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A Review of Environmental Microplastic Detection Methods

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Abstract: Microplastic detection provides an essential basis for investigating environmental contamination, elucidating transport patterns, assessing human exposure, and evaluating ecological risks. In recent years, research has progressed from early visual screening and single-particle identification to an integrated analytical framework that combines sample collection, matrix separation, chemical composition confirmation, particle counting, polymer mass quantification, and automated data processing. This review summarizes recent advances in microplastic detection over the past five years and discusses the collection and pretreatment of samples from water, sediments, soils, organisms, and air. Particular attention is given to the principles, applicability, and limitations of micro-Fourier transform infrared spectroscopy, micro-Raman spectroscopy, pyrolysis-gas chromatography-mass spectrometry, scanning electron microscopy, fluorescence staining, and flow cytometry. Particle number concentration and polymer mass concentration, minimum detectable particle size, analytical throughput, degree of automation, and applicability to complex matrices are further compared. Current studies show that micro-infrared and micro-Raman techniques are suitable for particle-level identification and morphological analysis, whereas pyrolysis mass spectrometry is more appropriate for polymer mass quantification in complex matrices. Fluorescence-based and flow-cytometric methods also show potential for rapid screening and high-throughput detection. Future work should further standardize sampling, pretreatment, blank control, recovery assessment, and result reporting, and establish a complementary multi-technique system that provides comparable data and is suitable for routine monitoring.

Keywords: Microplastics, micro-Fourier transform infrared spectroscopy, micro-Raman spectroscopy, pyrolysis mass spectrometry, fluorescence detection, automated analysis.

1. Introduction

Microplastics are generally defined as plastic particles smaller than 5 mm. They include primary microplastics released directly from industrial production and consumer products, as well as secondary microplastics formed through the gradual fragmentation of larger plastic products under mechanical abrasion, ultraviolet radiation, oxidative aging, and biological processes. Because microplastics differ substantially in polymer type, particle size, color, morphology, density, and degree of aging, their detection involves more than simply determining whether plastic is present in a sample. It also requires a series of analytical steps, including particle separation, morphological characterization, polymer identification, and content determination.

For microplastic detection in clean water samples, Schymanski et al. systematically reviewed key aspects of sample preparation, spectroscopic analysis, and quality assurance, and noted that sampling volume, filter type, laboratory background contamination, and the minimum detectable particle size can all affect the analytical results [1]. From an environmental analysis perspective, Huang Z et al. compared spectroscopic, thermal, and microscopic imaging methods and concluded that particle number, particle size, and mass concentration obtained using different methods are not equivalent [2]. Sharma et al. further considered sampling, separation, and characterization as an integrated analytical chain and emphasized that particle loss during pretreatment may be as important as instrumental error [3]. Huang M et al. reviewed the transition from qualitative to quantitative analysis and noted that microplastic detection is progressing from single-particle identification toward integrated characterization of particle abundance, polymer mass, and particle-size distribution [4]. In the field of drinking water, the World Health Organization summarized analytical methods for microplastics and issues related to data interpretation [5]. Belz et al. further discussed methods for measuring microplastics in drinking water in a technical report of the European Commission Joint Research Centre and emphasized the need to clearly specify the analyzed particle-size range, polymer types, reporting units, and quality-control procedures [6]. These studies indicate that even when different laboratories analyze the same type of water sample, the measured microplastic abundance may vary considerably if the sampling volume, filter pore size, and instrumental resolution differ.

As research has advanced, increasing attention has been paid to blank contamination, recovery assessment, and method validation in microplastic analysis. Prata et al. recommended minimizing the use of plastic consumables and including field blanks, transport blanks, and procedural blanks. For automated spectral identification, the source of the spectral library, matching threshold, and manual verification procedure should also be reported [7]. In regulatory practice, the California State Water Resources Control Board has promoted standardized monitoring of microplastics in drinking water [8], and the European Commission Joint Research Centre has established a corresponding measurement framework [9].

Overall, current microplastic detection mainly addresses four questions: whether plastics are present in a sample and which polymers they contain; the number, size, and morphology of the particles; the mass concentration of each polymer; and whether results are comparable across laboratories and environmental matrices. Because a single instrument cannot provide all of this information, current

research has shifted from seeking a single "best method" toward developing complementary analytical systems according to the sample matrix, target particle size, and analytical purpose.

2. Sample Collection and Pretreatment

2.1. Collection of Samples from Different Environmental Matrices

The sampling procedure determines the spatial range and particle-size range covered by subsequent analysis. Surface marine waters are commonly sampled by net towing, which enables floating particles to be collected rapidly over a relatively large area. However, because the minimum particle size retained is limited by the mesh size, this approach is generally more suitable for microplastics larger than several hundred micrometers. For freshwater, drinking water, and other low-turbidity samples, pump sampling, grab sampling, and large-volume online filtration can cover smaller particle sizes and improve the probability of detecting target particles in low-concentration samples. De Frond et al. organized an interlaboratory comparison of microplastics in drinking water. Their results showed that even when similar samples and basic analytical procedures were used, differences in sample filtration, spectral scanning, particle identification, and data processing among laboratories still affected the final results [10]. Therefore, sampling volume and the minimum analyzed particle size should be reported together with the detection results; otherwise, direct comparisons of particle abundance may be misleading.

Sediment and soil samples are generally collected using grab samplers, corers, or stainless-steel shovels. The sampled area, depth, wet mass, and dry mass should be recorded so that the results can be converted to consistent units. For biological samples, gastrointestinal contents should be distinguished from other tissues according to the research objective to avoid confusing ingested particles with particles present within tissues. Hagelskjær et al. analyzed drinking water using an automated Raman method and found that a considerable proportion of the particles were smaller than 20 μm, indicating that methods covering only larger particle sizes may systematically underestimate the number of microplastics in water samples [11]. Miserli et al. combined Fourier transform infrared spectroscopy (FTIR), micro-Raman spectroscopy, and scanning electron microscopy-energy-dispersive spectroscopy (SEM-EDS) to screen microplastics in fish, mussels, and aquaculture water. Their study showed that biological samples often require cross-validation using multiple techniques to reduce misidentification caused by tissue residues and inorganic particles [12]. Airborne microplastics are mainly collected by passive deposition or active air filtration. Passive sampling reflects particle deposition over a given period, whereas active sampling provides the particle concentration per unit volume of air. Because laboratory air, clothing, and plastic equipment may release fibers, air samples generally require stricter control of sampling media and background contamination than water samples.

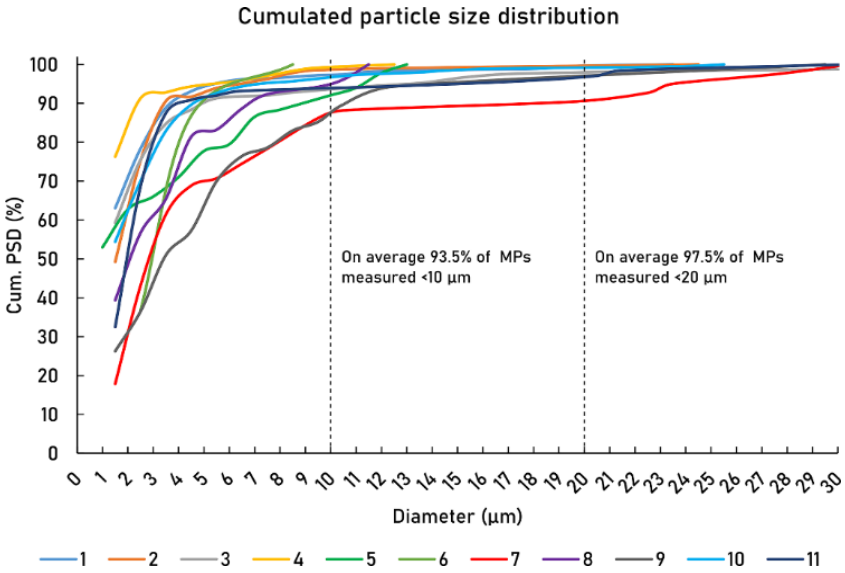

Figure 1. Cumulative particle-size distribution of microplastics in drinking water [11]

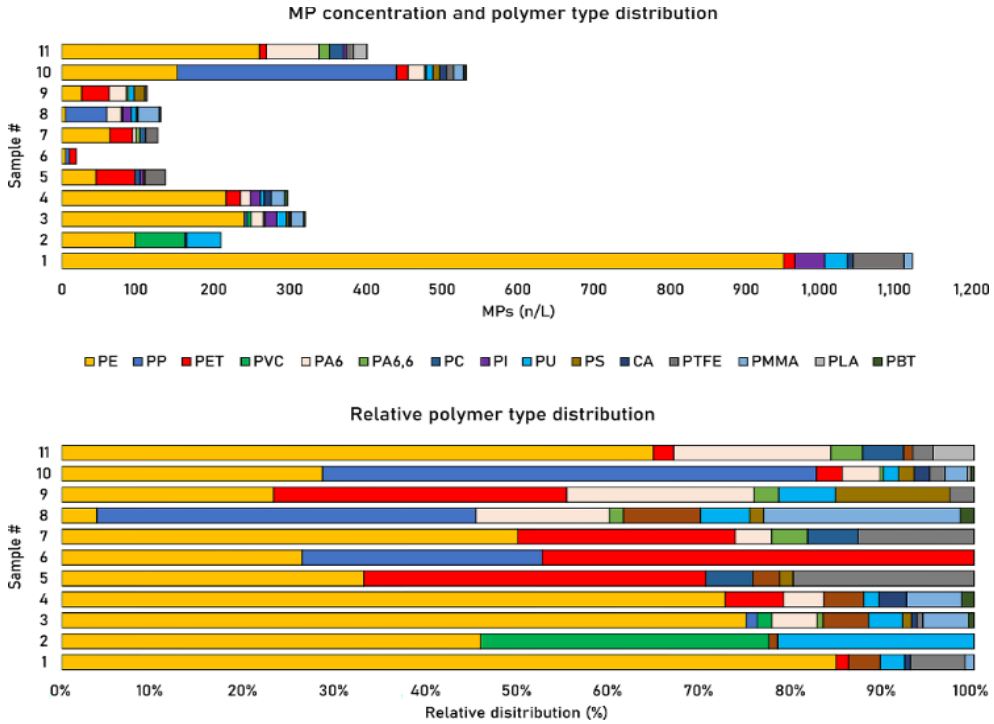

Figure 2. Microplastic concentrations and polymer-type distributions in different drinking water samples [11]

2.2. Separation, Digestion, and Enrichment

The main purpose of microplastic pretreatment is to remove the environmental matrix while preserving the chemical structure and morphology of the polymers as far as possible, rather than simply obtaining a completely clean filter surface. Drinking water and clean water samples with low organic-matter contents can generally be filtered directly and then analyzed spectroscopically, whereas samples containing larger amounts of algae, suspended particles, or humic substances require moderate digestion [2-3]. Microplastics in sediments, soils, and sludge generally require density separation. NaCl solution is inexpensive and relatively low in toxicity and is suitable for separating low-density plastics such as polyethylene and polypropylene, but it is less effective for high-density polymers such as polyvinyl chloride and polyethylene terephthalate. $ZnCl_2$ and NaI solutions have higher densities and can improve the recovery of high-density plastics, but they are more expensive and corrosive and create additional challenges for waste-liquid treatment. The separation solution should therefore be selected according to the target polymer rather than solely for operational convenience.

Organic matter is commonly digested using H_2O_2, Fenton reagent, KOH, acid or alkaline digestion, or enzymatic treatment [2-3]. H_2O_2 and Fenton systems effectively remove natural organic matter and provide a relatively good balance between digestion efficiency and polymer preservation. KOH is commonly used to treat biological tissues such as fish and shellfish, although excessively high temperatures or prolonged treatment may affect some polymers. Enzymatic digestion better preserves particle morphology and chemical structure and is suitable for biological samples and samples with high organic-matter contents. However, its high cost and long processing time have limited its routine use in many laboratories.

More pretreatment steps do not necessarily produce better results. Repeated transfer, filtration, centrifugation, and washing can lead to cumulative particle losses, particularly for small particles and fibers [22]. For complex samples, spiked-recovery experiments should therefore be used to evaluate separation and digestion, and the recovery of different particle sizes, morphologies, and polymer types should be assessed separately [3, 7]. The use of only regular spherical particles as spiking materials may not accurately represent the losses of environmental fibers and irregular fragments during pretreatment.

2.3. Contamination Control and Quality Assurance

Because microplastics are widely present in laboratory air, plastic containers, and synthetic-fiber clothing, quality control should cover the entire process of sampling, transport, pretreatment, and instrumental analysis [1, 7, 10]. Glass, metal, and aluminum-foil materials should be used whenever possible; reagents and laboratory water should be filtered in advance; and sample containers should be thoroughly cleaned and kept covered before use. Laboratory personnel should wear cotton laboratory coats and minimize the time for which samples are exposed to air [1, 7]. At a minimum, field blanks and procedural blanks should be included. For low-concentration samples such as air and drinking water, transport blanks and laboratory deposition blanks should also be used. Blank results should not only indicate whether the laboratory environment is sufficiently clean but should also be used to evaluate the reliability of the sample results. If the number of particles in the blanks is close to that in the samples, the reported concentration should be interpreted cautiously even when polymer identification has been completed. Quality control for automated spectral identification should also include the source of the spectral library, spectral preprocessing, the matching threshold, the proportion of unidentified particles, and the manual verification procedure [34]. Comparability among studies can be improved only when sampling error, pretreatment loss, and instrumental identification error are included within the same quality-assurance framework.

Table 1. Sampling and pretreatment of different sample matrices

Sample type	Common sampling methods	Pretreatment steps	Major interferences
Marine water and freshwater	Net towing, pump sampling, grab sampling, and large-volume filtration	Filtration, oxidative digestion, and refiltration	Algae, organic debris, and salt crystallization
Drinking water	Online filtration and large-volume filtration	Direct filtration or mild oxidation	Low concentration and laboratory background contamination
Sediments and soils	Grab sampling, coring, and stratified sampling	Density separation, digestion, and filtration	Mineral particles and high-density plastics
Sludge and wastewater	Fixed-volume sampling and centrifugation	Digestion, centrifugation or filtration, and concentration	Organic matter, particle overlap, and additives
Biological samples	Isolation of the gastrointestinal tract or tissues	KOH digestion, enzymatic digestion, and oxidation	Lipids, proteins, and tissue residues
Air samples	Passive deposition and active filtration	Filter-based enrichment and direct thermal analysis when necessary	Fiber background and sampler contamination

3. Detection and Characterization Techniques

3.1. Optical Microscopy and Vibrational Spectroscopy

Optical and stereomicroscopes are mainly used for preliminary screening based on particle color, transparency, shape, and surface characteristics. These methods are simple and inexpensive and are suitable for rapid counting of relatively large particles, but they cannot directly identify polymer types. Natural fibers, plant fragments, mineral particles, and coating debris may resemble microplastics in appearance. Visual identification can therefore only be used as a prescreening step and cannot replace chemical confirmation. Micro-Fourier transform infrared spectroscopy identifies polymers from characteristic absorption bands associated with molecular vibrations. Conventional single-point micro-FTIR is suitable for examining suspected particles individually, whereas focal plane array Fourier transform infrared (FPA-FTIR) imaging, laser direct infrared (LDIR) imaging, and whole-filter scanning can simultaneously provide particle position, size, morphology, and chemical composition, thereby improving the efficiency of large sample sets. Primpke et al. compared hyperspectral FTIR imaging and pyrolysis-gas chromatography-mass spectrometry (Py-GC/MS) for microplastic analysis. FTIR imaging retained information on particle number, size, and morphology, whereas Py-GC/MS was more suitable for polymer mass determination, indicating that the two techniques provide complementary information [13]. Maurizi et al. compared μFTIR and μRaman for microplastics in drinking water and found that the minimum detectable particle size of each instrument directly affected the measured particle abundance and polymer composition. The results should therefore be interpreted within the particle-size range covered by the method [14].

Machine learning has increasingly been used to address the large volume of spectral data and the time required for manual identification of environmental microplastics. Zhu et al. applied deep learning to automated microplastic recognition in FPA-μFTIR images, enabling the model to extract polymer features from large spectral datasets and reducing the effects of particle thickness and spectral variation on classification [15]. Hufnagl et al. combined μFTIR imaging with machine learning to establish a computer-assisted analytical workflow, thereby improving the efficiency of suspected-particle screening and spectral classification in environmental samples [16]. For Raman analysis, Weber et al. trained a machine-learning model using μ-Raman data obtained from environmental samples, reducing the dependence of conventional spectral-library matching on manual experience [17]. Zhang Z et al. combined μ-FTIR and μ-Raman analysis, using infrared spectroscopy for rapid analysis of larger particles and Raman spectroscopy to confirm smaller or difficult-to-identify particles [18]. This strategy combines the higher throughput of FTIR with the higher spatial resolution of Raman spectroscopy and can reduce particle-size bias associated with the use of a single method.

Micro-Raman spectroscopy generally covers smaller particles than micro-FTIR and is less affected by water, making it suitable for drinking water, air samples, and fine microplastics. However, Raman spectra are readily affected by fluorescence from dyes, aged surface layers, biofilms, and natural organic matter. In their evaluation of μ-Raman spectroscopy as a routine method for determining microplastics in drinking water, Barchiesi et al. noted that, despite its high spatial resolution, a balance must still be achieved among spectral quality, scanning time, degree of automation, and misidentification rate [19].

3.2. Thermal Analysis and Mass Spectrometry

Thermal methods identify and quantify polymers by decomposing them at high temperature and analyzing their characteristic pyrolysis products. Py-GC/MS is currently one of the most widely used mass-based analytical techniques. Unlike microspectroscopic methods, Py-GC/MS does not require particles to retain their original morphology and is less sensitive to particle size and color, making it particularly suitable for complex matrices such as sludge, sediments, airborne particles, and nanoplastics. Zhang J et al. reviewed the use of mass spectrometry for environmental microplastic detection and noted that high-resolution mass spectrometry and non-target screening are extending the scope of conventional Py-GC/MS, potentially allowing the simultaneous analysis of polymers, additives, and other coexisting chemicals [20]. However, thermal analysis provides the total mass of each polymer in a sample and cannot simultaneously provide particle number, size, and morphology. To obtain both types of information, Lee et al. developed a sequential FTIR and Py-GC/MS method in which microplastics in municipal wastewater were first counted and identified by polymer type and were then quantified by mass [21]. Their study demonstrated that number concentration and mass concentration can be obtained from the same sample by combining methods, although mass estimated from particle dimensions may differ from that measured by pyrolysis, particularly when irregular and small particles are abundant. Pretreatment also has an important influence on pyrolysis-

based quantification. When analyzing polypropylene and polystyrene in wastewater, Lykkemark et al. found that excessive purification and transfer steps could cause losses of the target polymers [22]. Pretreatment for Py-GC/MS should therefore prioritize sample enrichment and removal of major interferences rather than continuously adding processing steps to obtain a visually clean sample.

For airborne microplastics, Pipkin et al. used an optimized pyrolysis-gas chromatography-mass spectrometry method to analyze passively sampled indoor airborne particles and achieved nanogram-level mass detection [23]. Torres-Agullo et al. used pyrolysis-gas chromatography-Orbitrap high-resolution mass spectrometry (Pyr-GC-Orbitrap-MS) for targeted and non-targeted analysis of air samples, allowing known polymers and unknown pyrolysis products to be screened on the same platform [24]. For microplastics and nanoplastics in aquatic environments, Xu et al. used Py-GC/MS to assess changes in polymer mass across different treatment units of wastewater treatment plants and analyzed their removal from a mass-balance perspective [25]. Okoffo and Thomas combined ultrafiltration, digestion, and Py-GC/MS to quantify nanoplastics in environmental and potable waters [26]. These studies show that when particles fall below the reliable size range of microscopic imaging, pyrolysis mass spectrometry can complement microscopic methods by providing total polymer mass.

3.3. Fluorescence, Flow Cytometry, and Electron Microscopy

Fluorescence staining and flow cytometry represent important directions for rapid screening and high-throughput counting of microplastics. Lipophilic dyes such as Nile Red can bind to the surfaces of some plastics and generate strong fluorescence under specific excitation conditions, thereby increasing the contrast between plastic particles and the background. However, fluorescent dyes may also bind to lipids, natural organic matter, and other hydrophobic particles, and identification based only on fluorescence signals can therefore produce false positives. Li J et al. experimentally optimized the separation and flow-cytometric analysis of microplastics and nanoplastics. Flow cytometry can rapidly count individual particles in a liquid from the scattering and fluorescence signals generated as they pass through the detection region and has the potential to analyze submicrometer particles [27]. However, scattering intensity is also influenced by particle size, morphology, and refractive index, and cannot independently provide accurate polymer identification. Bianco et al. combined Nile Red staining with flow cytometry for the rapid detection of nanoplastics and small microplastics [28]. The fluorescence signal improved the distinction between target particles and the background and increased the detection efficiency for small particles, although dye concentration, reaction time, and interference from natural organic matter still required careful control. Li C et al. further developed a high-throughput flow-cytometric method for microplastics in water, enabling multiple samples to be counted within a short period and demonstrating its potential for screening large numbers of water samples [29].

In contrast to these individual method studies, Morgana et al. broadly reviewed fluorescence-based methods for the analysis of microplastics and nanoplastics and concluded that fluorescence imaging, fluorescent probes, and flow-cytometric analysis can increase detection speed but still cannot independently confirm polymer identity [30]. A more appropriate workflow is therefore to use fluorescence or flow cytometry for rapid screening and then confirm representative particles by FTIR, Raman spectroscopy, or thermal analysis. Scanning electron microscopy can reveal surface cracks, pores, abrasion marks, biofilms, and attached materials, while energy-dispersive spectroscopy provides elemental information. Webb et al. used simultaneous backscattered-electron and X-ray imaging (SEM-BEX) to analyze microplastics and other contaminant particles in complex samples and demonstrated its advantages for in situ morphological screening and elemental characterization [31]. However, elemental composition does not directly represent polymer molecular structure, and SEM-EDS or SEM-BEX cannot generally be used alone for definitive plastic identification. For microplastic and nanoplastic detection in biological samples, Zhao et al. identified incomplete tissue digestion, small particle size, and complex biological matrices as major factors affecting analytical reliability [32]. For such samples, cross-validation among microspectroscopy, electron microscopy, and thermal analysis is more effective than reliance on a single technique for excluding tissue residues and inorganic particles.

Table 2. Performance comparison of major microplastic detection techniques

Detection technique	Typical particle size or sensitivity	Main advantages	Main limitations
Optical microscopy	Generally applicable to particles larger than 20–50 μm	Low cost and rapid operation	Cannot confirm polymer identity; relatively high misidentification rate
Micro-FTIR/FPA-FTIR	Generally applicable to particles larger than approximately 10–20 μm	Whole-filter scanning and a relatively high degree of automation	Limited identification of very small and overlapping particles
Micro-Raman spectroscopy	Down to approximately 1 μm	High spatial resolution and suitability for small particles	Strong fluorescence interference and long scanning time
Py-GC/MS	Mass detection at the ng–μg level	Suitable for complex matrices and nanoplastics	Destructive analysis; no particle morphology or number information
Fluorescence staining	Screening from the micrometer to submicrometer range	Rapid and relatively inexpensive	Nonspecific staining; further confirmation is required
Flow cytometry	Can cover particles of approximately 200 nm and larger	High throughput and suitability for liquid-phase analysis	Limited ability to identify polymer types
SEM-EDS	Micrometer-scale morphological analysis	High resolution and the ability to observe aging features	Cannot independently identify polymers

4. Comparison and Discussion

The selection of a microplastic detection method should be guided primarily by the required reporting endpoint rather than by instrumental sensitivity alone. Particle-resolved techniques, including micro-FTIR and micro-Raman spectroscopy, provide information on particle abundance, size, morphology, and polymer identity, whereas Py-GC/MS measures the mass of individual polymers. These outputs are complementary rather than interchangeable because particle number cannot be reliably converted into mass without assumptions regarding particle geometry and density. Bouzid et al. demonstrated this distinction by sequentially applying micro-FTIR imaging and Py-GC/MS to river sediments. Although the two methods produced broadly consistent contamination patterns, their responses differed for fibers, irregular fragments, and small particles [33].

Data comparability is also determined by the fraction of the particle population that an analytical workflow can observe. Differences in filter pore size, lower particle-size limit, image segmentation, spectral preprocessing, and library-matching thresholds may alter both the measured abundance and the apparent polymer composition. Studies should therefore report the minimum analyzed and reported particle sizes, the proportion of unidentified particles, and the principal data-processing criteria. Cowger et al. showed that image processing and spectral-classification procedures are not neutral post-processing steps but important sources of analytical variability [34]. This issue is particularly relevant to machine-learning models, whose performance may decline when aged, colored, or matrix-coated particles are poorly represented in the training data.

No analytical method is universally superior. Microspectroscopy is more informative when particle-level exposure metrics are required, whereas thermal analysis is better suited to polymer mass balances and highly complex matrices. Rapid screening methods can reduce the analytical workload, but positive results generally require chemical confirmation. Electrochemical approaches may support miniaturized field analysis, although limitations in selectivity, matrix interference, and quantitative calibration remain unresolved [35]. More importantly, the interlaboratory comparison reported by Ciornii et al. showed that nominally similar μFTIR, μRaman, and LDIR workflows may still produce different results because of variations in scanning strategy, spectral libraries, and identification thresholds [36]. Method performance should therefore be evaluated in terms of fitness for purpose, transparent reporting, and interlaboratory reproducibility rather than detection limit alone.

5. Current Challenges and Future Directions

Current microplastic detection still faces several challenges, including inconsistent sampling and reporting practices, difficult pretreatment of complex matrices, insufficient capability for detecting small particles, and poor comparability among laboratories. Studies differ substantially in sampling volume, mesh size, filter pore size, particle-size classification, and reporting units, making direct comparison among regions and methods difficult. During pretreatment, intensive digestion may reduce interference from organic matter and biological tissues but can also fragment particles, alter surface structures, or cause polymer loss, whereas overly mild treatment may leave matrix residues that interfere with subsequent spectral identification. Small microplastics and nanoplastics remain particularly difficult to detect. Micro-FTIR is limited by spatial resolution, micro-Raman is susceptible to fluorescence background, and thermal analysis can determine total polymer mass but cannot retain information on particle number, size, and morphology. Automated identification and machine-learning methods are also limited by the coverage of their training data and their applicability to environmental samples, and their actual performance under conditions involving aged plastics, colored particles, and complex backgrounds still requires further validation.

Future microplastic detection systems should be improved through method standardization, complementary techniques, and intelligent data analysis. Basic requirements for sampling information, blank control, recovery assessment, minimum detectable particle size, and reporting units should be unified, and spiked-recovery tests should be conducted separately for different polymers, particle sizes, and morphologies. A tiered analytical workflow should also be established. Micro-FTIR or micro-Raman can first be used to obtain particle number, size, morphology, and polymer type, after which representative samples can be analyzed by Py-GC/MS for mass quantification. High-resolution mass spectrometry, field-flow fractionation, and fluorescence detection can further improve the analysis of small particles. Standard spectral libraries and reference materials should be expanded to include environmentally aged plastics, irregular fragments, fibers, and materials containing additives, and machine-learning models should be validated using real environmental samples and interlaboratory studies. Standardized sampling, complementary analytical techniques, harmonized spectral libraries, and interlaboratory quality assessment can gradually improve the accuracy, stability, and comparability of microplastic detection.

6. Conclusion

Microplastic analysis is a chain of coupled decisions rather than a single instrumental measurement. Sampling design, matrix removal, particle recovery, chemical confirmation, and data processing jointly determine the reliability of the final result. Particle-resolved microspectroscopy and mass-based thermal analysis answer different analytical questions and should therefore be regarded as complementary rather than competing approaches. Rapid screening and automated classification can improve analytical throughput, but they do not remove the need for chemical confirmation, quality control, and transparent reporting.

No single method is suitable for every environmental matrix or analytical objective. Progress toward routine monitoring will depend less on further reductions in instrumental detection limits than on the harmonization of sampling, blank control, recovery assessment, particle-size reporting, and data-processing criteria. A tiered workflow combining standardized sampling, particle-level screening, and targeted mass confirmation provides a practical route to more comparable results. Ultimately, interlaboratory reproducibility will determine whether microplastic measurements can reliably support pollution assessment, ecological risk evaluation, and environmental management.

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