



Microplastics in the Environment: Sources, Detection Techniques, and Analytical Challenges

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Abstract

The environmental concerns about microplastics are growing among scientists, policymakers, and the public. Plastic particles smaller than 5 mm are known as microplastics, and they come from a variety of primary sources, including personal care products and industrial abrasives. Additionally, they come from secondary sources like the degradation of larger plastics. A growing number of these particles have been found in soil, aquatic systems, and even atmospheric samples, highlighting the urgent need for further research into the origin, fate, and impacts of these particles. In this review, we provide an overview of the sources, types, and processes that lead to microplastics entering soil and water systems, focusing primarily on how wastewater, agricultural inputs, and industrial discharges contribute to their growth. Several sampling strategies are discussed across environmental matrices, highlighting the challenges associated with collecting samples that are representative and contamination-free. In the section on microplastics, a wide range of analytical techniques is discussed, including microscopy, spectroscopy, thermal analysis, chromatography, and mass spectrometry. Although significant progress has been made, several analytical challenges remain, including the lack of standardized methods, the identification of nanoplastics, and the limitations of quantification. Various emerging technologies, including machine learning, biosensors, and advanced imaging tools, are also explored for their potential to enhance current approaches. With this review, we aim to synthesize recent advancements and persistent challenges to support the development of more robust, sensitive, and harmonized approaches to microplastic analysis, ultimately leading

to improvements in environmental monitoring and risk assessment.

Subject Areas

Environmental Chemistry

Keywords

Analytical Techniques, Micro- and Nano-Plastics, Sampling Methods, Detection, Challenges

1. Introduction

As plastic pollution has become a pressing environmental issue in recent decades, microplastics are one of its most challenging aspects. A microplastic is a fragment of plastic less than 5 millimeters in size, which is produced either by the degradation of larger plastic debris or by industrial and consumer products. Environmental concerns over their impact on human health and the environment have been raised in marine, freshwater, terrestrial, and even atmospheric environments [1] [2]. A primary microplastic, which is used in cosmetics or industrial abrasives, is deliberately made small, whereas a secondary microplastic is produced when larger plastic items are exposed to sunlight, wind, and water currents that mechanically and chemically degrade [3]. Since microplastics are small and persistent, they are difficult to remove from the environment where they can contaminate water and soil. They are also capable of adsorbing and transporting hazardous chemicals [4]. While awareness is growing, microplastics have not been sampled or analyzed universally. Techniques have varied widely across studies, affecting their comparability and reliability. Nanoplastic identification is often difficult using traditional methods, such as optical microscopy. There are new methods that offer greater precision, but are also more expensive and technically demanding, including FTIR and Raman spectroscopy, thermal spectrometry, and mass spectrometry. For effective environmental policies and the advancement of the field, robust, standardized methods are crucial [5]. In this review, we provide a comprehensive overview of microplastic sources, sampling methods, and current analytical techniques. In addition, it examines emerging solutions that promise to improve the accuracy and consistency of microplastic detection across studies, as well as the challenges associated with microplastic analysis.

2. Sources and Types of Microplastics

Microplastics are broadly categorized into primary and secondary microplastics based on their origin.

2.1. Sources of Primary Microplastics

Primary microplastics come from a variety of sources and are a major cause of

environmental contamination. Microbeads are frequently found in toothpaste, face washes, and cosmetics, making personal care items one of the main sources. Synthetic textiles also play a substantial role, with fibers shed from clothing and fabric products accounting for approximately 35% of primary microplastic pollution. Vehicle tires are another important source, accounting for around 28% of all primary microplastics. These tires emit microplastic particles as a result of wear and tear while driving. The environmental impact of microplastics is increased in industrial settings by the frequent release of plastic pellets employed in production processes. Other sources include marine coatings, where boat paints and protective coatings contribute to plastic pollution in aquatic habitats, and road markings, where microplastic particles are released when road paint deteriorates. Microplastic pieces from artificial turf, building coatings, and urban infrastructure are also present in city dust. Agricultural products also contribute, as plastic-based coatings on seeds, fertilizers, and pesticides introduce microplastics into soil and water systems. There is growing concern about microplastic pollution, particularly with glitter and other purpose-made pieces of plastic. To reduce this pollution, we need to understand its source [6] [7].

2.2. Sources of Secondary Microplastics

Secondary microplastics are a common type of plastic pollution that comes from the breakdown of bigger plastic waste in the environment. Unlike primary microplastics, which are made small on purpose for use in products, secondary microplastics are broken pieces of larger plastic items. When exposed to environmental factors, these plastics undergo a breakdown process that reduces them to smaller, often microscopic, pieces. The fragmentation occurs through various mechanisms, including UV radiation from sunlight, mechanical abrasion from wind, waves, and currents, chemical degradation, temperature fluctuations, and microbial action [8]. Major sources of secondary microplastics include terrestrial and marine waste, such as discarded fishing gear, shipping waste, and other plastic debris. Over time, these plastics lose their structural integrity, making them more susceptible to breaking into smaller fragments. As the plastics break down, their carbon structure may also undergo chemical changes, contributing to the release of CO₂ and further impacting the environment. Bio-fragmentation, assimilation by organisms, and biodeterioration also contribute to the formation of secondary microplastics [9].

As of 2024, global plastic waste generation has reached approximately 220 million tons, averaging 28 kilograms per person annually. Of this, about one-third, or 69.5 million tons, is mismanaged and ends up in the natural environment [10] [11]. Recent studies suggest the problem may be even worse, with research on the UK's fishing fleet estimating that between 326 million and 17 billion microplastic pieces enter the ocean annually from this source alone [12]. While much attention is given to marine plastic pollution, a significant portion of plastic waste remains in terrestrial environments, where its sources, degradation pathways, and long-

term impacts are still not fully understood. These microplastics have a significant negative impact on the environment, as they are found in ecosystems ranging from remote mountain summits to vast oceans. Due to their low density, microplastics can float on water surfaces and be carried by winds, leading to their presence in groundwater, stormwater runoff, and the atmosphere. They are not inert, they can form toxic biofilms and adsorb pollutants, making them harmful to wildlife and potentially humans. The widespread and persistent presence of secondary microplastics in the environment has made them a significant source of microplastic contamination worldwide. The continuous degradation of larger plastics complicates efforts to address this growing environmental issue [13].

2.3. Sources of Microplastics in Soil

Soil is crucial for storing carbon, purifying water, and growing food, but plastic pollution is putting it at risk. Microplastics have been found everywhere, from forests to farms, and can harm soil fertility, crop growth, and microbes by changing the soil's structure and chemistry. Human activities, such as agriculture, urbanization, and waste disposal, introduce microplastics into the soil, where they remain for a long time because they do not break down easily. Due to the increasing use of plastic products in farming methods, agricultural activities constitute a major source of microplastics in soil. The many types of plastic materials used in agriculture have the following effects on soil contamination [14]. In intensive farming, plastic mulch films are widely used to boost crop yields, control weeds, and retain moisture. However, over time, these films break down due to weather, wear, and UV exposure, turning into microplastics. Research shows that these films contribute 33% to 56% of the total microplastics found in agricultural soils [15]. For instance, given the large volumes of plastic used annually in Chinese agriculture, plastic film mulching has emerged as a critical technique. More than 18.14 million hectares were covered by 1.41 million tons of plastic film in 2014. As the films decompose over time into microplastics, the widespread usage of plastic mulch adds to soil contamination. An exact figure of 300,000 tons is not stated in the search results, but the extensive use of plastic mulch in China and its effects on the environment are widely known, underscoring the necessity of efficient management and recycling techniques to reduce microplastic contamination in agricultural soils [16] [17].

2.3.1. Synthetic Fertilizers and Pesticides

They are significant contributors to microplastic pollution in soils, primarily through the intentional inclusion of microplastics in their formulations. Some controlled-release fertilizers, for example, use microplastics to encapsulate nutrients, allowing them to be gradually released over time. This practice, often marketed as sustainable, leads to the gradual accumulation of microplastics in the soil. Similarly, some pesticides contain microplastics to control the release of active ingredients, which can persist in the environment and negatively affect soil health. Additionally, while less common, microplastics can also be present in the packaging

of synthetic fertilizers and pesticides, as well as in additives used in these products. Another key source is plastic-coated seeds, which are used to protect seeds from pests. As these coatings degrade, they release microplastics into the soil. These practices underscore the need for regulatory action to limit the intentional use of microplastics in agricultural products. Ongoing efforts to develop microplastic-free alternatives, such as biodegradable seed coatings, aim to mitigate this issue and reduce the long-term environmental impact [18] [19].

2.3.2. Urban and Industrial Activities

Improper waste disposal, including littering of plastic products, leads to plastics breaking down into microplastics through weathering and fragmentation. Over time, these smaller plastic fragments make their way into urban soils via storm-water runoff, illegal dumping, or wind-blown debris. The presence of plastic waste in parks, playgrounds, and green spaces increases the likelihood that microplastics will be incorporated into the soil through human or animal activities [20] [21]. Construction activities contribute significantly to microplastic pollution in soils due to the widespread use of plastic materials. Plastics like PVC pipes, packing materials, insulation, and protective films are commonly used in construction. When exposed to weathering and wear during building and demolition, these materials can break down into microplastics. In addition, construction and demolition generate large amounts of plastic waste. For example, Auckland, New Zealand, produces at least 25,000 tons of plastic waste each year from these activities. If not properly disposed of, this waste can contaminate the soil around construction sites with microplastics [22]. At various phases of construction, including demolition (mainly from packaging materials), exterior and weatherproofing (using shrink-wrap and packaging), and services and cladding (where PVC pipes and fittings are major contributors), microplastics are produced. Plastic waste and the production of microplastics can be decreased with the use of mitigation techniques, such as implementing circular economy principles that prioritize recycling and material reuse. By recovering and repurposing valuable construction materials, material recovery plants can also help minimize environmental consequences at demolition sites [23].

2.3.3. Microplastics in Wastewater and Sludge

Another significant source of microplastics in soil comes from the disposal of treated wastewater and sewage sludge, primarily due to the presence of microplastics in household products, personal care items, and synthetic fabrics. Microplastics from sources such synthetic fibers shed from clothes and microbeads in cosmetics are frequently found in wastewater treated by wastewater treatment facilities (WWTPs). Although WWTPs are capable of removing a significant amount of these microplastics, their effectiveness is not 100%. Despite the fact that secondary and tertiary WWTPs can remove an average of 88% and 94% of microplastics, respectively, millions of microplastic particles are nevertheless released into the environment every day because of the sheer volume of wastewater they

treat [24]. Sewage sludge is the accumulation of the microplastics that are still present in wastewater. Approximately 1% of the weight of sewage sludge is made up of microplastic particles, which can number up to 24 per gram [25]. A study conducted at a wastewater treatment plant in Newport, South Wales, reported up to 24 microplastic particles per gram of sewage sludge (Cardiff University & University of Manchester, 2020). While this is not a global average, the researchers extrapolated the findings to estimate that 31,000 to 42,000 tons of microplastics are applied to European soils annually through sludge-based fertilizers [25]. Furthermore, contamination can extend beyond the initial application's location due to their ability to be carried by wind or water [26]. Because more than half of the biosolids from WWTPs in the US are used on agricultural land, significant amounts of microplastics are added to the soil. This emphasizes the necessity for wastewater treatment systems to employ more efficient microplastic removal techniques and to investigate alternate applications for sewage sludge in order to lessen its negative effects on soil pollution [27].

All the above elements make it possible for microplastics to enter soil systems and have detrimental effects on fertility, soil health, and the ecosystem as a whole. The change in soil fertility and structure is a major concern. Plastic particle buildup can alter the texture of the soil, decrease water retention and affect aeration, which can therefore impair root development and soil structure and perhaps lower agricultural productivity [28]. Furthermore, certain microplastics have the ability to bind to pesticides and heavy metals, which can concentrate these dangerous chemicals in the soil and endanger plant health. Additionally, soil microbial communities which are critical for nutrient cycling, organic matter breakdown, and preserving soil fertility are adversely impacted by microplastics [29]. In addition to obstructing nutrition exchange and clogging pores, the physical presence of these particles might further suppress microbial activity. The food chain may bioaccumulate microplastics when they build up in the soil because they can be absorbed by plants and eaten by herbivores. Humans and animals may be impacted directly or indirectly by this transfer, particularly as microplastics may go up the food chain. Microplastics can damage the environment by harming soil-dwelling species like earthworms, which can then transfer the particles up the food chain [30].

2.4. Sources of Microplastics in Aquatic Systems

Agricultural activities, runoff from urban and industrial areas, wastewater discharges, and road debris contribute to microplastic pollution. Synthetic fibers shed by clothes also enter the waterways during washing. Microplastics are introduced to the seas through fishing and aquaculture operations, offshore industries, and marine tourism. A flood, storm, or typhoon accelerates microplastic transport from land to water. Additionally, atmospheric deposition and plastic fragmentation contribute to their widespread spread. Understanding these diverse sources will help develop effective mitigation strategies.

In addition to land-based microplastic pollution, urban runoff enters water bodies through drains and sewage, as well as household and industrial plastics. Microcapsule fertilizers used by farmers contribute to the pollution. Synthetic fibers from textile washing are shed into the waterways. All of these items wash into the aquatic systems during rain or runoff, including tires, road markings, and personal care products [31].

Synthetic ropes and nets, commonly used by modern fishing fleets, are a significant source of microplastic pollution in aquatic environments. Made from durable plastics like nylon, polyethylene, and polypropylene, these materials are favored for their strength and longevity. However, their durability also means they break down slowly, releasing microplastic fragments over time. While these materials improve efficiency in the fishing industry, they contribute to the growing problem of plastic pollution in oceans and waterways. Research shows that new synthetic ropes can release around 20 microplastic fragments per yard hauled, while older ropes, due to wear and tear, can shed up to 760 microplastic fragments per yard [12]. In practice, this means that a single fishing vessel using 220 yards of rope per haul could release between 2000 and 40,000 microplastic fragments in one operation. These microplastics are often too small to be easily detected or recovered, making them a persistent threat to marine life [32].

Over time, as ropes and nets degrade, they fragment into smaller pieces, which are then consumed by marine organisms ranging from plankton to larger fish and seabirds, posing serious risks to their health and survival [33]. The shift from natural fibers like hemp and cotton to synthetic materials in the maritime industry, driven by the need for durability and cost-effectiveness, has increased the environmental challenge. Unlike natural fibers, which biodegrade, synthetic ropes and nets persist in marine environments for decades, continuously releasing microplastics into the water [34].

Furthermore, Abandoned or lost fishing gear, commonly referred to as “ghost gear,” is one of the most harmful contributors to microplastic pollution. Ghost gear includes synthetic ropes, nets, and lines that are left behind or discarded in the ocean. These materials not only entangle marine life but also degrade over time, shedding microplastic fibers into the water. Studies indicate that ghost gear can remain intact and continue releasing microplastics for up to 30 years. Globally, an estimated 5.7% of fishing nets, 8.6% of traps and pots, and 29% of fishing lines are lost or abandoned annually. This makes ghost gear a substantial source of marine plastic pollution [35].

The equipment used in aquaculture operations contributes significantly to microplastic pollution. As plastic-based items degrade in harsh marine conditions, they release microplastic fragments into the water. Moreover, fish farms can damage habitat and degrade nearby ecosystems through direct physical damage and effluent runoff. When used to clean ponds, tanks, and equipment, chlorine and copper can also harm aquatic life [36]. While copper and chlorine are not directly linked to microplastic formation, their presence in effluents may influence

microplastic behavior or toxicity. However, direct evidence of such interactions is currently limited [36]. Depending on the farming method, aquaculture equipment and practices have different environmental impacts. The use of open-net cages in coastal areas results in antibiotic, pesticide, and fish feces pollution of the surrounding water. It is common for flow-through systems to discharge dissolved and solid waste without proper regulation, further polluting local waterways [37] [38]. To mitigate these environmental impacts, the aquaculture industry is increasingly adopting more sustainable practices. This includes siting fish farms in areas with strong currents to help disperse effluent, periodically moving farms to prevent long-term ecosystem damage, and developing land-based aquaculture systems that have minimal impact on local habitats. Additionally, recirculating and zero water exchange systems are being implemented to reduce environmental harm while maintaining high production densities. As aquaculture continues to grow, addressing these environmental challenges is essential for ensuring sustainable production of farmed seafood [39].

3. Sampling Methods

3.1. Sampling Methods for Environmental Samples

The compartment of interest has a significant impact on the sampling of microplastics in the aquatic environment. Generally speaking, this can be distinguished from sampling of the sediment phase (shoreline sediments, riverbed, or lakebed sediments) and the aqueous phase (surface water, water column) [40]. The choice of the studied area is where the variations in microplastic studies for coastal sediment sampling begin. Shore sediments are gathered at various distances from the shoreline, either randomly or parallel to or perpendicular to it. In the majority of the studies, grid samples of the upper sediment layer were collected at sampling depths of 2 to 5 cm [41]. According to other studies, the sampling is based on the water body's lowest flotsam line [3] [42]. Between the water line and the lowest flotsam line, sediment samples are taken from the shorelines of the Rhine and Main rivers at 35 to 40 randomly selected locations within a 10- to 15-meter radius. After removing big objects, the samples are taken using a stainless-steel spoon down to a depth of 2 - 3 cm. To evaluate variability, three duplicates are gathered at locations R4 and R8. Following three days of drying at 50°C, the silt is sieved into various fractions, and a modified density separation technique is used to separate the microplastics. A saturated sodium chloride solution is added to the sediment, stirred, and left to settle for the night. Plastic particles are isolated after floating particles are screened. Following an overnight treatment with a hydrogen peroxide and sulfuric acid mixture to eliminate natural debris, the fractions are then vacuum filtered, washed, and dried. Weighing the glass fiber filters both before and after filtration allows to calculate the final plastic weight [43] [44].

3.2. Sampling Methods for Soil Samples

There is growing concern about microplastic contamination in soils, and removing

these synthetic particles from complex soil matrixes requires specialized extraction techniques. Microplastics have diverse physical properties and are challenging to collect due to soil heterogeneity, organic matter interference, and soil heterogeneity. Listed below are key techniques and best practices for collecting microplastics from soil samples. Typically, soil samples are dried (by air or freeze drying) and sieved (with a mesh of between 2 - 5 mm) to remove large debris and homogenize the material. For microplastics smaller than 1 mm, extra steps are needed, including dispersing soil aggregates using surfactants or ultrasonic baths. A sample quantity of 1 - 1000 g, dry weight, is preferred to analyze soils with low concentrations for spectroscopic analysis and large volumes for soils with higher concentrations for spectroscopic analysis [45]-[47]. Most plastics are separated using density-based separation according to their buoyancy, for example, polyethylene, polypropylene, in high-density solutions: These are cost-effective and common solutions, such as sodium chloride (NaCl, $\rho = 1.2 \text{ g}\cdot\text{cm}^{-3}$), zinc chloride (ZnCl_2 , $\rho = 1.5$ to $1.7 \text{ g}\cdot\text{cm}^{-3}$), and sodium iodide (NaI) [48]. It has been found that the use of optimized approaches, including a mixture of saturated NaCl and NaI solutions (1:1) with aeration, improves recovery rates for a wide range of polymers (PE, PET, PP, PVC, PS) [49]. This oil-based separation method is less sensitive to soil texture and organic content, achieving 95% to 98% recovery in agricultural soils on average. Also, canola oil extracted low density polymer (LDPE, PP) easier than salt solutions. Detection of microplastics is complicated by residual organic matter (e.g., plant debris, humus). Among the effective oxidation methods are hydrogen peroxide (H_2O_2), which provides minimal polymer degradation, as well as Fenton's reagent, which may damage sensitive polymers like PVC [50]. After treatment, residual reagents are removed from samples by filtering (e.g., glass fiber filters) and rinsing with ethanol [51].

3.3. Sampling Methods for Aquatic Matrices

In aquatic environments, microplastic collection presents unique challenges due to the unique matrices of water and sediment. Field practicality and laboratory precision are combined in effective methods, which aim to minimize contamination and maximize recovery rates. It is still preferred to use plankton nets (typically 330cm) when sampling large quantities of surface water, considering the likelihood of microplastics being shed into the samples by manta trawls with fine mesh nets. A simpler alternative to grab sampling is field-filtered grab sampling with stainless-steel buckets or telescopic poles (10 to 30 L capacity), which sieve water through stainless steel filters 20 to 500 m for particle retention immediately. In-situ filtration systems reduce contamination risk during transportation of water in deeper waters with submersible pumps [51]. A Kristineberg Microplastic Sediment Separator (KMSS) enhanced by a steep incline and glass sediment chamber facilitates the separation of dense solutions such as Zinc Chloride. In comparison to traditional funnel systems, valveless glass separators achieve 94% to 98% particle recovery rates utilizing NaI or ZnCl_2 solutions [52] [53].

3.4. Sampling Methods for Atmospheric Samples

As a result of particle mobility, varying deposition rates, and contamination risks, collecting microplastics in atmospheric environments can be challenging. Airborne particles are captured efficiently while maintaining polymer integrity to be analyzed later. Below are the main approaches and considerations for sampling atmospheric microplastics. Air can be collected effectively using active sampling methods using powered devices, which can draw air through a trap or to a collection medium. These methods are ideal for sampling high volumes of air over a short period of time. Air samples collected with high volume air samplers (HVAS) are typically glass fiber or quartz fiber filters. These samplers can collect fine particles in PM10, PM2.5, and smaller sizes. A cascade impactor is a particle separator that uses multiple stages to separate particles depending on their aerodynamic diameter. It is effective for sampling particles based on their size. In electrostatic precipitators, particles are captured electrostatically, making them ideal for collecting nanoparticles. The deposition of particles onto a collection medium occurs naturally over a longer period, usually days to months, as a result of passive samplers. The simplest way to collect atmospheric fallout particles is to use collection plates, petri dishes, or glass trays. Using polyurethane foam (PUF) samplers, airborne MPs can be collected by trapping particles within porous foam matrixes. A standardized device used to measure atmospheric deposition of particles, the Sigma-2 Sampler [54]-[56]. There are different types of filters for different particle sizes and types of particles. Glass Fiber Filters (GFF) are suitable for larger MPs [57] [58]. In addition to their chemical inertness, polytetrafluoroethylene (PTFE) and polycarbonate (PC) filters are applied to both MPs and NPs for filtration. Nanodisc filters have nanoscale pores, making them ideal for collecting NPs [58] [59].

4. Analytical Techniques for Microplastics

4.1. Microscopic Techniques

Microscopic techniques are crucial for detecting microplastics and nanoplastics, which are becoming a major environmental concern. These tiny particles, with different shapes and sizes, are difficult to detect using standard methods. Following are various techniques used to study microplastics and nanoplastics.

4.1.1. Optical Microscopy for Microplastics Detection

Optical microscopy is a widely used technique for the detection and analysis of microplastics (MPs) due to its simplicity, cost-effectiveness, and accessibility. It relies on visible light and optical lenses to magnify and visualize microplastic particles, making it an accessible and economical method for analyzing microplastics, particularly those larger than 20 to 100 μm . The technique involves observing microplastics through light microscopes, which reveal features such as size, shape, and color. To enhance detection, fluorescence staining with hydrophobic dyes like Nile Red is often used. These dyes adsorb onto the hydrophobic surfaces of plastics,

causing them to fluoresce under specific wavelengths of light, such as blue light. The fluorescence intensity helps differentiate microplastics from non-plastic particles in a sample [60] [61]. Digital imaging systems can capture magnified images of stained particles, which are then analyzed manually or with software like ImageJ to quantify and classify microplastics based on their size, shape, and fluorescence properties. Optical microscopy is often combined with supplementary techniques, such as infrared (IR) spectroscopy or electron microscopy, to validate findings and reduce error rates associated with visual identification. However, ordinary light microscopy has limited resolution, making it difficult to detect particles smaller than 100 μm . Environmental factors, sample impurities, and subjectivity in visual analysis can also lead to error rates exceeding 20% for opaque microplastics and 70% for transparent ones. Additionally, false positives may occur due to the staining of organic materials or interference from natural fluorescence in environmental samples [62] [63].

4.1.2. Electron Microscopy

Electron microscopy is a powerful imaging method that visualizes things at the nanoscale scale by using an electron beam rather than light to achieve incredibly high magnifications and resolutions. In contrast to optical microscopes, which are constrained by the visible light wavelength, electron microscopes offer broad details about the composition, surface texture, and shape of materials. The two most important of these for identifying and describing microplastics (MPs) at the nanoscale scale are scanning electron microscopy (SEM) and transmission electron microscopy (TEM). These methods allow for thorough elemental and morphological analysis, which is crucial for understanding MPs' sources, modifications, and effects on the environment.

1) Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a powerful imaging technique widely used for analyzing the surface morphology of microplastics (MPs). The working principle of SEM involves directing a focused beam of electrons onto the sample surface. These electrons interact with the sample, producing secondary electrons and backscattered electrons [64]. The resulting high-resolution images allow researchers to examine the roughness, porosity, and degradation patterns of MPs at the micro and nanoscale levels. The capability of SEM to identify small surface textures that can reveal weathering and degradation processes including cracking, pitting, and biofilm formation is one of its main advantages in the identification of microplastics [65]. Knowing this information is essential to understanding how MPs deteriorate under various environmental circumstances.

Furthermore, SEM is very helpful for examining how interactions with pollutants or living organisms alter the surface of microplastics [66]. SEM is frequently used in conjunction with Energy-Dispersive X-ray Spectroscopy (EDS) to improve microplastic characterization even more. By identifying the characteristic X-rays released from a material upon electron beam interaction, EDS makes elemental composition analysis possible. Based on their elemental signatures, scientists

can now distinguish between naturally occurring particles and synthetic polymers. Overall, SEM, in combination with EDS, is an essential tool for microplastic analysis, providing detailed morphological and compositional insights that aid in understanding their environmental fate, sources, and potential ecological impacts [67].

2) Transmission Electron Microscopy (TEM)

Transmission Electron Microscopy (TEM) offers even higher resolution than SEM, making it an invaluable tool for studying the internal structure of microplastics and nanoplastics. Unlike SEM, which captures surface details, TEM works by transmitting a focused beam of electrons through an ultra-thin sample. As electrons pass through the sample, they interact with its internal structures, forming high-resolution images that reveal fine details at the nanometer scale [68].

When analyzing microplastics that have undergone extensive fragmentation and broken down into nanoscale particles, TEM is especially helpful. The ability of High-Resolution TEM (HRTEM) to identify and describe nanoparticles as small as a few nanometers offers vital information about how MPs degrade with time. This is especially vital for research on nanoplastics, which are more dangerous to the environment and to life because of their small size and high surface reactivity. Moreover, TEM makes it possible to do thorough research on how microplastics interact with other particles, contaminants, and biological systems. For example, TEM imaging can reveal whether MPs act as carriers for heavy metals or organic pollutants, providing insights into their role in environmental contamination. It also helps assess how microplastics are internalized by cells or accumulate in biological tissues, aiding in toxicological studies [69].

Recent Advancements in Electron Microscopy for Microplastic Detection

a. Cryo-SEM and Cryo-TEM for Preserving Sample Integrity: Traditional SEM and TEM often require sample preparation steps that may alter microplastic morphology. Cryogenic techniques such as Cryo-SEM and Cryo-TEM help preserve MPs in their native state by freezing them before analysis. This method is particularly useful for studying microplastics in biological samples, water, and sediments without introducing artifacts [70].

b. Automated Image Analysis and AI Integration: Recent advancements in machine learning and artificial intelligence (AI) have enhanced the efficiency of microplastic detection. AI-driven image analysis tools can automatically classify microplastics based on their shape, size, and texture in SEM and TEM images. This reduces human error and accelerates large-scale MP characterization [71].

c. Enhanced Elemental Mapping with Advanced EDS: New-generation EDS detectors with improved sensitivity allow for more precise elemental characterization of microplastics. This is particularly useful in distinguishing polymer types based on their elemental signature, aiding in source identification and environmental forensics [72].

d. Electron Tomography for 3D Imaging of Microplastics: Recent developments in electron tomography enable three-dimensional (3D) reconstruction of

microplastic structures. This technique provides valuable insights into the internal composition, degradation patterns, and interaction of microplastics with environmental particles or biological systems [73].

4.1.3. Confocal Laser Scanning Microscopy (CLSM)

It has emerged as a powerful analytical technique for identifying and analyzing microplastics in environmental samples. By utilizing a focused laser beam and capturing emitted fluorescence through a pinhole aperture, CLSM effectively eliminates out-of-focus light, enabling high-resolution imaging and three-dimensional reconstruction. This capability makes it particularly advantageous for distinguishing different materials and visualizing surface topography in microplastic studies [74]. Advanced CLSM systems, such as the Keyence VK-X1000, have been employed in soil and aquatic environments to assess particle morphology and optical properties with high precision. When integrated with spectroscopic techniques like Raman spectroscopy (CM-ESHRS), CLSM not only provides structural visualization but also facilitates chemical characterization, improving the accuracy of polymer identification. This combination enhances microplastic research by offering both morphological and compositional insights [75]. One of the key strengths of CLSM is its non-destructive nature, allowing samples to remain intact for complementary analyses. Its superior spatial resolution is particularly beneficial for examining small microplastic particles at the microscale. Depth profiling capabilities enable the study of microplastics embedded in complex matrices, such as sediments and biological tissues, while minimal sample preparation reduces contamination risks. These attributes make CLSM an indispensable tool for advancing environmental monitoring and microplastic research [76].

Despite its advantages, CLSM has certain limitations. Fluorescence labeling, commonly used to enhance detection, may introduce variability due to differing dye affinities among polymers. Autofluorescence from natural organic materials can also interfere with accurate differentiation between microplastics and organic matter. Additionally, the technique is constrained by sample size, typically analyzing only small quantities, and requires significant time, with a single scan potentially taking up to two hours at high magnification. Moreover, the complexity of CLSM operation necessitates specialized expertise, which can limit its accessibility and widespread application [77] [78].

4.2. Spectroscopy Techniques

Spectroscopy techniques have become powerful tools for analyzing microplastics (MPs), offering non-destructive and highly sensitive methods for their identification and characterization.

4.2.1. Fourier Transform Infrared (FTIR)

Fourier Transform Infrared (FTIR) is a commonly used technique for analyzing polymers. It identifies the molecular and functional groups in plastic polymers by measuring the infrared (IR) radiation absorbed by microplastic samples. This

creates a unique spectral fingerprint, where absorption peaks represent vibrations of bonds within the polymer structure. Since each polymer has a different composition, no two compounds produce the same spectrum, making FTIR an important tool for identifying polymers. The IR spectrum is divided into three regions: Near-Infrared (NIR), Mid-Infrared (MIR), and Far-Infrared (FIR). The Near-Infrared (NIR) region, ranging from 14,000 to 4000 cm^{-1} , is sensitive to overtone and combination vibrations. The Mid-Infrared (MIR) region, spanning 4000 to 400 cm^{-1} , is commonly used for microplastic characterization due to its ability to study fundamental vibrations. The Far-Infrared (FIR) region, from 400 to 10 cm^{-1} , is primarily used for studying rotational vibrations [79].

FTIR spectroscopy plays a crucial role in identifying microplastics (MPs) collected from various environmental sources, even when they are contaminated with organic pollutants. Since MPs often interact with different substances, purification is necessary before analysis. This is done through chemical digestion methods, including oxidation (using hydrogen peroxide or sodium hypochlorite), acid (nitric or hydrochloric acid), alkaline (sodium hydroxide or potassium hydroxide), or enzymatic treatments (using trypsin or proteinase K). These methods help remove organic material while preserving the polymer structure of MPs for accurate identification.

Once purified, MPs are analyzed using FTIR, which detects the unique vibrational patterns of chemical bonds within the plastic polymers [80]. The choice of FTIR technique depends on particle size. Micro-FTIR (μ FTIR) is used for MPs smaller than 20 μm due to its high spatial resolution, while Attenuated Total Reflectance FTIR (ATR-FTIR) is employed for larger MPs as it enables direct measurement [81]. Beyond polymer identification, FTIR detects weathering-related chemical changes, such as hydroxyl (-OH) groups (3100 to 3700 cm^{-1}), alkenes (1600 to 1680 cm^{-1}), and carbonyl (C=O) groups (1690 to 1810 cm^{-1}), which indicate environmental degradation. The Carbonyl Index (CI) is often used to assess photo-oxidation, particularly in polyethylene, as its value increases with prolonged exposure to sunlight [82]. Advanced techniques such as Micro-FTIR allow precise identification of MPs down to the micron level. This technique integrates infrared spectroscopy with optical microscopy, enabling the simultaneous quantification and classification of MPs, including fibers released from washing textiles [83].

4.2.2. Raman Spectroscopy (RS)

Raman spectroscopy (RS) is a powerful vibrational spectroscopy technique based on inelastic light scattering, providing detailed molecular structural information. When a sample is illuminated with laser light of a specific wavelength, most photons are scattered elastically without a change in energy (Rayleigh scattering). The Raman effect, often referred to as Raman scattering, is the process by which just a small fraction of photons experiences inelastic scattering, where they either gain or lose energy as a result of molecular vibrations. Each molecule has its own distinct vibrational energy changes, which provide a distinctive Raman spectrum that

acts as a molecular fingerprint. It typically spanning from 0 to 4000 cm^{-1} , contains peaks corresponding to specific bond stretching and bending vibrations, making it an invaluable tool for chemical identification. The technique is highly sensitive, non-destructive, and requires minimal sample preparation, making it widely applicable across various scientific disciplines.

Given its excellent spatial resolution, which enables the analysis of tiny plastic particles, Raman spectroscopy is becoming more and more popular in the study of MPs. Since plastic pollution is becoming a bigger environmental concern, the numerical abundance of MPs is thought to be especially significant in aquatic systems. The Marine Strategy Framework Directive Technical Subgroup on Marine Litter in European Seas recommends RS and Fourier Transform Infrared (FT-IR) spectroscopy as the most widely used of the many methods used for polymer identification. In addition to being non-destructive and needing small amounts of sample with little preparation, these methods help distinguish between plastic and natural particles, lowering false positives.

Notably, RS offers significant advantages over FT-IR, including a higher spatial resolution (down to 1 μm , compared to 10 to 20 μm for FT-IR), broader spectral coverage, higher sensitivity to non-polar functional groups, lower water interference, and narrower spectral bands. The use of RS in identifying individual MPs in tap water was illustrated in a proof-of-concept study that examined microplastic suspensions including common polymers like PS, PET, PE and PP [84] [85]. A study has shown the potential for real-time monitoring of microplastics in drinking water and surface water sources by passing water through a specially designed flow cell at a rate of 1 L/h and adding particulate and fluorescent pollutants. This method can be used to detect microplastics in various water systems, including freshwater, groundwater, drinking water, oceanic waters, sea water, wastewater, and sewage systems [86].

Recent advancements in RS have further improved its capabilities for MPs analysis. AI-assisted machine learning (ML) has revolutionized microplastic detection using Raman spectroscopy by improving data processing, feature extraction, and classification accuracy. By optimizing parameters like laser intensity and exposure time, AI ensures the acquisition of high-quality spectral data, which is then refined through preprocessing techniques such as noise reduction and baseline correction. AI automates feature extraction, identifying spectral patterns that are crucial for detecting microplastics. Machine learning models, including Random Forest (RF), Support Vector Machine (SVM), and neural networks, are trained on labeled datasets to classify microplastics based on spectral features, enabling real-time monitoring of environmental samples. Additionally, AI integrates multi-dimensional data, combining chemical, physical, and environmental parameters to offer a holistic analysis. The integration of Convolutional Neural Networks (CNNs) for imaging analysis further enhances classification by linking visual and spectral traits. As AI models continuously learn, they remain adaptable to new microplastic types, while validation mechanisms improve model reliability, making

the process more efficient, scalable, and accurate for both environmental and industrial applications [87] [88].

4.2.3. Nuclear Magnetic Resonance (NMR)

Nuclear Magnetic Resonance (NMR) spectroscopy is a powerful analytical technique that detects and quantifies compounds based on the interaction of atomic nuclei with an external magnetic field. This interaction provides detailed structural and compositional information, making NMR an invaluable tool in various scientific fields. Specifically, it is highly effective for analyzing microplastics (MPs) due to its ability to provide high specificity and sensitivity, even in complex environmental and biological samples. Detecting and quantifying MPs in environmental samples requires precise, reliable, and non-destructive techniques. NMR is particularly suited for this task because different polymer types exhibit unique proton signals in their NMR spectra. These distinct signals allow for the accurate identification and quantification of various microplastic types [89].

In recent advancements, NMR has been applied for the analysis of microplastics by dissolving them in deuterated solvents such as CDCl_3 or THF-d_8 . The addition of an internal standard allows for precise quantification by comparing the signal intensities of the microplastics to the known concentration of the standard. Two key quantification methods are employed in NMR-based microplastic analysis: 1) Proton Signals with Internal Standard, which achieves lower detection limits (0.2 to 8 $\mu\text{g/mL}$) and higher accuracy by directly comparing the polymer proton signals with the internal standard, and 2) Signal-to-Noise Ratio (SNR), which offers a higher detection range (1 to 10 $\mu\text{g/mL}$) but with slightly less precision. Statistical methods such as Mean Absolute Percentage Error (MAPE) and ANOVA confirm the strong correlation between measured and nominal polymer concentrations.

One of the key advantages of NMR, is its minimal sample preparation requirements, making it a cost-effective and efficient alternative. Moreover, NMR enables the simultaneous analysis of polymer size distribution and concentration in a single sample. Its non-destructive nature preserves sample integrity, allowing for further analysis if needed. These benefits make NMR spectroscopy an invaluable tool for microplastic research, offering high precision, reproducibility, and reliability in detecting and quantifying MPs in environmental and biological samples. In conclusion, NMR spectroscopy, particularly through quantitative methods like qNMR, provides a highly efficient, accurate, and scalable approach to microplastic detection. It addresses the growing need for reliable techniques in environmental monitoring, offering unique insights into the presence, concentration, and degradation of microplastics in ecosystems [90]. NMR differentiates polymers like PET and PE by their unique ^1H chemical shifts, allowing individual identification even in mixtures. However, in complex environmental samples, careful sample preparation is essential to remove matrix interferences and avoid signal overlap. Studies have shown that with appropriate solvents and purification steps, NMR enables reliable quantification of coexisting polymers [89] [90].

4.3. Thermal Analysis

Thermal analysis refers to a group of techniques that measure changes in the physical and chemical properties of a material as a function of temperature. It has gained increasing attention as an alternative and complementary approach for MPs characterization. This method relies on identifying polymers based on their degradation products and thermal properties, making it particularly valuable for analyzing MPs with low solubility and additives that are difficult to extract or hydrolyze. It encompasses a range of analytical techniques, including Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), and Pyrolysis-Gas Chromatography Mass Spectrometry (Py-GC-MS). Additionally, hyphenated techniques such as TGA-Mass Spectrometry (TGA-MS) and TGA-Thermal Desorption-GC-MS further enhance the analytical capabilities. These methods offer several advantages, including rapid analysis, minimal sample requirements, and high sensitivity in differentiating various polymers based on their thermal behavior [91].

4.3.1. Pyrolysis Gas Chromatography-Mass Spectrometry (Py-GC-MS)

Pyrolysis Gas Chromatography-Mass Spectrometry (Py-GC-MS) is a highly effective analytical technique that integrates the processes of pyrolysis, gas chromatography (GC), and mass spectrometry (MS) for in-depth characterization of complex macromolecular substances, including both synthetic and natural polymers. It offers valuable insights into the composition and degradation products of polymers, making it an essential tool in environmental science, materials research, and forensic investigations. In Py-GC-MS, the process begins with the thermal degradation of the sample under an inert atmosphere, typically at temperatures ranging from 300°C to 1000°C. This pyrolysis step breaks down large macromolecules into smaller volatile compounds. These products are then separated by gas chromatography based on their volatility and interaction with the stationary phase of the GC column. The separated fragments are subsequently ionized and analyzed by the mass spectrometer, providing detailed structural and compositional information about the polymer and its degradation products [92].

One of the most significant applications of Py-GC-MS is in the analysis of microplastics in environmental samples. This technique efficiently decomposes complex polymer structures into identifiable molecular fragments, enabling the detection of even trace amounts of microplastics and nanoplastics. Due to its high sensitivity, it can precisely identify and quantify various polymer types, making it indispensable for understanding pollution sources, assessing environmental impacts, and conducting regulatory evaluations of plastic contamination. Additionally, its compatibility with various extraction methods minimizes interference from organic matter, making it ideal for the analysis of microplastic particles in complex environmental matrices. Furthermore, Py-GC-MS does not require sample pre-treatment, unlike traditional solvent extraction procedures, thus reducing both time and costs. For example, an automated Py-GC-MS method has been

proposed for analyzing polymer types and organic plastic additives (OPAs) in marine microplastic particles, offering a streamlined, solvent-free alternative to conventional methods. However, a key limitation of Py-GC-MS in microplastic characterization is the inability to provide information regarding the particle size. Despite this, the technique remains invaluable for its sensitivity, speed, and ability to analyze complex polymeric materials with high precision [93].

4.3.2. DSC-TGA

Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) are powerful thermal analysis techniques commonly integrated into a single instrument known as Simultaneous DSC-TGA. This approach simultaneously measures both heat flow and mass changes of a sample as it undergoes temperature variations in a controlled environment. DSC detects thermal transitions such as melting points, glass transitions, and crystallization by comparing the heat flow between the sample and a reference, while TGA monitors mass loss during heating or cooling, providing valuable insights into the thermal stability, decomposition, and other changes in mass due to temperature variations. This technique is highly effective for detecting and analyzing microplastics and nanoplastics in environmental samples. By analyzing the unique thermal characteristics of different plastic types, such as their melting and decomposition temperatures, this method helps to identify and differentiate plastics. TGA also allows for the quantification of microplastic content through the measurement of mass loss during decomposition. Additionally, the size-dependent thermal behavior of plastics enables the differentiation between microplastics and nanoplastics. This technique requires minimal sample preparation and can analyze complex environmental samples, enhancing the accuracy of plastic identification and characterization. Plastics exhibit unique thermal characteristics, including melting points, glass transition temperatures, and decomposition temperatures, which can be measured to identify and classify different types. The thermal response of plastics is influenced by particle size, enabling TGA-DSC to potentially distinguish between microplastics and nanoplastics. While TGA measures mass changes and provides decomposition profiles that can quantify plastic content, DSC reveals subtle differences in thermal transitions that may vary with particle size. However, the technique may face challenges in detecting smaller particles, such as nanoplastics (<1 μm), due to its resolution limits. Nevertheless, advancements in instrumentation and methodology continue to improve the sensitivity of TGA-DSC. For comprehensive plastic particle characterization, this technique is often used in combination with other analytical methods like FTIR or Raman spectroscopy, particularly for complex environmental samples containing both microplastics and nanoplastics [94] [95].

4.4. High-Performance Liquid Chromatography (HPLC)

HPLC is a widely used analytical technique for separating, identifying, and quantifying chemical compounds in complex mixtures, particularly useful for analyzing

non-volatile, polar, and thermally unstable compounds. As a result of a stationary phase and mobile phase, HPLC allows the separation of a liquid sample in accordance with chemical affinity. In addition to UV, fluorescence, and mass spectrometry, HPLC can also be used to identify microplastics and nanoplastics. Microplastics (MPs) and nanoplastics (NPs) can harm the environment and humans, whether they originate as small plastic particles or are degraded into larger plastics. HPLC evaluates these particles' chemical composition, the adsorption and desorption of organic contaminants, and their breakdown products. As a result, toxic substances such as phthalates and bisphenol A (BPA) can be detected [96].

HPLC is employed to detect and quantify specific polymeric microplastics, such as polyamides (nylon 6 and nylon 6.6) and polyester (PET). The process begins with depolymerizing these microplastics through hydrolysis, acid-catalyzed hydrolysis for polyamides, breaking them down into monomers like 6-aminohexanoic acid (AHA) and hexamethylene diamine (HMDA), and alkaline hydrolysis for PET, yielding monomers such as terephthalic acid (TPA). These monomers are derivatized with a fluorescent tag to enhance sensitivity before undergoing reversed-phase HPLC separation using a nonpolar stationary phase with a polar mobile phase, ensuring efficient separation, while fluorescence detection enables quantification at ppm and ppb levels. Calibration curves for AHA and HMDA facilitate accurate concentration measurements in environmental samples such as wastewater sludge, aiding in pollution assessment. It is also utilized to study chemical additives leached from microplastics into aquatic environments. The process involves preconcentration and cleanup using a monolithic silica column (C₁₈) to remove the seawater matrix and concentrate the analytes, simulating environmental conditions by continuously pumping seawater through microplastic materials to collect leachates under realistic conditions. The leachate undergoes on-line micro solid-phase extraction before being injected into the HPLC system, where an optimized acetonitrile/water gradient is used for separation on a monolithic column (Onyx C18HD), and UV detection at 240 nm ensures selective identification of target analytes, including dimethyl phthalate (DMP), diethyl phthalate (DEP), and BPA. The comprehensive and highly sensitive methodology allows for precise quantification of polymeric microplastics and chemical additives in complex matrices such as wastewater sludge and seawater, providing valuable insights into microplastic contamination and its broader environmental implications, ultimately supporting efforts to mitigate its effects on ecosystems and human health [97].

4.5. Mass Spectrometry (MS)

It is a powerful analytical technique used to separate and identify ions (charged atoms or molecules) based on their mass-to-charge ratio (m/z). It operates by converting a sample into gas-phase ions, which are then accelerated through an electric or magnetic field and detected. This process enables the determination of molecular weight, and, in many cases, the structure of the molecules presents in a

sample [98]. In recent years, mass spectrometry has become an indispensable tool for detecting and quantifying microplastics (MPs) and nanoplastics (NPs) in complex environmental and biological samples. Given the widespread presence and potential environmental and health risks of these small plastic particles, effective detection is essential for understanding and addressing plastic pollution. MS-based techniques are particularly valuable in identifying and analyzing the composition of MPs and NPs, with various methods offering distinct advantages depending on the sample type and the nature of the particles.

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is one of the leading MS techniques used for surface analysis of MPs and NPs. This method utilizes ion bombardment to eject secondary ions from the sample surface, providing detailed information on the polymer composition of the particles. TOF-SIMS is particularly useful for smaller MPs and NPs, offering high spatial resolution and sensitivity. Matrix-Assisted Laser Desorption/Ionization Time-of-Flight Mass Spectrometry (MALDI-TOF) MS uses a laser to ionize the sample with minimal fragmentation. It is a soft ionization method, making it suitable for detecting both low- and high-molecular-weight polymers in complex polymer mixtures. MALDI-TOF MS allows for precise identification of polymer types without significantly degrading the sample [99].

Orbitrap and quadrupole mass spectrometry are widely employed for microplastic analysis. Orbitrap MS excels at analyzing low-molecular-weight MPs (under 50,000 Da) and provides high-resolution mass analysis, making it ideal for distinguishing between polymers based on their molecular weight. Quadrupole MS, on the other hand, aids in studying thermal or depolymerized products, which can reveal information about MPs based on their chemical breakdown [100].

4.6. Alternative Approaches for Microplastic Detection

Gas chromatography-mass spectrometry (GC-MS), when coupled with MS, provides an alternative approach for analyzing MPs. This method uses thermal desorption or pyrolysis to introduce polymer degradation products and additives into the GC-MS system, generating a “chemical fingerprint” that can be used to trace the sources of microplastic pollution. However, using GC-MS with low-resolution quadrupole mass spectrometers presents some limitations, including long separation times (30 to 60 minutes) and challenges in separating complex pyrolysis mixtures. These constraints can hinder the speed and comprehensiveness of the analysis. To overcome these limitations, high-resolution mass spectrometry (HRMS) offers a more refined approach. HRMS allows for the determination of thousands of elemental compositions, providing a more detailed and informative fingerprint compared to standard GC-MS. This enables the differentiation of microplastics with similar polymer types, offering better insights into their sources and environmental impact. Additionally, ambient ionization techniques such as Direct Analysis in Real Time (DART) have emerged as rapid methods for polymer

characterization. DART allows for the direct analysis of microplastics with minimal sample preparation, facilitating quick identification and source tracing in environmental samples [101].

5. Challenges in Microplastics Analysis

5.1. Identification and Quantification Issues

The variety of polymers, sizes, shapes (fibers, films, spheres), and chemical additives of microplastics make it difficult for them to be identified accurately. Although visual microscopy is cost-effective, it misclassifies up to 70% of particles, particularly those that are transparent or small enough to be subtracted. Although Pyrolysis-GC/MS allows for the detection of nano-plastics, it requires calibration curves specific to polymers and struggles with fibers of low mass. FTIR and Raman vibration spectroscopy are limited in their application in complex matrices like soil and biofilms, due to interference from organic residues and fluorescence. A mass-based approach lacks particle-specific data, while a particle-based approach lacks sensitivity to rare polymers [102] [103].

5.2. Contamination Control

As microplastic studies are typically confounded by airborne fibers, lab plastics, and synthetic clothing, procedure blanks play an important role in removing background contamination [104]. In order to reduce contact with plastic, use metal tools, glass containers, and cotton lab coats. You can also isolate work areas with HEPA filters to prevent airborne particles from entering your workplace. The validation of organic digestion protocols to prevent polymer degradation is particularly important in Arctic studies where logistical constraints exacerbate contamination risks, requiring meticulous quality assurance/quality control measures [105].

5.3. Standardization of Methods

Microplastics analysis protocols are not universally accepted, resulting in inconsistencies between studies. Differences in sampling, processing, and analysis techniques result in variability. The process of selecting filters, digesting samples, and analyzing results must be standardized in order to achieve comparable and reproducible results [105].

5.4. Limitations of Current Techniques

Nanoplastics are very small and relatively light, so current analytical techniques often have difficulty detecting them. A number of FTIR and Raman spectroscopy techniques require extensive sample preparation and may not be able to detect particles below detection limits due to extensive sample preparation. Developing advanced, high-resolution analytical tools with better sensitivity and accuracy is vital [102]-[104] [106].

5.5. Data Interpretation and Reporting

The accuracy of microplastic data remains challenging due to variations in particle size classifications, unit expressions, and polymer identification criteria. It is possible to increase the reliability of results by incorporating comprehensive quality control measures and standardized reporting formats [57] [102]-[107].

6. Recent Advances in Microplastics Research

6.1. Emerging Analytical Techniques for Microplastics Detection

Research on microplastics has seen significant advancements in analytical techniques, addressing the challenges of detecting, identifying, and quantifying microplastics. In order to better understand microplastics' environmental, ecological, and public health impacts, these innovations are crucial.

6.1.1. Mass Spectrometry Imaging (MSI) Techniques

The combination of MALDI with time-of-flight or Orbitrap mass analyzers has been developed for precise in situ imaging of microplastics in biological samples using Mass Spectra Imaging. Using this technique, it is possible to analyze at high resolution the chemical composition of microplastics without labeling, which provides valuable insight into their accumulation and transformation in living organisms [108].

6.1.2. Spectroscopic Methods

In Raman spectroscopy technique, plastic particles as small as 1 μm can be detected with high sensitivity. Its advantages include its non-destructive nature, and the minimal sample preparation required for real-time analysis. Microplastics can be identified with Fourier Transform Infrared spectroscopy (FTIR), but opaque samples and particles smaller than 20 μm are difficult. FTIR has improved over the years, such as focal plane array FT-IR (FPA-FT-IR) [109].

6.1.3. Fluorescence-Based Techniques

Fluorescent staining of microplastics with hydrophobic dyes like Nile Red has proven to be an effective method for detecting microplastics under microscopy. As a result of this method, identification processes are streamlined; however, false positives must be reduced with further development [110].

6.1.4. Machine Learning and Automation

The evolution of machine learning has made it possible to detect microplastics more efficiently and accurately through spectroscopic and microscopic techniques. In addition to reducing analysis time and increasing precision, models such as PlasticNet are highly accurate at identifying microplastics from spectral data [111] [112].

6.1.5. Biosensors and Electrochemical Sensing

Microplastics can be detected using electrochemical biosensors developed in recent years. Despite their potential for low-cost, rapid detection of a wide range of

environmental samples, these sensors require further optimization before being applied widely [111].

6.2. Innovations in Sampling and Sample Preparation

In order to detect and analyze microplastics accurately in various environmental matrices, innovative sampling and preparation methods must be used. A number of advancements have been made in developing standard protocols, improving isolation methods, and improving the effectiveness of organic matter removal.

6.2.1. Development of Standardized Protocols for Environmental Matrices

To ensure consistency and comparability across multiple studies, standardizing sampling protocols is crucial. It is being developed that standardized methods will be used to collect soil, water, and air samples. Microplastic sampling methods, such as ambient water, stormwater, sediment, and aquatic life, have been standardized in California by a group of international experts [113].

6.2.2. Use of Novel Filtration and Density Separation Techniques

The isolation of microplastics from complex environmental samples has been improved using filtration and density separation techniques. Microplastics from sediments and soils are often extracted using density separation techniques such as sodium chloride and zinc chloride. In recent years, the use of sodium dihydrogen phosphate (NaH_2PO_4) has become increasingly popular due to its high extraction efficiency, cost-effectiveness, and non-hazardous nature [114]. In addition, specialized equipment like the Kristineberg Microplastic Sediment Separator (KMSS) has been designed to enhance density separation [53].

6.2.3. Application of Enzymatic Digestion for Biological Samples

Microplastics can be extracted from biological samples by enzymatic digestion, which has emerged as a powerful tool for removing organic matter from samples. Microplastics have been thoroughly digested with proteolytic enzymes such as trypsin without damaging them, demonstrating high digestive efficiency. It has been demonstrated that trypsin can achieve up to 88% digestion efficiency at low concentrations, which makes it an efficient and cost-effective way to extract microplastics from biological samples [115]. Marine organisms have a great deal of benefit from this approach when analyzing microplastic intake.

7. Conclusion

Microplastics are a growing global concern due to their persistence, widespread presence, and potential impacts on ecosystems and human health. This review highlights the diverse sources contributing to microplastic pollution, from agricultural and industrial activities to consumer products and wastewater. While advancements in analytical techniques such as spectroscopy, microscopy, thermal analysis, and mass spectrometry have improved detection capabilities, significant challenges remain. These include the lack of standardized methods, difficulties in

Table 1. Comparative overview of advanced analytical techniques for microplastic detection and characterization.

Analytical Techniques	Microplastic Type	Size Detection Range	Limitations	Reference
FTIR	Most polymers	>20 μm	struggles with opaque/black MPs; affected by sample heterogeneity and environmental factors	[106]
Raman Spectroscopy	Most polymers	$\geq 1 \mu\text{m}$	Prone to fluorescence interference; low signal-to-noise ratio; sample heating may cause degradation	[106]
Micro-FTIR	Polymer composites	$\sim 10 \mu\text{m}$	Resolution-dependent size artefacts; particle pre-selection required for single-pixel detectors	[115]
Pyrolysis-GC/MS	Polymer identification	N/A (bulk analysis)	Destructive method; cannot provide particle size or physical properties	[106]
SEM-EDX	Surface characterization	Wide range	Complex pretreatment; high cost; cannot distinguish colors	[106]
NMR (qNMR)	PE, PET, PS, PB, PI, PVC, PU, PLA (in solution)	Not size-dependent (bulk)	Overlapping signals in mixtures; low concentrations challenging; requires sample dissolution; not particle-specific	[116]
DSC-TGA	PP, PA, HDPE, LDPE, PET (semi-crystalline); PS, PVC (amorphous)	Not size-dependent (bulk)	Glass transition signal weak; interference from organic/inorganic matter; not for individual particle analysis	[117]
Mass Spectrometry (MS, ASAP-MS)	Polyolefins, polyaromatics, polyacrylates, (bio)polyesters, polyamides, polycarbonates, polyacrylonitriles	$\geq 5 \mu\text{m}$ (selected ion mode), $\geq 30 \mu\text{m}$ (full scan)	Destructive; requires individual particle handling; limited for routine environmental screening	[118]
Confocal Laser Scanning Microscopy (CLSM)	Most polymers	Micrometer to sub-millimeter	Requires labeling/fluorescence; limited chemical specificity; not for routine quantification	[119]
HPLC	Not standard for microplastics; some polymer additives	Not applicable	Not designed for polymer particle analysis; mainly detects additives or degradation products	[109]

nanoplastic identification, and analytical limitations in complex environmental matrices (Table 1). Addressing these challenges requires collaborative efforts across scientific disciplines and regulatory sectors. Standardization of sampling and analytical protocols is essential to ensure data comparability and reliability. Policymakers must also consider stricter regulations on plastic use in agriculture and industry, as well as guidelines for the reuse of wastewater sludge and biosolids. Investment in advanced technologies, sustainable alternatives, and public awareness initiatives will be critical to minimizing microplastic pollution. Ultimately, a harmonized global approach is needed to monitor, assess, and mitigate the risks posed by microplastics to environmental and human health.

Conflicts of Interest

The authors declare no conflicts of interest.

References

- [1] Cole, M., Lindeque, P., Halsband, C. and Galloway, T.S. (2011) Microplastics as Contaminants in the Marine Environment: A Review. *Marine Pollution Bulletin*, **62**, 2588-2597. <https://doi.org/10.1016/j.marpolbul.2011.09.025>
- [2] Geyer, R., Jambeck, J.R. and Law, K.L. (2017) Production, Use, and Fate of All Plastics Ever Made. *Science Advances*, **3**, e1700782. <https://doi.org/10.1126/sciadv.1700782>
- [3] Browne, M.A., Galloway, T.S. and Thompson, R.C. (2010) Spatial Patterns of Plastic Debris along Estuarine Shorelines. *Environmental Science & Technology*, **44**, 3404-3409. <https://doi.org/10.1021/es903784e>
- [4] Rochman, C.M., Hoh, E., Hentschel, B.T. and Kaye, S. (2013) Long-Term Field Measurement of Sorption of Organic Contaminants to Five Types of Plastic Pellets: Implications for Plastic Marine Debris. *Environmental Science & Technology*, **47**, 1646-1654. <https://doi.org/10.1021/es303700s>
- [5] K ppler, A., Fischer, D., Oberbeckmann, S., Schernewski, G., Labrenz, M., Eichhorn, K., *et al.* (2016) Analysis of Environmental Microplastics by Vibrational Microspectroscopy: FTIR, Raman or Both? *Analytical and Bioanalytical Chemistry*, **408**, 8377-8391. <https://doi.org/10.1007/s00216-016-9956-3>
- [6] (2025) Where Do Microplastics Come from? <https://www.horiba.com/usa/scientific/resources/science-in-action/where-do-microplastics-come-from/>
- [7] Illinois Environmental Protection Agency (2025) Microplastics. <https://epa.illinois.gov/topics/water-quality/microplastics.html>
- [8] Britannica (2025) Microplastics. Definition, Properties, & Plastic Pollution. <https://www.britannica.com/technology/microplastic>
- [9] Embers, C. (2022) What Are Microplastics and How Are They Formed? Cruz Foam. <https://www.cruzfoam.com/post/what-are-microplastics-and-how-are-they-formed/>
- [10] (2025) Understanding Microplastics: Causes, Risks, and Mitigation Strategies. Microplastic Free. <https://microplasticfree.org/blogs/news/understanding-microplastics-causes-risks-and-mitigation-strategies>
- [11] (2024) Plastic Overshoot Day 2024: Global Waste Crisis Surpasses Management Capacity. SAFE—Safe Food Advocacy Europe. <https://www.safefoodadvocacy.eu/plastic-overshoot-day-2024-global-waste-crisis-surpasses-management-capacity/>
- [12] Kindy, D. (2021) With Ropes and Nets, Fishing Fleets Contribute Significantly to Microplastic Pollution. Smithsonian. <https://www.smithsonianmag.com/smart-news/fishing-fleets-contribute-significantly-to-microplastic-pollution-study-says-180978760/>
- [13] (2025) Sources of Microplastics and Their Distribution in the Environment. <https://www.firstsentier-mufg-sustainability.com/insight/sources-of-microplastics-and-their-distribution-in-the-environment.html>
- [14] Moeck, C., Davies, G., Krause, S. and Schneidewind, U. (2022) Microplastics and Nanoplastics in Agriculture—A Potential Source of Soil and Groundwater Contamination? *Grundwasser*, **28**, 23-35. <https://doi.org/10.1007/s00767-022-00533-2>

- [15] Li, S., Ding, F., Flury, M., Wang, Z., Xu, L., Li, S., *et al.* (2022) Macro- and Microplastic Accumulation in Soil after 32 Years of Plastic Film Mulching. *Environmental Pollution*, **300**, Article ID: 118945. <https://doi.org/10.1016/j.envpol.2022.118945>
- [16] Yang, H., Hu, Z., Wu, F., Guo, K., Gu, F. and Cao, M. (2023) The Use and Recycling of Agricultural Plastic Mulch in China: A Review. *Sustainability*, **15**, Article 15096. <https://doi.org/10.3390/su152015096>
- [17] Long, B., Li, F., Wang, K., Huang, Y., Yang, Y. and Xie, D. (2023) Impact of Plastic Film Mulching on Microplastic in Farmland Soils in Guangdong Province, China. *Heliyon*, **9**, e16587. <https://doi.org/10.1016/j.heliyon.2023.e16587>
- [18] Erickson, B.E. (2022) Stop Delivering Agrochemicals with Microplastics, Group Says. Chemical & Engineering News. <https://cen.acs.org/environment/pesticides/Stop-delivering-agrochemicals-microplastics-group/100/i19>
- [19] SGBLOG (2025) Biodegradable Seed Coatings—A Viable Solution for Microplastic Pollution! Sathguru Management Consultants. <https://blog.sathguru.com/agribusiness/biodegradable-seed-coatings-a-viable-solution-for-microplastic-pollution/>
- [20] Monkul, M.M. and Özhan, H.O. (2021) Microplastic Contamination in Soils: A Review from Geotechnical Engineering View. *Polymers*, **13**, Article 4129. <https://doi.org/10.3390/polym13234129>
- [21] Büks, F. and Kaupenjohann, M. (2020) Global Concentrations of Microplastics in Soils—A Review. *SOIL*, **6**, 649-662. <https://doi.org/10.5194/soil-6-649-2020>
- [22] Hernandez, G., Low, J., Nand, A., Bu, A., Wallis, S.L., Kestle, L., *et al.* (2022) Quantifying and Managing Plastic Waste Generated from Building Construction in Auckland, New Zealand. *Waste Management & Research: The Journal for a Sustainable Circular Economy*, **41**, 205-213. <https://doi.org/10.1177/0734242x221105425>
- [23] (2025) Building a Greener Future: Reducing Plastic Waste in Construction. <https://www.4ocean.com/blogs/construction/building-a-greener-future-reducing-plastic-waste-in-construction>
- [24] Iyare, P.U., Ouki, S.K. and Bond, T. (2020) Microplastics Removal in Wastewater Treatment Plants: A Critical Review. *Environmental Science: Water Research & Technology*, **6**, 2664-2675. <https://doi.org/10.1039/d0ew00397b>
- [25] Lavars, N. (2022) Fertilizer Turning Europe's Farms into Massive Reservoirs of Microplastics. New Atlas. <https://newatlas.com/environment/fertilizer-sewage-sludge-europe-farmlands-microplastic-reservoirs/>
- [26] Tagg, A.S., Brandes, E., Fischer, F., Fischer, D., Brandt, J. and Labrenz, M. (2022) Agricultural Application of Microplastic-Rich Sewage Sludge Leads to Further Uncontrolled Contamination. *Science of the Total Environment*, **806**, Article ID: 150611. <https://doi.org/10.1016/j.scitotenv.2021.150611>
- [27] (2024) Microplastics from Natural Fertilizers Are Blowing in the Wind More Often Than Once Thought. American Chemical Society. <https://www.acs.org/pressroom/presspacs/2024/january/microplastics-from-natural-fertilizers-are-blowing-in-the-wind.html>
- [28] Jia, L., Liu, L., Zhang, Y., Fu, W., Liu, X., Wang, Q., *et al.* (2023) Microplastic Stress in Plants: Effects on Plant Growth and Their Remediations. *Frontiers in Plant Science*, **14**, Article 1226484. <https://doi.org/10.3389/fpls.2023.1226484>
- [29] Aralappanavar, V.K., Mukhopadhyay, R., Yu, Y., Liu, J., Bhatnagar, A., Praveena,

- S.M., *et al.* (2024) Effects of Microplastics on Soil Microorganisms and Microbial Functions in Nutrients and Carbon Cycling—A Review. *Science of the Total Environment*, **924**, Article ID: 171435. <https://doi.org/10.1016/j.scitotenv.2024.171435>
- [30] Moreno-Jiménez, E., Leifheit, E.F., Plaza, C., Feng, L., Bergmann, J., Wulf, A., *et al.* (2021) Effects of Microplastics on Crop Nutrition in Fertile Soils and Interaction with Arbuscular Mycorrhizal Fungi. *Journal of Sustainable Agriculture and Environment*, **1**, 66-72. <https://doi.org/10.1002/sae2.12006>
- [31] Wang, J., Bucci, K., Helm, P.A., Hoellein, T., Hoffman, M.J., Rooney, R., *et al.* (2022) Runoff and Discharge Pathways of Microplastics into Freshwater Ecosystems: A Systematic Review and Meta-Analysis. *FACETS*, **7**, 1473-1492. <https://doi.org/10.1139/facets-2022-0140>
- [32] Williams, A. (2021) Maritime Rope Could Be Adding Billions of Microplastics to the Ocean Every Year. University of Plymouth. <https://www.plymouth.ac.uk/news/maritime-rope-could-be-adding-billions-of-microplastics-to-the-ocean-every-year>
- [33] Kaliszewicz, A., Panteleeva, N., Karaban, K., Runka, T., Winczek, M., Beck, E., *et al.* (2023) First Evidence of Microplastic Occurrence in the Marine and Freshwater Environments in a Remote Polar Region of the Kola Peninsula and a Correlation with Human Presence. *Biology*, **12**, Article 259. <https://doi.org/10.3390/biology12020259>
- [34] (2025) The Impact of Fishing Gear as a Source of Marine Plastic Pollution: A Global Ghost Gear Initiative (GGGI) Information Paper to Support Negotiations in Preparation for UNEA 5.2. https://oceanconservancy.org/wp-content/uploads/2022/06/UNEA5.2_GGGI.pdf
- [35] (2025) Briefing Untangled Plastics Treaty Fishing Gear OC. https://www.oceancare.org/wp-content/uploads/2023/05/briefing-untangled-plastics-treaty-fishing-gear_OC_2023_long.pdf
- [36] (2025) Aquaculture. Oceana. <https://oceana.org/aquaculture/>
- [37] (2025) Environmental Impacts: Open Net-Pen Salmon Farms Benefit from the Ocean but the Ocean Doesn't Benefit from Salmon Farms. Living Oceans. <https://www.livingoceans.org/initiatives/salmon-farming/issues/environmental-impacts-open-net-pen-salmon-farms-benefit-the-ocean>
- [38] US EPA (2025) Best Management Practices for Flow-Through Aquaculture. <https://www.epa.gov/npdes/best-management-practices-flow-through-aquaculture>
- [39] Cassidy, M. (2019) What Is the Environmental Impact of Aquaculture? Global Seafood Alliance. <https://www.globalseafood.org/blog/what-is-the-environmental-impact-of-aquaculture/>
- [40] Stock, F., Kochleus, C., Bansch-Baltruschat, B., Brennholt, N. and Reifferscheid, G. (2019) Sampling Techniques and Preparation Methods for Microplastic Analyses in the Aquatic Environment—A Review. *TrAC Trends in Analytical Chemistry*, **113**, 84-92. <https://doi.org/10.1016/j.trac.2019.01.014>
- [41] Barcelo, D. and Kostianoy, A.G. (2025) The Handbook of Environmental Chemistry. Springer. <https://www.springer.com/series/0698>
- [42] Imhof, H.K., Ivleva, N.P., Schmid, J., Niessner, R. and Laforsch, C. (2013) Contamination of Beach Sediments of a Subalpine Lake with Microplastic Particles. *Current Biology*, **23**, R867-R868. <https://doi.org/10.1016/j.cub.2013.09.001>
- [43] Klein, S., Worch, E. and Knepper, T.P. (2015) Occurrence and Spatial Distribution of Microplastics in River Shore Sediments of the Rhine-Main Area in Germany. *Environmental Science & Technology*, **49**, 6070-6076.

- <https://doi.org/10.1021/acs.est.5b00492>
- [44] Corcoran, P.L., Biesinger, M.C. and Grifi, M. (2009) Plastics and Beaches: A Degrading Relationship. *Marine Pollution Bulletin*, **58**, 80-84. <https://doi.org/10.1016/j.marpolbul.2008.08.022>
- [45] Thomas, D., Schütze, B., Heinze, W.M. and Steinmetz, Z. (2020) Sample Preparation Techniques for the Analysis of Microplastics in Soil—A Review. *Sustainability*, **12**, Article 9074. <https://doi.org/10.3390/su12219074>
- [46] Lee, H., Kim, S., Sin, A., Kim, G., Khan, S., Nadagouda, M.N., *et al.* (2023) Pretreatment Methods for Monitoring Microplastics in Soil and Freshwater Sediment Samples: A Comprehensive Review. *Science of the Total Environment*, **871**, Article ID: 161718. <https://doi.org/10.1016/j.scitotenv.2023.161718>
- [47] Möller, J.N., Löder, M.G.J. and Laforsch, C. (2020) Finding Microplastics in Soils: A Review of Analytical Methods. *Environmental Science & Technology*, **54**, 2078-2090. <https://doi.org/10.1021/acs.est.9b04618>
- [48] Han, X., Lu, X. and Vogt, R.D. (2019) An Optimized Density-Based Approach for Extracting Microplastics from Soil and Sediment Samples. *Environmental Pollution*, **254**, Article ID: 113009. <https://doi.org/10.1016/j.envpol.2019.113009>
- [49] Razeghi, N., Hamidian, A.H., Wu, C., Zhang, Y. and Yang, M. (2021) Microplastic Sampling Techniques in Freshwaters and Sediments: A Review. *Environmental Chemistry Letters*, **19**, 4225-4252. <https://doi.org/10.1007/s10311-021-01227-6>
- [50] Radford, F., Zapata-Restrepo, L.M., Horton, A.A., Hudson, M.D., Shaw, P.J. and Williams, I.D. (2021) Developing a Systematic Method for Extraction of Microplastics in Soils. *Analytical Methods*, **13**, 1695-1705. <https://doi.org/10.1039/d0ay02086a>
- [51] Kononov, A., Hishida, M., Suzuki, K. and Harada, N. (2022) Microplastic Extraction from Agricultural Soils Using Canola Oil and Unsaturated Sodium Chloride Solution and Evaluation by Incineration Method. *Soil Systems*, **6**, Article 54. <https://doi.org/10.3390/soilsystems6020054>
- [52] Mattsson, K., Ekstrand, E., Granberg, M., Hassellöv, M. and Magnusson, K. (2022) Comparison of Pre-Treatment Methods and Heavy Density Liquids to Optimize Microplastic Extraction from Natural Marine Sediments. *Scientific Reports*, **12**, Article No. 15459. <https://doi.org/10.1038/s41598-022-19623-5>
- [53] Nakajima, R., Tsuchiya, M., Lindsay, D.J., Kitahashi, T., Fujikura, K. and Fukushima, T. (2019) A New Small Device Made of Glass for Separating Microplastics from Marine and Freshwater Sediments. *PeerJ*, **7**, e7915. <https://doi.org/10.7717/peerj.7915>
- [54] Habibi, N., Uddin, S., Fowler, S.W. and Behbehani, M. (2022) Microplastics in the Atmosphere: A Review. *Journal of Environmental Exposure Assessment*, **1**, 6. <https://doi.org/10.20517/jeea.2021.07>
- [55] Luo, X., Wang, Z., Yang, L., Gao, T. and Zhang, Y. (2022) A Review of Analytical Methods and Models Used in Atmospheric Microplastic Research. *Science of the Total Environment*, **828**, Article ID: 154487. <https://doi.org/10.1016/j.scitotenv.2022.154487>
- [56] Jarosz, K., Janus, R., Wądrzyk, M., Wilczyńska-Michalik, W., Natkański, P. and Michalik, M. (2022) Airborne Microplastic in the Atmospheric Deposition and How to Identify and Quantify the Threat: Semi-Quantitative Approach Based on Kraków Case Study. *International Journal of Environmental Research and Public Health*, **19**, Article 12252. <https://doi.org/10.3390/ijerph191912252>
- [57] Allen, S., Allen, D., Phoenix, V.R., Le Roux, G., Durántez Jiménez, P., Simonneau, A., *et al.* (2019) Atmospheric Transport and Deposition of Microplastics in a Remote Mountain Catchment. *Nature Geoscience*, **12**, 339-344.

- <https://doi.org/10.1038/s41561-019-0335-5>
- [58] Cai, L., Wang, J., Peng, J., Tan, Z., Zhan, Z., Tan, X., *et al.* (2017) Characteristic of Microplastics in the Atmospheric Fallout from Dongguan City, China: Preliminary Research and First Evidence. *Environmental Science and Pollution Research*, **24**, 24928-24935. <https://doi.org/10.1007/s11356-017-0116-x>
- [59] Dris, R., Gasperi, J., Saad, M., Mirande, C. and Tassin, B. (2016) Synthetic Fibers in Atmospheric Fallout: A Source of Microplastics in the Environment? *Marine Pollution Bulletin*, **104**, 290-293. <https://doi.org/10.1016/j.marpolbul.2016.01.006>
- [60] Gao, Z., Wontor, K. and Cizdziel, J.V. (2022) Labeling Microplastics with Fluorescent Dyes for Detection, Recovery, and Degradation Experiments. *Molecules*, **27**, Article 7415. <https://doi.org/10.3390/molecules27217415>
- [61] Maes, T., Jessop, R., Wellner, N., Haupt, K. and Mayes, A.G. (2017) A Rapid-Screening Approach to Detect and Quantify Microplastics Based on Fluorescent Tagging with Nile Red. *Scientific Reports*, **7**, Article No. 44501. <https://doi.org/10.1038/srep44501>
- [62] Pimpke, S., Christiansen, S.H., Cowger, W., De Frond, H., Deshpande, A., Fischer, M., *et al.* (2020) Critical Assessment of Analytical Methods for the Harmonized and Cost-Efficient Analysis of Microplastics. *Applied Spectroscopy*, **74**, 1012-1047. <https://doi.org/10.1177/0003702820921465>
- [63] Foster, B. (2024) Methods for Microplastics Detection. Applied Sciences from Technology Networks. <http://www.technologynetworks.com/applied-sciences/articles/methods-for-microplastics-detection-394279>
- [64] Abd Mutalib, M., Rahman, M.A., Othman, M.H.D., Ismail, A.F. and Jaafar, J. (2017) Scanning Electron Microscopy (SEM) and Energy-Dispersive X-Ray (EDX) Spectroscopy. In: Hilal, N., *et al.*, Eds., *Membrane Characterization*, Elsevier, 161-179. <https://doi.org/10.1016/b978-0-444-63776-5.00009-7>
- [65] Sun, X., Xiang, H., Xiong, H., Fang, Y. and Wang, Y. (2023) Bioremediation of Microplastics in Freshwater Environments: A Systematic Review of Biofilm Culture, Degradation Mechanisms, and Analytical Methods. *Science of the Total Environment*, **863**, Article ID: 160953. <https://doi.org/10.1016/j.scitotenv.2022.160953>
- [66] Furfaro, G., D'Elia, M., Mariano, S., Trainito, E., Solca, M., Piraino, S., *et al.* (2022) SEM/EDX Analysis of Stomach Contents of a Sea Slug Snacking on a Polluted Seafloor Reveal Microplastics as a Component of Its Diet. *Scientific Reports*, **12**, Article No. 10244. <https://doi.org/10.1038/s41598-022-14299-3>
- [67] Girão, A.V., Caputo, G. and Ferro, M.C. (2017) Application of Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDS). *Comprehensive Analytical Chemistry*, **75**, 153-168. <https://doi.org/10.1016/bs.coac.2016.10.002>
- [68] Tang, C.Y. and Yang, Z. (2017) Transmission Electron Microscopy (TEM). In: Hilal, N., *et al.*, Eds., *Membrane Characterization*, Elsevier, 145-159. <https://doi.org/10.1016/b978-0-444-63776-5.00008-5>
- [69] Mariano, S., Tacconi, S., Fidaleo, M., Rossi, M. and Dini, L. (2021) Micro and Nanoplastics Identification: Classic Methods and Innovative Detection Techniques. *Frontiers in Toxicology*, **3**, Article 636640. <https://doi.org/10.3389/ftox.2021.636640>
- [70] Carreras, H.Z. (2024) Cryo Electron Microscopy: Principle, Strengths, Limitations and Applications. Analysis & Separations from Technology Networks. <http://www.technologynetworks.com/analysis/articles/cryo-electron-microscopy-principle-strengths-limitations-and-applications-377080>
- [71] Jin, H., Kong, F., Li, X. and Shen, J. (2024) Artificial Intelligence in Microplastic

- Detection and Pollution Control. *Environmental Research*, **262**, Article ID: 119812. <https://doi.org/10.1016/j.envres.2024.119812>
- [72] Olivatto, G.P., Ando, R.A., Fernandes, R.F., de Moraes, N.G. and Tornisielo, V.L. (2024) A Critical Comparison of the Main Characterization Techniques for Microplastics Identification in an Accelerated Aging Laboratory Experiment. *Water Emerging Contaminants & Nanoplastics*, **3**, Article 7. <https://doi.org/10.20517/wecn.2023.69>
- [73] Cholewinski, A., Wortman, J., Hayashida, M., Anderson, W.A. and Zhao, B. (2024) 3D Imaging Photocatalytically Degraded Micro- and Nanoplastics. *Nanotechnology*, **35**, Article ID: 395706. <https://doi.org/10.1088/1361-6528/ad5dc5>
- [74] Yılmaz, E., Özgür, E., Akgönüllü, S., Özbek, M.A., Bereli, N., Yavuz, H., *et al.* (2024) Atomic Force Microscopy and Scanning Tunneling Microscopy of Live Cells. In: Denizli, A., Ed., *Biophysics At the Nanoscale*, Elsevier, 183-202. <https://doi.org/10.1016/b978-0-443-15359-4.00010-3>
- [75] Zeyer, T. and Fiener, P. (2020) Detection and Quantification of Microplastic in Soils Using a 3D Laser Scanning Confocal Microscope. *EGU General Assembly 2020*, 4-8 May 2020, EGU2020-3612. <https://doi.org/10.5194/egusphere-egu2020-3612>
- [76] Kumari, A., Yadav, M. and Bhatia, R. (2025) Analytical and Detection Techniques for Microplastics. In: Kataria, N., Garg, V.K., Han, C. and Rene, E.R., Eds., *Microplastic Pollution*, CRC Press, 147-166. <https://doi.org/10.1201/9781032706573-9>
- [77] Mir, D.H. and Rather, M.A. (2024) Advantages and Limitations of the Biofilm Study Methods. *Applied Biochemistry and Microbiology*, **60**, 264-279. <https://doi.org/10.1134/s000368382402011x>
- [78] Pipintakos, G., Hasheminejad, N., Lommaert, C., Bocharova, A. and Blom, J. (2021) Application of Atomic Force (AFM), Environmental Scanning Electron (ESEM) and Confocal Laser Scanning Microscopy (CLSM) in Bitumen: A Review of the Ageing Effect. *Micron*, **147**, Article ID: 103083. <https://doi.org/10.1016/j.micron.2021.103083>
- [79] Mukherjee, S. and Gowen, A. (2015) A Review of Recent Trends in Polymer Characterization Using Non-Destructive Vibrational Spectroscopic Modalities and Chemical Imaging. *Analytica Chimica Acta*, **895**, 12-34. <https://doi.org/10.1016/j.aca.2015.09.006>
- [80] Veerasingam, S., Ranjani, M., Venkatachalapathy, R., Bagaev, A., Mukhanov, V., Litvinyuk, D., *et al.* (2020) Contributions of Fourier Transform Infrared Spectroscopy in Microplastic Pollution Research: A Review. *Critical Reviews in Environmental Science and Technology*, **51**, 2681-2743. <https://doi.org/10.1080/10643389.2020.1807450>
- [81] Okoffo, E.D., O'Brien, S., O'Brien, J.W., Tschärke, B.J. and Thomas, K.V. (2019) Wastewater Treatment Plants as a Source of Plastics in the Environment: A Review of Occurrence, Methods for Identification, Quantification and Fate. *Environmental Science: Water Research & Technology*, **5**, 1908-1931. <https://doi.org/10.1039/c9ew00428a>
- [82] Brandon, J., Goldstein, M. and Ohman, M.D. (2016) Long-Term Aging and Degradation of Microplastic Particles: Comparing in Situ Oceanic and Experimental Weathering Patterns. *Marine Pollution Bulletin*, **110**, 299-308. <https://doi.org/10.1016/j.marpolbul.2016.06.048>
- [83] Corami, F., Rosso, B., Bravo, B., Gambaro, A. and Barbante, C. (2020) A Novel Method for Purification, Quantitative Analysis and Characterization of Microplastic Fibers Using Micro-FTIR. *Chemosphere*, **238**, Article ID: 124564.

- <https://doi.org/10.1016/j.chemosphere.2019.124564>
- [84] Chakraborty, I., Banik, S., Biswas, R., Yamamoto, T., Noothalapati, H. and Mazumder, N. (2022) Raman Spectroscopy for Microplastic Detection in Water Sources: A Systematic Review. *International Journal of Environmental Science and Technology*, **20**, 10435-10448. <https://doi.org/10.1007/s13762-022-04505-0>
- [85] Nava, V., Frezzotti, M.L. and Leoni, B. (2021) Raman Spectroscopy for the Analysis of Microplastics in Aquatic Systems. *Applied Spectroscopy*, **75**, 1341-1357. <https://doi.org/10.1177/00037028211043119>
- [86] Kniggendorf, A., Wetzel, C. and Roth, B. (2019) Microplastics Detection in Streaming Tap Water with Raman Spectroscopy. *Sensors*, **19**, Article 1839. <https://doi.org/10.3390/s19081839>
- [87] Hu, B., Dai, Y., Zhou, H., Sun, Y., Yu, H., Dai, Y., *et al.* (2024) Using Artificial Intelligence to Rapidly Identify Microplastics Pollution and Predict Microplastics Environmental Behaviors. *Journal of Hazardous Materials*, **474**, Article ID: 134865. <https://doi.org/10.1016/j.jhazmat.2024.134865>
- [88] Sunil, M., Pallikkavaliyaveetil, N., N, M., Gopinath, A., Chidangil, S., Kumar, S., *et al.* (2024) Machine Learning Assisted Raman Spectroscopy: A Viable Approach for the Detection of Microplastics. *Journal of Water Process Engineering*, **60**, Article ID: 105150. <https://doi.org/10.1016/j.jwpe.2024.105150>
- [89] Peez, N., Janiska, M. and Imhof, W. (2018) The First Application of Quantitative ¹H NMR Spectroscopy as a Simple and Fast Method of Identification and Quantification of Microplastic Particles (PE, PET, and PS). *Analytical and Bioanalytical Chemistry*, **411**, 823-833. <https://doi.org/10.1007/s00216-018-1510-z>
- [90] Schmidt, J., Haave, M., Underhaug, J. and Wang, W. (2024) Unlocking the Potential of NMR Spectroscopy for Precise and Efficient Quantification of Microplastics. *Microplastics and Nanoplastics*, **4**, Article No. 17. <https://doi.org/10.1186/s43591-024-00095-5>
- [91] Peñalver, R., Arroyo-Manzanares, N., López-García, I. and Hernández-Córdoba, M. (2020) An Overview of Microplastics Characterization by Thermal Analysis. *Chemosphere*, **242**, Article ID: 125170. <https://doi.org/10.1016/j.chemosphere.2019.125170>
- [92] Picó, Y. and Barceló, D. (2020) Pyrolysis Gas Chromatography-Mass Spectrometry in Environmental Analysis: Focus on Organic Matter and Microplastics. *TrAC Trends in Analytical Chemistry*, **130**, Article ID: 115964. <https://doi.org/10.1016/j.trac.2020.115964>
- [93] Fischer, M. and Scholz-Böttcher, B.M. (2019) Microplastics Analysis in Environmental Samples—Recent Pyrolysis-Gas Chromatography-Mass Spectrometry Method Improvements to Increase the Reliability of Mass-Related Data. *Analytical Methods*, **11**, 2489-2497. <https://doi.org/10.1039/c9ay00600a>
- [94] Khan, T. (2023) Assessing Microplastic Exposure with Thermal Analysis. AZoNano. <https://www.azonano.com/article.aspx?ArticleID=6382>
- [95] Majewsky, M., Bitter, H., Eiche, E. and Horn, H. (2016) Determination of Microplastic Polyethylene (PE) and Polypropylene (PP) in Environmental Samples Using Thermal Analysis (TGA-DSC). *Science of the Total Environment*, **568**, 507-511. <https://doi.org/10.1016/j.scitotenv.2016.06.017>
- [96] Fu, W., Min, J., Jiang, W., Li, Y. and Zhang, W. (2020) Separation, Characterization and Identification of Microplastics and Nanoplastics in the Environment. *Science of the Total Environment*, **721**, Article ID: 137561. <https://doi.org/10.1016/j.scitotenv.2020.137561>
- [97] Castelvetro, V., Corti, A., Ceccarini, A., Petri, A. and Vinciguerra, V. (2021) Nylon 6

- and Nylon 6,6 Micro- and Nanoplastics: A First Example of Their Accurate Quantification, along with Polyester (PET), in Wastewater Treatment Plant Sludges. *Journal of Hazardous Materials*, **407**, Article ID: 124364. <https://doi.org/10.1016/j.jhazmat.2020.124364>
- [98] Hoffmann, E. and de Stroobant, V. (2007) *Mass Spectrometry: Principles and Applications*. John Wiley & Sons.
- [99] Lin, Y., Huang, X., Liu, Q., Lin, Z. and Jiang, G. (2020) Thermal Fragmentation Enhanced Identification and Quantification of Polystyrene Micro/Nanoplastics in Complex Media. *Talanta*, **208**, Article ID: 120478. <https://doi.org/10.1016/j.talanta.2019.120478>
- [100] Zhang, J., Fu, D., Feng, H., Li, Y., Zhang, S., Peng, C., *et al.* (2024) Mass Spectrometry Detection of Environmental Microplastics: Advances and Challenges. *TrAC Trends in Analytical Chemistry*, **170**, Article ID: 117472. <https://doi.org/10.1016/j.trac.2023.117472>
- [101] Zhang, X., Mell, A., Li, F., Thaysen, C., Musselman, B., Tice, J., *et al.* (2020) Rapid Fingerprinting of Source and Environmental Microplastics Using Direct Analysis in Real Time-High Resolution Mass Spectrometry. *Analytica Chimica Acta*, **1100**, 107-117. <https://doi.org/10.1016/j.aca.2019.12.005>
- [102] Adhikari, S., Kelkar, V., Kumar, R. and Halden, R.U. (2022) Methods and Challenges in the Detection of Microplastics and Nanoplastics: A Mini-Review. *Polymer International*, **71**, 543-551. <https://doi.org/10.1002/pi.6348>
- [103] Jenkins, T., Persaud, B.D., Cowger, W., Szigeti, K., Roche, D.G., Clary, E., *et al.* (2022) Current State of Microplastic Pollution Research Data: Trends in Availability and Sources of Open Data. *Frontiers in Environmental Science*, **10**, Article 912107. <https://doi.org/10.3389/fenvs.2022.912107>
- [104] Prata, J.C., Padrão, J., Khan, M.T. and Walker, T.R. (2024) Do's and Don'ts of Microplastic Research: A Comprehensive Guide. *Water Emerging Contaminants & Nanoplastics*, **3**, Article 8. <https://doi.org/10.20517/wecn.2023.61>
- [105] Pimpke, S., Booth, A.M., Gerdts, G., Gomiero, A., Kögel, T., Lusher, A., *et al.* (2023) Monitoring of Microplastic Pollution in the Arctic: Recent Developments in Polymer Identification, Quality Assurance and Control, and Data Reporting. *Arctic Science*, **9**, 176-197. <https://doi.org/10.1139/as-2022-0006>
- [106] Huang, Z., Hu, B. and Wang, H. (2022) Analytical Methods for Microplastics in the Environment: A Review. *Environmental Chemistry Letters*, **21**, 383-401. <https://doi.org/10.1007/s10311-022-01525-7>
- [107] Ivleva, N.P. (2021) Chemical Analysis of Microplastics and Nanoplastics: Challenges, Advanced Methods, and Perspectives. *Chemical Reviews*, **121**, 11886-11936. <https://doi.org/10.1021/acs.chemrev.1c00178>
- [108] Jin, M., Liu, J., Yu, J., Zhou, Q., Wu, W., Fu, L., *et al.* (2022) Current Development and Future Challenges in Microplastic Detection Techniques: A Bibliometrics-Based Analysis and Review. *Science Progress*, **105**, 1-22. <https://doi.org/10.1177/00368504221132151>
- [109] Workman, J. (2024) A Review of Spectroscopic Techniques Used for the Quantification and Classification of Microplastics and Nanoplastics in the Environment. *Spectroscopy*. <https://doi.org/10.56530/spectroscopy.ac7567r4>
- [110] Awada, A., Potter, M., Aherne, J., Lavoie-Bernstein, S., Diamond, M.L., Helm, P.A., *et al.* (2025) Facile Detection of Microplastics from a Variety of Environmental Samples with Conjugated Polymer Nanoparticles. *Environmental Science: Advances*, **4**, 270-

278. <https://doi.org/10.1039/d4va00239c>
- [111] Rivera-Rivera, D.M., Quintanilla-Villanueva, G.E., Luna-Moreno, D., Sánchez-Álvarez, A., Rodríguez-Delgado, J.M., Cedillo-González, E.I., et al. (2025) Exploring Innovative Approaches for the Analysis of Micro- and Nanoplastics: Breakthroughs in (Bio)sensing Techniques. *Biosensors*, **15**, Article 44. <https://doi.org/10.3390/bios15010044>
- [112] Murugan, P., Sivaperumal, P., Balu, S., Arya, S., Atchudan, R. and Sundramoorthy, A.K. (2023) Recent Advances on the Methods Developed for the Identification and Detection of Emerging Contaminant Microplastics: A Review. *RSC Advances*, **13**, 36223-36241. <https://doi.org/10.1039/d3ra05420a>
- [113] Ang, A.L.T., Jose, C.M.D., Del Rosario, C.I.P., Uy, O.L.T. and Garcia, J. (2022) Recent Advances on Density Separation Techniques for Microplastic Recovery from Sediments. *Sinaya: A Philippine Journal for Senior High School Teachers and Students*, **1**, Article 1. <https://doi.org/10.59588/3027-9283.1010>
- [114] Courtene-Jones, W., Quinn, B., Murphy, F., Gary, S.F. and Narayanaswamy, B.E. (2017) Optimisation of Enzymatic Digestion and Validation of Specimen Preservation Methods for the Analysis of Ingested Microplastics. *Analytical Methods*, **9**, 1437-1445. <https://doi.org/10.1039/c6ay02343f>
- [115] Wang, S., Mintenig, S.M., Wu, J. and Koelmans, A.A. (2026) Implications of Method-And Instrument-Based Size Detection Limits in MFTIR-Based Microplastic Analysis. *Talanta*, **296**, Article ID: 128417. <https://doi.org/10.1016/j.talanta.2025.128417>
- [116] Papini, G., Petrella, G., Cicero, D.O., Boglione, C. and Rakaj, A. (2024) Identification and Quantification of Polystyrene Microplastics in Marine Sediments Facing a River Mouth through NMR Spectroscopy. *Marine Pollution Bulletin*, **198**, Article ID: 115784. <https://doi.org/10.1016/j.marpolbul.2023.115784>
- [117] Sorolla-Rosario, D., Llorca-Porcel, J., Pérez-Martínez, M., Lozano-Castelló, D. and Bueno-López, A. (2022) Study of Microplastics with Semicrystalline and Amorphous Structure Identification by TGA and DSC. *Journal of Environmental Chemical Engineering*, **10**, Article ID: 106886. <https://doi.org/10.1016/j.jece.2021.106886>
- [118] Vitali, C., Janssen, H., Ruggeri, F.S. and Nielen, M.W.F. (2022) Rapid Single Particle Atmospheric Solids Analysis Probe-Mass Spectrometry for Multimodal Analysis of Microplastics. *Analytical Chemistry*, **95**, 1395-14001. <https://doi.org/10.1021/acs.analchem.2c04345>
- [119] Lamprecht, A., Schäfer, U. and Lehr, C. (2000) Structural Analysis of Microparticles by Confocal Laser Scanning Microscopy. *AAPS PharmSciTech*, **1**, 10-19. <https://doi.org/10.1208/pt010317>