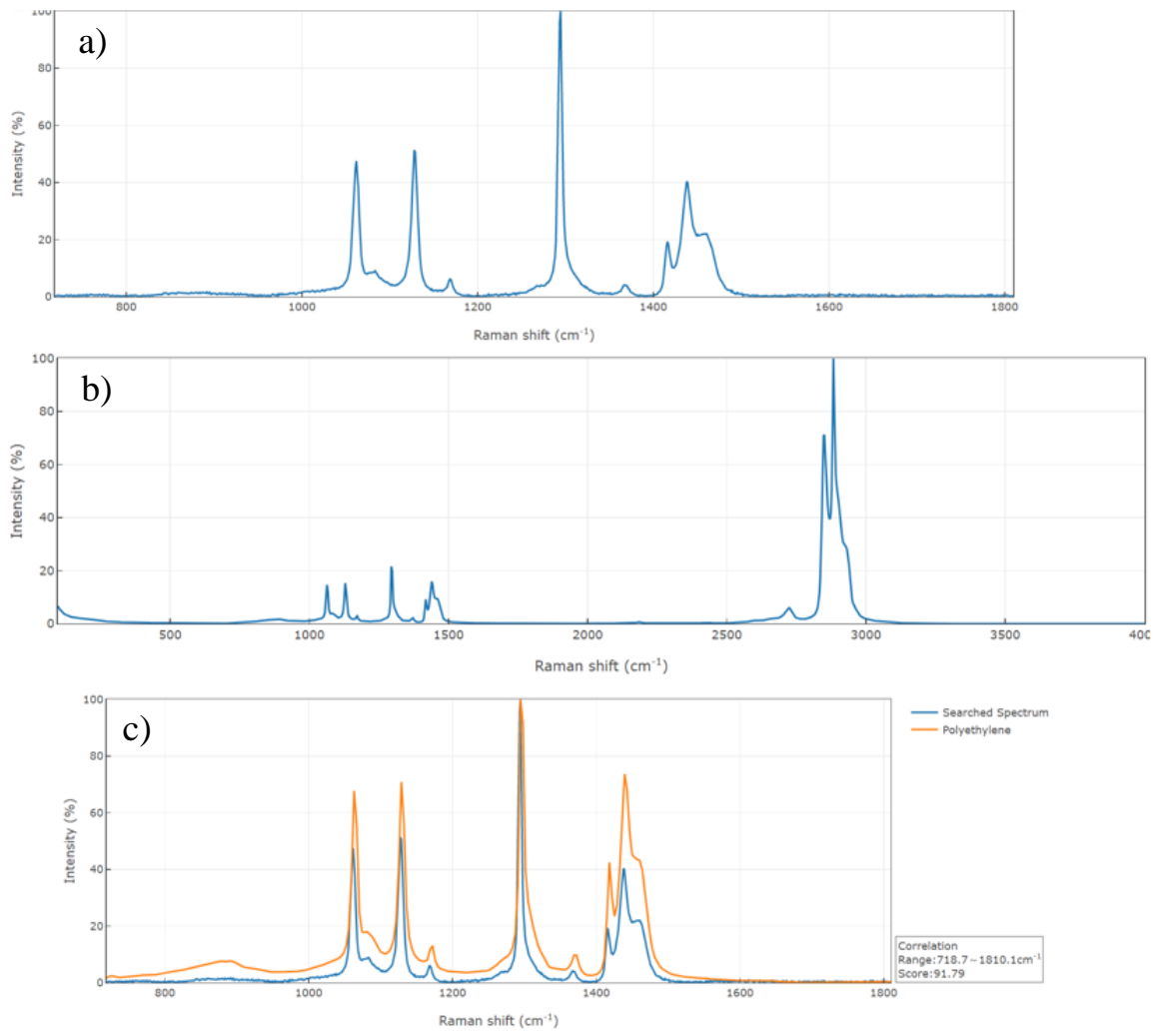


Figure 48 Three large samples (specs) from SO-1_Leave_2, all classified as polyethylene.

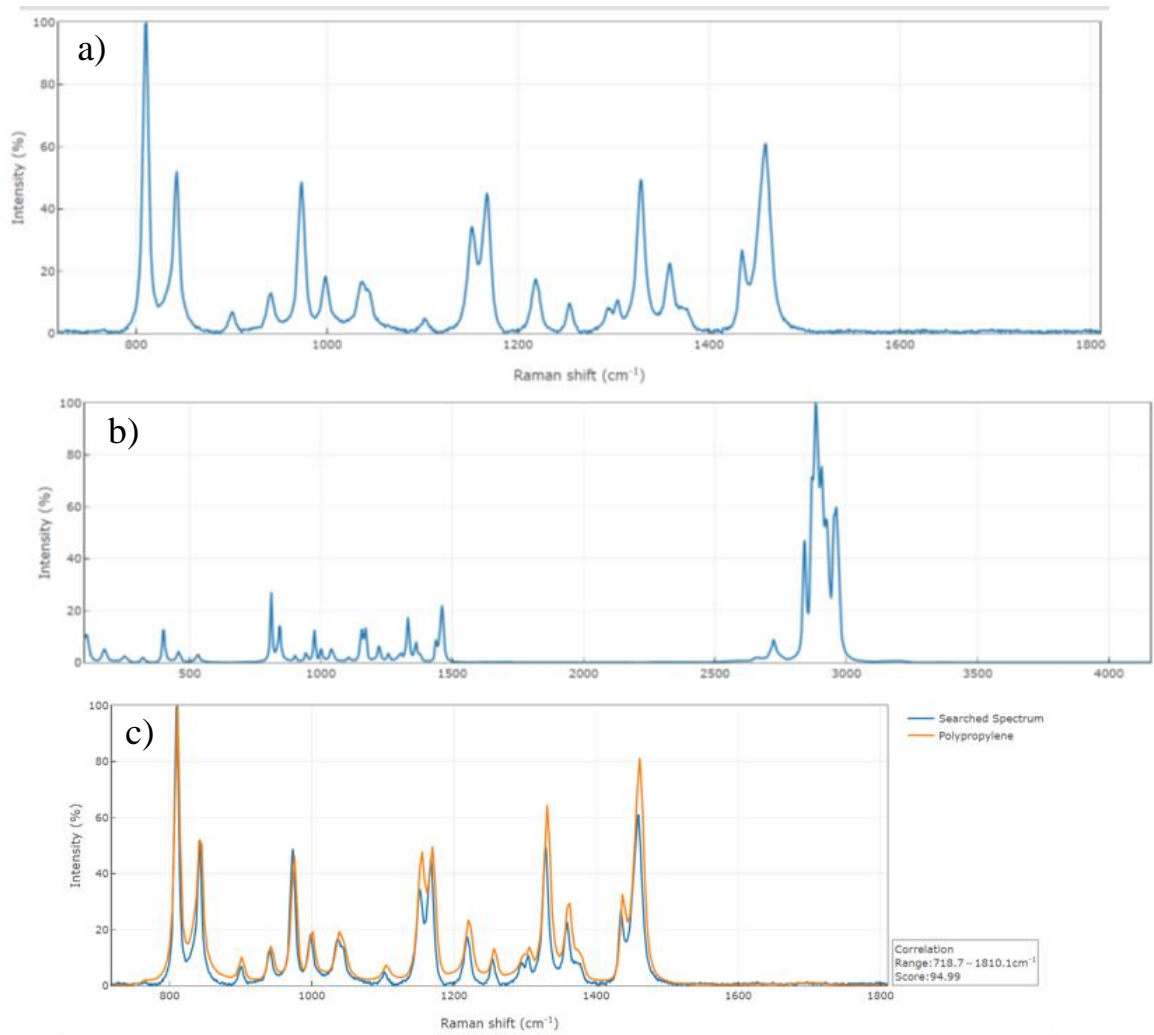


Figure 49 Three large samples (specs) from SO-2_2, all classified as polypropylene.

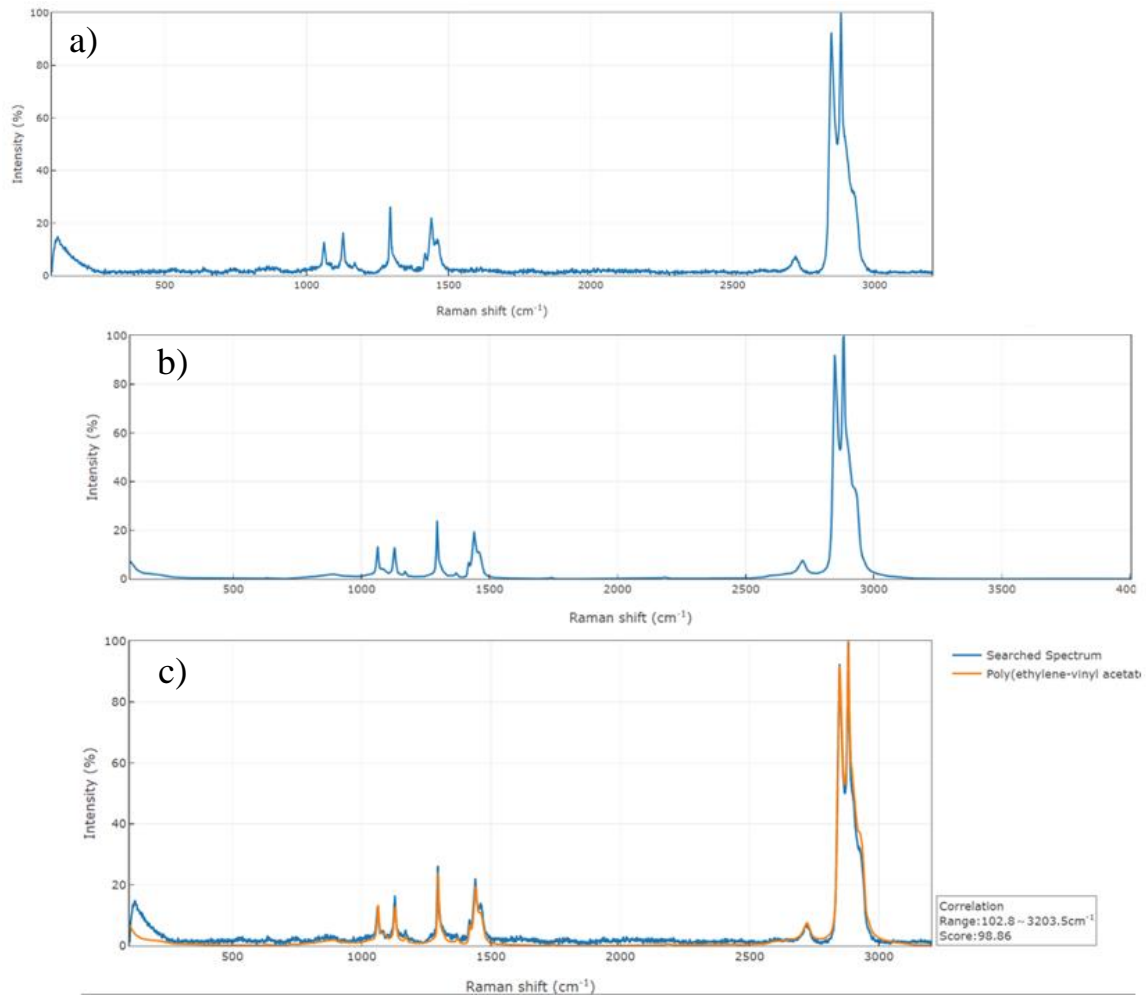


Figure 50 Three large samples (specs) from BO-1_3, all classified as poly (ethylene-vinyl acetate).

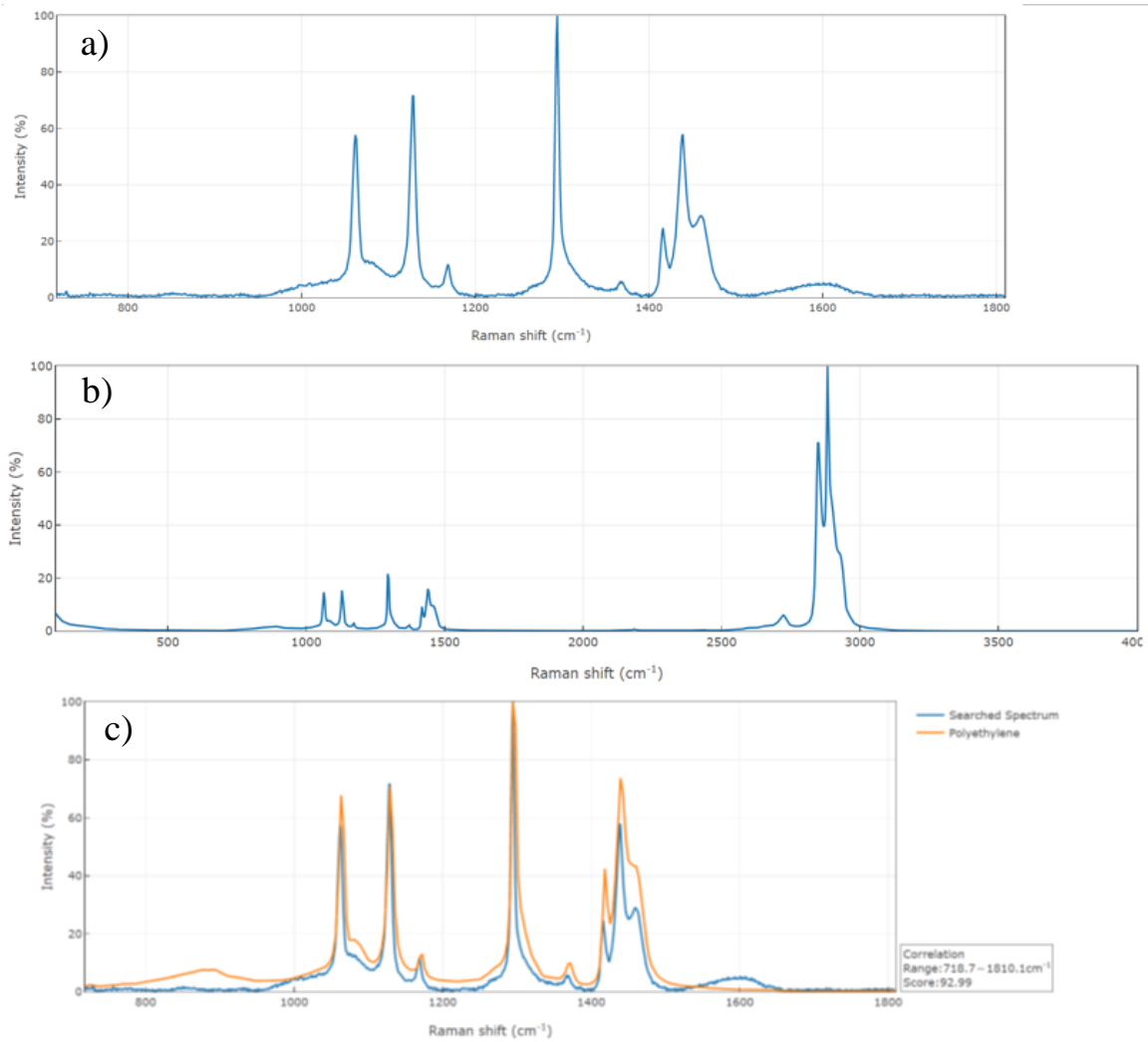


Figure 51 Three large samples (specs) from BO-1_Leave_3, all classified as polyethylene.

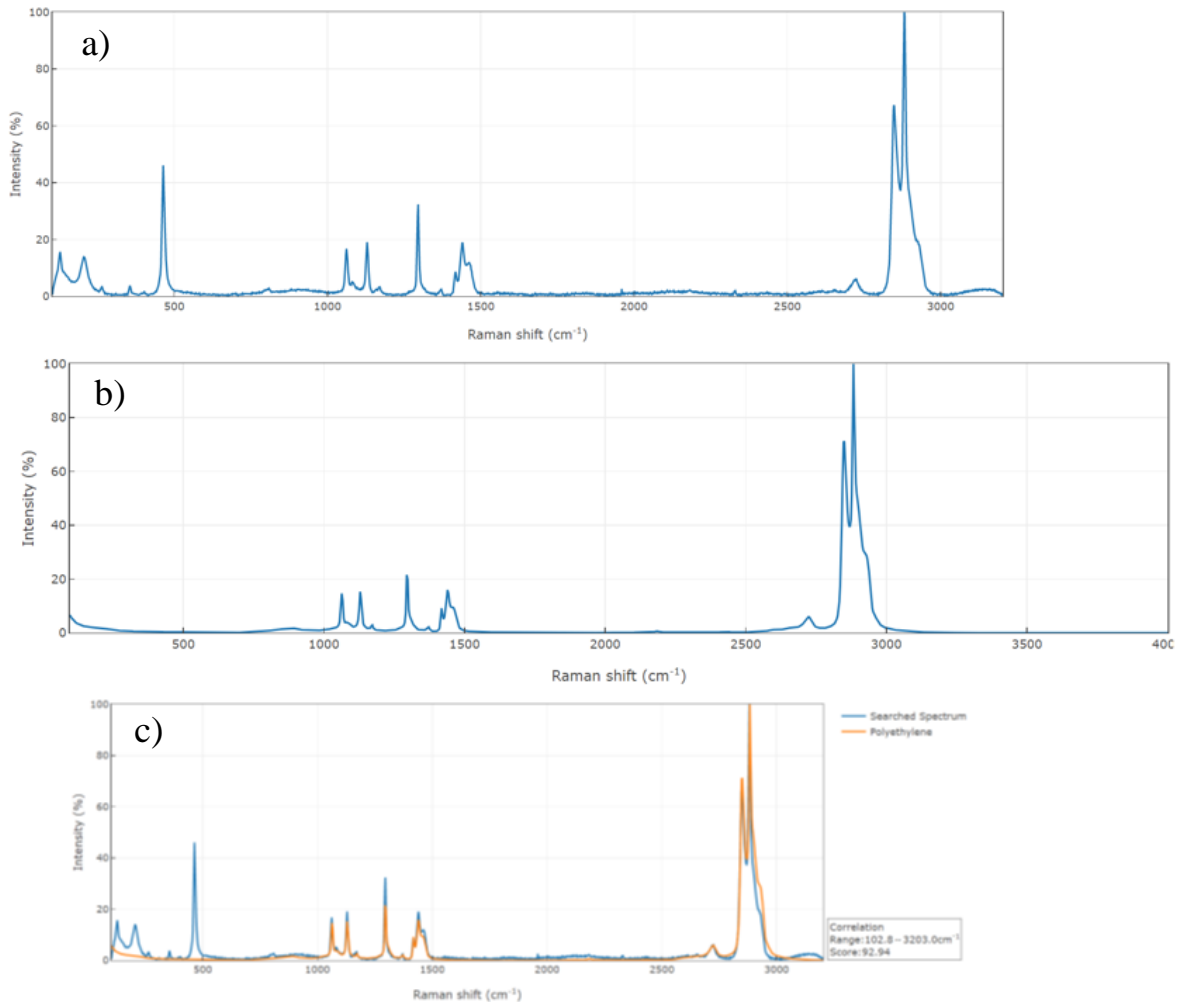


Figure 52 Three large samples (specs) from BO-1_250-125 μm, all classified as polyethylene.

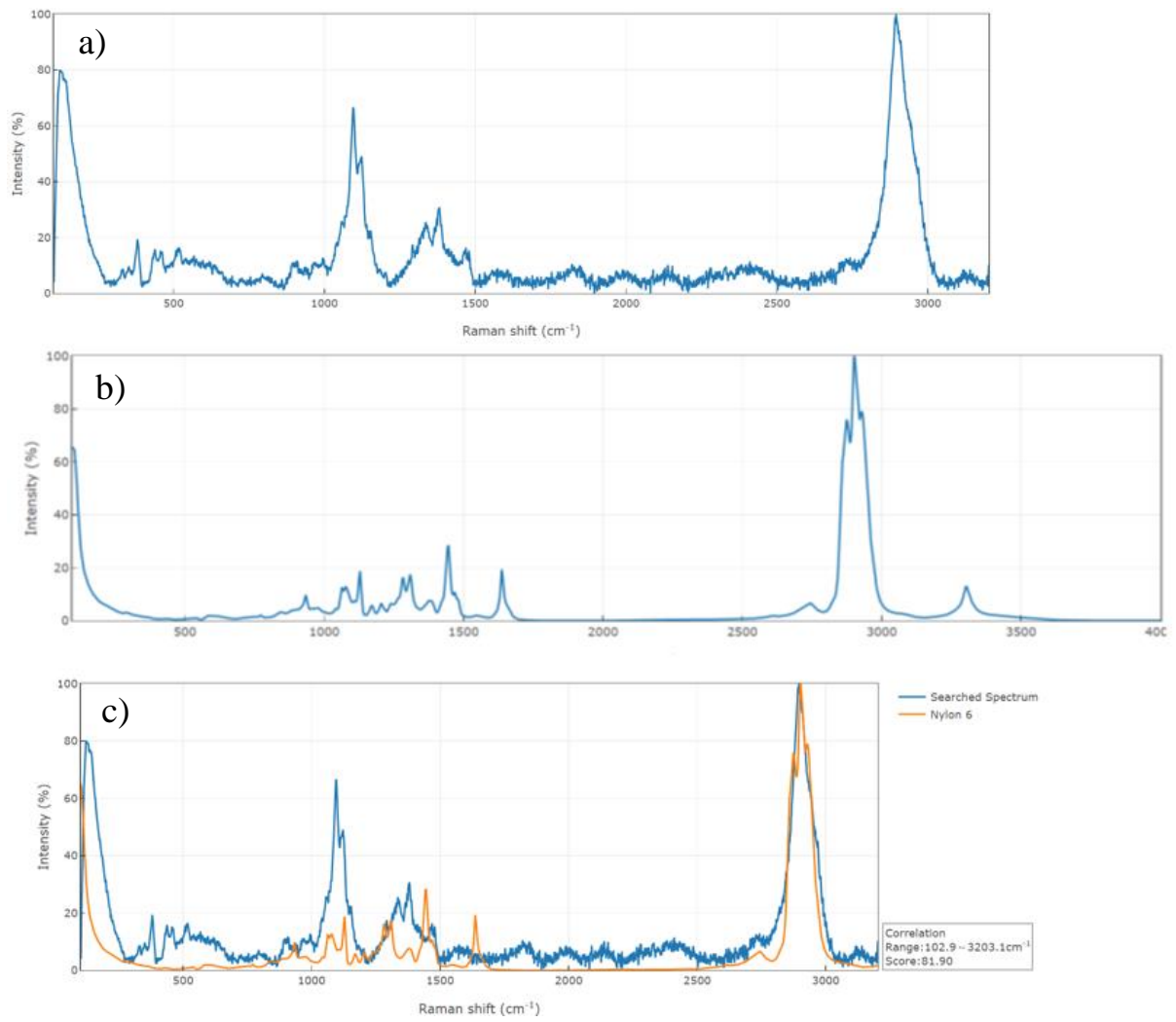


Figure 53 Three large samples (specs) from SS-1_500-250 μm , all classified as Nylon6.

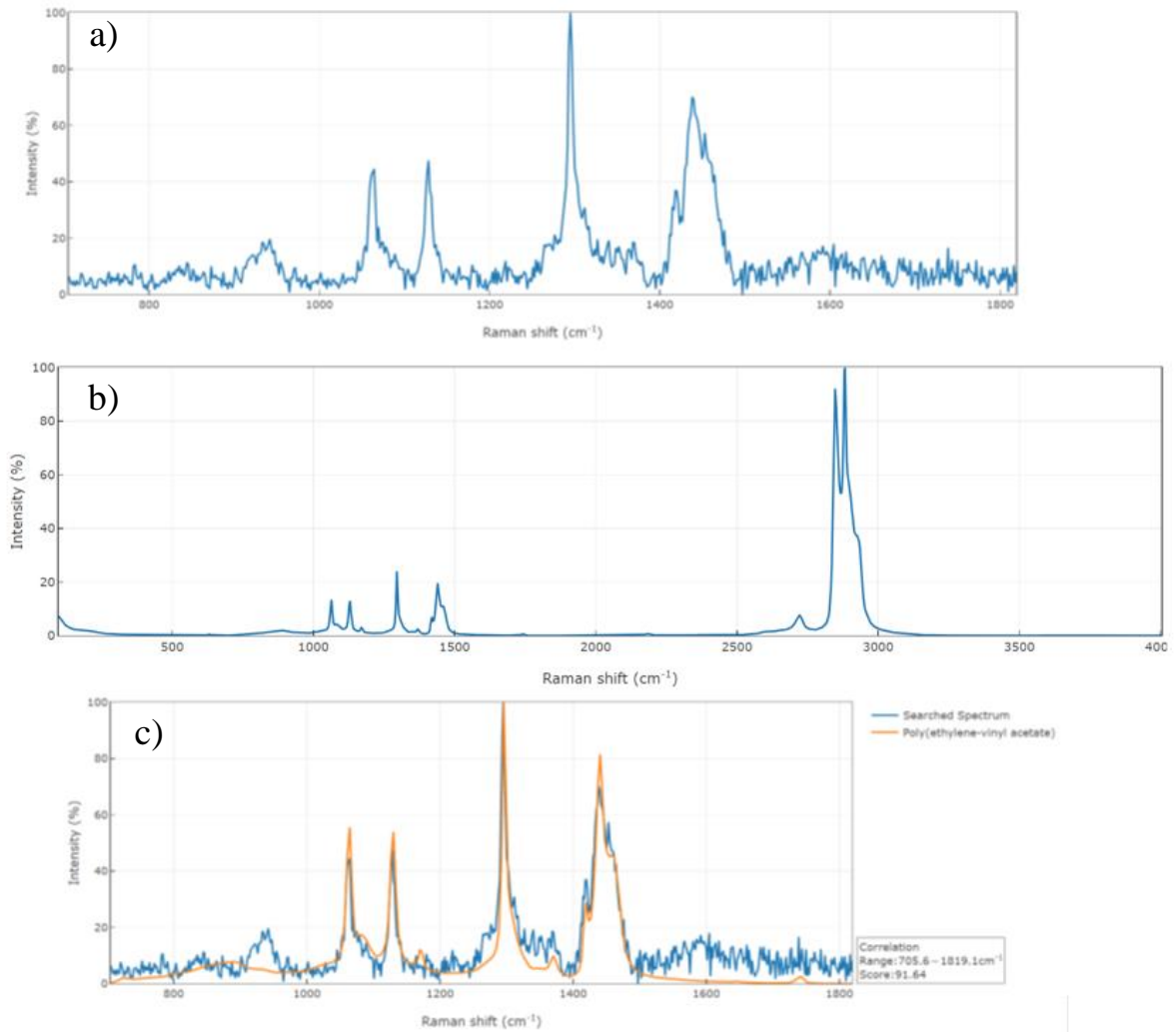


Figure 54 Three large samples (specs) from SS-2_500-250 μm, all classified as poly (ethylene-vinyl acetate).

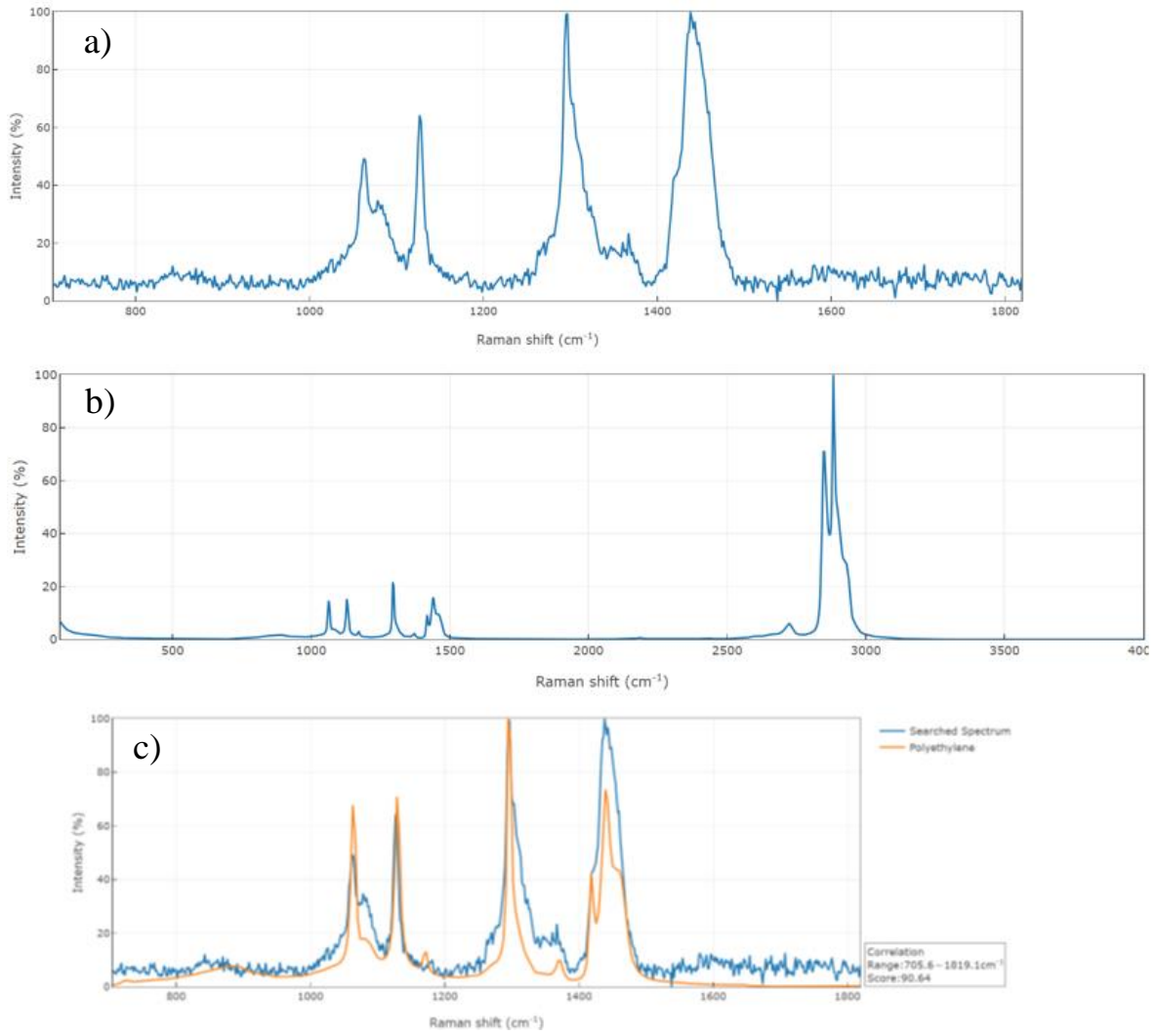


Figure 55 Three large samples (specs) from SS-2_2_500-250 μm , all classified as polyethylene.

6.4.3 Comparison of results from UiS with Milano

To compare the spectra and verify them, the results of the collected plastic samples were tested at the University of Milano-Bicocca, (Laboratory for Provenance Studies, Department of Earth and Environmental Sciences). Aim was to identify possible differences and validate spectra we measured at UiS. Fig 56-64 shows the obtained spectra at UiS (a) compared with the obtained spectra for the same sample at University of Milano-Bicocca (b). As seen the spectra are basically the same.

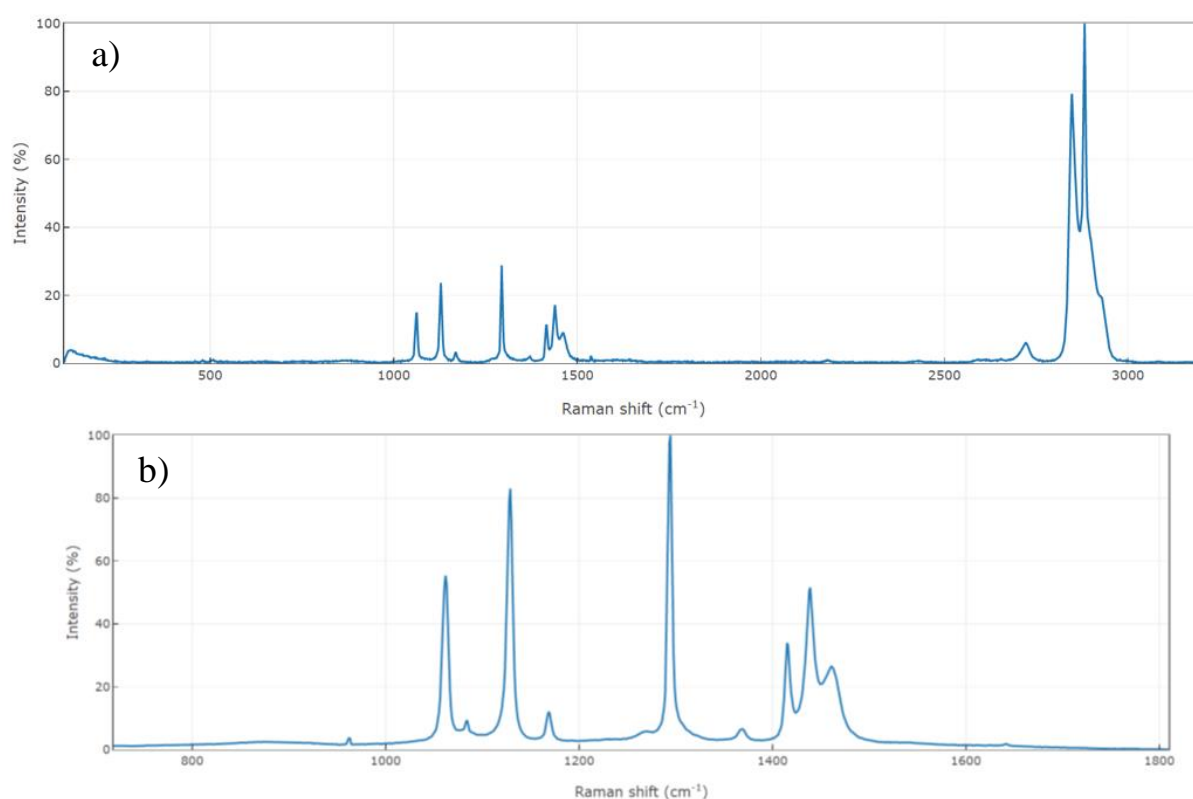


Figure 56 Two large samples (specs) from SO-1_1, both classified as poly (ethylene-vinyl acetate).

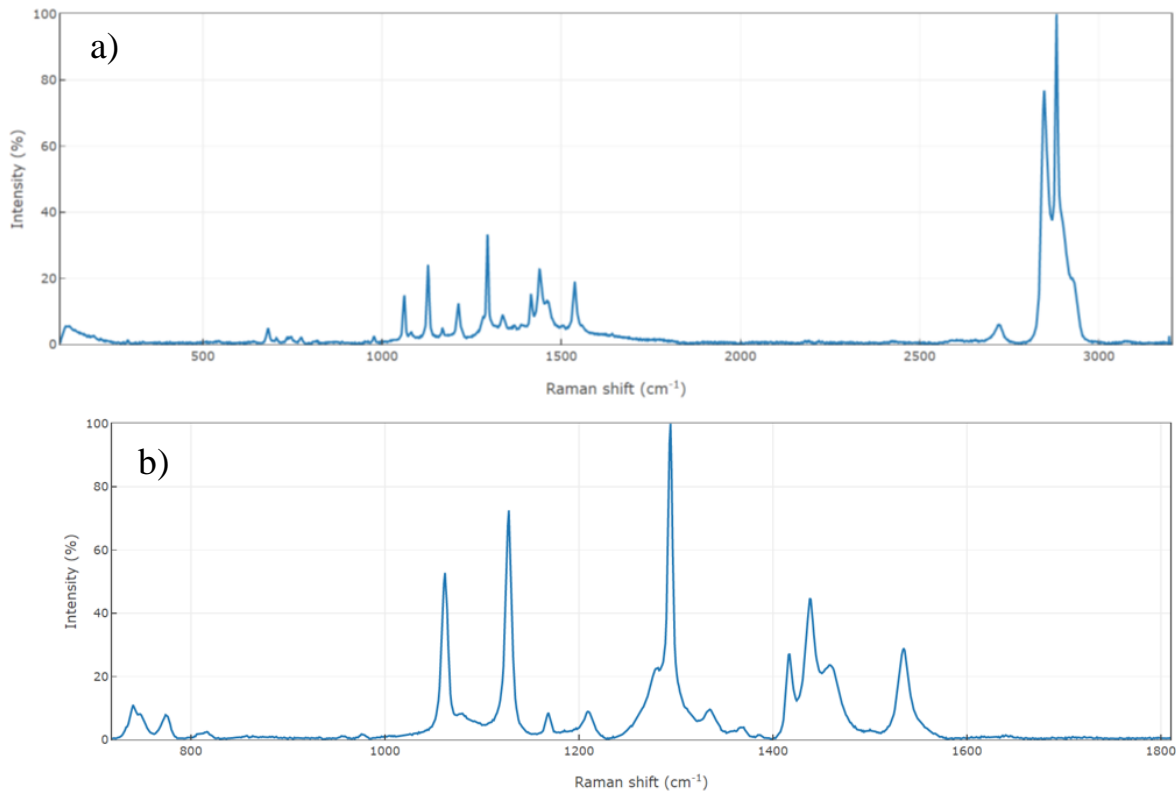


Figure 57 Two large samples (specs) from SO-1_2, both classified as poly (ethylene-vinyl acetate).

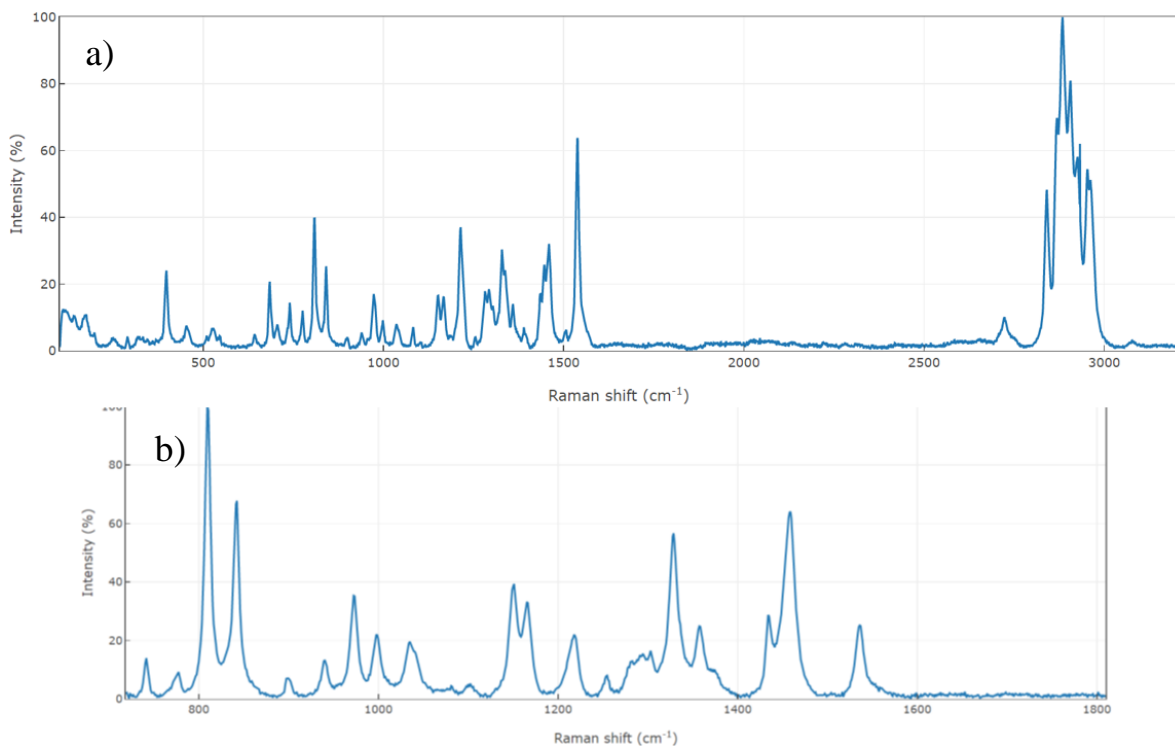


Figure 58 Two large samples (specs) from SO-1_Leave_1, both classified as polypropylene.

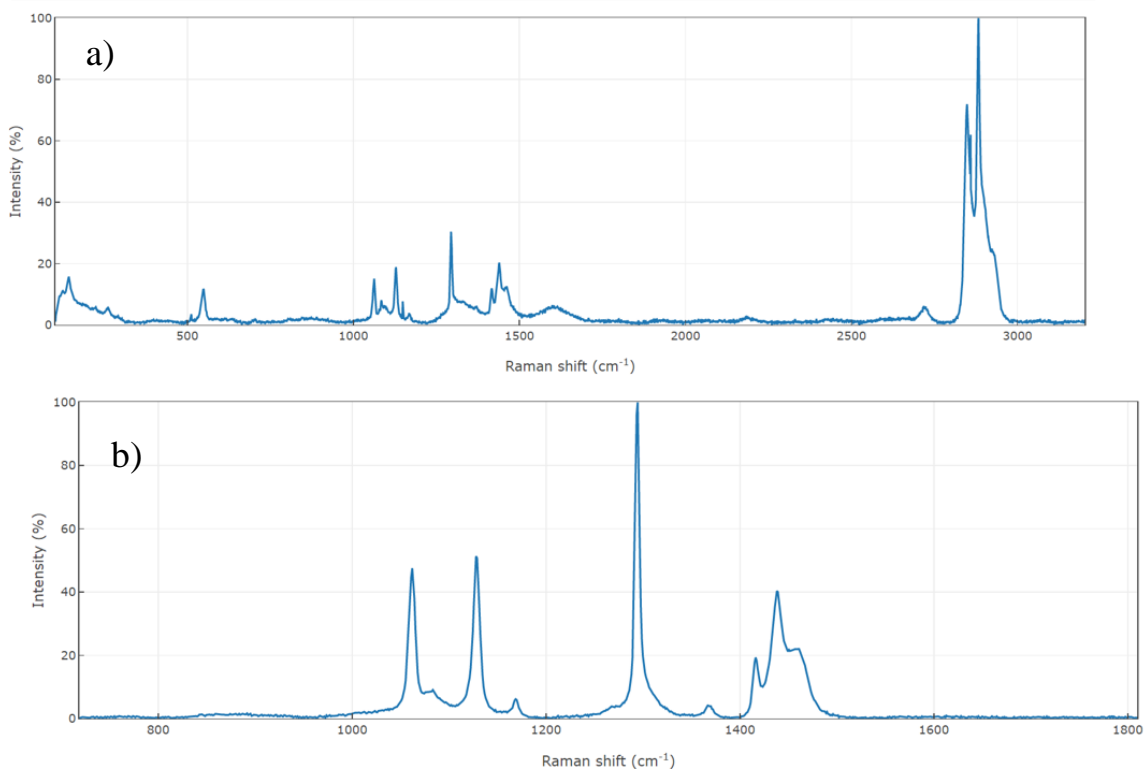


Figure 59 Two large samples (specs) from SO-1_Leave_2, both classified as polyethylene.

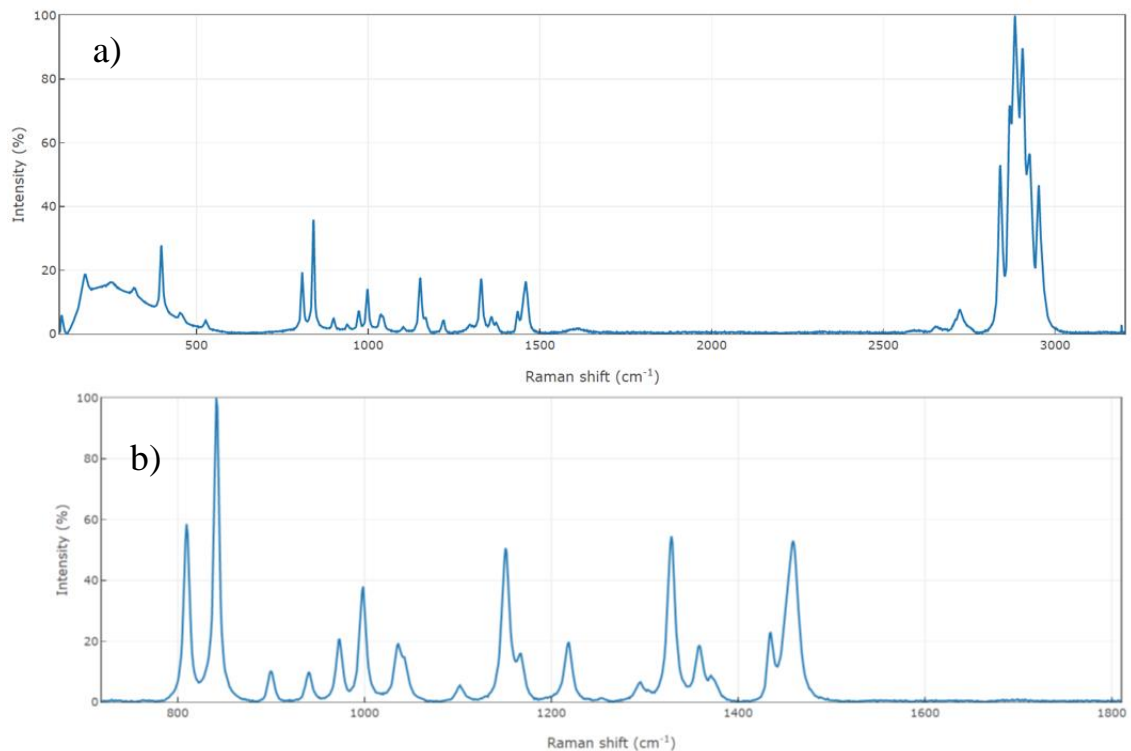


Figure 60 Two large samples (specs) from SO-1_Leave_3, both classified as polypropylene.

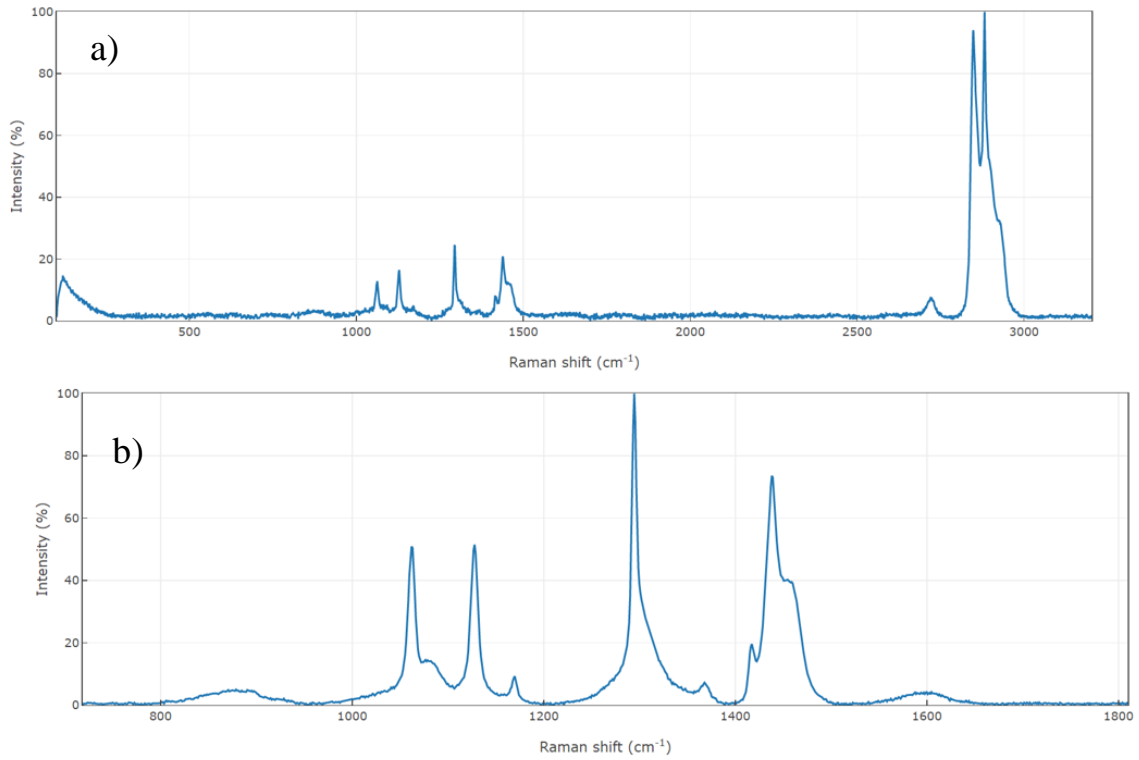


Figure 61 Two large samples (specs) from BO-1_1, both classified as poly (ethylene-vinyl acetate).

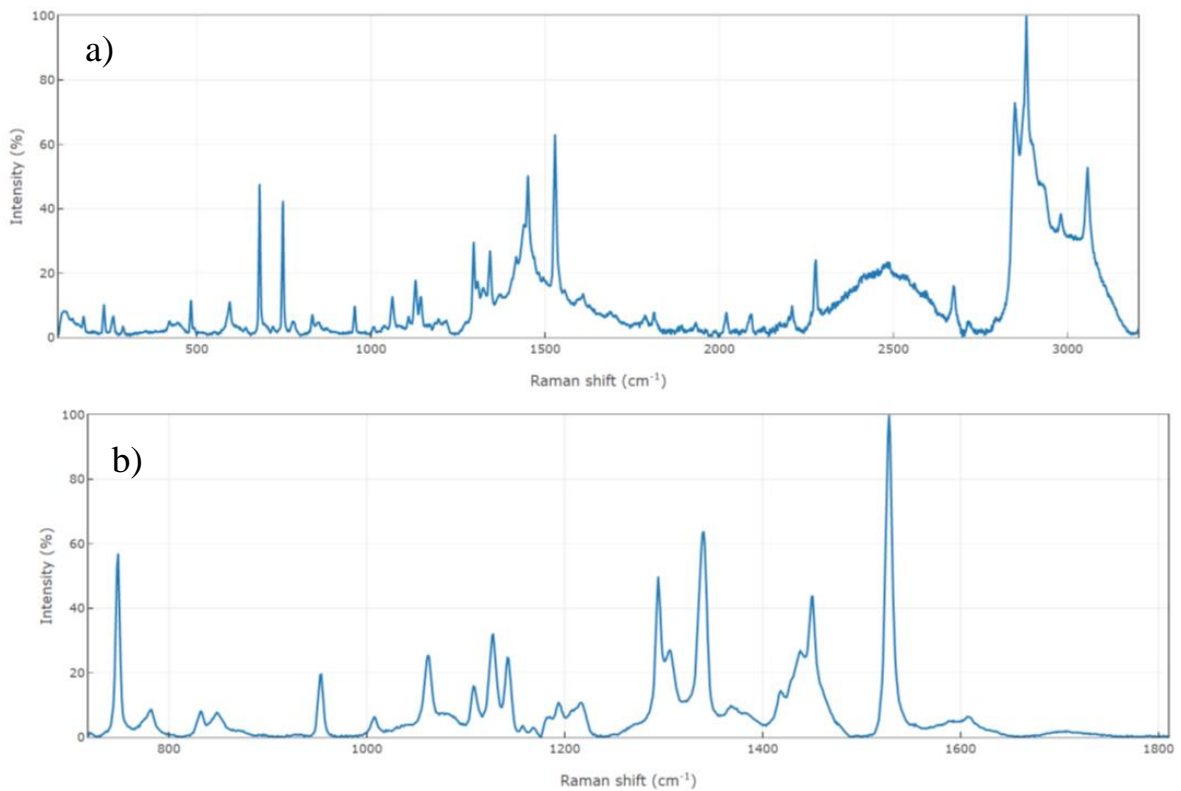


Figure 62 Two large samples (specs) from BO-1_2, both classified as polyethylene.

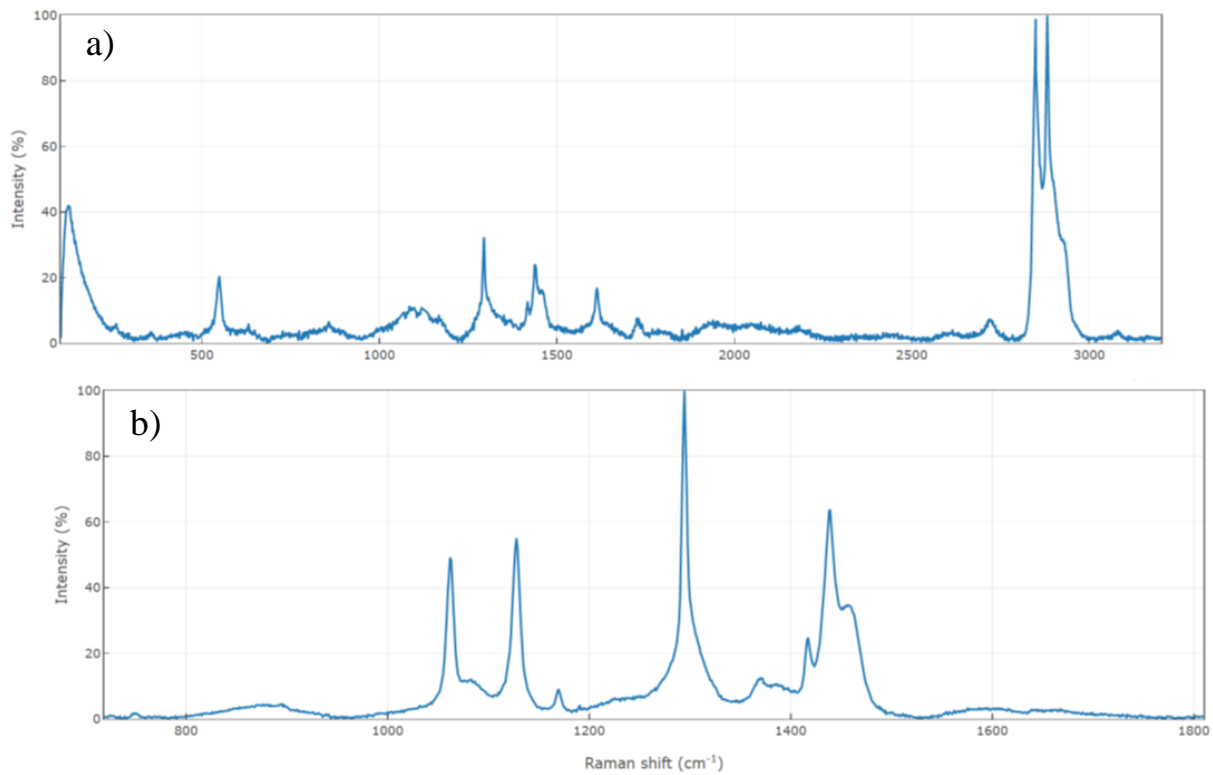


Figure 63 Two large samples (specs) from BO-1_3, both classified as poly (ethylene-vinyl acetate).

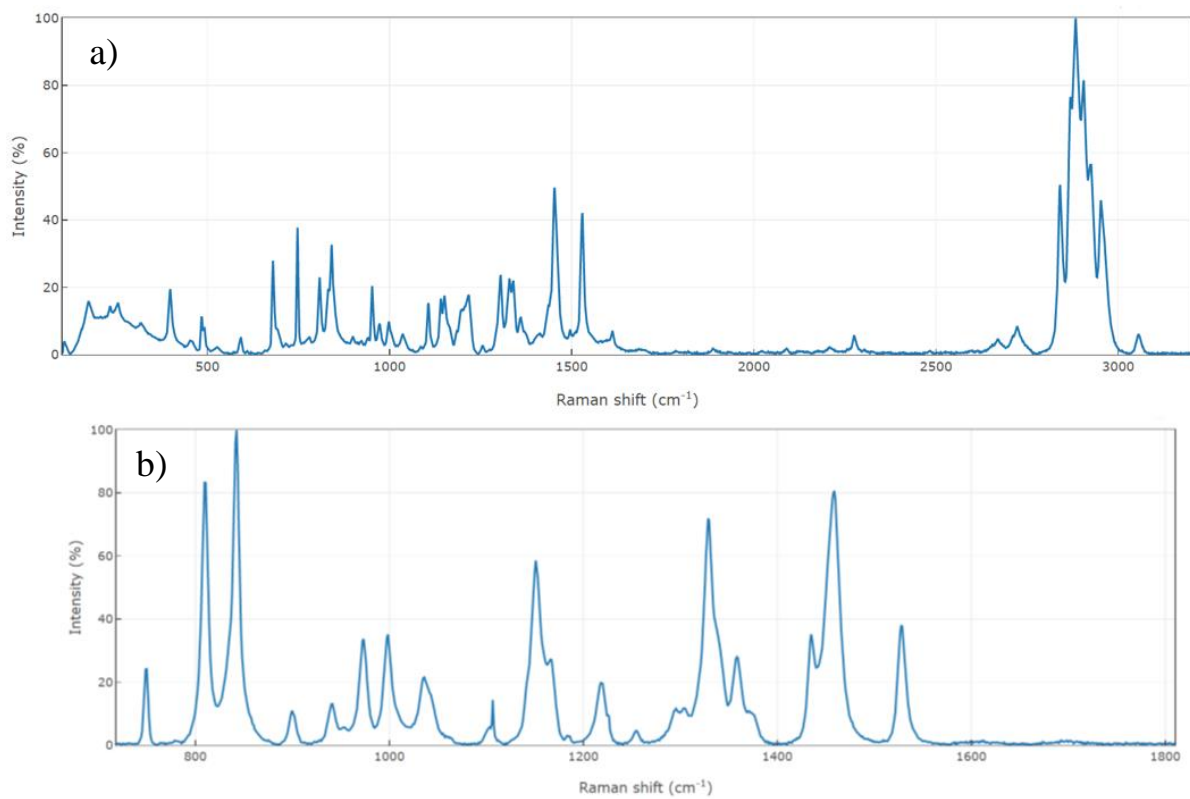


Figure 64 Two large samples (specs) from BO-1_Leave_2, both classified as polypropylene.

6.4.4 Identified microplastics

Identified microplastic types in sample SO, BO and SS are shown in Fig 65-72 were the obtained spectrum for the sample (searched spectra) compared with the open Raman spectral database (PublicSpectra 2019).

Results for SO sample:

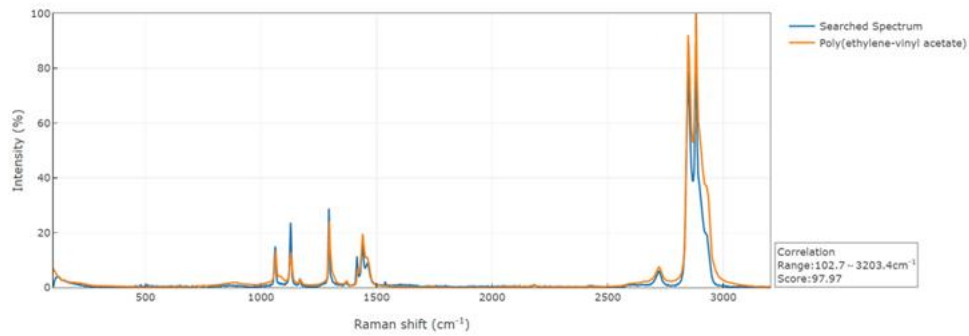


Figure 65 Obtained spectra for sample SO-1_1 (searched spectra), with 97.97 % match for Poly (ethylene-vinyl acetate). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

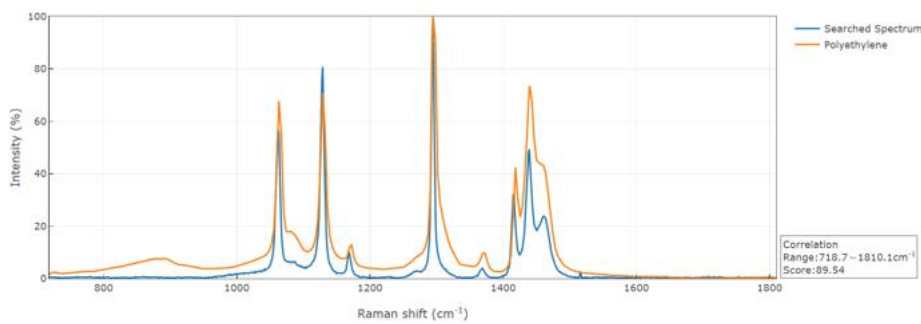


Figure 66 Obtained spectra for sample SO-2_1 (searched spectra), with 89.54 % match for polyethylene. Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

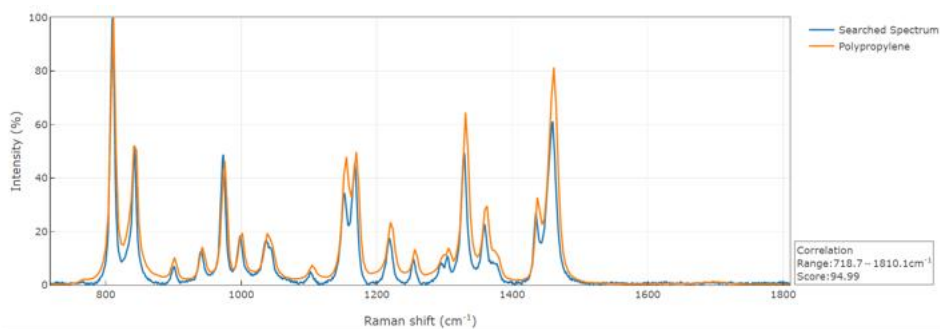


Figure 67 Obtained spectra for sample SO-2_2 (searched spectra), with 94.99 % match for Polypropylene. Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

Results for BO sample:

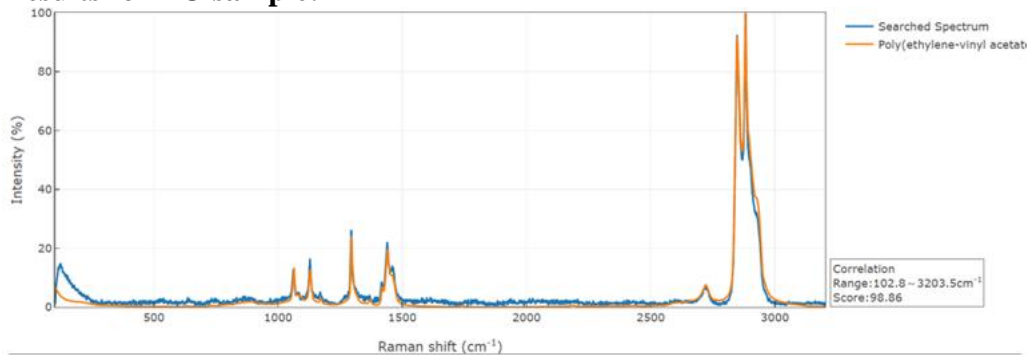


Figure 68 Obtained spectra for sample BO-1_3 (searched spectra), with 98.86 % match for Poly (ethylene-vinyl acetate). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

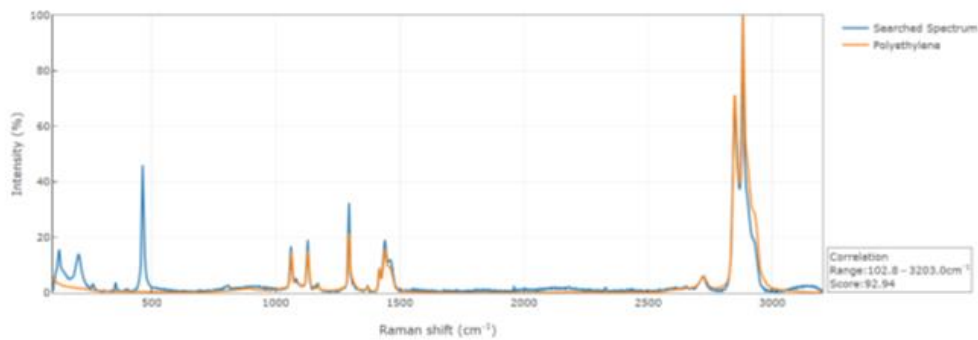


Figure 69 Obtained spectra for sample BO-1_250-125 (searched spectra), with 92.94 % match for Polyethylene. Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

Results for SS sample:

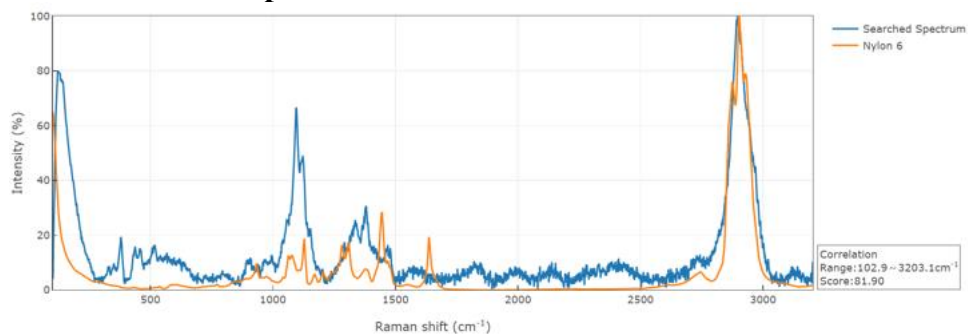


Figure 70 Obtained spectra for sample SS-1_500-250 (searched spectra); with 81.90 % match for Nylon6. Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

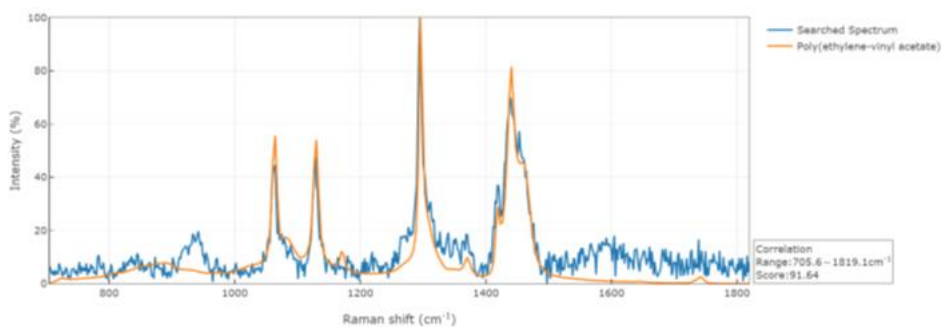


Figure 71 Obtained spectra for sample SS-2_500-250 μm (searched spectra), with 91.64 % match for Poly (ethylene-vinyl acetate). Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019)

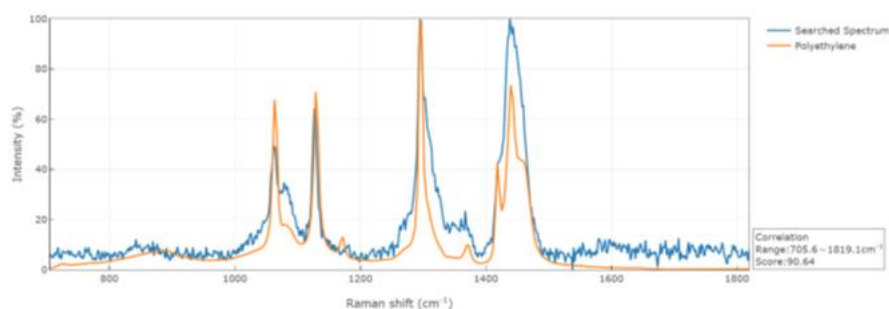


Figure 72 Obtained spectra for sample SS-2_500-250 μm (searched spectra), with 90.64 % match for Polyethylene. Searched spectra is the unknown, the plastic type is from (PublicSpectra 2019).

7 Discussion

In this study only particles larger than 32 μm were considered as it was already sufficient material, and the aim of the study was to develop a methodology for sampling and identification of microplastic. Moreover, the sample material had to be compared with an external laboratory to verify the results at UiS. A method to measure with Raman was developed. The basic setup used the extended scan with a laser source of 633 nm, 10 seconds exposure time with the laser fixed at 50 % and 5 accumulations. The setup worked well for the identification of microplastic, although spectra for fibres was not obtained. All lasers available at UiS were used 457, 532 and 633 nm with no results for fibres. Unclassified particles have been fabrics, collected at home and fibres with sizes below 1000 μm in the collected samples. Identified and not identified samples were also tested in Milano (see Methodology chapter 4.5). The set up at Milano used static scan with a laser source of 785 nm, 1 second exposure time was used with the laser fixed at 10 % and 30 accumulations. With the latter set up, the time used to search for spectra were considerably reduced and spectra were obtained also for the fibres. However, the fibres were not identified as plastic but cellulose (Fig N a), in appendix G. In total three white fibres were tested, and all had the required peaks for cellulose. One red and one blue fibre were also tested. Spectra were obtained but not identified. Reasons for this might be that the library was not large enough, as the entire plastic research is still building up, as mentioned above.

The study shows that white and transparent plastic gives a nearly identical spectra with its match as seen in Fig 41 and 42 where a white nylon strip has a 96.19 % match, and a white polyester rope has a 93.14 % match without additional peaks. Plastic that are coloured as seen in Fig 38 and 40 shows the matching peaks but also additional peaks. This can be explained by the colour that is added to the plastic, as the colour chemistry influences the spectrum and

manipulates the signal. However, this aspect is yet not intensively understood. The study also showed that it is easier to obtain Raman spectrum for higher density plastic. The lower density and the ones that appear to be composed of several types of plastic or maybe recycled, were more complicated to analyze. Most of the low-density samples tested at UiS, had a poor signal to noise ratio, meaning that there has been significant fluorescence. Yet, this effect was not possible to suppress.

After the experiences and knowledge gain from Milano, a suggestion would be to change the set up used at UiS to use static scan and centering the spectrum range on 1300 Raman shift cm^{-1} together with a laser power of 633 nm, an exposure time of 1 sec with the laser fixed at 10 % and 30 accumulations. Fluorescence commonly occurs in complex organic molecules as found in plastics and as a laser power of 532 nm has the tendency to cause sample fluorescence which again can swamp the faint Raman signal. A laser power of 785 nm will be preferred for plastic studies as it can eliminate sample fluorescence from most organic molecules (Tedesco and Slater 2000). Alternatively, the suggested setup could be adjusted with changing the laser percentage to 50 % and accumulations to 50 for better results and less fluorescence, because higher accumulations will reduce fluorescence. However, there will be the risk of burning the sample with the laser percentage set to 50 % and need to be monitored for every sample starting at 10 %.

Within the samples here tested, the surface sample had a considerably higher contamination of microplastic than the bottom sample at SO and BO. Plastic on surface can have been transported by wind or by people's waste but it is unsure how the plastic has been deposited at the beaches. This is, however out of the scope for this study here. The surface sample at Bore also had a lot of microplastic nurdles Fig 73 a), which is small plastic pellets that are used to make nearly all the plastic products we use. "These pieces span a continuum of colours, shapes, sizes, and densities with high variability that could impact clean-up efforts, alter transport in the ocean, and potentially affect wildlife" (Vos, et al. 2021). These nurdles are common along beaches and billions are used each year. Fig 73 b) shows nurdles collected from Mt. Lavinia.

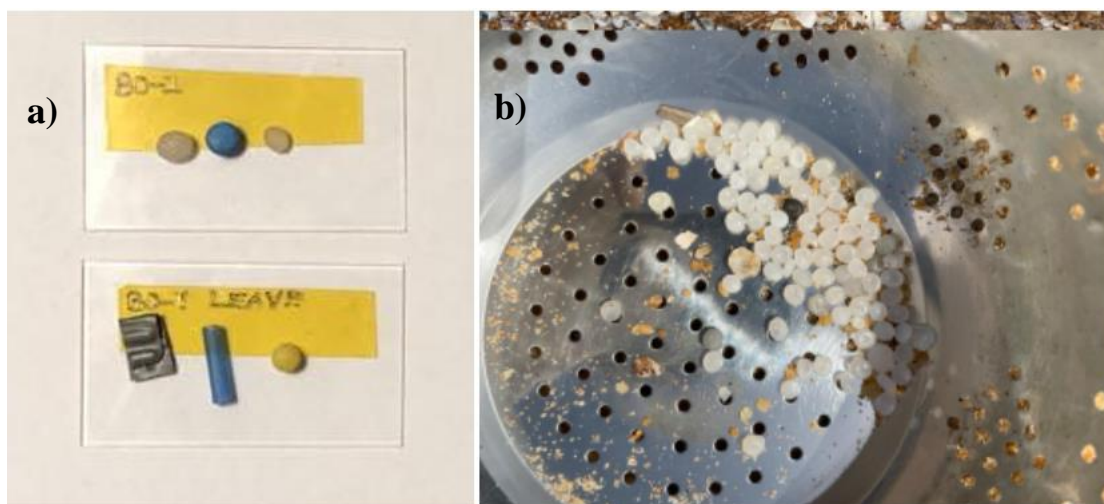


Figure 73 Photos of nurdles a) Nurdles collected at Bore beach; b) Photo of nurdle spill from Mt. Lavinia (Vos, et al. 2021).

The surface sample from Sola (SO-1) had a lot more plastic than the surface sample BO-1 and SS-1. In total 10 microplastic particles larger than 2000 μm were identified in SO-1, with 6 identified in BO-1. In BO-1 also 1 microplastic particle smaller than 1000 μm were identified. The sample from the school (SS) did not have any microplastic particles found at the surface and none microplastic particles larger than 1000 μm were identified. However, the SS-2 sample had a higher abundance of microplastic particles smaller than 1000 μm . In total 3 microplastic particles were identified in the SS sample, as seen in Fig 70-72.

SO, BO are quite well sorted but SO is finer than BO. This means with ideal packing drainage should be much lower in SO than in BO. The identified microplastic particle smaller than 1000 μm in the BO sample can be explained by this as the drainage is higher in BO than SO, hence in BO small plastic particles should be able to move easier. The study shows that for material larger than 32 μm there were no significant drainage of microplastic from the surface to the deeper parts for SO and BO. The plastic identified were mostly larger than 1000 μm which is much larger than the general grain size (230-245 μm) for SO and (315-250 μm) for BO. Meaning that the plastic would not be able to move through the pore spaces between the sediment particles. Maybe smaller material is able to be moved and has to be studied in a follow up study. Here, the objective was to determine larger plastic components; hence, only particles larger than 32 μm were studied. This means that in the future it would be necessary to study particles smaller than 10 μm or even smaller. The general size of plastic fibres is 5-10 μm and therefore possibly small enough to move through the pore spaces between the

sediments here studied. This would be of importance as the smaller the plastic component the higher the possibility to be incorporated into organic tissue.

Surprisingly very few plastic particles have been found in the surface samples here, but even 10 particles within an amount of 190.9 g of sediment would amount to several kg in large amounts of sediments. Sola beach extends for 2.3 km by maybe 500 m which is about 1.15 km², immediately then the magnitude of contamination only in the category of microplastic is imaginable. In this methodological study, mainly polyethylene and polypropylene type of plastic has been found, a very common type of plastic used in beverage bottles, food container, fleece, bottle caps, rope, outdoor furniture etc. Follow up studies should try to quantify the types of plastic and investigate the provenance of this material to reduce possible usage of those plastic container or objects. This should be combined with stability knowledge of these specific most abundant plastic components added by some research of decay of these types. The study had demonstrated that such research is possible at UiS and will be hopefully carried out in the future to enhance the living quality in Rogaland.

8 Conclusions

In this study three different public exposure in Rogaland were compared in regard of the contamination by plastic in sediments. This was part of the main objective for the thesis project, to develop a sound methodology using in-house RAMAN for the identification of microplastic. Therefore, samples have been analysed at UiS and at a world renowned laboratory for Raman spectroscopy at the University of Milano-Bicocca. Samples have been taken on the surface of the sediment and at approximately 30 cm depth to gain access to the abundance and variation of microplastic in comparison. Two outcrops have been of natural origin, beach sediments, while one outcrop has been represented by artificial sand bought at a specific hardware shop in Rogaland (Siddis Hus og Hageservice), but the sand from a school sandbox has been exposed to urban contamination likewise. Results show that the surface sample had a considerably higher contamination of microplastic than the bottom sample at both Sola beach (SO) and Bore beach (BO). Microplastic is here defined as plastic particles smaller than 5 mm to 1 µm in size along its longest dimension. Polyethylene and poly (ethylene-vinyl acetate) were identified in all sample locations. Polypropylene was found at Sola beach (SO) and Bore beach (BO) and nylon at the school (SS). The surface sample from Sola beach (SO) had higher abundance of plastic than Bore beach (BO) and the school (SS), although these are estimations as a definite quantification was not the scope of the research. The sample from the

school (SS) did not have any microplastic particles found at the surface when performing selective sampling, which refers to the sampling of visible plastic components. Maybe because staff at the school permanently clean the material. The study show that they certainly don't contaminate the reservoir as humans in other circumstances. This is obviously a field of future research.

Based on the research strategy it can be concluded that a well working method for sampling, separation, and identification of microplastic in sediments was developed for the analytical facility at UiS. The preferred sampling technique is to use a combination of bulk and selective sampling. For the separation process a fluid with the density of 1.4 g/cm^3 is sufficient as the most abundant plastic has a density ranging between 0.808 and 1.238 g/cm^3 fine to medium grained sediment, as her it is the case. For finer sediment possibly more plastic can be envisaged, this needs to be tackled in the future. Furthermore, the composition of the fraction below $32 \text{ }\mu\text{m}$. Raman spectroscopy for identification of microplastic is a non-destructive technic used that also has minimal necessary preparation of the samples to be tested and is the preferred method for the identification. For this study an inVia™ confocal Raman microscope was used. A laser power of 785 nm would be preferred for plastic studies to avoid unnecessary fluorescence and to get the best balance between scattering efficiency and influence of fluorescence. Further the laser fixed at 10% with 30 accumulations and 1 second exposure time. However, a method to measure with Raman that worked well without the use of a laser source of 785 nm was developed. A laser source of 633 nm also works well with the exposure time set to $1-2$ second, the laser fixed at $10-50 \%$ and $30-50$ accumulations, depending on the plastic to be analysed and the degree of fluorescence. The higher the power, measured in percentage, of the laser, the better would the spectral result be but there is a very high risk in burning of the sample, wherefore for the sake of repeatability a lower percentage of the laser power is recommended. A static scan and centring the spectra range on 1300 Raman shift cm^{-1} works ideal to decrease the time used for analyses but generating push able results using the mentioned parameters. Therefore, future research at UiS in regards of plastic can easily adopt the here presented techniques for sampling and preparation as well as the analytical set-up. Future studies can take care of then quantification of plastic particles and focus on smaller fraction, wherefore further sampling, sample preparation and possible analytical techniques should be developed to tailor it to the laboratories at UiS.

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Appendix

Appendix A

Sieving analyses for each sample:

Table 1 Sample name, sample weight before sieving, total time sieved, vibration intensity, intervals, grain size, mass per sieve (g), mass per sieve (%) and cumulative mass (%).

SO-1			
Masse før sikting (g)	Tid (min)	Intensitet	Intervall (s)
190,9	25	6	3
Korn størrelse (μm)	masse per sikt (g)	masse per sikt (%)	Kumulativ masse (%)
>2000	0,4	0,21 %	0,21 %
2000-1000	0,1	0,05 %	0,26 %
1000-500	0,2	0,10 %	0,37 %
500-250	10	5,24 %	5,61 %
250-125	177,6	93,03 %	98,64 %
125-63	2	1,05 %	99,69 %
63-32	0,1	0,05 %	99,74 %
<32	0,1	0,05 %	99,79 %
Sum	190,5	99,79 %	
Avvik	0,4		

Table 2 Sample name, sample weight before sieving, total time sieved, vibration intensity, intervals, grain size, mass per sieve (g), mass per sieve (%) and cumulative mass (%).

SO-2			
Masse før sikting (g)	Tid (min)	Intensitet	Intervall (s)
148,9	25	6	3
Korn størrelse (μm)	masse per sikt (g)	masse per sikt (%)	Kumulativ masse (%)
>2000	0,1	0,07 %	0,07 %
2000-1000	0,1	0,07 %	0,13 %
1000-500	0,1	0,07 %	0,20 %
500-250	8,9	5,98 %	6,18 %
250-125	136,8	91,87 %	98,05 %
125-63	2,4	1,61 %	99,66 %
63-32	0,1	0,07 %	99,73 %
<32	0	0,00 %	99,73 %
Sum	148,5	99,73 %	
Avvik	0,4		

Table 3 Sample name, sample weight before sieving, total time sieved, vibration intensity, intervals, grain size, mass per sieve (g), mass per sieve (%) and cumulative mass (%).

BO-1			
Masse før sikting (g)	Tid (min)	Intensitet	Intervall (s)
189,7	25	6	3
Korn størrelse (μm)	masse per sikt (g)	masse per sikt (%)	Kumulativ masse (%)
>2000	0,7	0,37 %	0,37 %
2000-1000	0,1	0,05 %	0,42 %
1000-500	2,9	1,53 %	1,95 %
500-250	119,2	62,84 %	64,79 %
250-125	64,1	33,79 %	98,58 %
125-63	2,3	1,21 %	99,79 %
63-32	0,1	0,05 %	99,84 %
<32	0	0,00 %	99,84 %
Sum	189,4	99,84 %	
Avvik	0,3		

Table 4 Sample name, sample weight before sieving, total time sieved, vibration intensity, intervals, grain size, mass per sieve (g), mass per sieve (%) and cumulative mass (%).

BO-2			
Masse før sikting (g)	Tid (min)	Intensitet	Intervall (s)
168,5	25	6	3
Korn størrelse (μm)	masse per sikt (g)	masse per sikt (%)	Kumulativ masse (%)
>2000	0	0,00 %	0,00 %
2000-1000	0	0,00 %	0,00 %
1000-500	0,5	0,30 %	0,30 %
500-250	91	54,01 %	54,30 %
250-125	75,3	44,69 %	98,99 %
125-63	1,6	0,95 %	99,94 %
63-32	0	0,00 %	99,94 %
<32	0	0,00 %	99,94 %
Sum	168,4		
Avvik	0,1		

Table 5 Sample name, sample weight before sieving, total time sieved, vibration intensity, intervals, grain size, mass per sieve (g), mass per sieve (%) and cumulative mass (%).

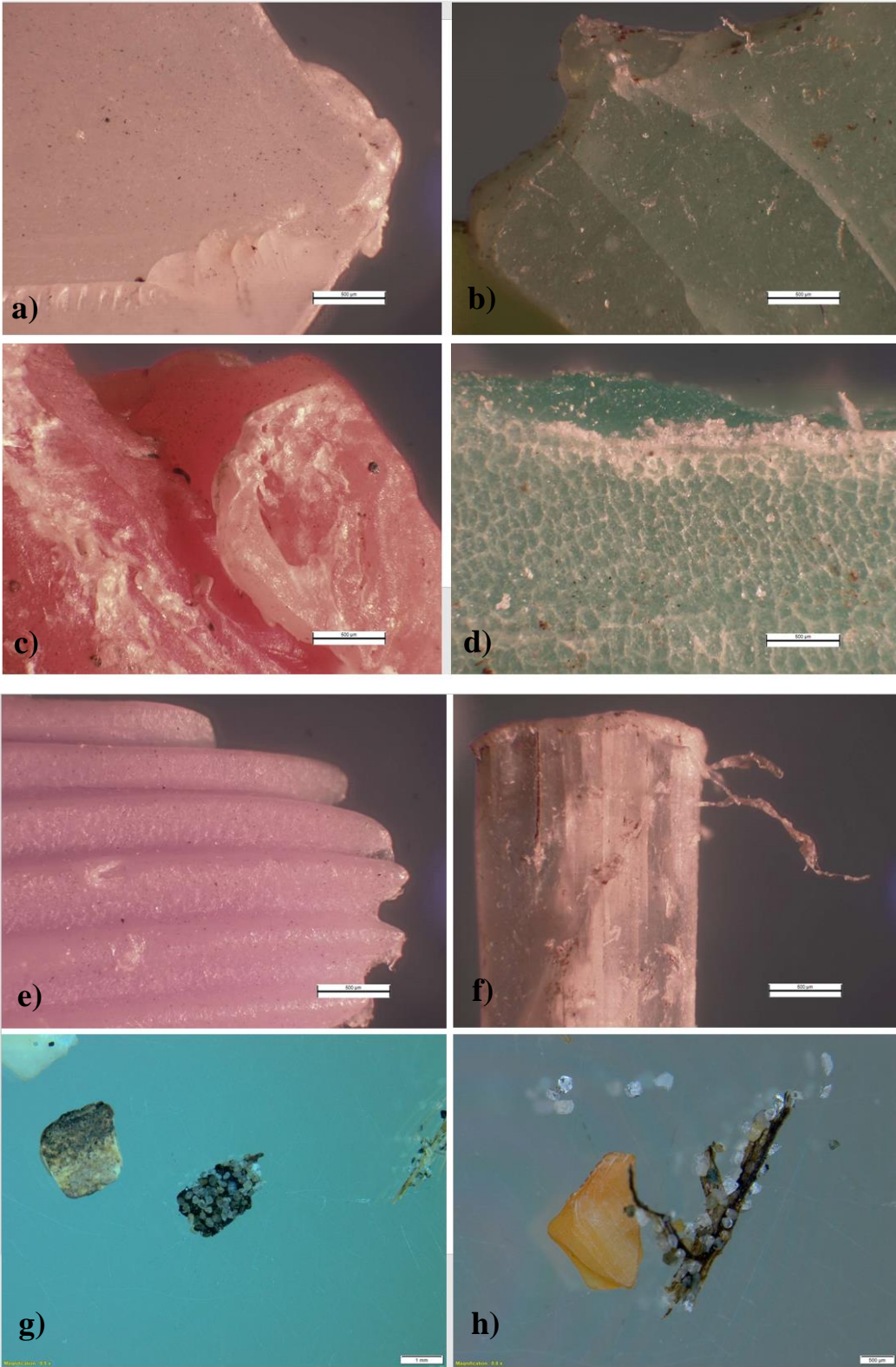
SS-1			
Masse før sikting (g)	Tid (min)	Intensitet	Intervall (s)
179	25	6	3
Korn størrelse (μm)	masse per sikt (g)	masse per sikt (%)	Kumulativ masse (%)
>2000	41,3	23,07 %	23,07 %
2000-1000	42,6	23,80 %	46,87 %
1000-500	38,5	21,51 %	68,38 %
500-250	33,5	18,72 %	87,09 %
250-125	15,9	8,88 %	95,98 %
125-63	4,5	2,51 %	98,49 %
63-32	1,4	0,78 %	99,27 %
<32	0,5	0,28 %	
Sum (g)	178,2	99,55 %	
Avvik (g)	0,8		

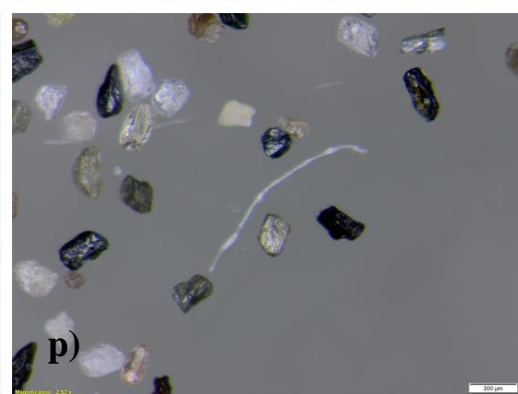
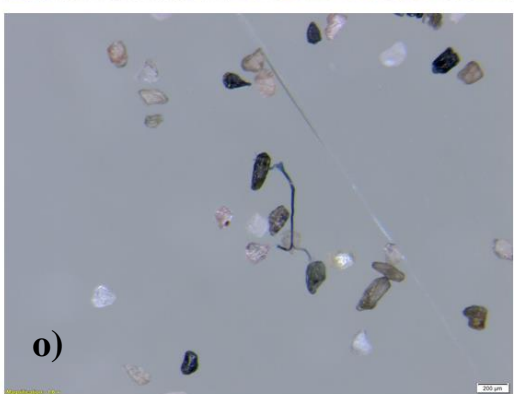
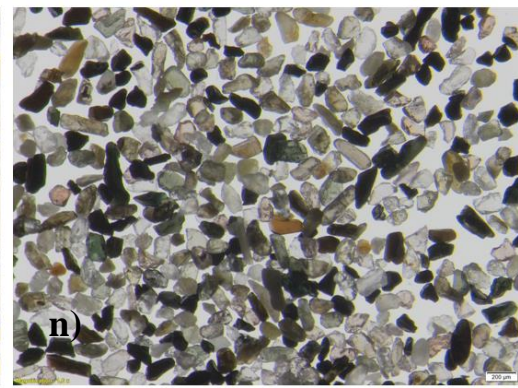
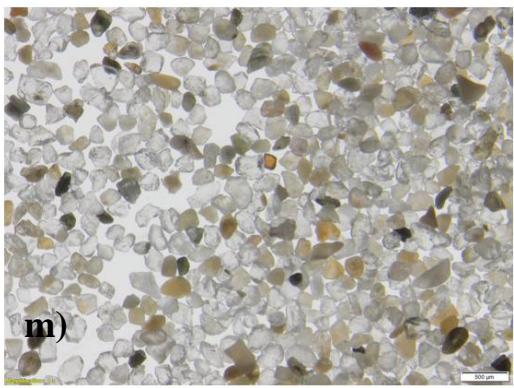
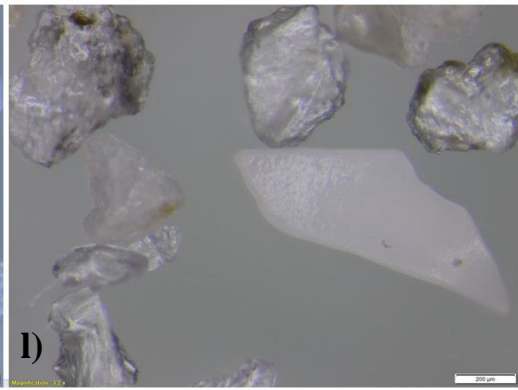
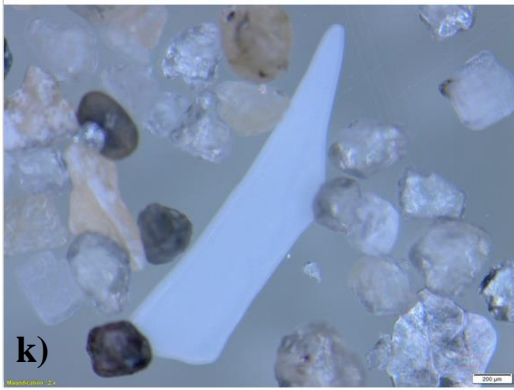
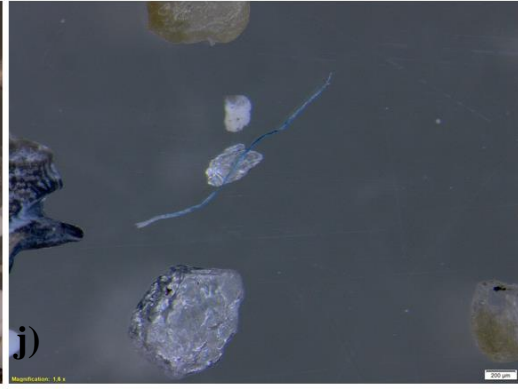
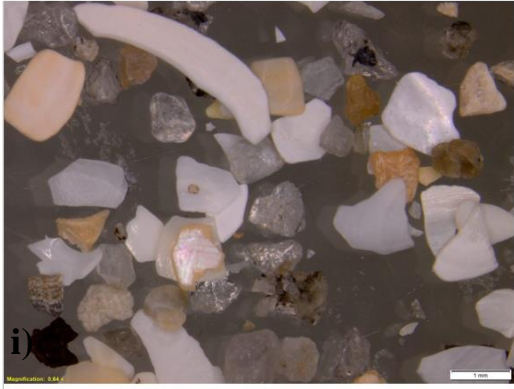
Table 6 Sample name, sample weight before sieving, total time sieved, vibration intensity, intervals, grain size, mass per sieve (g), mass per sieve (%) and cumulative mass (%).

SS-2	216,1		
Masse før sikting (g)	Tid (min)	Intensitet	Intervall (s)
	25	6	3
Korn størrelse (μm)	masse per sikt (g)	masse per sikt (%)	Kumulativ masse (%)
>2000	39,4	18,23 %	18,23 %
2000-1000	45,1	20,87 %	39,10 %
1000-500	49,5	22,91 %	62,01 %
500-250	61,2	28,32 %	90,33 %
250-125	16,6	7,68 %	98,01 %
125-63	1,6	0,74 %	98,75 %
63-32	0,5	0,23 %	98,98 %
<32	0,6	0,28 %	99,26 %
Sum	214,5	99,26 %	99,26 %
Avvik	1,6		

Appendix B

Additional optical light microscopy images before separation:





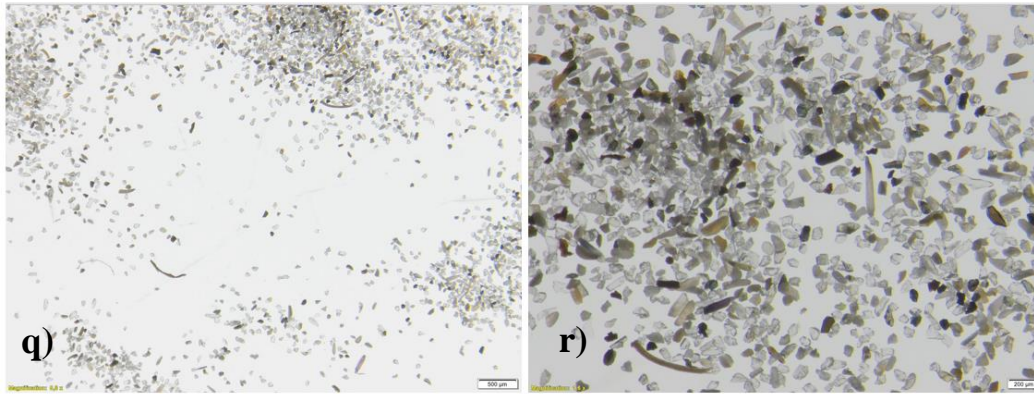
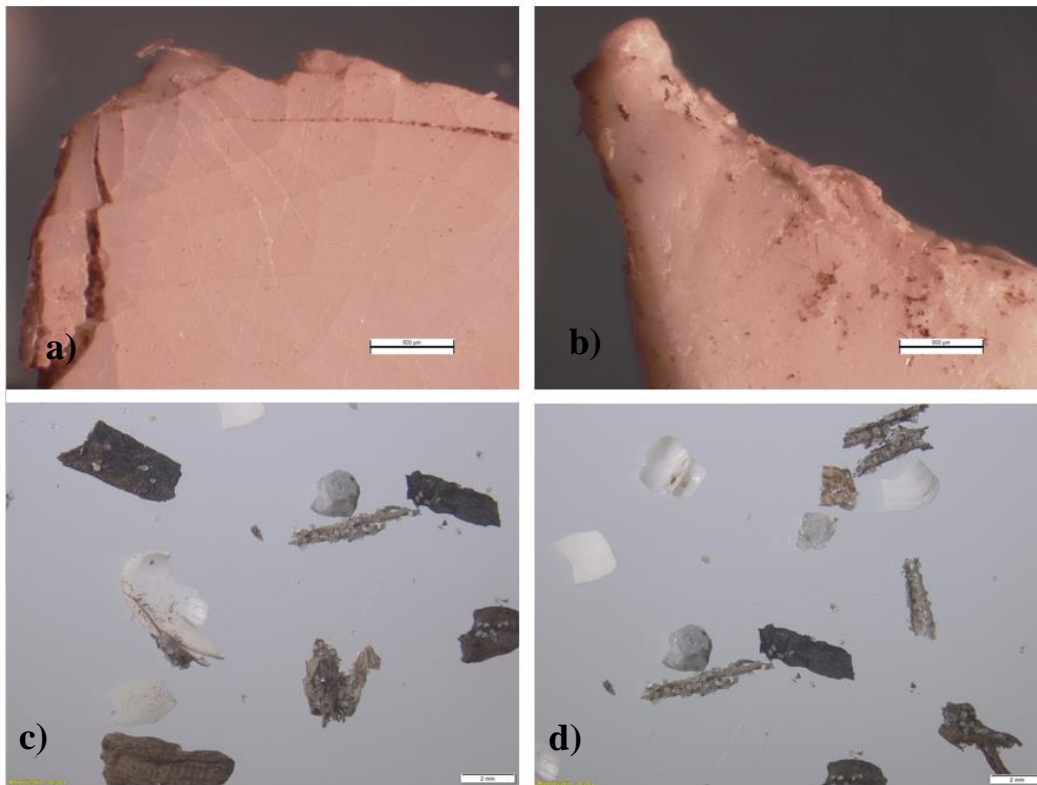
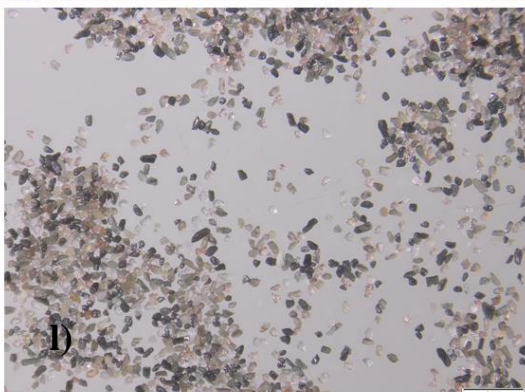
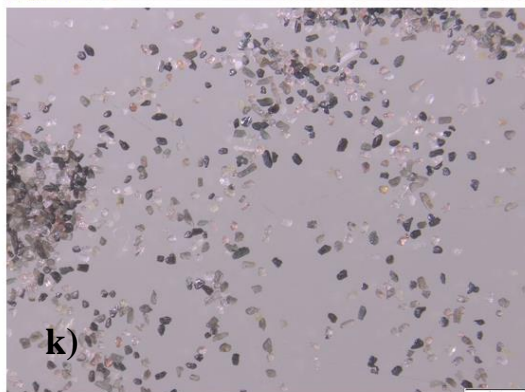
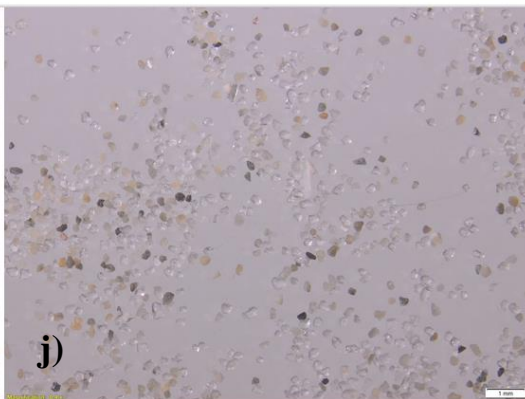
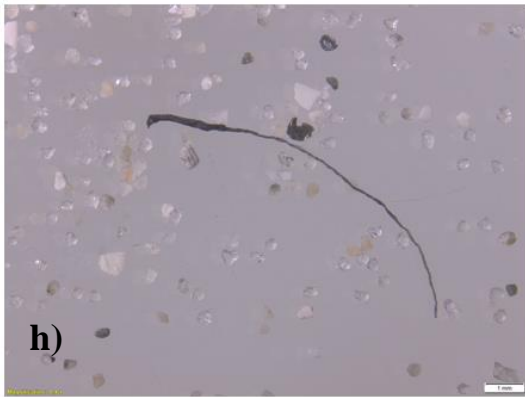
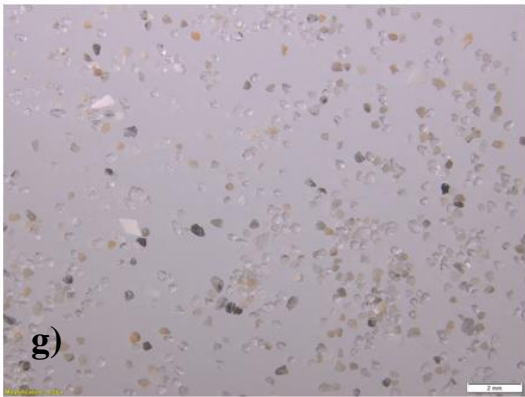
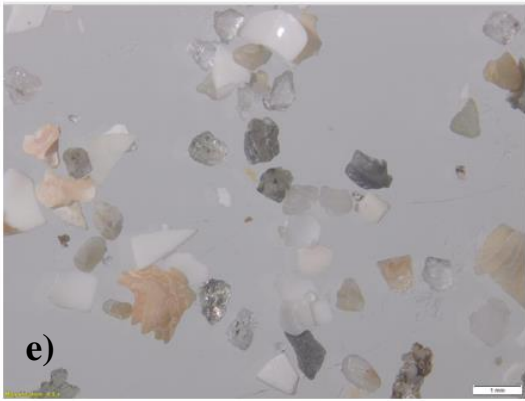


Figure A Optical pictures before separation for surface sample SO-1 a) SO-1_1 white colors plastic with visible fractures showing how microplastics are formed; b) SO-1_2 light blue colors plastic showing fractures and other attached fibers; c) SO-1_3 pink partly deformed plastic piece; d) SO-1_Leave_1 nice view of a light blue plastic looking like a beach; e) SO-1_Leave_2 irregular pinkish piece of plastic; f) SO-1_Leave_3 an particle with attached fibers; g) SO-1_2000-1000 µm smaller grains attached to a larger one; h) SO-1_2000-1000 µm smaller grains attached to possibly some organic matter; i) SO-1 1000-500 µm overview of the sample; j) SO-1 1000-500 µm blue fiber; k) SO-1 500-250 µm larger white particle possibly plastic or a shell fragment in the middle of several smaller grains; l) SO-1 500-250 µm white particle possibly plastic or a shell fragment; m) SO-1 250-125 µm overview of the sample; n) SO-1 125-63 µm overview of the sample; o) SO-1 125-63 µm blue fiber; p) SO-1 125-63 µm white fiber; q) SO-1 63-32 µm overview of the sample; r) SO-1 63-32 µm overview of the sample.





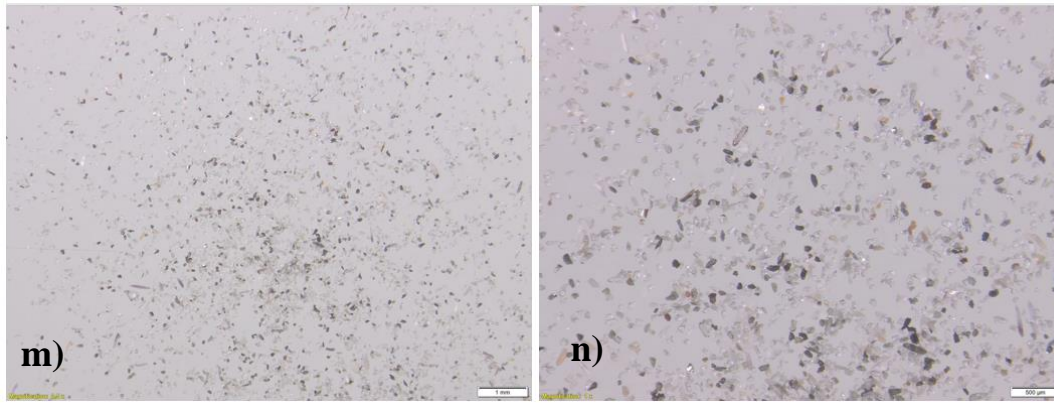
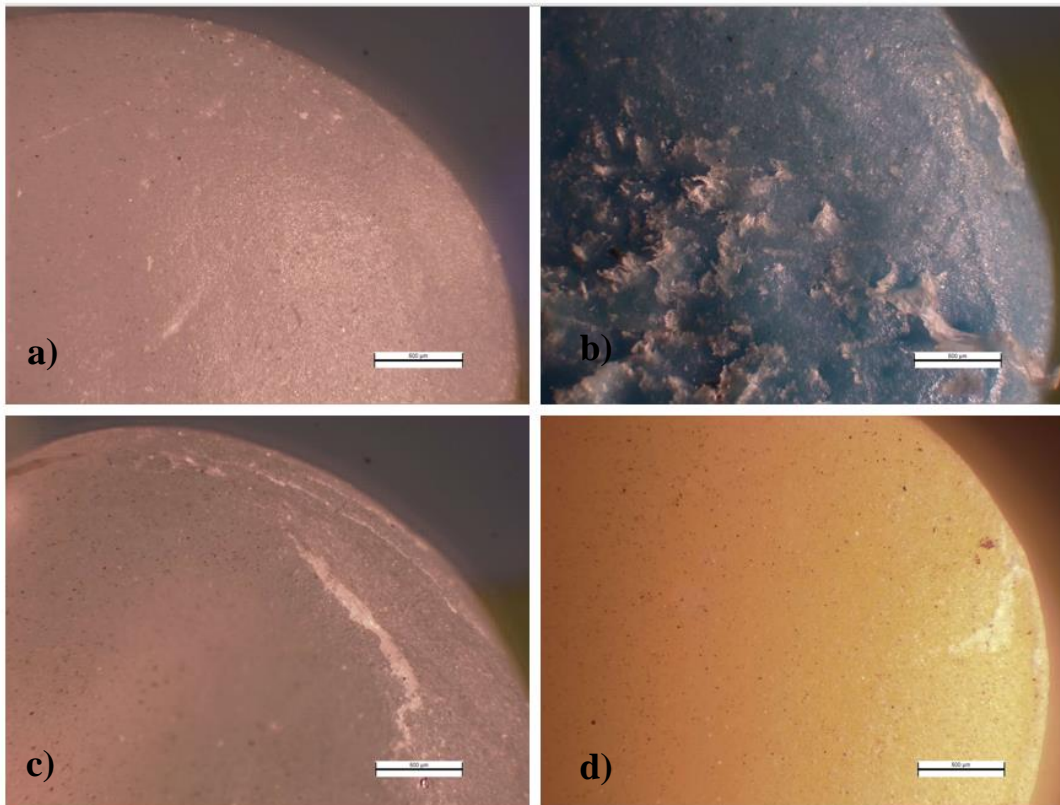


Figure B Optical pictures before separation for subsurface sample SO-2 a) SO-2_1 white piece of plastic with several fractures; b) SO-2_2 white piece of plastic; c) SO-2 2000-1000 μm overview of sample; d) SO-2 2000-1000 μm overview of sample; e) SO-2 1000-500 μm overview of sample; f) SO-2 1000-500 μm probably organic matter with grains attached and a white/transparent fiber surround it; g) SO-2 500-250 μm overview of the sample; h) SO-2 500-250 μm blackish fiber; i) SO-2 250-125 μm overview of the sample; j) SO-2 250-125 μm overview of the sample; k) SO-2 125-63 μm overview of the sample; l) SO-2 125-63 μm overview of the sample; m) SO-2 63-32 μm overview of the sample; n) SO-2 63-32 μm overview of the sample.



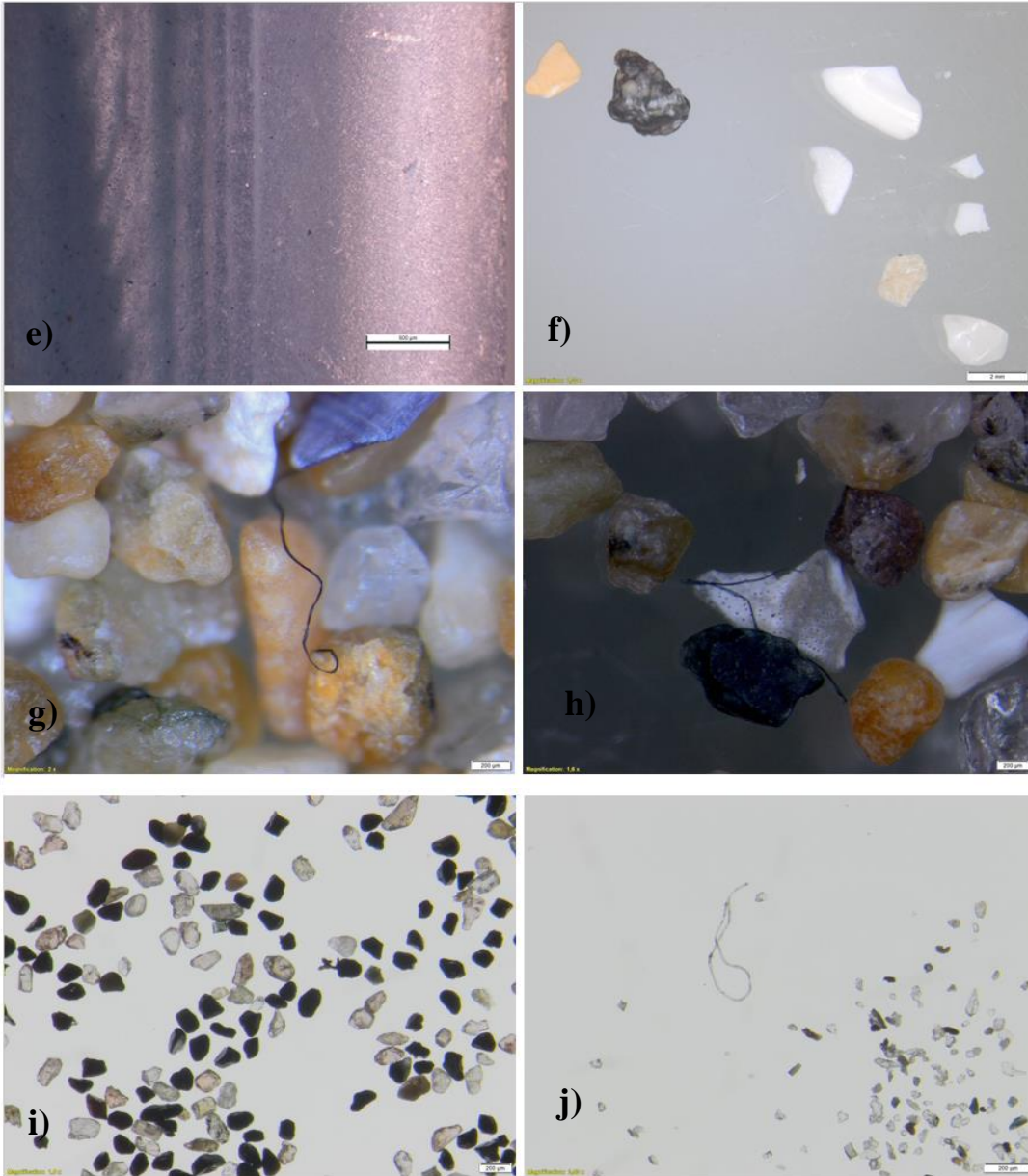


Figure C Optical pictures before separation for surface sample BO-1 a) BO-1_1 white plastic nurdle; b) BO-1_2 blue plastic nurdle looking like a planet with other smaller fiber attached to it; c) BO-1_3 white/blueish plastic nurdle; d) BO-1_Leave_3 yellow nurdle looking like the sun; e) BO-1_Leave_2 blueish plastic particle looking like the ocean; f) BO-1 2000-1000 μm several shell fragments; g) BO-1 500-250 μm black fiber; h) BO-1 500-250 μm blue fiber; i) BO-1 125-63 μm overview of the sample; j) BO-1 63-32 μm white/transparent fiber.

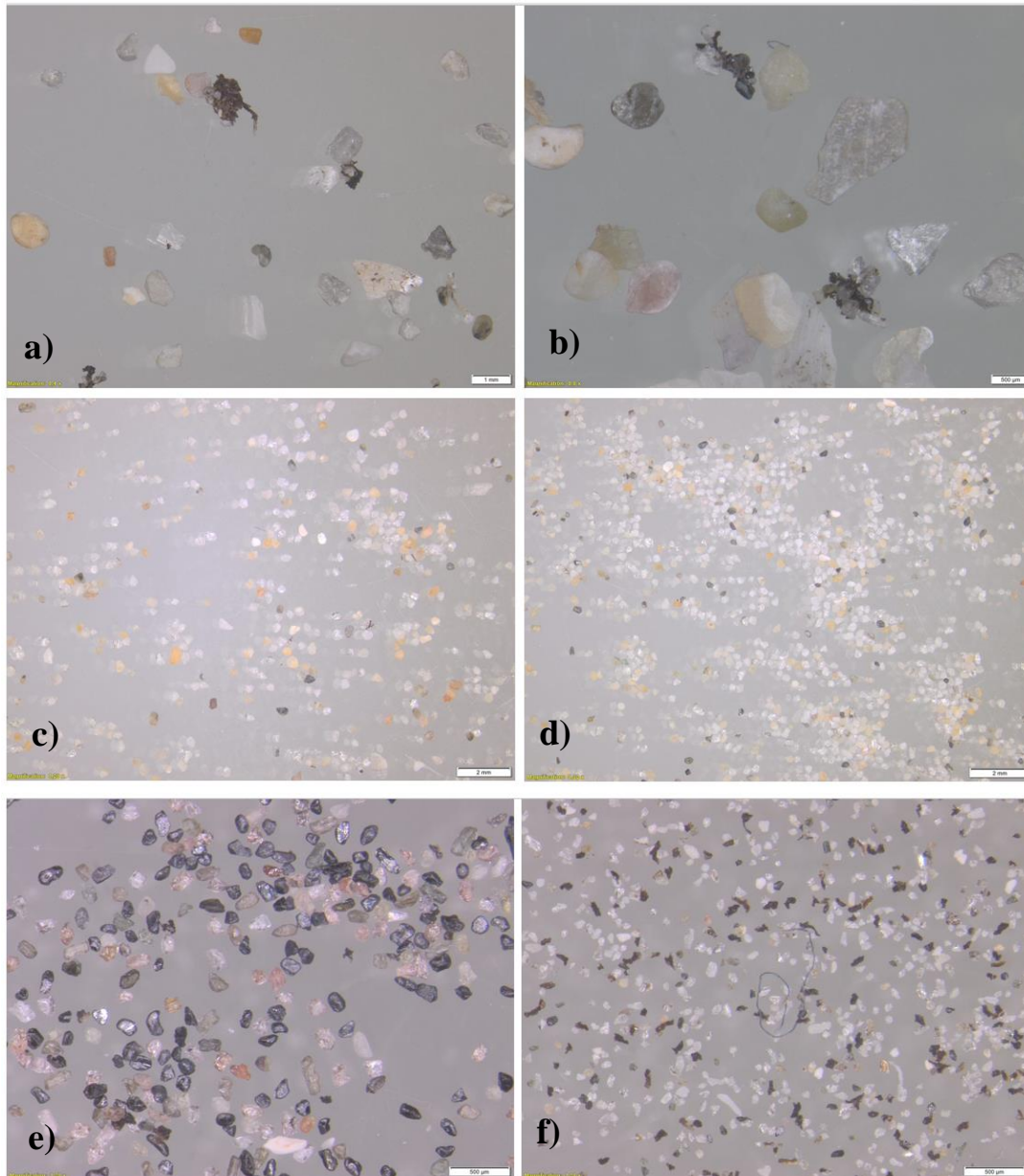
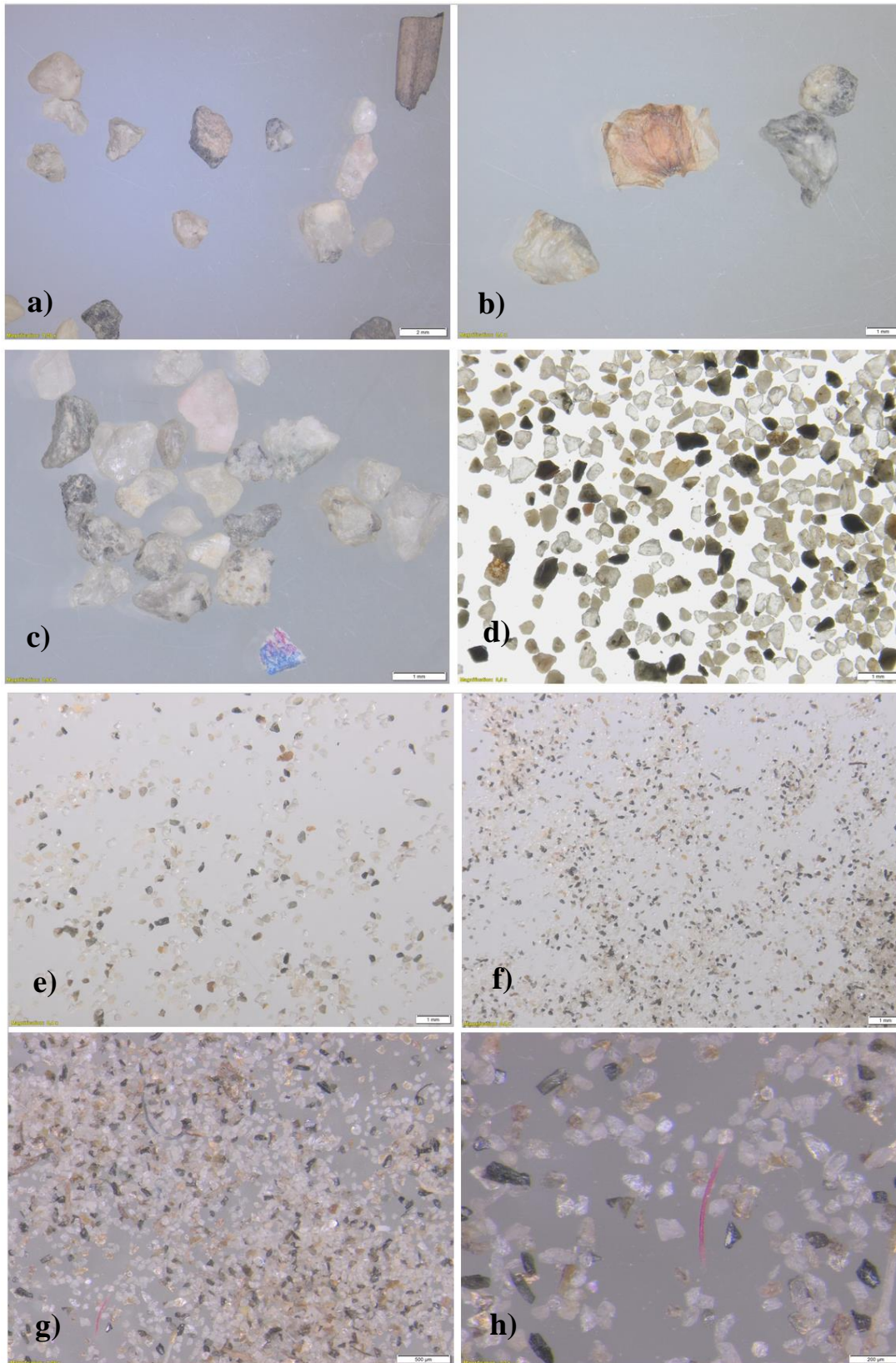


Figure D Optical pictures before separation for subsurface sample BO-2 a) BO-2 1000-500 μm overview of the sample; b) BO-2 1000-500 μm overview of the sample; c) BO-2 500-250 μm overview of the sample; d) BO-2 250-125 μm overview of the sample; e) BO-2 125-63 μm overview of the sample; f) BO-2 63-32 μm overview of the sample with a blue fiber in the middle.



Optical pictures before separation for surface sample SS-1 a) SS-1 2000-1000 μm overview of the sample; b) SS-1 2000-1000 μm overview of the sample; c) SS-1 1000-500 μm interesting pink and blue particle; d) SS-1 500-250 μm overview of the sample; e) SS-1 250-125 μm overview of the sample; f) SS-1 125-63 μm overview of the sample; g) SS-1 63-32 μm overview of the sample showing several fibers; h) SS-1 63-32 μm red fiber.

Figure E

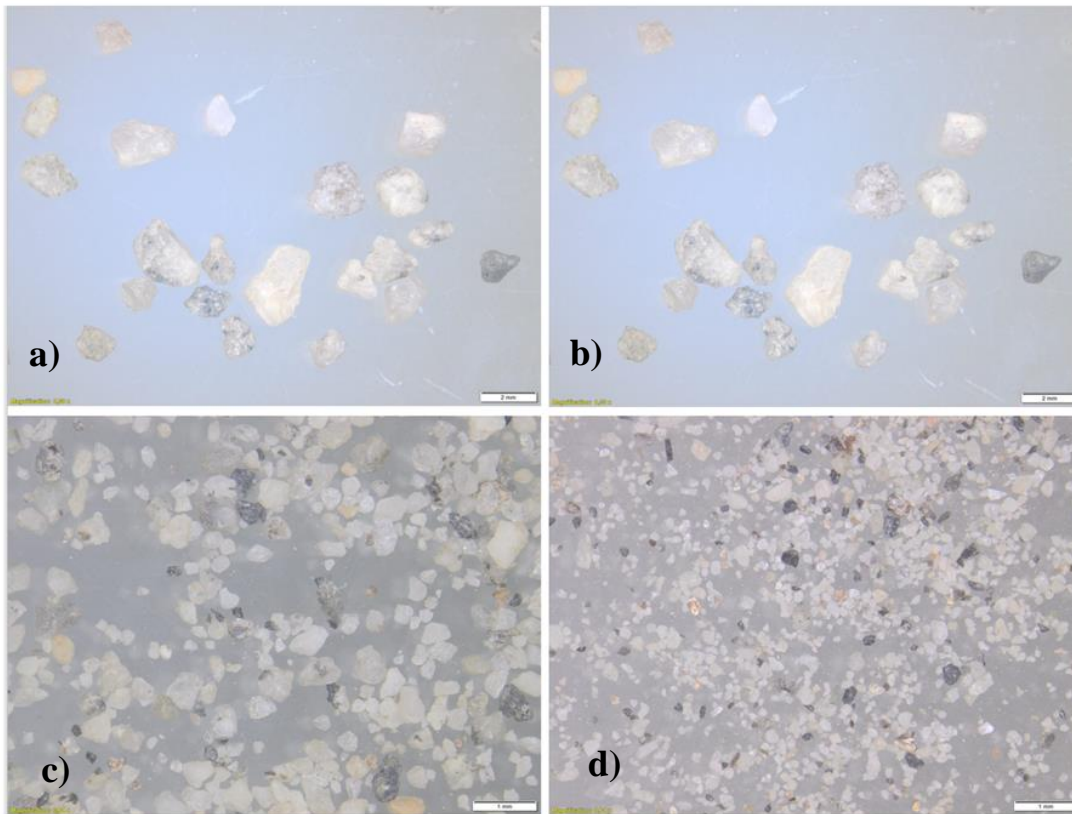


Figure F Optical pictures before separation for subsurface sample SS-2 a) SS-2 2000-1000 μm overview of the sample; b) SS-2 1000-500 μm overview of the sample; c) SS-2 500-250 μm overview of the sample; d) SS-2 250-125 μm overview of the sample.

Appendix C

Weight of the filters before and after separation:

Table 7 Sample name, fraction, weight of filter before separation, weight of filter after separation with sample, collected sample weight.

SO-1			
Fraction (μm)	Weight filter (g)	Weight filter with sample (g)	Diff (=sample weight) (g)
500-250	1,499	1,506	0,007
250-125	1,352	1,410	0,058
125-63	1,346	1,349	0,003

Table 8 Sample name, fraction, weight of filter before separation, weight of filter after separation with sample, collected sample weight.

SO-2			
Fraction (μm)	Weight filter (g)	Weight filter with sample (g)	Diff (=sample weight) (g)
500-250	0,923	0,931	0,008
250-125	1,240	1,306	0,066
125-63	0,965	0,974	0,009

Table 9 Sample name, fraction, weight of filter before separation, weight of filter after separation with sample, collected sample weight.

BO-1			
Fraction (μm)	Weight filter (g)	Weight filter with sample (g)	Diff (=sample weight) (g)
500-250	1,440	1,449	0,009
250-125	1,340	1,342	0,002
125-63	1,879	1,889	0,011

Table 10 Sample name, fraction, weight of filter before separation, weight of filter after separation with sample, collected sample weight.

BO-2			
Fraction (μm)	Weight filter (g)	Weight filter with sample (g)	Diff (=sample weight) (g)
500-250	1,059	1,068	0,009
250-125	1,046	1,091	0,045
125-63	1,110	1,119	0,009

Table 11 Sample name, fraction, weight of filter before separation, weight of filter after separation with sample, collected sample weight.

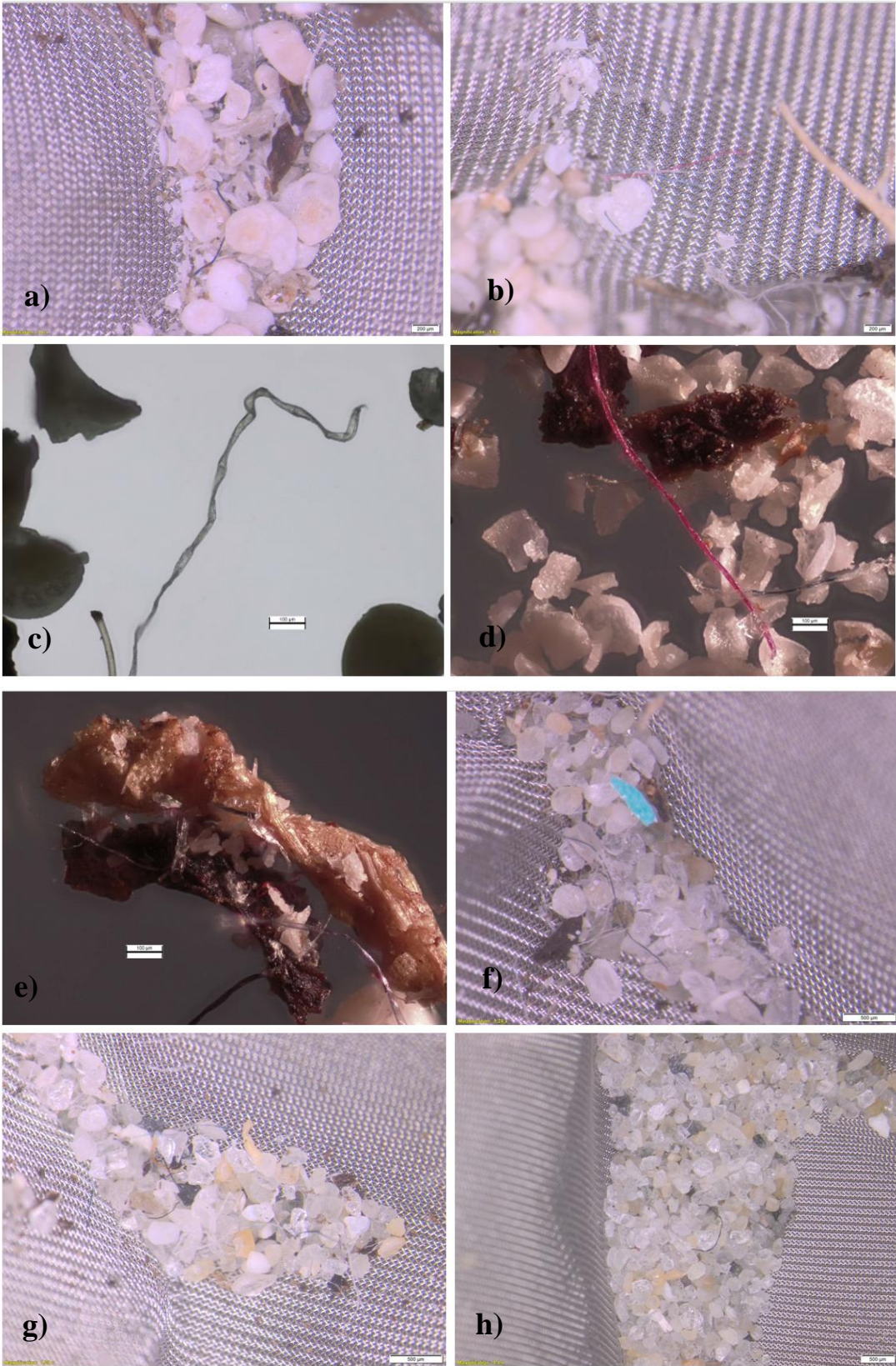
SS-1			
Fraction (μm)	Weight filter (g)	Weight filter with sample (g)	Diff (=sample weight) (g)
500-250	1,273	1,309	0,037
250-125	1,264	1,279	0,015
125-63	1,307	1,323	0,016

Table 12 Sample name, fraction, weight of filter before separation, weight of filter after separation with sample, collected sample weight.

SS-2			
Fraction (μm)	Weight filter (g)	Weight filter with sample (g)	Diff (=sample weight) (g)
500-250	1,099	1,104	0,005
250-125	1,126	1,132	0,006

Appendix D

Additional optical light microscopy images after separation:



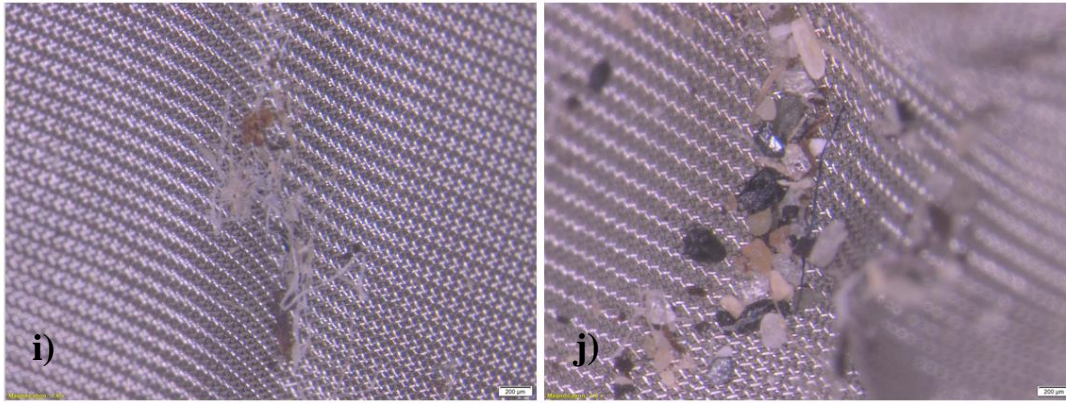


Figure G Optical pictures after separation for surface sample SO-1 a) SO-1 500-250 μm fibers in multiple colors; b) SO-1 500-250 μm several fibers in multiple colors; c) SO-1 500-250 μm transparent fiber, photo credit (University of Milano-Bicocca, Italy n.d.); d) SO-1 500-250 μm red fiber, photo credit (University of Milano-Bicocca, Italy n.d.); e) SO-1 500-250 μm two larger grain probably organic matter with other grains attached, photo credit (University of Milano-Bicocca, Italy n.d.); f) SO-1 250-125 μm interesting light blue particle and a black fiber; g) SO-1 250-125 μm overview of the sample; h) SO-1 250-125 μm overview of the sample; i) SO-1 125-63 μm several white fibers; j) SO-1 125-63 μm black fiber.



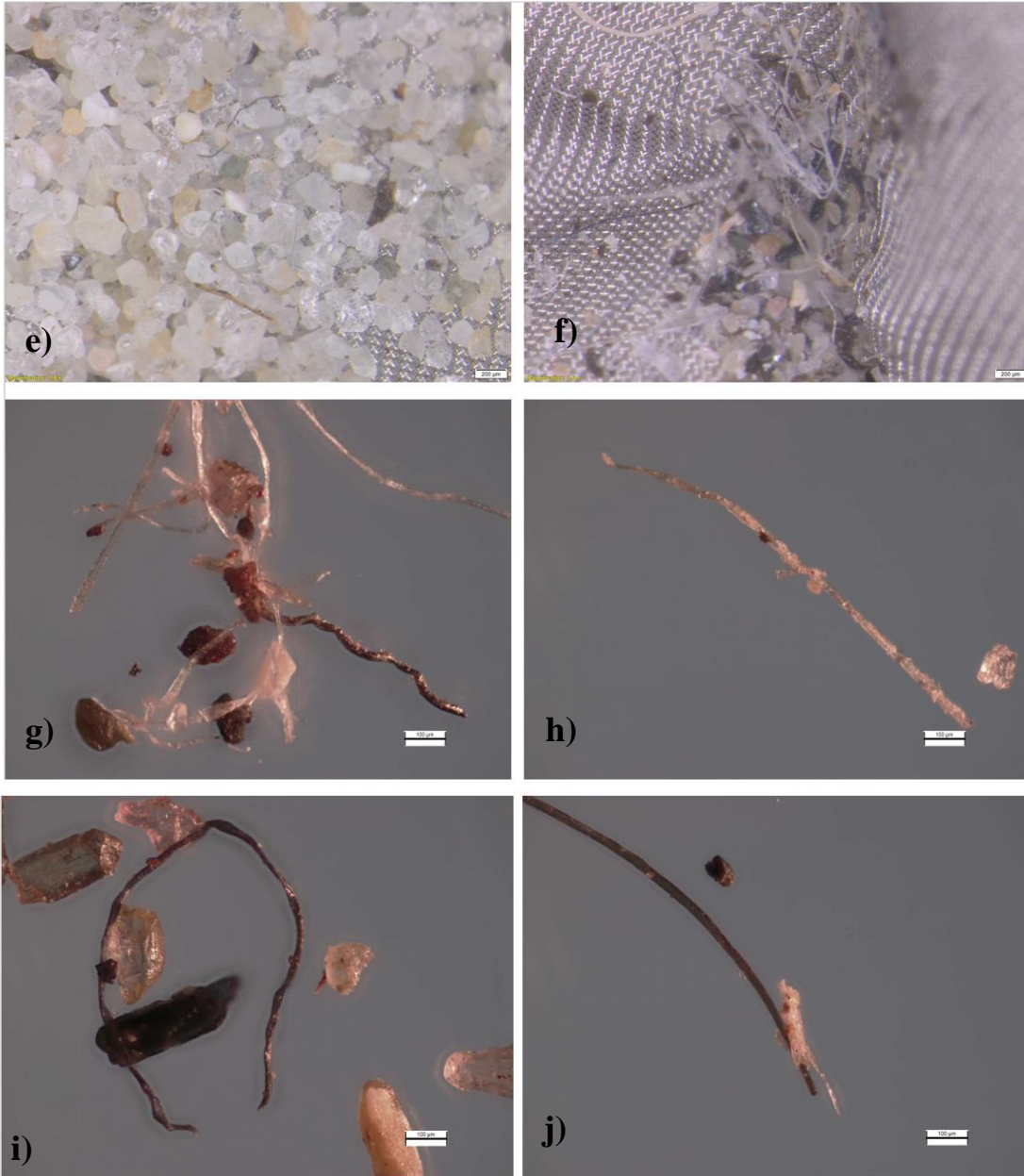


Figure H Optical pictures after separation for subsurface sample SO-2 a) SO-2 500-250 μm white and black fibers; b) SO-2 500-250 μm white, red and blue fibers; c) SO-2 500-250 μm overview of the sample; d) SO-2 250-125 μm overview of the sample; e) SO-2 250-125 μm overview of the sample; f) SO-2 125-63 μm several fibers; g) SO-2 125-63 μm transparent fibers, photo credit (University of Milano-Bicocca, Italy n.d.); h) SO-2 125-63 μm possibly fiber with even smaller grains attached, photo credit (University of Milano-Bicocca, Italy n.d.); i) SO-2 125-63 μm possibly blackish fiber, photo credit (University of Milano-Bicocca, Italy n.d.); j) SO-2 125-63 μm fiber or organic matter with other grains and fiber attached, photo credit (University of Milano-Bicocca, Italy n.d.).

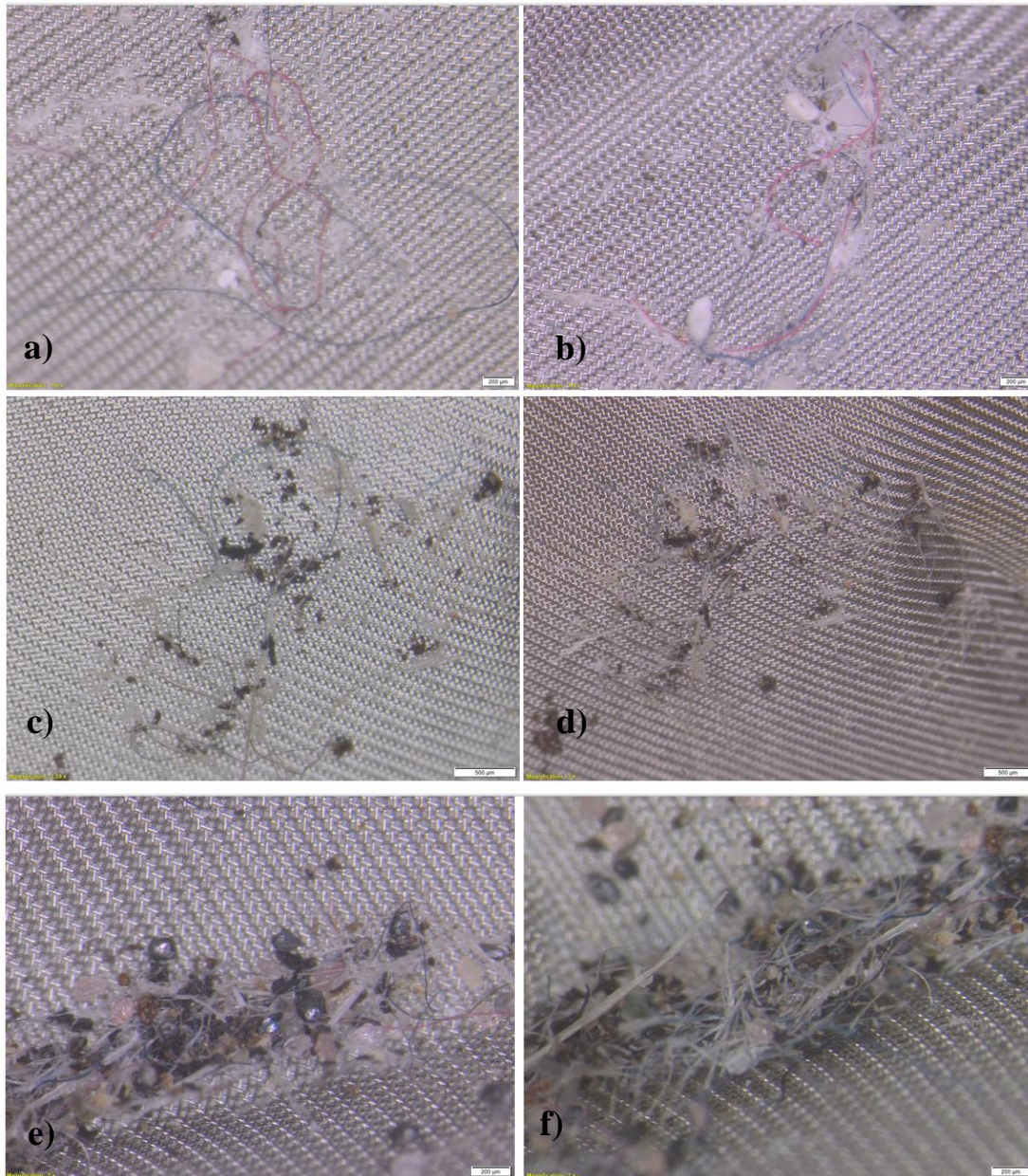
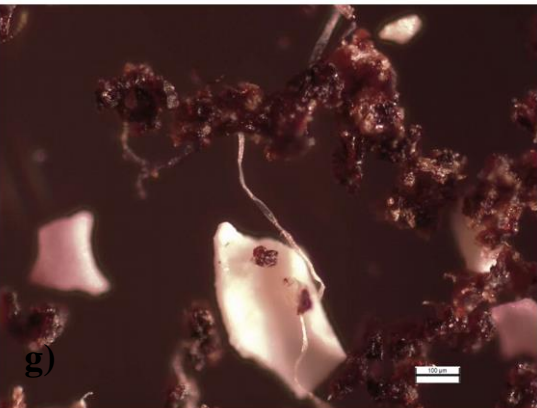
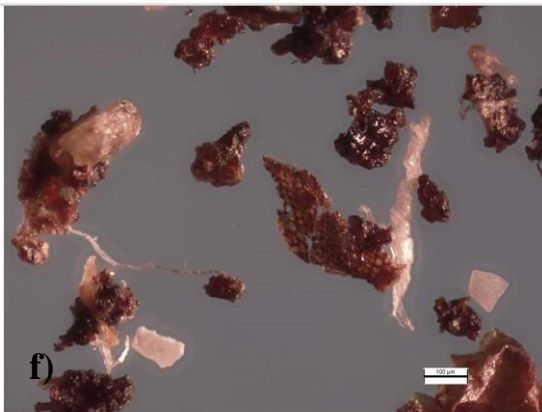
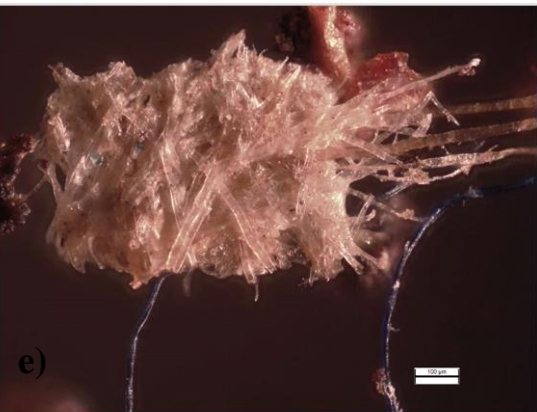
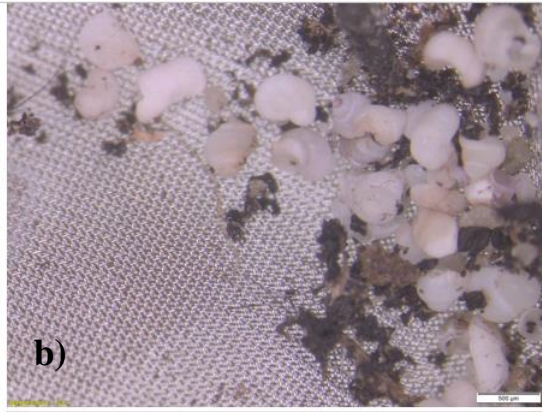
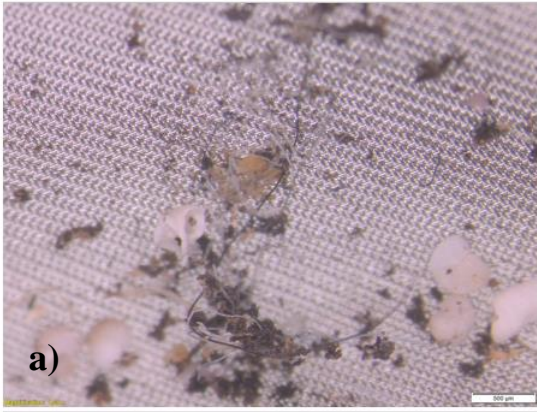


Figure I Optical pictures after separation for surface sample BO-1 a) BO-1 250-125 μm overview of the sample showing several fibers; b) BO-1 250-125 μm overview of the sample showing several fibers; c) BO-1 250-125 μm overview of the sample showing several fibers; d) BO-1 250-125 μm overview of the sample showing several fibers; e) BO-1 125-63 μm overview of the sample showing several fibers; f) BO-1 125-63 μm overview of the sample showing several fibers.



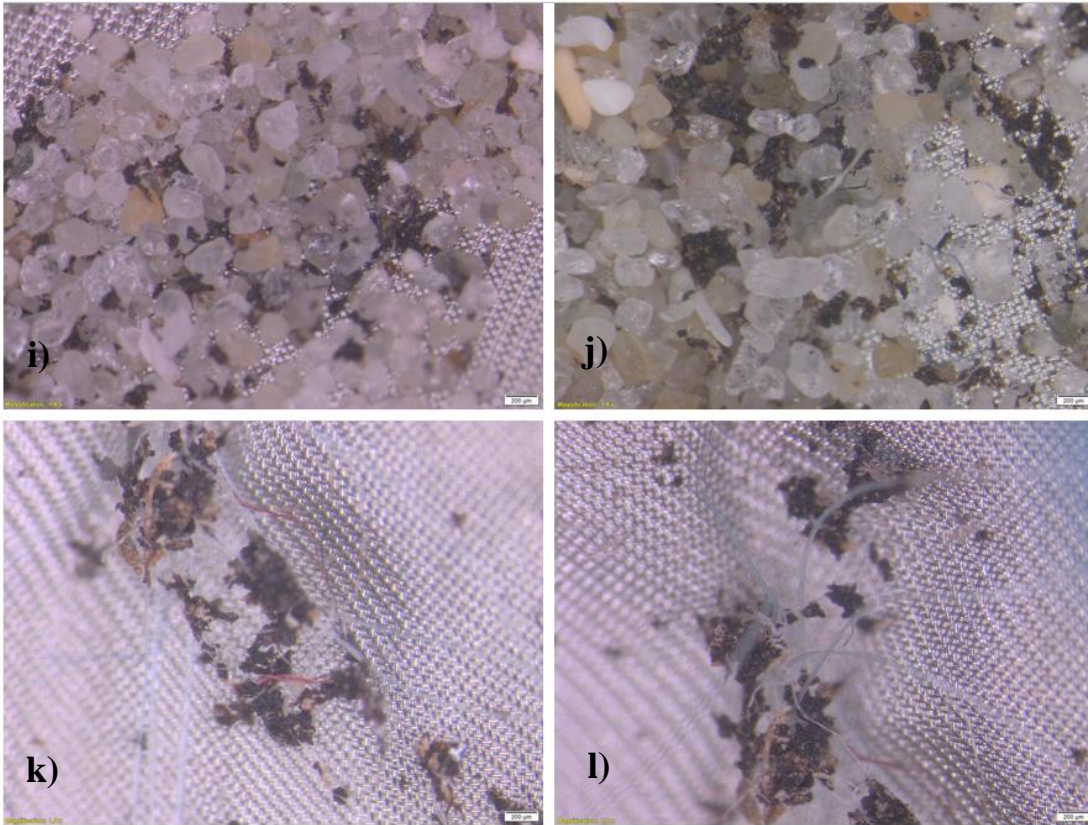


Figure J Optical pictures after separation for subsurface sample BO-2 a) BO-2 500-250 μm overview of the sample; b) BO-2 500-250 μm overview of the sample; c) BO-2 500-250 μm transparent fiber, photo credit (University of Milano-Bicocca, Italy n.d.); d) BO-2 500-250 μm transparent and black fiber, photo credit (University of Milano-Bicocca, Italy n.d.); e) BO-2 500-250 μm interesting collecting of several fibers, photo credit (University of Milano-Bicocca, Italy n.d.); f) BO-2 500-250 μm smaller white fiber and a larger twisted possible fiber, photo credit (University of Milano-Bicocca, Italy n.d.); g) BO-2 500-250 μm white fiber and other grains, photo credit (University of Milano-Bicocca, Italy n.d.); h) BO-2 500-250 μm fiber , photo credit (University of Milano-Bicocca, Italy n.d.); i) BO-2 250-125 μm overview of the sample; j) BO-2 250-125 μm overview of the sample; k) BO-2 125-63 μm overview of the sample showing several fibers; l) BO-2 125-63 μm overview of the sample showing several fibers.

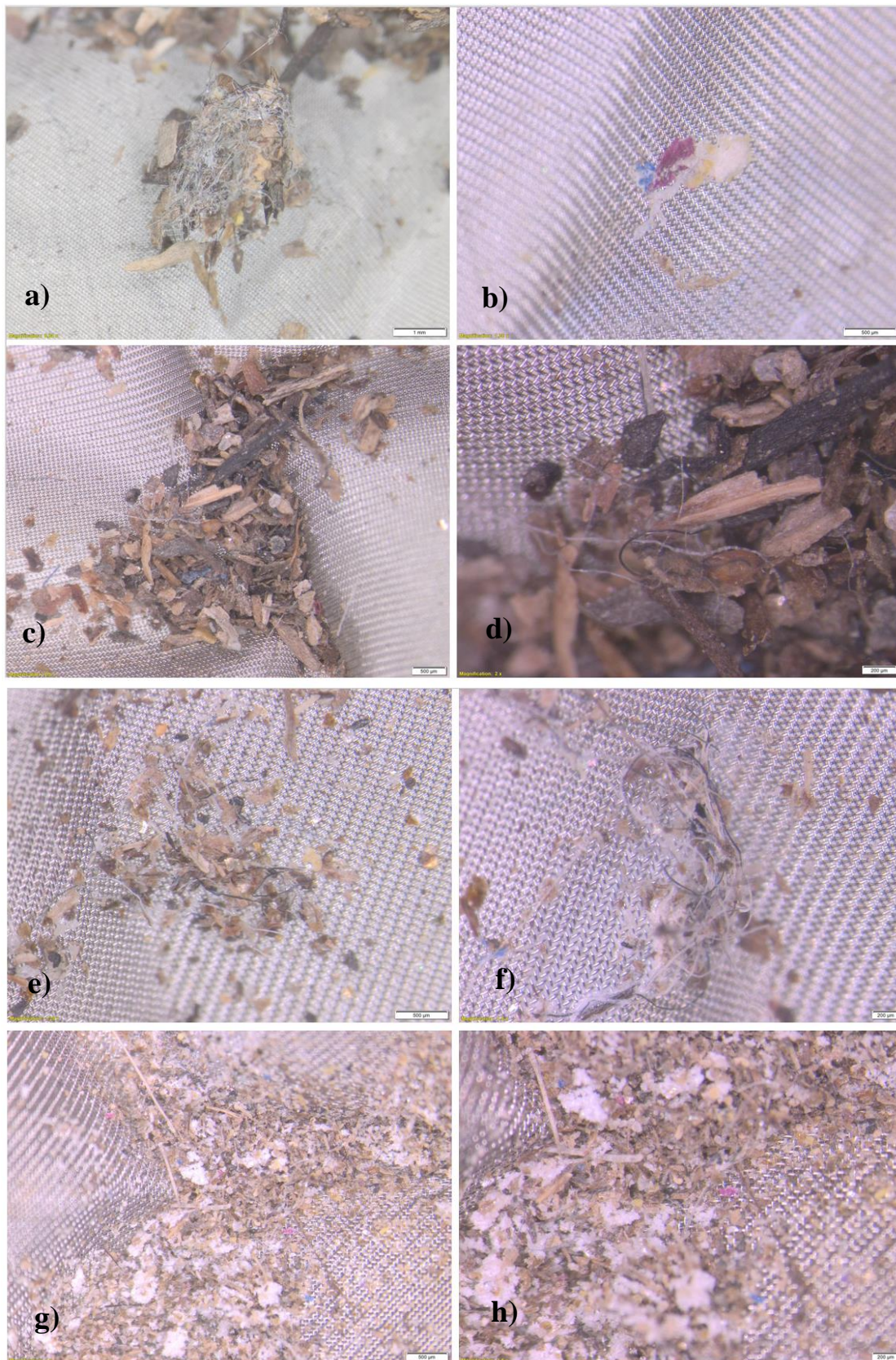


Figure K Optical pictures after separation for surface sample SS-1 a) SS-1 500-250 μm several fibers rolled together like a ball; b) SS-1 500-250 μm interesting pink and blue particle; c) SS-1 250-125 μm overview of the sample; d) SS-1 250-125 μm overview of the sample; e) SS-1 125-63 μm overview of the sample; f) SS-1 125-63 μm overview of the sample; g) SS-1 63-32 μm overview of the sample showing several smaller pink and blue particles; h) SS-1 63-32 μm overview of the sample showing several smaller pink and blue particles.

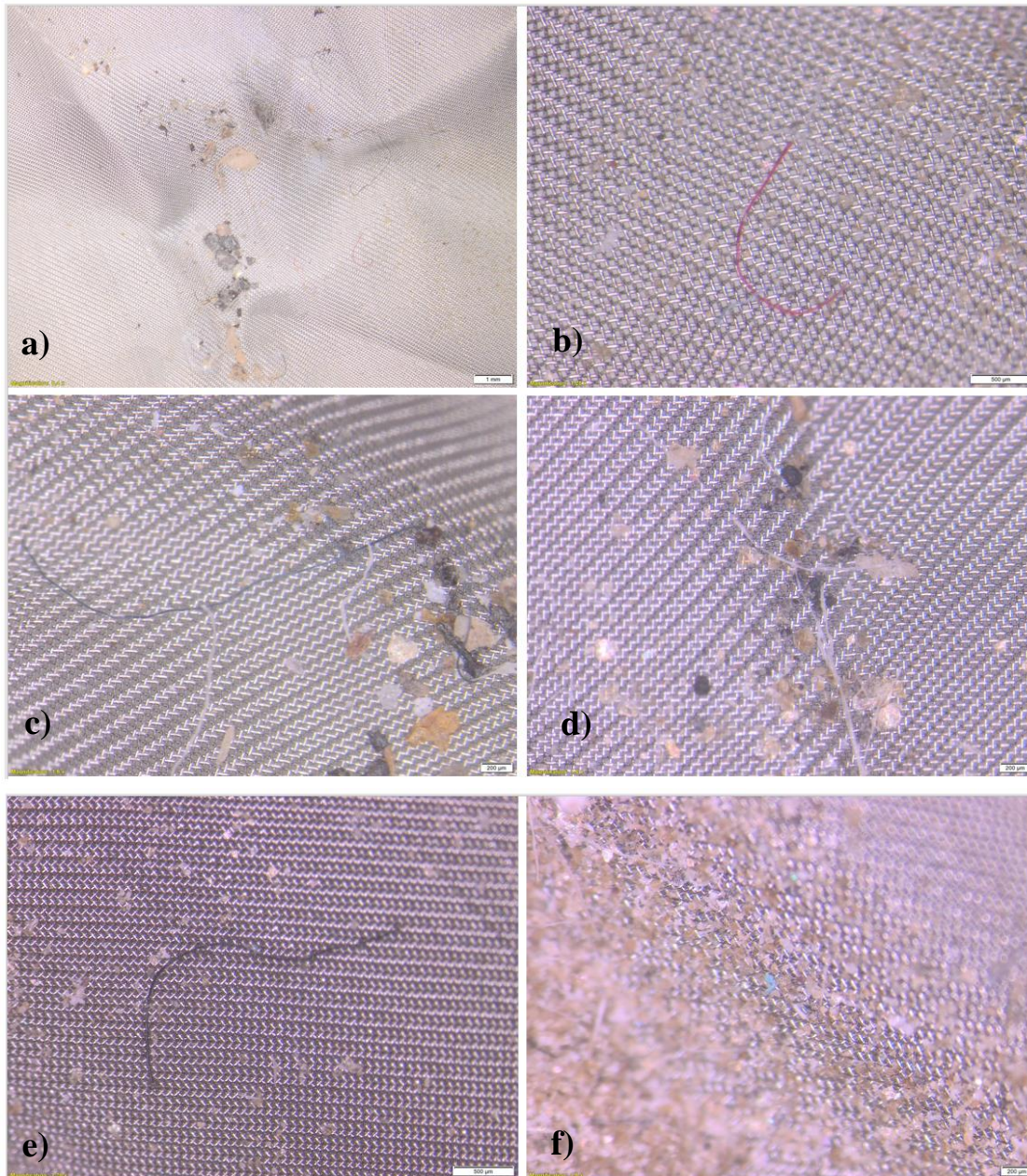


Figure L Optical pictures after separation for subsurface sample SS-2 a) SS-2 500-250 μm overview of the sample; b) SS-2 500-250 μm red fiber; c) SS-2 250-125 μm blue and white fibers; d) SS-2 250-125 μm white fibers; e) SS-2 62-32 μm black fiber; f) SS-2 62-32 μm light blue particle.

Appendix E

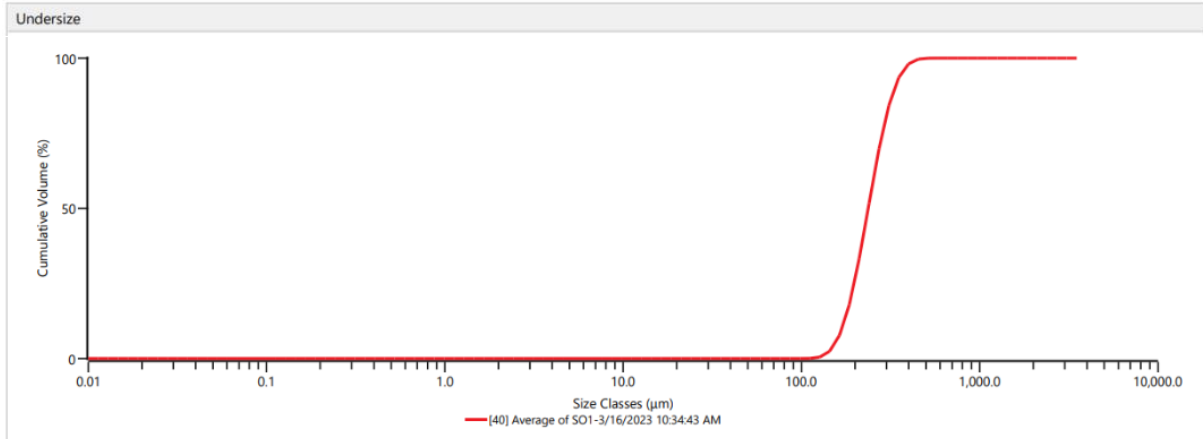
Full PSD analysis for all the samples:

Analysis

Malvern Panalytical



Measurement Details Operator Name caroline Sample Name Average of SO1 SOP File Name 240223 - Christine sand.msop	Measurement Details Analysis Date Time 3/16/2023 10:34:43 AM Measurement Date Time 3/16/2023 10:34:43 AM Result Source Averaged
Analysis Particle Name Quartz Particle Refractive Index 1.543 Particle Absorption Index 0.010 Dispersant Name Dry dispersion Dispersant Refractive Index 1.000 Scattering Model Mie Analysis Model General Purpose Weighted Residual 0.55 % Laser Obscuration 4.74 %	Result Concentration 0.1440 % Span 0.704 Uniformity 0.217 Specific Surface Area 26.17 m ² /kg D [3,2] 229 µm D [4,3] 245 µm Dv (10) 168 µm Dv (50) 237 µm Dv (90) 335 µm



Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In
0.0100	0.00	0.0463	0.00	0.214	0.00	0.991	0.00	4.58	0.00	21.2	0.00	98.1	0.03	454	0.31
0.0114	0.00	0.0526	0.00	0.243	0.00	1.13	0.00	5.21	0.00	24.1	0.00	111	0.42	516	0.02
0.0129	0.00	0.0597	0.00	0.276	0.00	1.28	0.00	5.92	0.00	27.4	0.00	127	1.95	586	0.00
0.0147	0.00	0.0679	0.00	0.314	0.00	1.45	0.00	6.72	0.00	31.1	0.00	144	5.30	666	0.00
0.0167	0.00	0.0771	0.00	0.357	0.00	1.65	0.00	7.64	0.00	35.3	0.00	163	10.25	756	0.00
0.0189	0.00	0.0876	0.00	0.405	0.00	1.88	0.00	8.68	0.00	40.1	0.00	186	15.32	859	0.00
0.0215	0.00	0.0995	0.00	0.460	0.00	2.13	0.00	9.86	0.00	45.6	0.00	211	18.40	976	0.00
0.0244	0.00	0.113	0.00	0.523	0.00	2.42	0.00	11.2	0.00	51.8	0.00	240	18.07	1110	0.00
0.0278	0.00	0.128	0.00	0.594	0.00	2.75	0.00	12.7	0.00	58.9	0.00	272	14.49	1260	0.00
0.0315	0.00	0.146	0.00	0.675	0.00	3.12	0.00	14.5	0.00	66.9	0.00	310	9.30	1430	0.00
0.0358	0.00	0.166	0.00	0.767	0.00	3.55	0.00	16.4	0.00	76.0	0.00	352	4.57	1630	0.00
0.0407	0.00	0.188	0.00	0.872	0.00	4.03	0.00	18.7	0.00	86.4	0.00	400	1.57	1850	0.00

Analysis

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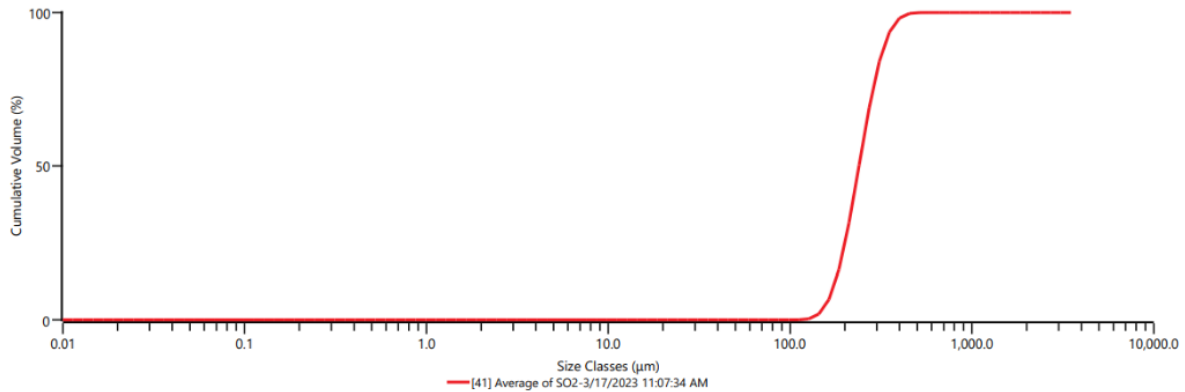
Measurement Details	
Operator Name	caroline
Sample Name	Average of SO2
SOP File Name	240223 - Christine sand.msop

Measurement Details	
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Measurement Date Time	3/17/2023 11:07:34 AM
Result Source	Averaged

Analysis	
Particle Name	Quartz
Particle Refractive Index	1.543
Particle Absorption Index	0.010
Dispersant Name	Dry dispersion
Dispersant Refractive Index	1.000
Scattering Model	Mie
Analysis Model	General Purpose
Weighted Residual	0.57 %
Laser Obscuration	4.02 %

Result	
Concentration	0.1231 %
Span	0.686
Uniformity	0.211
Specific Surface Area	25.89 m ² /kg
D [3,2]	232 μm
D [4,3]	247 μm
Dv (10)	171 μm
Dv (50)	239 μm
Dv (90)	335 μm

Undersize



Result													
Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In
0.0100	0.00	0.0463	0.00	0.214	0.00	0.991	0.00	4.58	0.00	21.2	0.00	98.1	0.02
0.0114	0.00	0.0526	0.00	0.243	0.00	1.13	0.00	5.21	0.00	24.1	0.00	111	0.29
0.0129	0.00	0.0597	0.00	0.276	0.00	1.28	0.00	5.92	0.00	27.4	0.00	127	1.58
0.0147	0.00	0.0679	0.00	0.314	0.00	1.45	0.00	6.72	0.00	31.1	0.00	144	4.72
0.0167	0.00	0.0771	0.00	0.357	0.00	1.65	0.00	7.64	0.00	35.3	0.00	163	9.73
0.0189	0.00	0.0876	0.00	0.405	0.00	1.88	0.00	8.68	0.00	40.1	0.00	186	15.19
0.0215	0.00	0.0995	0.00	0.460	0.00	2.13	0.00	9.86	0.00	45.6	0.00	211	18.77
0.0244	0.00	0.113	0.00	0.523	0.00	2.42	0.00	11.2	0.00	51.8	0.00	240	18.71
0.0278	0.00	0.128	0.00	0.594	0.00	2.75	0.00	12.7	0.00	58.9	0.00	272	15.04
0.0315	0.00	0.146	0.00	0.675	0.00	3.12	0.00	14.5	0.00	66.9	0.00	310	9.56
0.0358	0.00	0.166	0.00	0.767	0.00	3.55	0.00	16.4	0.00	76.0	0.00	352	4.59
0.0407	0.00	0.188	0.00	0.872	0.00	4.03	0.00	18.7	0.00	86.4	0.00	400	1.51
												454	0.27
												516	0.01
												586	0.00
												666	0.00
												756	0.00
												859	0.00
												976	0.00
												1110	0.00
												1260	0.00
												1430	0.00
												1630	0.00
												1850	0.00
												2100	0.0
												2390	0.0
												2710	0.0
												3080	0.0
												3500	0.0



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240223 - Christine sand recovered

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Instrument Serial No: MAL1282973

21CFR Mode: Inactive

Record Number: 41

Created: 3/17/2023 11:07 AM

Printed: 3/23/2023 2:14 PM

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Analysis

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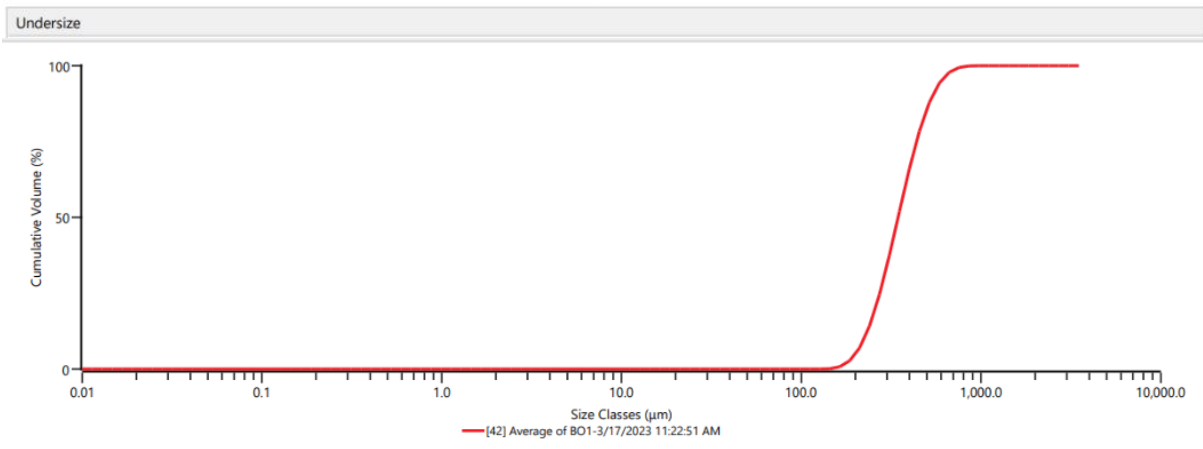


Measurement Details	
Operator Name	caroline
Sample Name	Average of BO1
SOP File Name	240223 - Christine sand.msop

Measurement Details	
Analysis Date Time	3/17/2023 11:22:51 AM
Measurement Date Time	3/17/2023 11:22:51 AM
Result Source	Averaged

Analysis	
Particle Name	Quartz
Particle Refractive Index	1.543
Particle Absorption Index	0.010
Dispersant Name	Dry dispersion
Dispersant Refractive Index	1.000
Scattering Model	Mie
Analysis Model	General Purpose
Weighted Residual	0.80 %
Laser Obscuration	3.44 %

Result	
Concentration	0.1475 %
Span	0.913
Uniformity	0.282
Specific Surface Area	18.32 m ² /kg
D [3,2]	328 μm
D [4,3]	366 μm
Dv (10)	223 μm
Dv (50)	346 μm
Dv (90)	538 μm



Result															
Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In
0.0100	0.00	0.0463	0.00	0.214	0.00	0.991	0.00	4.58	0.00	21.2	0.00	98.1	0.00	454	9.52
0.0114	0.00	0.0526	0.00	0.243	0.00	1.13	0.00	5.21	0.00	24.1	0.00	111	0.00	516	6.38
0.0129	0.00	0.0597	0.00	0.276	0.00	1.28	0.00	5.92	0.00	27.4	0.00	127	0.10	586	3.59
0.0147	0.00	0.0679	0.00	0.314	0.00	1.45	0.00	6.72	0.00	31.1	0.00	144	0.64	666	1.59
0.0167	0.00	0.0771	0.00	0.357	0.00	1.65	0.00	7.64	0.00	35.3	0.00	163	1.98	756	0.49
0.0189	0.00	0.0876	0.00	0.405	0.00	1.88	0.00	8.68	0.00	40.1	0.00	186	4.25	859	0.07
0.0215	0.00	0.0995	0.00	0.460	0.00	2.13	0.00	9.86	0.00	45.6	0.00	211	7.25	976	0.00
0.0244	0.00	0.113	0.00	0.523	0.00	2.42	0.00	11.2	0.00	51.8	0.00	240	10.41	1110	0.00
0.0278	0.00	0.128	0.00	0.594	0.00	2.75	0.00	12.7	0.00	58.9	0.00	272	12.99	1260	0.00
0.0315	0.00	0.146	0.00	0.675	0.00	3.12	0.00	14.5	0.00	66.9	0.00	310	14.34	1430	0.00
0.0358	0.00	0.166	0.00	0.767	0.00	3.55	0.00	16.4	0.00	76.0	0.00	352	14.09	1630	0.00
0.0407	0.00	0.188	0.00	0.872	0.00	4.03	0.00	18.7	0.00	86.4	0.00	400	12.32	1850	0.00



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Instrument Serial No: MAL1282973

21CFR Mode: Inactive

Record Number: 42

Created: 3/17/2023 11:22 AM

Printed: 3/23/2023 2:17 PM

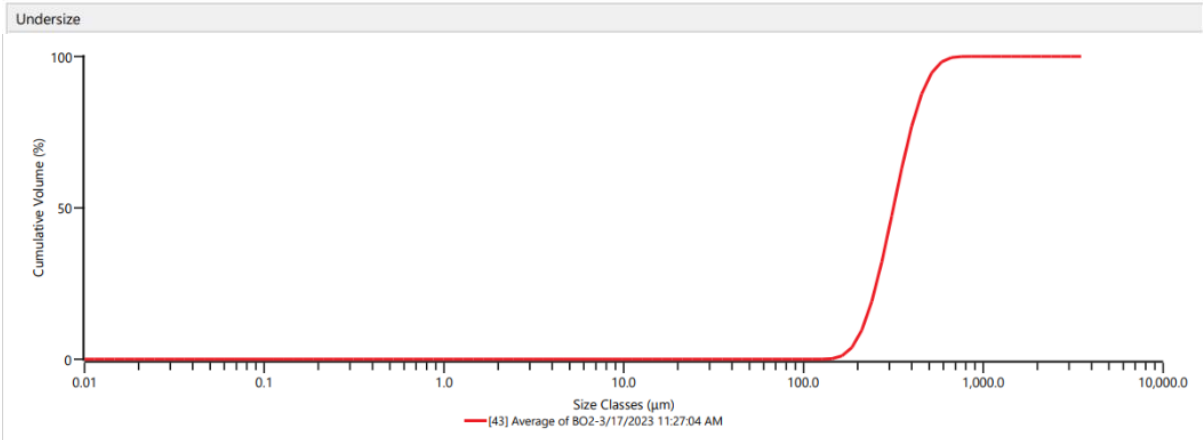
C:\ProgramData\Malvern Instruments\Mastersizer 3000\Workspace\Measurement Data\Christine bachelor 23\240223 - Christine sand recovered.mmes

Analysis

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Measurement Details Operator Name caroline Sample Name Average of BO2 SOP File Name 240223 - Christine sand.msop	Measurement Details Analysis Date Time 3/17/2023 11:27:04 AM Measurement Date Time 3/17/2023 11:27:04 AM Result Source Averaged
Analysis Particle Name Quartz Particle Refractive Index 1.543 Particle Absorption Index 0.010 Dispersant Name Dry dispersion Dispersant Refractive Index 1.000 Scattering Model Mie Analysis Model General Purpose Weighted Residual 0.76 % Laser Obscuration 3.49 %	Result Concentration 0.1383 % Span 0.829 Uniformity 0.256 Specific Surface Area 19.87 m ² /kg D [3,2] 302 µm D [4,3] 331 µm Dv (10) 212 µm Dv (50) 316 µm Dv (90) 474 µm



Result															
Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In
0.0100	0.00	0.0463	0.00	0.214	0.00	0.991	0.00	4.58	0.00	21.2	0.00	98.1	0.00	454	6.93
0.0114	0.00	0.0526	0.00	0.243	0.00	1.13	0.00	5.21	0.00	24.1	0.00	111	0.00	516	3.65
0.0129	0.00	0.0597	0.00	0.276	0.00	1.28	0.00	5.92	0.00	27.4	0.00	127	0.15	586	1.43
0.0147	0.00	0.0679	0.00	0.314	0.00	1.45	0.00	6.72	0.00	31.1	0.00	144	0.93	666	0.34
0.0167	0.00	0.0771	0.00	0.357	0.00	1.65	0.00	7.64	0.00	35.3	0.00	163	2.77	756	0.03
0.0189	0.00	0.0876	0.00	0.405	0.00	1.88	0.00	8.68	0.00	40.1	0.00	186	5.76	859	0.00
0.0215	0.00	0.0995	0.00	0.460	0.00	2.13	0.00	9.86	0.00	45.6	0.00	211	9.47	976	0.00
0.0244	0.00	0.113	0.00	0.523	0.00	2.42	0.00	11.2	0.00	51.8	0.00	240	13.00	1110	0.00
0.0278	0.00	0.128	0.00	0.594	0.00	2.75	0.00	12.7	0.00	58.9	0.00	272	15.30	1260	0.00
0.0315	0.00	0.146	0.00	0.675	0.00	3.12	0.00	14.5	0.00	66.9	0.00	310	15.64	1430	0.00
0.0358	0.00	0.166	0.00	0.767	0.00	3.55	0.00	16.4	0.00	76.0	0.00	352	13.92	1630	0.00
0.0407	0.00	0.188	0.00	0.872	0.00	4.03	0.00	18.7	0.00	86.4	0.00	400	10.69	1850	0.00



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240223 - Christine sand recovered

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Instrument Serial No: MAL1282973

21CFR Mode: Inactive
Record Number: 43

Created: 3/17/2023 11:27 AM
Printed: 3/23/2023 2:21 PM

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Analysis

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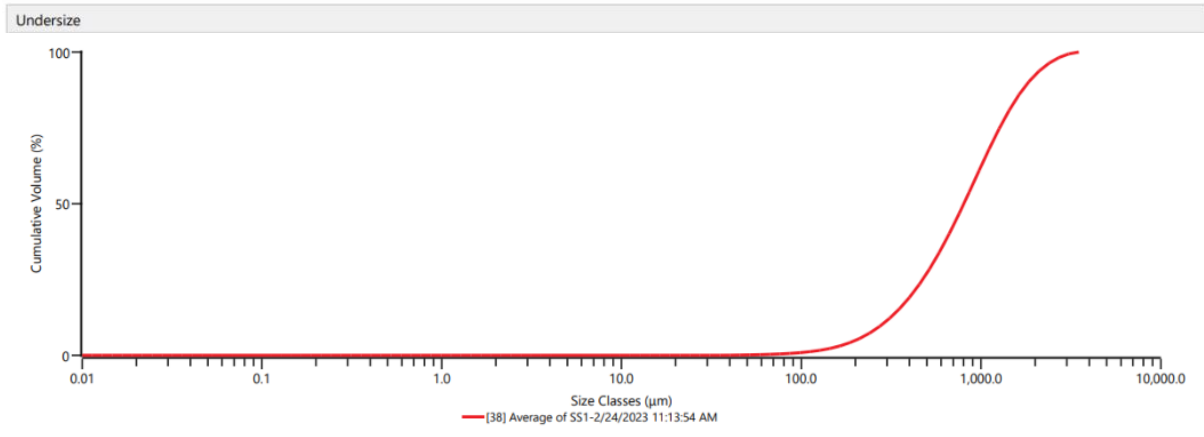


Measurement Details	
Operator Name	caroline
Sample Name	Average of SS1
SOP File Name	240223 - Christine sand.msop

Measurement Details	
Analysis Date Time	2/24/2023 11:13:54 AM
Measurement Date Time	2/24/2023 11:13:54 AM
Result Source	Averaged

Analysis	
Particle Name	Quartz
Particle Refractive Index	1.543
Particle Absorption Index	0.010
Dispersant Name	Dry dispersion
Dispersant Refractive Index	1.000
Scattering Model	Mie
Analysis Model	General Purpose
Weighted Residual	2.30 %
Laser Obscuration	3.34 %

Result	
Concentration	0.2385 %
Span	1.938
Uniformity	0.596
Specific Surface Area	10.86 m ² /kg
D [3,2]	552 μm
D [4,3]	948 μm
Dv (10)	279 μm
Dv (50)	802 μm
Dv (90)	1830 μm



Result															
Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In
0.0100	0.00	0.0463	0.00	0.214	0.00	0.991	0.00	4.58	0.00	21.2	0.00	98.1	0.33	454	4.95
0.0114	0.00	0.0526	0.00	0.243	0.00	1.13	0.00	5.21	0.00	24.1	0.00	111	0.45	516	5.58
0.0129	0.00	0.0597	0.00	0.276	0.00	1.28	0.00	5.92	0.00	27.4	0.00	127	0.61	586	6.17
0.0147	0.00	0.0679	0.00	0.314	0.00	1.45	0.00	6.72	0.00	31.1	0.00	144	0.82	666	6.67
0.0167	0.00	0.0771	0.00	0.357	0.00	1.65	0.00	7.64	0.00	35.3	0.02	163	1.08	756	7.03
0.0189	0.00	0.0876	0.00	0.405	0.00	1.88	0.00	8.68	0.00	40.1	0.05	186	1.40	859	7.17
0.0215	0.00	0.0995	0.00	0.460	0.00	2.13	0.00	9.86	0.00	45.6	0.06	211	1.77	976	7.05
0.0244	0.00	0.113	0.00	0.523	0.00	2.42	0.00	11.2	0.00	51.8	0.08	240	2.19	1110	6.67
0.0278	0.00	0.128	0.00	0.594	0.00	2.75	0.00	12.7	0.00	58.9	0.10	272	2.66	1260	6.04
0.0315	0.00	0.146	0.00	0.675	0.00	3.12	0.00	14.5	0.00	66.9	0.14	310	3.18	1430	5.23
0.0358	0.00	0.166	0.00	0.767	0.00	3.55	0.00	16.4	0.00	76.0	0.18	352	3.74	1630	4.33
0.0407	0.00	0.188	0.00	0.872	0.00	4.03	0.00	18.7	0.00	86.4	0.23	400	4.33	1850	3.43



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Instrument Serial No: MAL1282973
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Record Number: 38
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Printed: 3/23/2023 2:00 PM

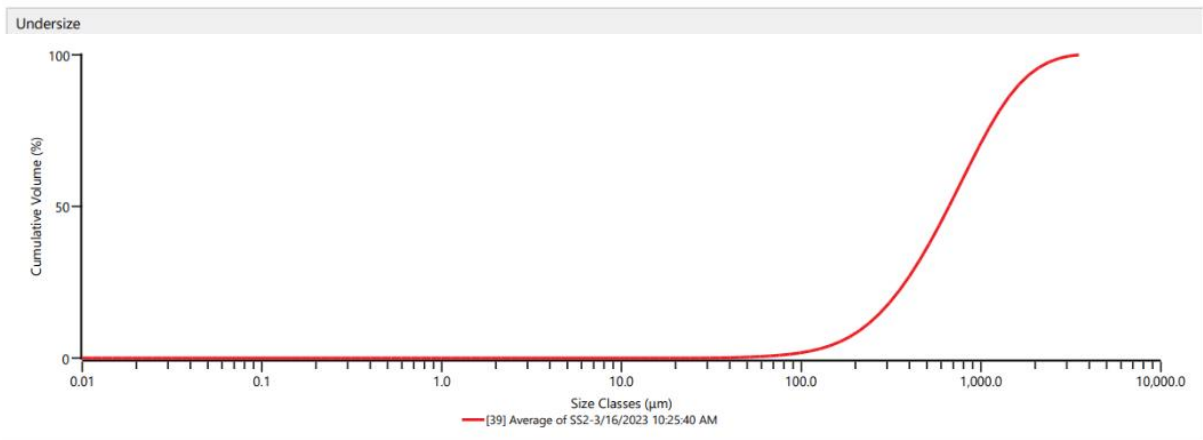
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Analysis

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Measurement Details Operator Name caroline Sample Name Average of SS2 SOP File Name 240223 - Christine sand.msop	Measurement Details Analysis Date Time 3/16/2023 10:25:40 AM Measurement Date Time 3/16/2023 10:25:40 AM Result Source Averaged
Analysis Particle Name Quartz Particle Refractive Index 1.543 Particle Absorption Index 0.010 Dispersant Name Dry dispersion Dispersant Refractive Index 1.000 Scattering Model Mie Analysis Model General Purpose Weighted Residual 1.70 % Laser Obscuration 3.50 %	Result Concentration 0.2059 % Span 2.099 Uniformity 0.655 Specific Surface Area 13.55 m ² /kg D [3,2] 443 μm D [4,3] 812 μm Dv (10) 221 μm Dv (50) 663 μm Dv (90) 1610 μm



Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In	Size (μm)	% Volume In
0.0100	0.00	0.0463	0.00	0.214	0.00	0.991	0.00	4.58	0.00	21.2	0.00	98.1	0.57	454	5.63
0.0114	0.00	0.0526	0.00	0.243	0.00	1.13	0.00	5.21	0.00	24.1	0.01	111	0.76	516	6.05
0.0129	0.00	0.0597	0.00	0.276	0.00	1.28	0.00	5.92	0.00	27.4	0.03	127	1.00	586	6.37
0.0147	0.00	0.0679	0.00	0.314	0.00	1.45	0.00	6.72	0.00	31.1	0.04	144	1.30	666	6.57
0.0167	0.00	0.0771	0.00	0.357	0.00	1.65	0.00	7.64	0.00	35.3	0.07	163	1.66	756	6.61
0.0189	0.00	0.0876	0.00	0.405	0.00	1.88	0.00	8.68	0.00	40.1	0.08	186	2.08	859	6.46
0.0215	0.00	0.0995	0.00	0.460	0.00	2.13	0.00	9.86	0.00	45.6	0.12	211	2.54	976	6.09
0.0244	0.00	0.113	0.00	0.523	0.00	2.42	0.00	11.2	0.00	51.8	0.15	240	3.05	1110	5.53
0.0278	0.00	0.128	0.00	0.594	0.00	2.75	0.00	12.7	0.00	58.9	0.20	272	3.57	1260	4.81
0.0315	0.00	0.146	0.00	0.675	0.00	3.12	0.00	14.5	0.00	66.9	0.25	310	4.11	1430	4.00
0.0358	0.00	0.166	0.00	0.767	0.00	3.55	0.00	16.4	0.00	76.0	0.33	352	4.64	1630	3.18
0.0407	0.00	0.188	0.00	0.872	0.00	4.03	0.00	18.7	0.00	86.4	0.43	400	5.15	1850	2.42



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240223 - Christine sand recovered

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Instrument Serial No: MAL1282973

21CFR Mode: Inactive
Record Number: 39

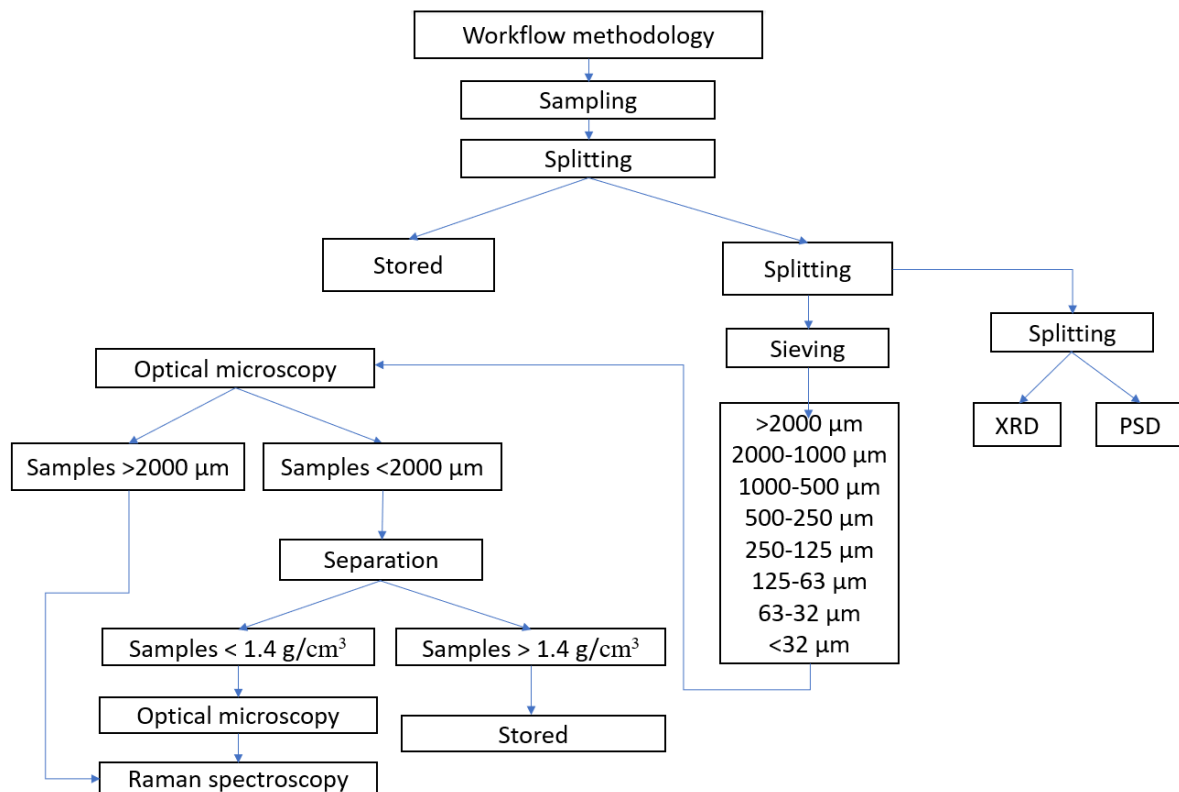
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Figure O Full PSD analysis showing the measurement details and results for each sample a) PSD analysis results for sample SO-1; b) PSD analysis results for sample SO-2; c) PSD analysis results for sample BO-1; d) PSD analysis results for sample BO-2; e) PSD analysis results for sample SS-1; f) PSD analysis results for sample SS-2.

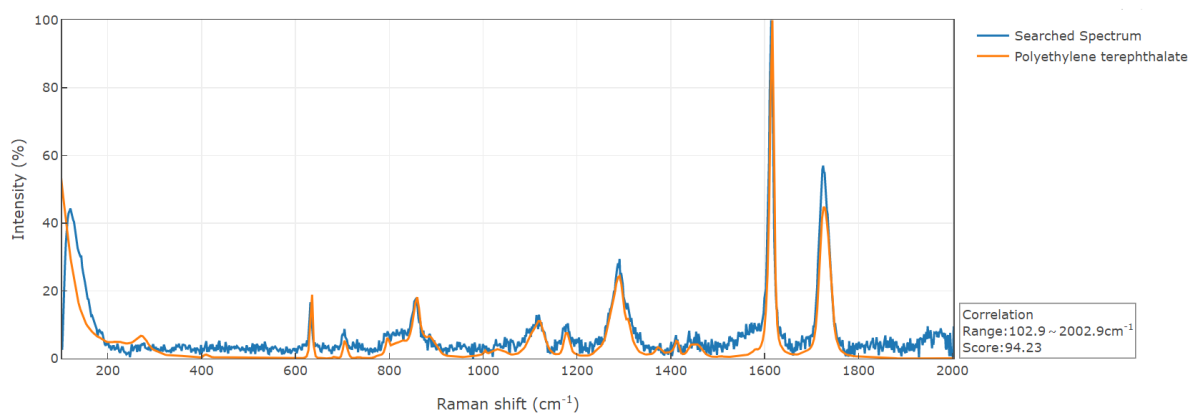
Appendix F

Workflow methodology:



Appendix G

Additional collected spectra:



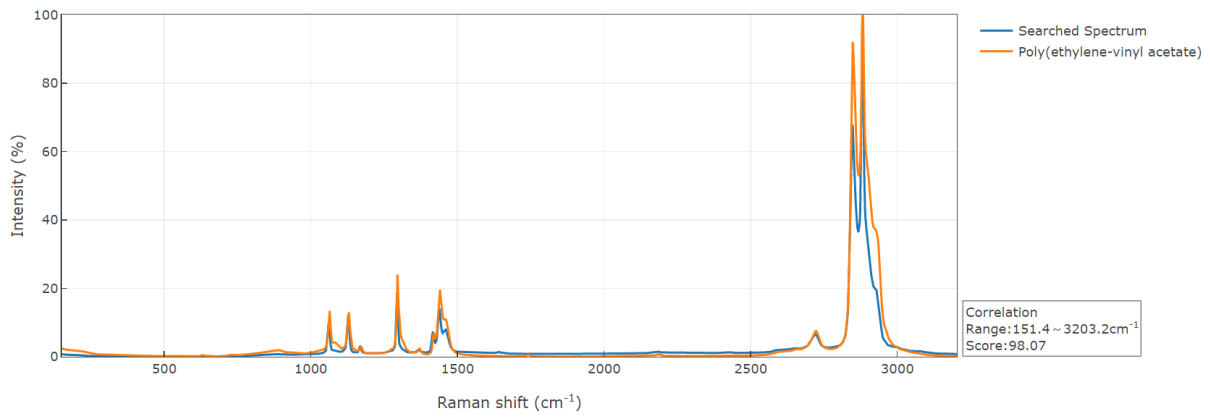
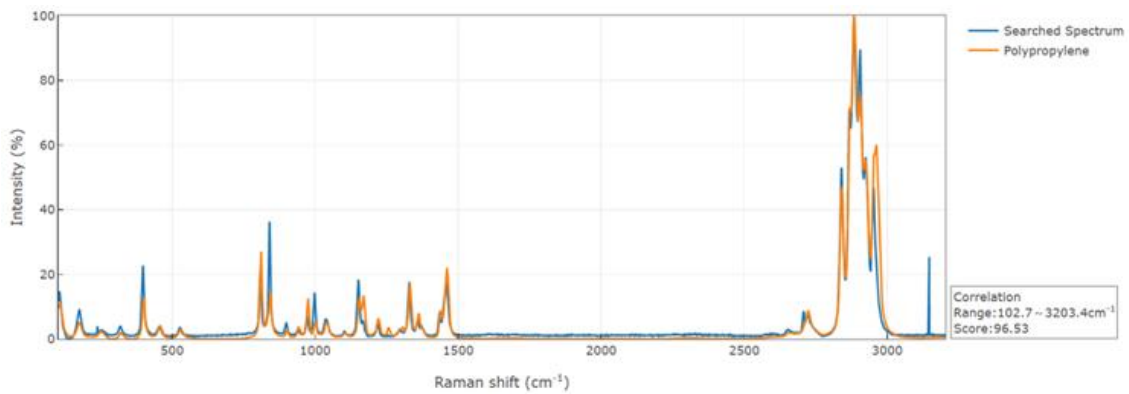
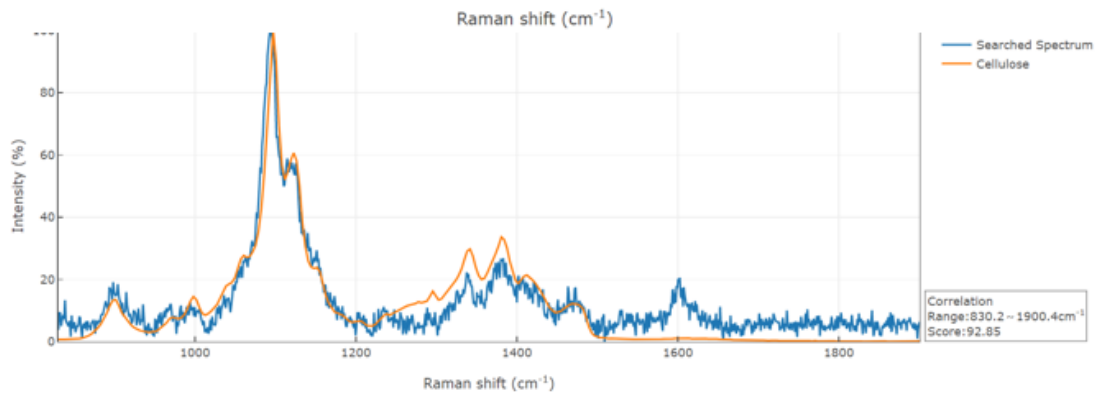
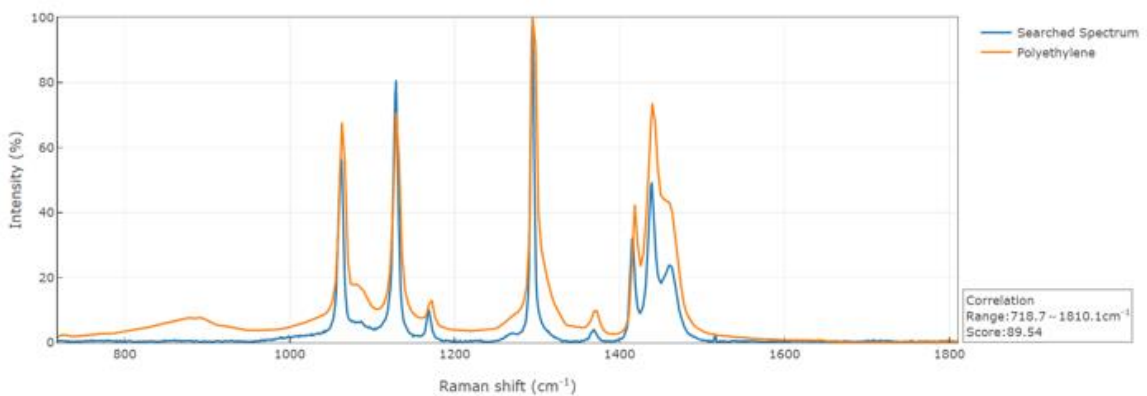
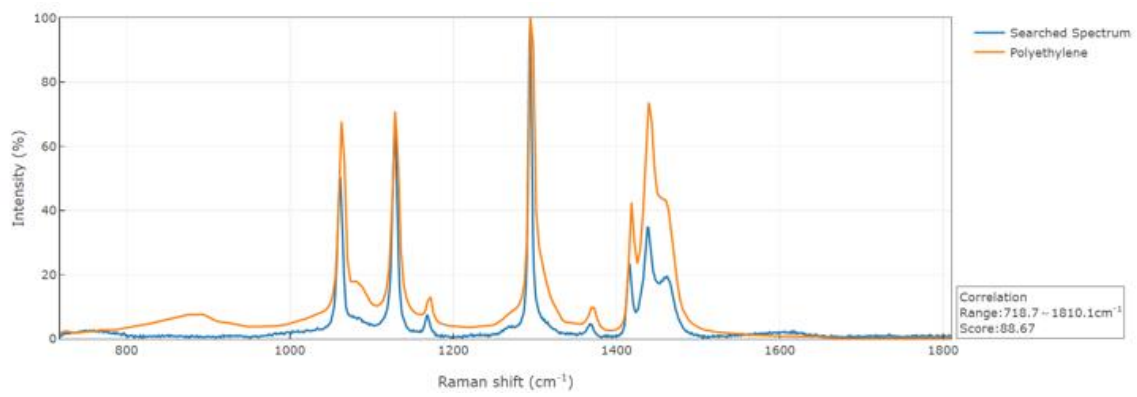
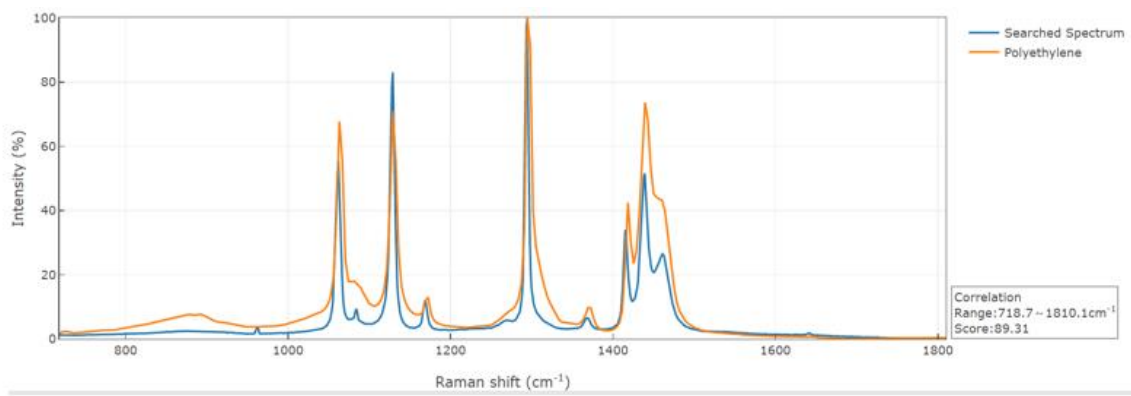
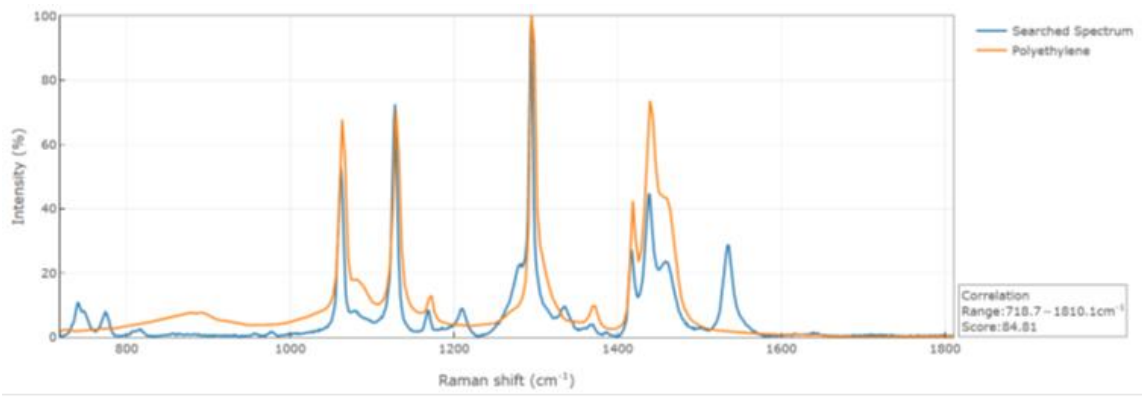
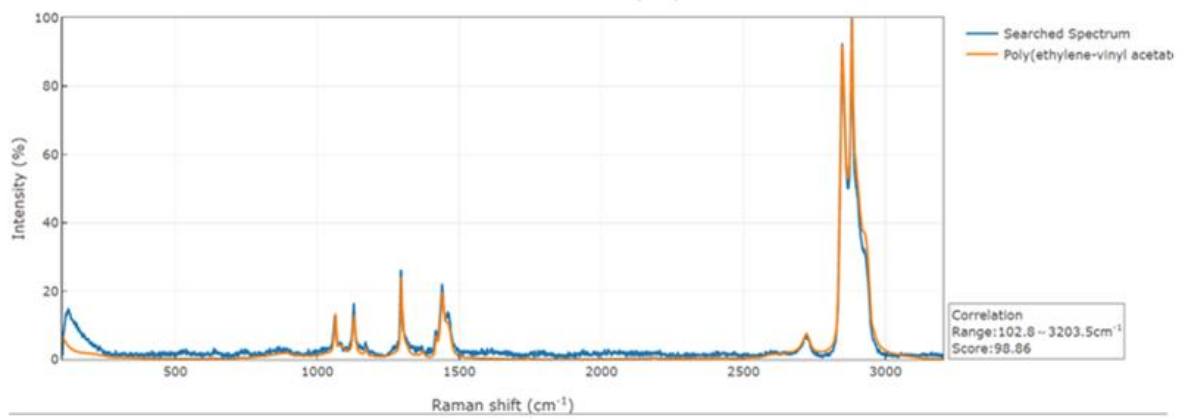
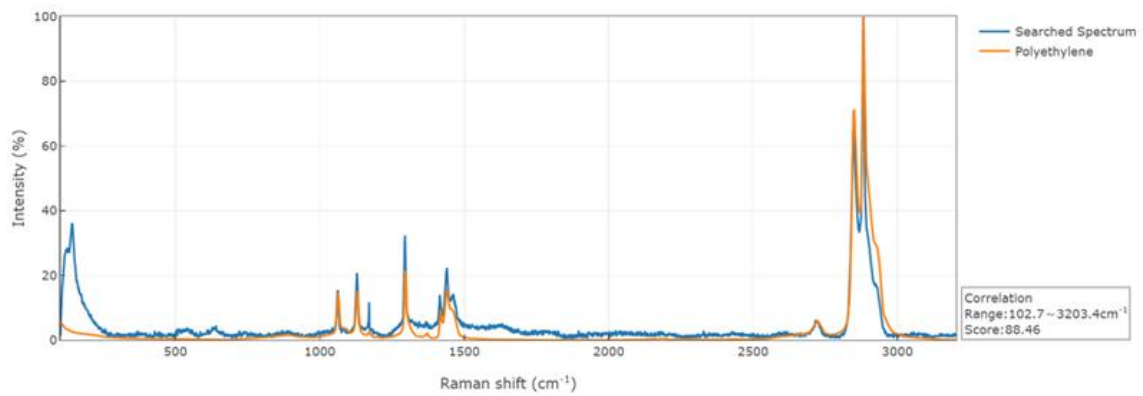
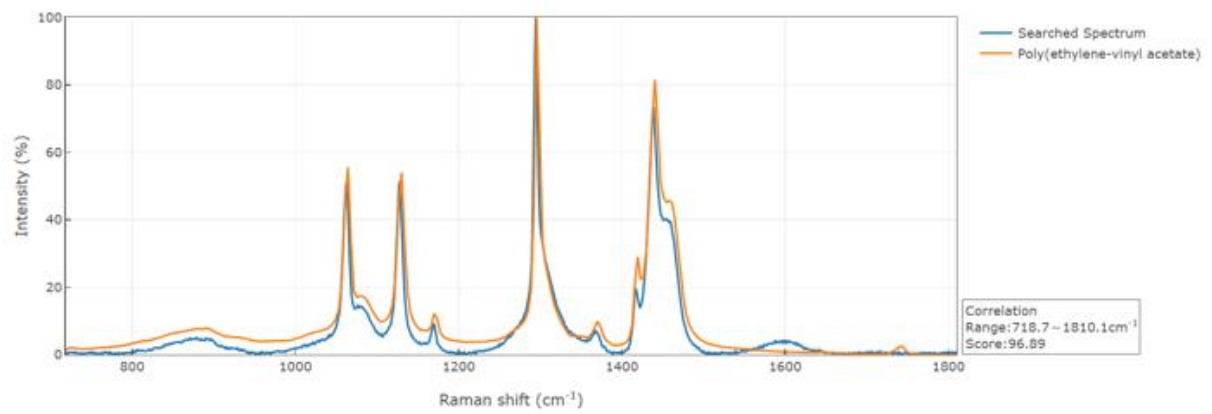
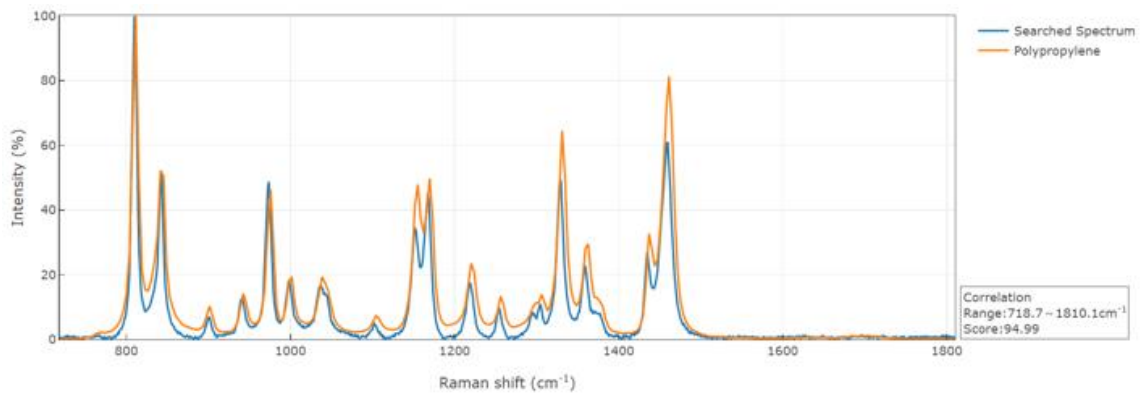


Figure M Additional obtained spectra from samples collected at home a) Grapes fruit container, Labeled R-PET with a 94.23 % match with polyethylene terephthalate; b) Caramel coloring container, Labeled HD-PE with a 98.07 % match Poly (ethylene-vinyl acetate).







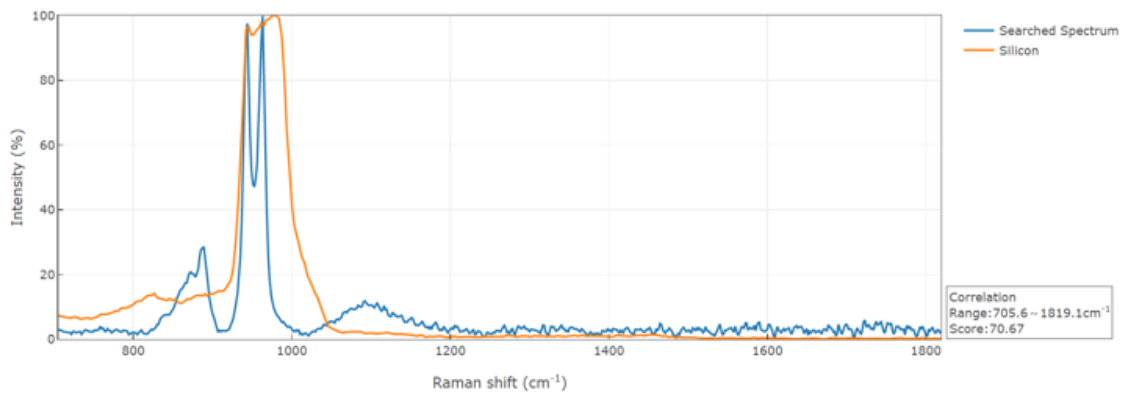
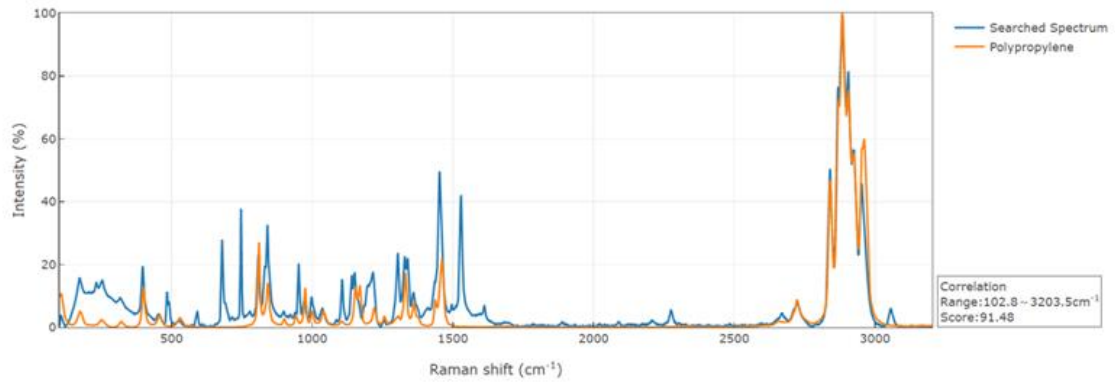


Figure N Additional obtained spectra from microplastics from collected samples a) SO-1_500-250_whitefiber_cellulose; b) SO-1_Leave_3_Polypropylene; c) SO_1_2_Polyethylene; d) SO_1_1_Polyethylene; e) SO_1_3_Polyethylene; f) SO-2_1_Polyethylene; g) SO-2_2_Polypropylene; h) BO-1_1_Poly (ethylene-vinyl acetate); i) BO-1_2_Polyethylene; j) BO-1_3_Poly (ethylene-vinyl acetate); k) BO-1_Leave_2_Polypropylene; l) SS-1_63-32_Silicon.