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Procedural Framework for Microplastic Recovery and Identification in Urban Catchment Deposits

IP2.02.01 Understanding Microplastics

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Procedural Framework for Microplastic Recovery and Identification in Urban Catchment Deposits

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The Sustainable Communities and Waste Hub acknowledges all Aboriginal and Torres Strait Islander Traditional Custodians of Country and recognises their continuing connection to land, sea, culture, and community. We pay our respects to Elders past, present, and emerging. We support Aboriginal and Torres Strait Islander peoples and their aspirations to maintain, protect and manage their culture, language, land and sea Country and heritage.

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Executive Summary

Microplastics, defined as plastic fragments less than 1 mm in size, are a significant and rising global environmental concern. This report presents a systematic approach to microplastic identification and characterisation in urban stormwater runoff through the implementation of sequential separation and size fractionation methodologies. The study examines samples collected from commercial, industrial, and residential catchments to identify predominant polymer types across different particle size distributions and different urban ecosystems.

A collaborative initiative with Ocean Protect employs their established OceanGuard technology, designed to capture pollution that runs into stormwater drains from strategically selected commercial, industrial, and residential discharge points across the study area. The partnership focuses on Ocean Protect's proven field deployment capabilities and sampling expertise, ensuring consistent sample collection protocols across various urban catchment types.

The specific objectives of this work are to develop a systematic methodological hierarchy to separate microplastics from urban runoff catchments, prepare a structured approach to identify the separated microplastics, and provide information on microplastic pollution in different zones across diverse ecosystems in Australia. The analytical framework employed enables the isolation of microplastic particles from complex environmental matrices whilst maintaining sample integrity for subsequent chemical characterisation and morphological analysis. The findings provide valuable insights into urban microplastic pollution, offering critical data to support the development of targeted mitigation strategies and inform stormwater management practices.

This methodology provides a reliable, adaptable workflow for isolating and identifying microplastics from catchment-area environmental samples. It integrates practical techniques (visual separation, flotation, digestion, centrifugation) with robust spectroscopic tools to allow confident polymer classification. While the method is not focused on complete recovery, it is well-suited for identification-based studies and can support broader research into microplastic degradation, transport, and environmental impact.

Contents

1. Introduction

Microplastics, defined as plastic fragments less than 1 mm in size, represent a significant and rising global environmental concern (AS ISO 2025). According to Standards Australia / International Organization for Standardisation AS ISO 24187:2025, microplastics are defined as solid plastic particles insoluble in water with dimensions between 1 µm and 1 mm, while large microplastics refer to solid plastic particles with dimensions between 1 mm and 5 mm. These particles may exhibit regular or irregular morphologies and can originate either as primary microplastics intentionally manufactured for use in products such as cosmetics, coatings, and paints, or as secondary microplastics generated through fragmentation and degradation of larger plastic materials and end-use products. These abundant particles are accumulating in vast quantities across all natural environments, urging extensive research into their diverse origins, widespread distribution, environmental persistence, and cumulative detrimental effects. Among the primary mechanisms of transferring microplastics to aquatic environments, including atmospheric deposition and wastewater effluent discharge, urban runoff from different sources like carparks, loading docks, hardstands, roadways, etc., is increasingly recognised as the most significant conduit. This process, involving the overland flow of water from rainfall, snowmelt, or stormwater in urban areas, acts as a major driver in mobilising and transporting substantial quantities of land-based microplastics and other pollutants into aquatic systems. As a diffuse source of pollution, urban runoff carries a complex mixture of contaminants, from visible debris like trash and sediment to unseen pollutants such as toxic chemicals and microplastics.

Urban stormwater runoff represents a significant pathway for microplastic contamination in aquatic environments, with commercial, industrial, and residential catchments contributing varying quantities and types of plastic particles to receiving water bodies. The increase of synthetic materials in urban infrastructure, consumer products, and industrial processes has resulted in the widespread distribution of microplastics ranging from nanometres to millimetre scales throughout urban drainage systems. The characterisation of microplastics in stormwater presents considerable analytical challenges due to the heterogeneous nature of urban runoff matrices, which contain complex mixtures of organic matter, sediments, and diverse contaminants that can interfere with particle identification and quantification. Traditional sampling and analysis methods often lack the specificity required to accurately differentiate microplastic particles from natural organic matter and other anthropogenic debris present in stormwater samples.

This report presents a systematic approach to microplastic identification and characterisation in urban stormwater runoff through the implementation of sequential separation and size fractionation methodologies. The study examines samples collected from commercial, industrial, and residential catchments to establish baseline contamination levels and identify predominant polymer types and particle size

distributions across different urban land uses. The analytical framework employed enables the isolation of microplastic particles from complex environmental matrices whilst maintaining sample integrity for subsequent chemical characterisation and morphological analysis.

The findings contribute valuable insights to the growing body of research on urban microplastic pollution. It can provide critical data to support the development of targeted mitigation strategies and inform stormwater management practices. A deeper understanding of the sources, distribution, and characteristics of microplastics in urban runoff is essential for shaping effective policy and engineering solutions aimed at reducing plastic pollution in Australian waterways.

2. Microplastics in the environment

Microplastics are pervasive pollutants in both natural environments and urban settings, originating from the breakdown of larger plastics and direct emissions from sources like household products, road traffic, and industrial activities. In natural ecosystems, including rivers, lakes, wetlands, and oceans, microplastics accumulate due to their persistence and resistance to degradation, posing significant risks to aquatic organisms through ingestion and entanglement, and ultimately entering the food web and human diets. Urbanisation intensifies microplastic pollution, with stormwater runoff identified as a major pathway for transporting microplastics from rigid surfaces, roads, and atmospheric deposition into waterways. During rain events, stormwater can discharge millions to billions of microplastic particles per outfall, often at concentrations exceeding those from treated wastewater, making urban runoff a dominant contributor to microplastic loads in receiving waters. These particles are found in various forms, such as fibres, fragments, and beads, across a range of sizes, with higher concentrations typically observed during rain events due to the wash-off effect and resuspension within stormwater systems. Effective mitigation requires a combination of preventive measures, decentralised treatment options such as stormwater retention ponds and street sweeping, and regulatory controls targeting key sources like laundry effluent, road runoff, and plastic pre-production facilities. Without comprehensive management, microplastics will continue to accumulate in both aquatic and terrestrial environments, threatening ecosystem health and food safety.

In urban environments, microplastics are predominantly secondary microplastics, which are primarily generated from the breakdown of larger pieces of plastics due to mechanical abrasion, photo-oxidation, and other weathering processes. Additional contributors include tire wear, urban dust, landfills, wastewater treatment discharges, construction materials, road markings, paints, plastic piping, industrial operations, and textile fibres released during domestic washing. According to estimates by Kole et al., approximately 5.9 million tons of tire-derived particles are emitted globally each year, with roughly 90% eventually entering stormwater systems (Kole, Löhr et al. 2017).

Fine microplastic particles can accumulate on urban surfaces and are readily mobilised by rainfall, entering stormwater drains and ultimately reaching aquatic systems. Apart from smaller particles, larger plastic debris may also be transported by runoff, either settling on urban surfaces or being discharged into water bodies. Once exposed to environmental conditions such as UV radiation, heat, and oxidative stress, these larger items progressively fragment into microplastics, further contributing to their widespread distribution and persistence in the environment.

3. Extraction of microplastics from the environment: Techniques and methods

Microplastic research typically employs a two-phase approach: initial extraction to separate and purify microplastic fragments from diverse environmental matrices (water, soil, air, and biota), followed by a separation process that categorises these extracted plastics by shape, size, and colour. For soil, sand, and sediments, microplastic extraction often begins with physical pretreatment, such as sieving to eliminate larger particles (typically >5 mm). Subsequent separation techniques frequently applied to sediments include visual separation, flotation, density separation, digestion to remove organic matter, and further sieving for size fractionation.

The accurate identification and characterisation of microplastics in environmental samples rely heavily on effective initial isolation and separation techniques. Three primary methods- sieving, visual sorting, and filtration- are widely employed to prepare samples for further analysis. Each technique contributes uniquely to the separation process, particularly when dealing with complex urban runoff matrices.

4. Need for systematic microplastic assessment in runoffs and waterways

The development of robust analytical methods for assessing microplastics in urban runoff and waterways has become increasingly important, as awareness grows around plastic pollution as a long-term environmental concern. Existing gaps in our understanding of how microplastics are distributed, transported, and behave in aquatic environments continue to hinder the effectiveness of current pollution control measures and regulatory policies.

Microplastics demonstrate distinct environmental behaviour compared to conventional pollutants, featuring high persistence, extensive distribution, and complex interactions with aquatic ecosystems. Unlike biodegradable contaminants, they accumulate continuously in sediments and water columns, creating long-term contamination that demands urgent quantification. These particles act as vectors for chemical pollutants like persistent organic compounds and heavy metals, potentially amplifying toxicity for aquatic organisms through heightened bioavailability.

Understanding their dynamics is crucial for predicting ecological consequences, including impacts on primary productivity, food web disruption, and human health risks via seafood, while assessing particle size, polymer composition, and contamination loads helps identify critical exposure pathways.

The current absence of standardised monitoring protocols and regulatory limits in Australia creates significant environmental protection gaps, necessitating robust analytical methodologies to inform evidence-based water quality guidelines and discharge regulations. Systematic source identification within urban catchments enables targeted intervention strategies, allowing prioritisation of management resources and cost-effective pollution prevention. Establishing standardised assessment protocols advances global scientific understanding while addressing knowledge gaps specific to Australian urban environments and climatic conditions.

5. Aims of the project

The analysis of microplastics in urban runoff is fundamental to understanding the scale and nature of plastic pollution entering Australian waterways through stormwater infrastructure. Microplastics pose significant ecological risks through bioaccumulation in aquatic food webs, potential transfer of adsorbed contaminants, and physical impacts on marine organisms. Furthermore, the persistence of these particles in the environment necessitates immediate action to measure current contamination levels and identify primary source categories. The development of a robust analytical roadmap is essential given the complexity of microplastic identification in environmental matrices. This project was designed based on the insights gained from our previous work, documented in the report “Methodology for Identifying Locations to Investigate Sources and Characteristics of Microplastics” (Way and Kamateros 2025). This report outlined a systematic methodology for selecting microplastic sampling sites, focusing on identifying key environmental sources and accumulation points. The initial collaboration with Ocean Protect during this phase laid the foundation for the current study by enabling access to strategically placed OceanGuard devices across diverse land use types. The system features a range of filtration bag liners designed to capture gross pollutants, total suspended solids, and associated contaminants. It can function either as a stand-alone treatment solution or be integrated into a broader treatment train (OceanProtect 2024).

Building on this foundation, the present collaborative initiative with Ocean Protect employs their established OceanGuard technology, designed to capture pollution that runs into stormwater drains. It can be installed within new and existing stormwater pits from strategically selected commercial, industrial, and residential discharge points across the study area. The partnership focuses on Ocean Protect's proven field deployment capabilities and sampling expertise, while ensuring consistent sample collection protocols across various urban catchment types. The project anticipates generating comprehensive baseline data on microplastic contamination

levels and compositional profiles within urban stormwater systems, establishing critical standards for ongoing monitoring programmes. These findings are expected to inform evidence-based stormwater management policies and support the development of targeted intervention strategies tailored to specific land use categories and their associated contamination modes.

In short, the specific objectives of this work are to:

- Develop a systematic methodological hierarchy to separate the microplastics from the urban runoff catchments that ensures reproducible results and supports regulatory compliance requirements.
- Prepare a structured approach to identify the separated microplastics, providing a foundation for future research initiatives.
- Provide information on microplastic pollution in different zones of Australia and serve as a framework for broader microplastic monitoring programmes across Australian urban catchments.

6. Schematic diagram of the methodology

Figure 1 presents the schematic workflow for isolating, separating, and identifying microplastics from urban catchment samples. The methodology combines physical separation, chemical digestion, and spectroscopic identification to recover microplastics from complex matrices such as soil, sediment, and organic debris.

This study followed the Australian Standard AS ISO 24187:2025, which outlines internationally harmonised principles for the analysis of microplastics in environmental matrices. The standard provides clear guidance on critical steps such as sample preparation (drying, contamination control), size fractionation, and polymer identification using spectroscopic techniques like FTIR. It ensures methodological consistency through defined particle size classifications, plastic-free equipment use, and data processing protocols.

By aligning with AS ISO 24187:2025, this report supports the comparability, traceability, and reproducibility of results across studies and jurisdictions. The standard's emphasis on quality assurance, representative sampling, and reliable detection enhances the scientific robustness of microplastic research and promotes its applicability in regulatory frameworks. This compliance strengthens confidence in the methodology and ensures the data can contribute meaningfully to national and international assessments of microplastic pollution.

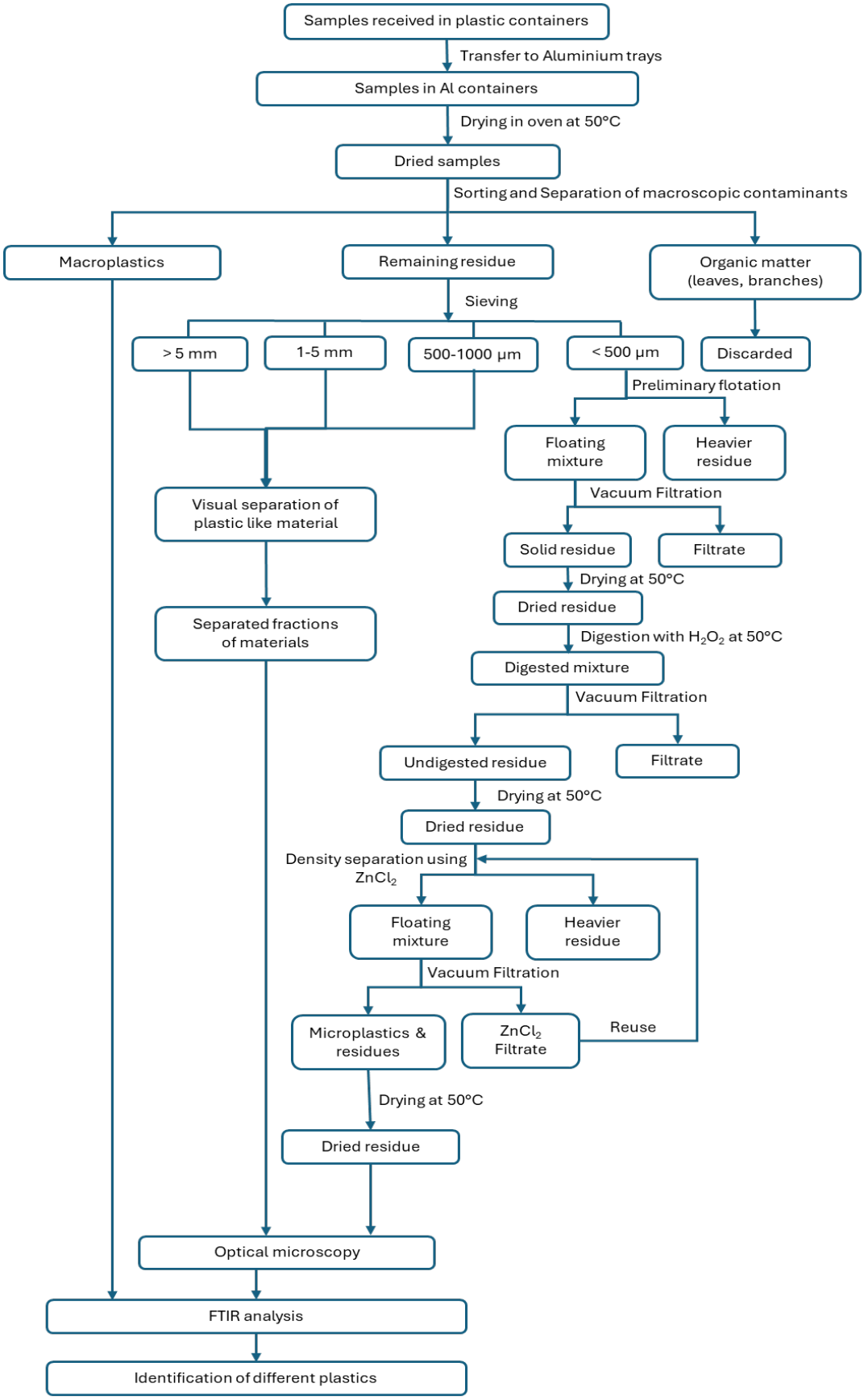

Figure 1: Schematic diagram of the experimental procedure for microplastic separation and identification

7. Experimental methods

7.1 Sample collection and pre-processing

The OceanGuards are located over different catchment zones. Inside the OceanGuard, a mesh bag of 200 µm pore size is placed. Any debris from the nearby areas gets accumulated inside this mesh bag. Every 3 to 4 months, these bags are carefully retrieved, and the collected material is emptied and transferred into clean plastic containers for transport to the laboratory for immediate processing. In some instances where excessive accumulation occurred, such as at heavily polluted sites, only a representative portion of the material could be collected. Figure 2 (a, b) represents the OceanGuard location with the mesh bag inside, and Figure 2 (c, d) shows the collected materials in the plastic containers with proper labelling.

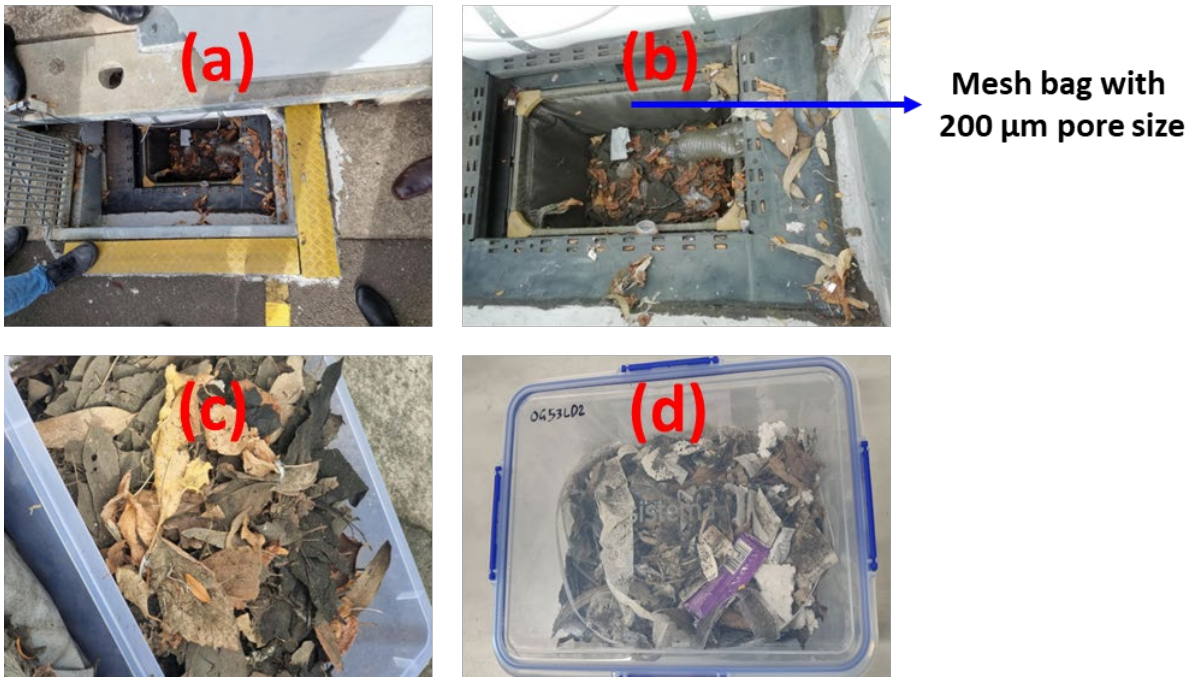

Figure 2: **(a, b)** Samples deposited in the mesh bag inside the OceanGuard, **(c, d)** Samples stored in plastic containers for easy transportation

Upon arrival at the laboratory, samples were promptly transferred to aluminium trays and appropriately labelled to ensure accurate tracking throughout subsequent processing steps. Initial drying was performed as a mandatory measure to minimise the risk of microbial contamination during handling. Drying was conducted in an oven maintained at 50°C, as higher temperatures could potentially degrade the plastic components within the samples. The drying process continued until a constant sample weight was achieved, indicating the complete removal of moisture, which is essential for precise quantification. Throughout this stage, comprehensive documentation was maintained, including the recording of initial weights and photographic documentation of the dried material. This foundational step ensures that all subsequent analyses are conducted on well-characterised and stable samples. **Figure 3** illustrates the drying

process, with images of samples in aluminium trays within the dedicated oven (a), as well as the resulting dried samples (b, c).

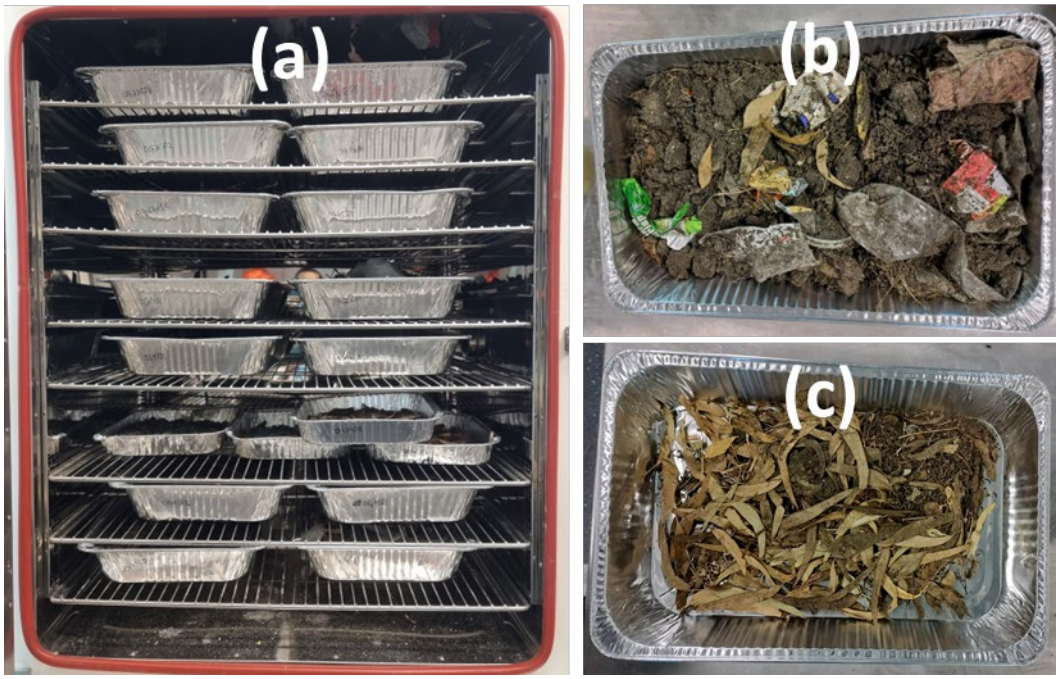

Figure 3. (a) Samples getting dried in the oven, **(b, c)** Dried samples

7.2 Preliminary separation and weighing

After drying, all the samples are weighed without any further processing and noted. Later, the samples undergo a thorough visual inspection to identify and manually separate any large plastic debris and organic matter, such as leaves or branches. This manual sorting process removes macroscopic contaminants, leaving a residue that contains the smaller particles of interest for further analysis. Figure 4 (a) indicates the dried samples before processing, (b, c) shows the sorting process, and (d) shows the separated macro plastics from the collected samples. These sorted macro plastics are collected and stored with proper labelling.

Figure 4. (a) Dried samples weighed and ready for processing, **(b-d)** preliminary sorting to separate large macro plastics, leaves and other organic materials

7.3 Size-based fractionation

The remaining residue is then subjected to sequential sieving, which divides the material into defined size fractions:

- >5 mm
- 1-5 mm
- 0.5-1 mm
- 0-0.5 mm

This size-based separation is essential for understanding the distribution of microplastics and for tailoring subsequent analytical techniques to each size fraction. **Figure 5** shows the sieved samples based on the mentioned size categories.

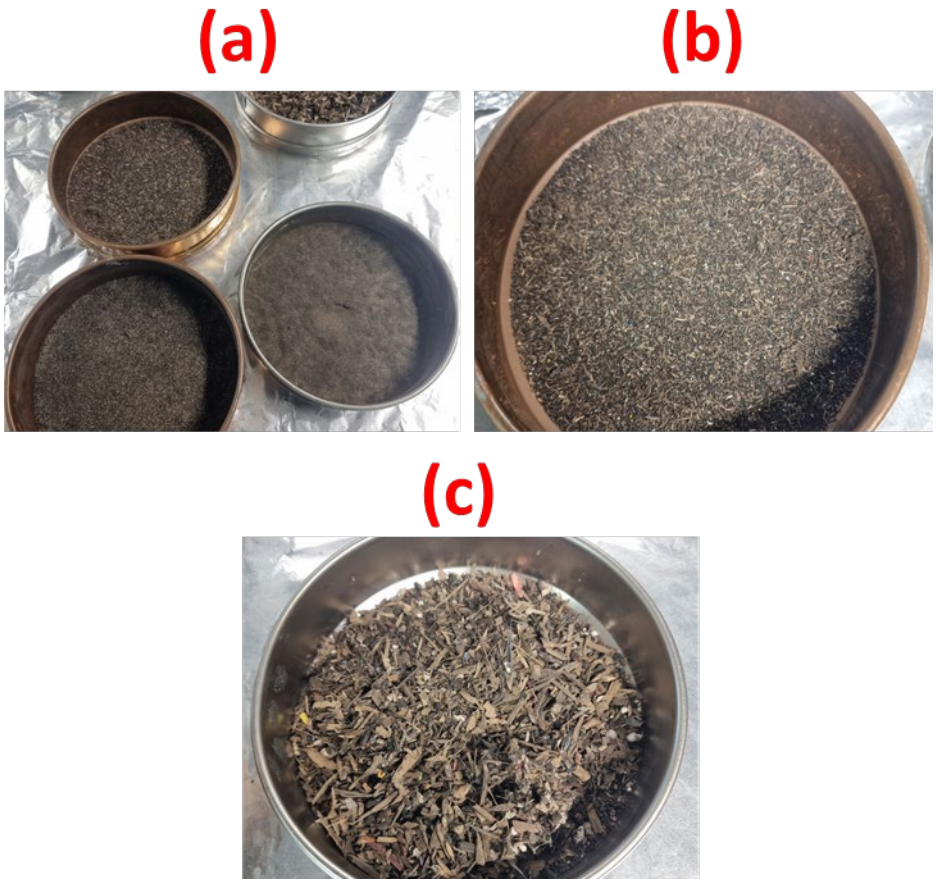

Figure 5. Sorting the samples into different size categories by sieving

7.4 Recording weight measurements

Each size fraction is weighed and photographed to document its characteristics and then stored in clearly labelled containers. This thorough documentation not only supports traceability and quality assurance but also provides a visual record that facilitates comparative analysis across sampling sites and periods. All fractions were stored in clearly labelled containers under contamination-controlled conditions to preserve sample integrity for subsequent chemical digestion and spectroscopic analysis.

7.5. Initial microplastic isolation

7.5.1 Visual separation for fractions >0.5 mm

Following size fractionation, visual sorting was conducted for all fractions exceeding 0.5 mm under natural lighting conditions. Suspected microplastic particles were identified based on morphological criteria, including colour, shape (such as fibre, fragment, or film), texture, and surface irregularities. Key indicators for identification included unnatural colouration, uniform thickness, and the absence of cellular structure. Spectroscopic confirmation was not performed at this stage. Suspected

particles were carefully collected using metal tweezers and placed into petri dishes, which were subsequently covered and stored in a clean environment to prevent contamination prior to further analysis. **Figure 6 (a, b)** depicts the visual sorting process and the collection of suspected microplastics.

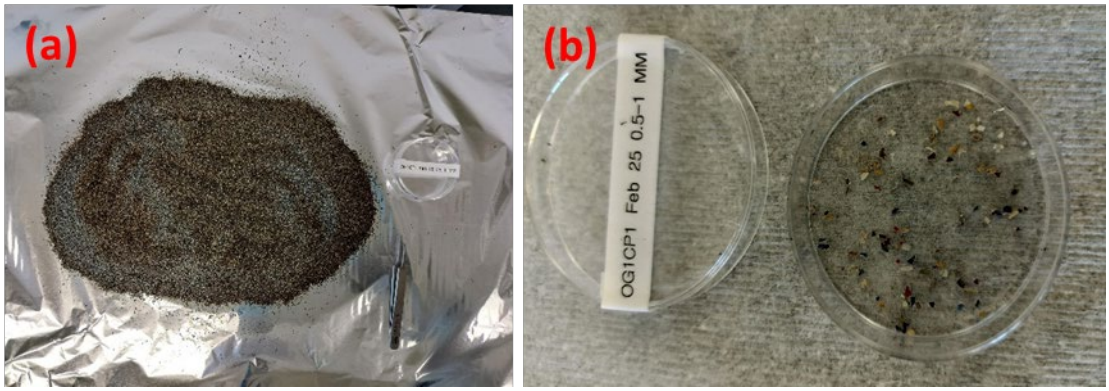

Figure 6. (a) Visual separation with tweezers, **(b)** Collected suspected microplastics

7.5.2 Sample handling and storage post-visual sorting

Sorted materials were handled exclusively with non-plastic instruments, such as metal tweezers, to minimise the risk of contamination. Samples were stored in covered petri dishes at room temperature within a desiccator to maintain sample integrity. All separated particles were placed in clean, covered petri dishes, each clearly labelled with the corresponding sample ID, size category, and collection date. **Figure 7** illustrates the labelling and storage procedures employed for the collected microplastics.

Figure 7. Labelling and storage procedures for isolated microplastics

7.6 Preliminary flotation for fractions <0.5mm

Due to the presence of high quantities of sand, silt, and fine organic debris in the <0.5 mm fraction, a preliminary flotation step using deionised (DI) water was introduced before digestion. Samples were mixed with DI water in a 1:10 ratio and then centrifuged at 5000 rpm for 5 min. **Figure 8 (a, b)** presents the suspended particles in DI water before centrifuging and the floated particles after centrifuging, respectively. The lighter, floating particles were collected by vacuum filtration using a 0.22 µm membrane filter, then dried at 50°C to ensure all moisture is removed. The dried samples were weighed and then used for subsequent chemical digestion.

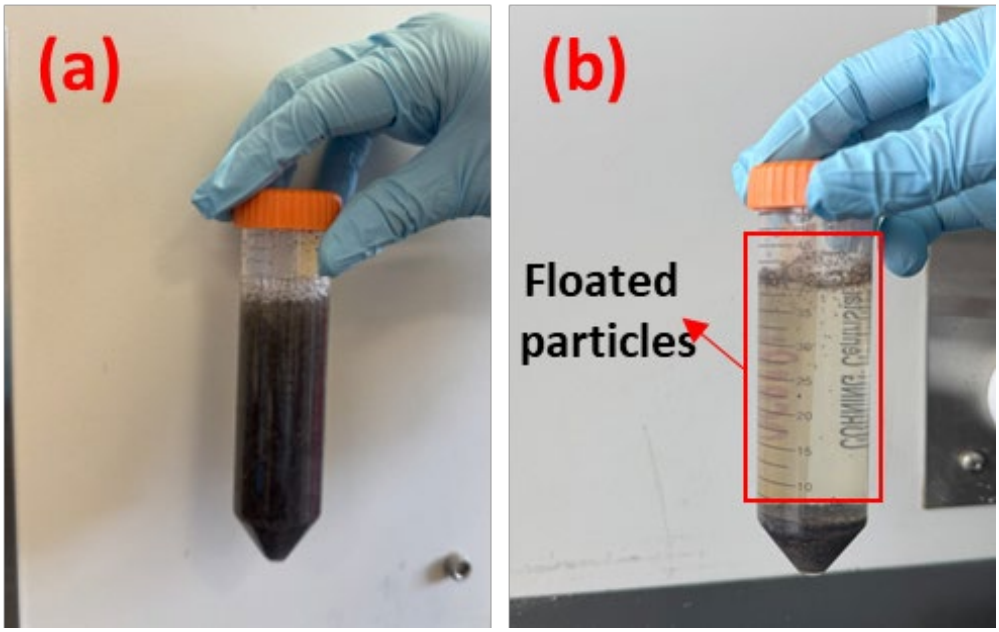

Figure 8. (a) Before preliminary flotation, **(b)** After flotation and centrifuging

DI water was chosen due to its simplicity, cost-effectiveness, and ability to recover low-density polymers like PE and PP. Literature supports the use of water for initial flotation in organic-rich samples. (Hidalgo-Ruz, Gutow et al. 2012). Denser solutions like NaCl, $ZnCl_2$ were not used at this stage to avoid interference with later digestion. **Table 1** indicates the common microplastics found in the environment and their respective densities.

Table 1: Density range of plastics

No.	Plastic	Density (g/cm^3)
1.	Expanded polystyrene	0.01-0.05
2.	Polypropylene (homopolymer)	0.90–0.91
3.	Low-density polyethylene	0.92–0.94
4.	High-density polyethylene	0.94–0.97
5.	Acrylonitrile butadiene styrene	1.00–1.10
6.	Nylon 6,6/ Polyamide	1.05–1.10
7.	Poly(methyl methacrylate)	1.10–1.20
8.	Polyethylene terephthalate	1.40–1.50
9.	Polyvinyl chloride	1.35–1.50

7.7. Chemical digestion of organic matter

Samples from catchment zones contained substantial organic matter that obscured microplastic visibility and interfered with FTIR analysis. For this purpose, a chemical digestion step is necessary to remove organic components while preserving plastic integrity. For chemical digestion, researchers have used different chemical solutions with their pros and cons. Table 2 includes some common digestion solutions and their advantages and limitations. It seems that considering the sample type, location, and other parameters, hydrogen peroxide (H_2O_2) is better suitable for this study. The digestion process is carried out under controlled temperature, with regular monitoring to ensure complete breakdown of organics without damaging microplastics.

For the digestion process, dried samples were immersed in 30% H_2O_2 and heated at 50°C to facilitate oxidation of organic matter. The presence of bubbling was indicative of ongoing organic material breakdown. Digestion was continued until bubbling subsided, and the solution became visibly clearer, signifying the completion of organic matter degradation. No catalysts were introduced during this procedure. Following digestion, samples were vacuum filtered using a 0.22 µm filter membrane, thoroughly rinsed with deionised water and ethanol, and subsequently dried at 50°C.

Table 2: Comparison of Digestion Solutions for Microplastic Separation

Reagents	Advantages	Drawbacks	Reference
Hydrogen peroxide (H_2O_2)	Mild oxidation; effective for organic matter destruction without severe polymer degradation	Slower digestion might leave residual organic debris in complex matrices	(Khan, Ghias et al. 2022)
Nitric acid (HNO_3)	Highly efficient for calcareous material (e.g., shells) and biological tissue elimination	Degrades polyamide (15-100% weight loss) and alters PET; not suitable for acid-sensitive polymers	(Pfeiffer and Fischer 2020)
Fenton's reagent (H_2O_2 +$FeSO_4$)	Rapid oxidation at low temperatures; effective for preserved tissues (e.g., fish intestines)	Obscures polymer identification via Raman spectroscopy; degrades PVC (29.5% Raman quality loss)	(Pfeiffer and Fischer 2020)
Sodium hypochlorite (NaOCl)	Optimal for soft/hard tissue destruction; minimal polymer damage at low concentrations	Ineffective for calcareous material; requires temperature control (40-70°C) for full efficacy	(Pfeiffer and Fischer 2020)
Enzymatic digestion	Polymer-friendly; preserves particle integrity for sensitive identification	Expensive; time-consuming; may obscure polymer signatures in spectroscopic analysis	(Miller, Motti et al. 2021)

Figure 9 (a, b, c) depicts the chemical digestion process, the solution post-digestion, and the floated particles following filtration, respectively.

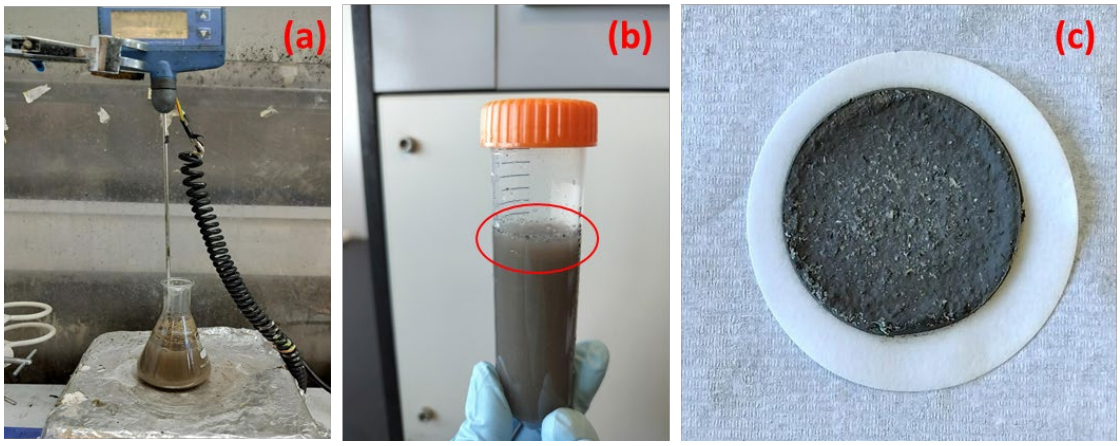

Figure 9: (a) Chemical digestion process, **(b)** after chemical digestion, and **(c)** after filtration

7.8 Density separation

Since chemical digestion with H_2O_2 leaves organic residue, it is necessary to use density separation using a high-density solution. Table 3 shows the density of different solutions suitable for different polymers with their advantages and disadvantages.

Table 3: Common salt solutions for density separation of microplastics

Salt Solution	Chemical Formula	Density (g/cm^3)	Suitability	Remarks
Sodium Chloride	NaCl	~1.20	PE, PP, EPS	Inexpensive, non-toxic, easy disposal, not suitable for high-density plastics
Zinc Chloride	$ZnCl_2$	~1.70	PE, PP, PET, PVC, PS, PA	High recovery efficiency, corrosive and hazardous, must be handled carefully
Sodium Iodide	NaI	~1.83	Almost all common polymers	Very effective but expensive, reusable with care, sensitive to moisture
Potassium Formate	HCOOK	~1.60	PE, PP, PET, PS, PA	Low toxicity alternative, expensive, hygroscopic
Calcium Chloride	$CaCl_2$	~1.40	PE, PP	Low cost, may precipitate in some conditions, limited effectiveness
Sodium Bromide	NaBr	~1.50	PE, PP, PS	Less common, moderate cost

In this method, zinc chloride ($ZnCl_2$) was employed for density separation. A $ZnCl_2$ solution with a density of 1.6 g/cm³ was prepared using deionised water, adhering to the appropriate mass-to-volume ratio. The dried residue obtained following chemical digestion was introduced into a centrifuge tube at a 1:10 ratio with the $ZnCl_2$ solution and centrifuged at 5000 rpm for 5 minutes. The microplastics that floated to the surface were subsequently recovered by vacuum filtration using a 0.22 µm membrane, rinsed with deionised water, and dried at 50°C. **Figure 10 (a–c)** illustrates the digested solution prior to and following density separation, as well as after vacuum filtration. The selected density of 1.6 g/cm³ facilitated the recovery of both low- and high-density plastics. The $ZnCl_2$ solution was reused up to five times, filtered between uses, and regularly monitored to ensure density stability.

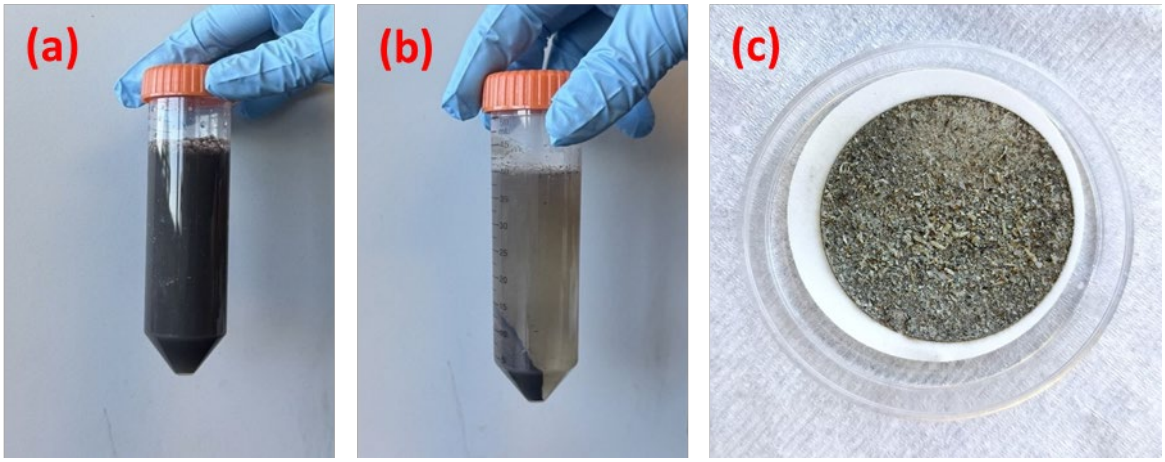

Figure 10: (a) Before $ZnCl_2$ separation, **(b)** After ZnCl2 separation, and (c) After filtration

7.9 Characterisation of isolated microplastics

7.9.1 Optical microscopy

All particles were initially assessed using an optical microscope (Nikon 600). Characteristics such as shape, colour, texture, and uniformity were recorded. This step was essential for screening before spectroscopic analysis. **Figure 11** shows the images received from the optical microscope.

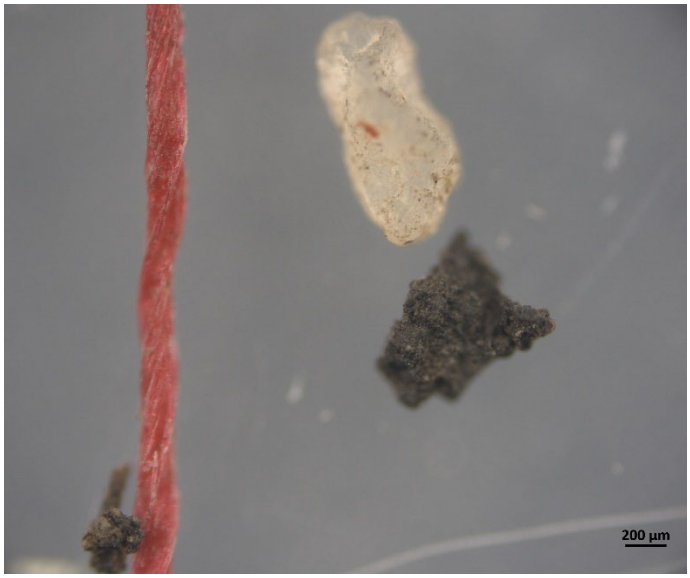

Figure 11: Microplastic particles observed in optical microscopy (0.5-1mm size category)

7.9.2 FTIR Spectroscopy

To confirm the polymer composition of suspected microplastics, the particles were analysed using a Fourier-transform infrared (FTIR) spectroscopy (Spectrum Two, PerkinElmer) with an ATR accessory. Spectra were acquired from 4000 to 450 cm^{-1} wavenumber, 4 cm^{-1} resolution, and an accumulation of 8. **Figure 12** shows the spectra received from the FTIR analysis. Spectra were archived with metadata including polymer match, sample ID, location, property type, and size.

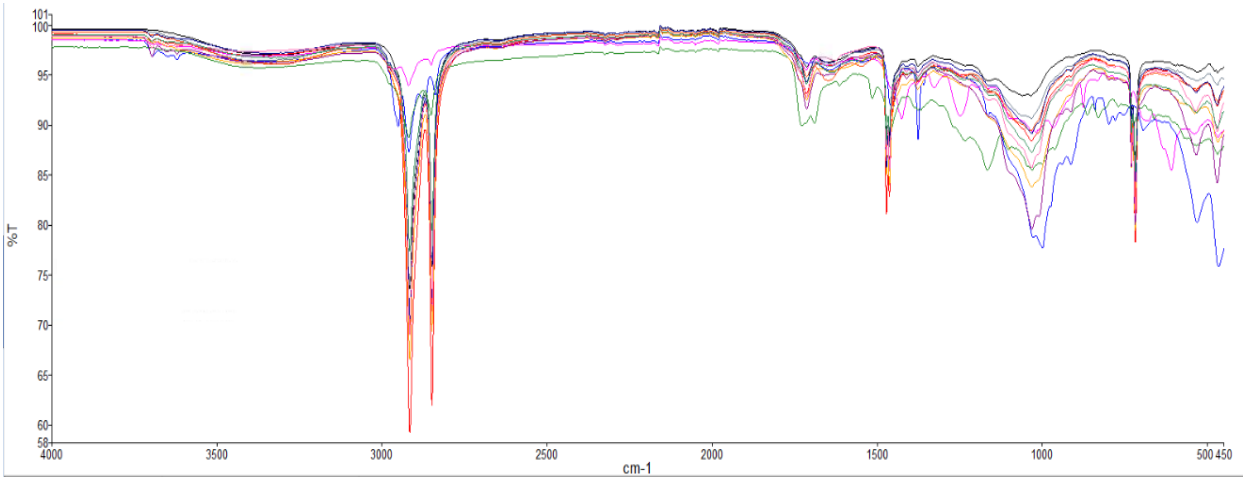

Figure 12: Polymer spectra analysed from FTIR

7.10 Identification of the microplastics

ATR-FTIR spectra were compared against the PerkinElmer reference library, with a 60% match threshold employed to tentatively identify polymer types. Particles exhibiting evidence of weathering or oxidation were either reanalysed or excluded from further analysis if identification remained inconclusive. All data were systematically recorded, including relevant metadata such as sample ID, collection location, date, and size category. **Figure 13** illustrates the process of matching sample spectra with the reference library.

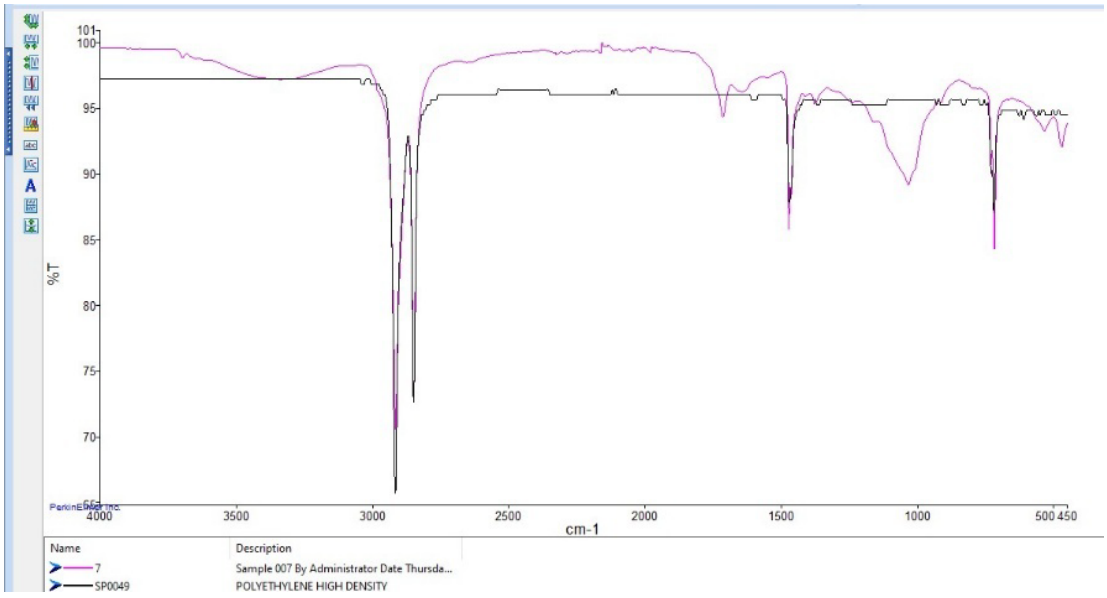

Figure 13: Spectra matching with the reference library to identify polymer type

8. Challenges and recommendations

The protocol successfully isolated and identified microplastics from complex matrices by integrating visual sorting, chemical digestion, density-based flotation, and FTIR spectroscopy, enabling reliable polymer identification while preserving microplastic integrity. However, several challenges were identified across both the field collection phase and the laboratory process.

From a collection standpoint, challenges stemmed from the nature and accessibility of field assets like OceanGuards. While not a major issue in this study, access to some sites, especially those on private property, is not always guaranteed for every sampling round. There is also a risk that property owners or site managers may have emptied the OceanGuards between visits, which could reduce sample consistency. This may have occurred at the commercial region, where notably less material was recovered during the collection. Moreover, in some cases, OceanGuards contained large volumes of water, and due to the limitations of the mesh bag design and transport containers, not all water was collected, potentially resulting in the loss

of suspended microplastics. Furthermore, standard service and cleaning of these assets, either by external contractors or site personnel, could lead to additional microplastic loss, particularly of fine particles not visible to the eye that may remain adhered to the mesh lining.

From the laboratory perspective, visual sorting proved to be the most labour-intensive and subjective step, potentially introducing operator-based variability. While preliminary flotation following sorting helped reduce sediment carryover, it was not entirely effective in removing all contaminants. Future studies are recommended to evaluate and improve recovery efficiency, ideally using spiked validation samples to better quantify losses. Investigating automated or semi-automated sorting approaches may also help enhance reproducibility and reduce processing time. To mitigate these issues, it is recommended that future studies coordinate closely with asset managers to avoid unplanned clean-outs between sampling intervals. Additionally, strategies should be developed to capture retained water from OceanGuards where feasible. Finally, microplastic retention within the mesh bag should be considered as a contributing factor in recovery efficiency estimates.

9. Conclusion

This methodology establishes a dependable, flexible workflow for isolating and identifying microplastics from environmental samples collected within catchment areas. The approach combines practical laboratory techniques, including visual separation, flotation, digestion, and centrifugation, with reliable spectroscopic analysis to enable confident polymer classification. Whilst the method prioritises identification over complete recovery, it proves well-suited for characterisation-based studies and effectively supports broader research programmes investigating microplastic degradation pathways, transport mechanisms, and environmental impacts across diverse catchment systems

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