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Nanotechnology-Based Analytical Techniques for Detection and Characterisation of Microplastics in Aquatic Environments

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Microplastic contamination in aquatic environments has emerged as one of the most emerging environmental challenges of the twenty-first century. Conventional analytical methods often fall short in sensitivity, throughput, and the capacity to detect nanoscale plastic fragments. Nanotechnology-based analytical techniques have recently demonstrated considerable promise in addressing these limitations. This paper presents a comprehensive technical review of nanotechnology-driven approaches for the detection and characterisation of microplastics in aquatic matrices, including surface-enhanced Raman scattering (SERS) using plasmonic nanomaterials, nano-biosensor platforms, nanomaterial-integrated electrochemical sensors, atomic force microscopy coupled with infrared spectroscopy (AFM-IR), nanoparticle tracking analysis (NTA), and total internal reflection fluorescence (TIRF) microscopy. The operational principles, detection limits, size-range capabilities, and comparative advantages of each technique are discussed. Key challenges including matrix interference, standardisation, and scalability are also addressed, followed by future research directions aimed at developing integrated, miniaturised sensor platforms for real-time aquatic monitoring.

Keywords: microplastics, nanotechnology, SERS, AFM-IR, nano-biosensors, aquatic pollution, electrochemical sensors.

Introduction

Microplastics, defined as plastic particles or fragments with a diameter of less than 5 mm, have been identified in virtually every aquatic habitat on Earth, from surface freshwater systems to deep-sea sediments and marine food webs. Their persistence, capacity to adsorb toxic organic pollutants, and potential to traverse biological membranes position them as a multidimensional ecological and public health threat. The accurate quantification and physicochemical characterisation of microplastics in water matrices is therefore a scientific and regulatory priority. Conventional analytical workflows, which combine physical separation steps such as density flotation, filtration, and visual sorting with spectroscopic identification by Fourier-transform infrared spectroscopy (FTIR) or Raman spectroscopy, have advanced the field considerably. Nevertheless, they suffer from inherent constraints when applied to nanoscale plastic particles (< 1 mm), present in complex water matrices laden with organic matter, suspended sediments, and biological debris. Detection sensitivity is limited, sample throughput is low, and many benchtop instruments preclude field deployment.

Nanotechnology offers a fundamentally different paradigm. By exploiting the unique optical, electronic, and physicochemical properties of nanomaterials, researchers have engineered sensor platforms capable of detecting microplastics at trace concentrations with high selectivity, often in real time and with the potential for miniaturisation into portable devices. This paper systematically reviews the most impactful nanotechnology-based techniques currently applied to microplastic detection and characterisation in aquatic environments, evaluates their comparative performance, and discusses the challenges and opportunities that define the next generation of aquatic microplastic monitoring.

Overview of Microplastic Contamination in Aquatic Systems

Microplastics enter aquatic systems through diverse pathways, including the fragmentation of macro-plastic debris, direct discharge of industrial pellets (nurdles), release of synthetic textile fibres during laundering, runoff of tire wear particles, and degradation of personal care product microbeads. Once introduced, their buoyancy, morphology, and density dictate their spatial distribution across surface waters, the water column, and bottom sediments. The polymer composition of microplastics encountered in aquatic matrices is heterogeneous, encompassing polyethylene (PE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC), polyethylene terephthalate (PET), and polyamide (PA), among others. Each polymer exhibits a distinct spectroscopic signature, which is exploited by nanotechnology-enhanced spectroscopic methods for unambiguous chemical identification. Size, shape, surface chemistry, and adsorbed contaminant load further define the environmental behaviour and toxicological relevance of a given microplastic particle, underscoring the need for multi-parametric characterisation platforms.

Nanotechnology-Based Detection Methods

Surface-Enhanced Raman Scattering (SERS)

Surface-enhanced Raman scattering exploits the electromagnetic field enhancement arising at the surface of plasmonic nanostructures, typically gold or silver nanoparticles or nanoarrays, to amplify the intrinsically weak Raman signal of analyte molecules by factors of up to 10^{10}. When microplastic particles are deposited onto SERS-active substrates, the vibrational fingerprint of the constituent polymer chains is dramatically enhanced, enabling detection at concentrations as low as 0.01 mg/L. Recent advances have seen the development of silver nanotriangle arrays and gold nanostar clusters as SERS substrates optimised for plastic polymer detection in complex water samples. Ge et al. demonstrated that gold nanorod-decorated filter membranes could simultaneously separate and identify PE, PP, and PS microplastics from spiked river water samples, achieving unambiguous polymer-type discrimination in under 30 minutes. The integration of SERS with portable handheld Raman spectrometers further positions this technique as a strong candidate for in-situ aquatic monitoring.

Nano-Biosensor Platforms

Nano-biosensors integrate biological recognition elements, such as antibodies, aptamers, or Molecularly Imprinted Polymers (MIPs), with nanomaterial-based transducers to achieve highly selective detection of microplastic particles in solution. Aptamer-functionalised gold nanoparticles have been engineered to bind selectively to polystyrene bead surfaces via hydrophobic and pi-stacking interactions, generating colorimetric or fluorescence signals proportional to microplastic concentration. Molecularly imprinted polymer nanoparticles represent a particularly versatile nano-biosensor strategy. MIP nanoparticles synthesised with surface cavities complementary to the geometry and chemistry of target plastic fragments offer antibody-like selectivity without the associated cost or stability limitations of biological antibodies. Upon binding, conformational changes or fluorescence quenching events generate quantifiable analytical signals, with reported detection limits in the sub-nanogram-per-millilitre range for nano-sized PS and PE fragments.

Nanomaterial-Enhanced Electrochemical Sensors

Electrochemical sensors incorporating carbon nanomaterials, including graphene oxide, carbon nanotubes (CNTs), and carbon quantum dots, as electrode modifiers have demonstrated high sensitivity toward microplastics through two distinct mechanisms: direct adsorption-based impedance shifts and indirect detection of polymer-associated organic contaminants (e.g., phthalates, bisphenol A). Graphene-oxide-modified screen-printed electrodes have enabled differential pulse voltammetric detection of PS microplastics at concentrations as low as 1 ppb in filtered seawater. The large surface area, high electrical conductivity, and functionalisation versatility of carbon nanomaterials facilitate robust sensor regeneration and multiple detection cycles without significant signal drift, making them attractive for autonomous moored sensor buoys. CNT-based impedimetric biosensors have additionally been reported for the simultaneous quantification of mixed polymer assemblages, a particularly relevant capability given the multi-polymer nature of real environmental samples.

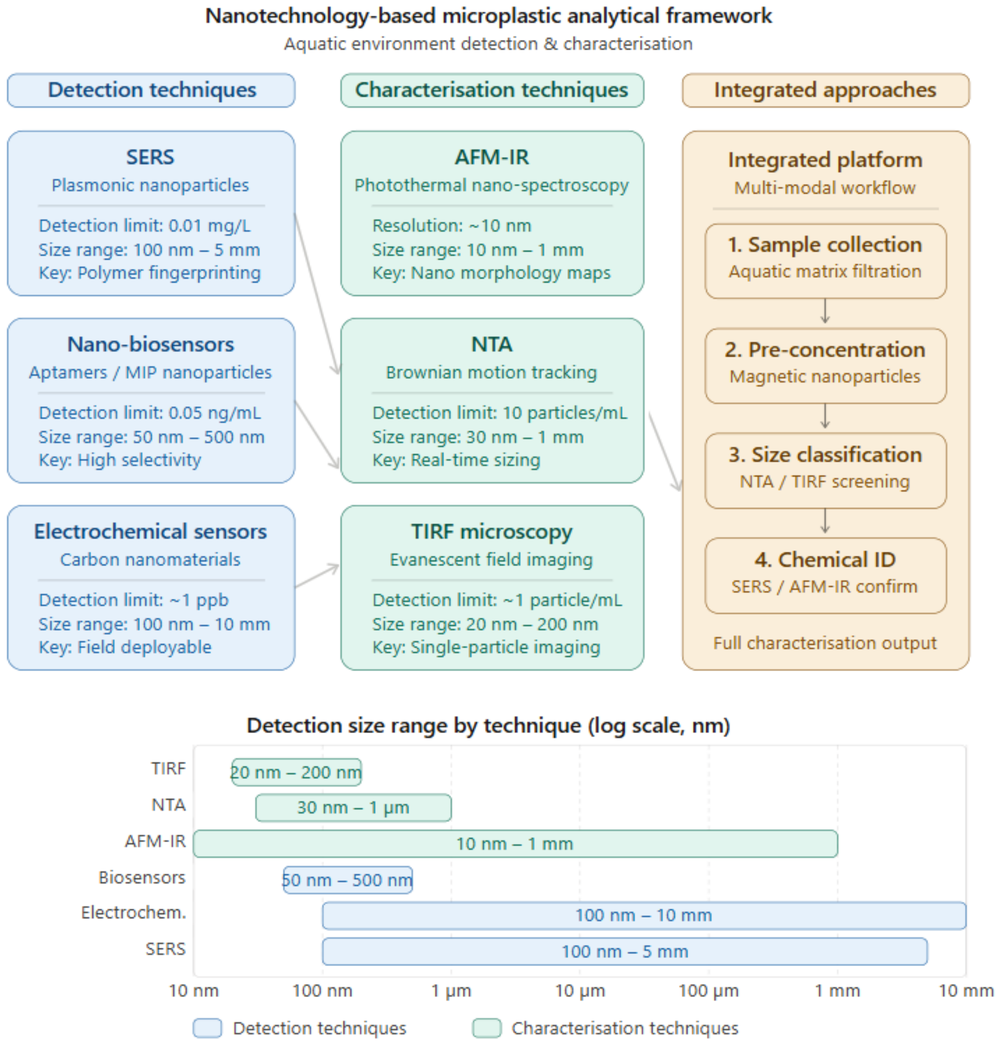

Figure 1. Framework of nanotechnology-based analytical techniques for microplastic detection and characterisation in aquatic environments, showing detection categories, integrated workflow, and size detection ranges on a logarithmic scale.

Nanotechnology-Based Characterisation Techniques

Atomic Force Microscopy Coupled with Infrared Spectroscopy (AFM-IR)

Atomic force microscopy coupled with infrared spectroscopy, also known as nano-IR or photothermal AFM-IR, overcomes the diffraction-limited spatial resolution of conventional FTIR by using a scanning cantilever tip to locally probe thermal expansion induced by infrared absorption at the nanometre scale. This technique provides simultaneous topographic and chemical composition maps of individual microplastic particles with spatial resolutions down to 10 nm, far surpassing the ~10 mm resolution ceiling of conventional attenuated total reflectance FTIR. AFM-IR has been applied to characterise weathered microplastic fragments collected from marine sediments, revealing surface oxidation gradients, adsorbed organic matter layers, and inorganic mineral coatings that standard spectroscopic techniques miss. The nanomechanical mapping capability of AFM further permits quantification of polymer elastic moduli and surface stiffness, parameters that correlate with degree of environmental degradation and are of direct relevance to ecotoxicological risk assessments.

Nanoparticle Tracking Analysis (NTA)

Nanoparticle tracking analysis illuminates dispersed particles in suspension with a laser beam and tracks the Brownian motion of each particle individually using a high-sensitivity video camera. The hydrodynamic diameter and number concentration of particles are then derived from the Stokes-Einstein equation. NTA is capable of resolving particles in the 30 nm to 1 mm size range in real time, providing size distribution histograms at concentrations as low as ten particles per millilitre. When NTA instruments are equipped with fluorescence modules and microplastics are labelled with Nile Red or other lipophilic fluorescent dyes, the technique gains polymer-selective capability, allowing discrimination of plastic particles from biotic debris in complex environmental matrices. NTA has been validated against electron microscopy for nanoplastic quantification in treated wastewater effluents, exhibiting good concordance in the 50-500 nm size window and demonstrating utility as a high-throughput screening tool prior to confirmatory spectroscopic analysis.

Total Internal Reflection Fluorescence (TIRF) Microscopy

Total internal reflection fluorescence microscopy exploits the evanescent electromagnetic field generated at a glass-water interface under total internal reflection conditions to excite fluorophores within approximately 200 nm of the surface, providing an extremely low background and single-particle detection sensitivity. Nanoplastic particles labelled with fluorescent probes or intrinsically fluorescent nanoplastics can be detected and counted at the single-particle level in thin aqueous films. TIRF-based platforms have achieved detection limits of one plastic particle per millilitre in deionised water, though matrix effects in environmental samples reduce this performance. Integration with microfluidic channels permits continuous flow-through analysis of water samples, enabling particle size distribution and concentration determination on timescales of minutes, substantially faster than conventional filtration-based workflows.

Comparative Analysis of Nanotechnology-Based Techniques

Table 1 presents a structured comparison of the key nanotechnology-based detection and characterisation techniques reviewed in this paper, summarising their detection limits, operational size ranges, and primary analytical advantages.

Table 1. Comparative Summary of Nanotechnology-Based Microplastic Analytical Techniques

Technique	Detection Limit	Size Range	Key Advantage
SERS	~0.01 mg/L	100 nm–5 mm	Chemical fingerprinting of polymer type
Nano-Biosensors	~0.05 ng/mL	50 nm–500 nm	High selectivity, real-time output
Electrochemical Sensors	~1 ppb	100 nm–10 mm	Portable, low-cost field deployment
AFM-IR	~10 nm resolution	10 nm–1 mm	Nanoscale morphology and chemistry
NTA	~10 particles/mL	30 nm–1 mm	Real-time particle size distribution
TIRF Microscopy	~1 particle/mL	20 nm–200 nm	Single-particle fluorescence imaging

From the comparison, SERS emerges as the most chemically informative technique, providing polymer-specific vibrational fingerprints that enable unambiguous identification of plastic type. AFM-IR extends chemical characterisation to the nanometre scale with concurrent morphological mapping, making it indispensable for nanoplastic research. NTA and TIRF excel in particle sizing and counting throughput, while electrochemical sensors offer the greatest potential for low-cost, field-deployable monitoring. Nano-biosensor platforms bridge selectivity and sensitivity, though their performance in complex environmental matrices requires further validation. No single technique provides a complete analytical solution. An integrated platform combining nano-biosensor-based pre-concentration, NTA-based sizing, and SERS-based chemical identification would address the full spectrum of analytical requirements for comprehensive aquatic microplastic surveillance.

Challenges and Future Perspectives

Matrix Interference and Sample Preparation

Aquatic environmental matrices contain a complex mixture of natural organic matter, humic and fulvic acids, biological colloids, and inorganic particles that can adsorb onto nanostructured sensor surfaces, occlude analytical signals, and generate false positives. Developing antifouling surface chemistries, selective pre-concentration strategies using magnetically recoverable nanoparticles, and microfluidic sample clean-up modules compatible with nanosensor platforms remains a central technical challenge.

Standardisation and Reference Materials

The absence of internationally certified reference materials for nano- and microplastics in water matrices impedes cross-laboratory validation and regulatory adoption of nanotechnology-based methods. Collaborative efforts between standards bodies, such as ISO and ASTM, and the research community are needed to develop certified plastic particle standards across the relevant size range and to establish standard operating protocols for nanotechnology-based analytical workflows.

Future Directions

Emerging research directions include the development of lab-on-chip microfluidic nanosensor arrays capable of simultaneous multi-parameter microplastic characterisation, the integration of machine learning algorithms with hyperspectral SERS imaging for automated polymer classification, and the deployment of wireless networked nano-electrochemical sensor buoys for continuous real-time aquatic monitoring at basin scale. The convergence of nanotechnology, microfluidics, and artificial intelligence is expected to yield a new generation of portable, autonomous, and highly sensitive microplastic monitoring instruments within the coming decade.

Conclusion

Nanotechnology-based analytical techniques represent a transformative advancement in the detection and characterisation of microplastics in aquatic environments. Surface-enhanced Raman scattering, nano-biosensors, electrochemical nanosensors, AFM-IR, NTA, and TIRF microscopy each offer distinct capabilities that collectively address the limitations of conventional methods, particularly in sensitivity, spatial resolution, and potential for field deployment. The complementary nature of these techniques argues strongly for integrated multi-modal analytical platforms as the future standard for aquatic microplastic surveillance. Continued investment in nanomaterial synthesis, sensor miniaturisation, and standardisation is essential to translate these laboratory advances into robust, regulatory-grade environmental monitoring tools.

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