Environmental
Science
Water Research & Technology

ROYAL SOCIETY
OF CHEMISTRY

PERSPECTIVE

View Article Online
View Journal

Cite this: DOI: 10.1039/d6ew00112b

Microplastics in water: challenges in measurement and regulation, and a path towards mass-based metrics

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Received 2nd February 2026,
Accepted 9th April 2026

DOI: 10.1039/d6ew00112b

rsc.li/es-water

Microplastics (MPs) have become a ubiquitous environmental pollutant, with water acting as the main vector for their transport and widespread dissemination across natural and engineered systems. Municipal wastewater treatment plants (WWTPs) play a pivotal role as both major sinks and continuous point sources of MPs, as incomplete removal during treatment, combined with high effluent flow rates and sludge reuse, leads to significant emissions to aquatic and terrestrial environments. Growing awareness of this issue is driving the development of increasingly stringent regulatory frameworks in the water sector. However, the absence of standardized and policy-relevant analytical methodologies has so far hindered the explicit regulation of MPs. Current monitoring strategies rely predominantly on particle-based approaches using visual and spectroscopic techniques, which are labor-intensive, observer-dependent and prone to substantial underestimation, particularly for small particles. Therefore, they are poorly suited for regulatory implementation and process control. While thermoanalytical techniques such as pyrolysis- and thermodesorption-gas chromatography coupled to mass spectrometry (Py-GC/MS and TD-GC/MS) provide valuable insights into polymer composition and mass-based information, they remain costly, time-consuming and limited in their ability to deliver rapid, comprehensive quantification of total plastic content in complex (waste)water matrices. This Perspective critically reviews the state of the art in MP quantification, highlights the urgent need for robust mass-based metrics compatible with regulatory and operational requirements, and introduces a practical, cost-effective mass-based method developed by our research group and Captoplastic S. L., and commercialized through the start-up Captoplastic S.L. Its applicability is finally demonstrated through a municipal WWTP case study.

Water impact

This work advances microplastics management by introducing a practical, mass-based quantification method suitable for regulation and wastewater treatment. It overcomes limitations of particle-based monitoring with a patented, cost-effective, scalable approach to measure total plastic mass in complex waters. The methodology supports standardized metrics, improves process control and compliance, and promotes sustainable sludge reuse and reduced environmental emissions.

1. Introduction

The ubiquitous occurrence of microplastics (MPs) in the environment has emerged as a critical global concern and a top research priority. MPs, *i.e.* tiny plastic particles measuring <5 mm in size, have been detected across all compartments of aquatic and terrestrial ecosystems.[1] Their remarkable stability, potential for bioaccumulation and uncertain toxicological effects pose a significant burden on the worldwide ecosystems and human health.

Although the environmental life cycle of MPs is highly complex, water is generally recognized as the main vector for their transport and dissemination. In the last years, municipal wastewater treatment plants (WWTPs) have been identified as a critical pathway within this cycle.[2,3] Large amounts of MPs are continuously introduced into WWTPs

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through domestic sewage, surface runoff and atmospheric sedimentation.[4] Relevant examples include polyester microfibers released during laundry processes,[5] fragments originating from the breakdown of household items,[6] and tire-wear particles generated by the abrasion of tires during road traffic.[7]

Although WWTPs are generally highly effective for MPs removal, they cannot warrant their complete elimination. On average, the overall MP abundance decreases by more than 90% after treatment.[8] Consequently, the release of these persistent particles from WWTPs remains highly significant considering the large effluent flow rates that are continuously discharged into receiving water bodies or reused as reclaimed water.[9] Extensive evidence indicates that WWTP effluents result in significantly higher MP abundances downstream compared to upstream in receiving rivers,[10,11] with quantitative assessments suggesting that daily discharges of MP particles into aquatic environments range from millions to billions globally.[12,13] Moreover, it should be noted that the MPs retained during wastewater treatment are predominantly transferred to sewage sludge,[14,15] which is often further processed into compost for agricultural fertilization. This practice ultimately contributes to the accumulation of MPs in soils and, potentially, in crops.[2,4] Consequently, WWTPs constitute important hotspots for MP pollution in the environment and thus represent critical control points for mitigation. In fact, it is estimated that over 90% of MP contamination could be prevented if all wastewater were effectively treated prior to discharge.[16]

Given the pivotal role of WWTPs as major sources of MP contamination, regulatory frameworks are expected to become increasingly stringent in the coming years. A representative example is the new European Urban Wastewater Treatment Directive (UWTD),[17] whose primary objective is preserving freshwater from micropollutants by both controlling emissions at major industrial sources and implementing appropriate treatments for their removal (*i.e.* quaternary treatments) in WWTPs. Micropollutants encompass a plethora of chemical compounds originating from pharmaceuticals, personal care products and numerous consumer goods containing, for example, per- and polyfluoroalkyl compounds (PFAS). Notably, MPs have not yet been regulated in the UWTD, primarily due to the absence of standardized methodologies for their quantification. In fact, the available data in the literature exhibits substantial variability, with no consensus on which polymers should be reported or the units in which MPs should be quantified. These challenges have been recognized across multiple regulatory frameworks and scientific organizations, including initiatives on drinking water quality,[18–20] water reuse,[21] and wastewater management,[17] all of which underscore the urgent need for reliable, robust, and standardized methods for MP quantification. Achieving this goal clearly represents the essential first step toward managing the widespread MPs discharge from WWTPs.

Analysis of MPs in (waste)water is a particularly challenging task due to the extreme heterogeneity of particle size, shape, color, density, and chemical composition. So far, most studies have relied on visual inspection and spectroscopic imaging techniques, such as Raman and FTIR spectroscopy, to characterize particle abundance, size, shape, coloration, and polymer type.[22–24] However, these methods are inherently limited by particle size, which represents a clear source of uncertainty and likely leads to underestimation of the total plastic content in samples. Moreover, they are observer-dependent and prone to misidentifying other solid particles as MPs, especially in complex matrices such as WWTP samples, thereby compromising both the accuracy and comprehensiveness of the analysis. Remarkably, these techniques quantify MPs based on particle count rather than total mass, which is not only highly error-prone due to the wide variability in particle dimensions, shapes and densities, but also impractical for regulatory purposes. Attempts to convert particle numbers into mass are subject to substantial uncertainty, making inter-study comparisons unreliable and hindering the development of standardized monitoring frameworks. In fact, visual-based methods have consistently underestimated plastic pollution when compared to mass-based approaches, which currently rely on thermoanalytical techniques such as pyrolysis-gas chromatography (Py-GC/MS)[25] or thermodesorption-gas chromatography-mass spectrometry (TD-GC/MS).[26] Therefore, transitioning toward mass-based quantification is essential to ensure accurate, reproducible, and policy-relevant assessments of MP pollution.

Despite the valuable insights provided by thermoanalytical methods regarding the polymeric composition of MPs, these techniques present important practical limitations. They are time-consuming, costly, and require specialized expertise. Furthermore, their accuracy can be compromised by the presence of organic interferences and by the aging of MPs, which can alter their spectral response and hinder identification. More critically, they typically yield bulk information on selected polymers rather than the total plastic content, which is a significant drawback given the wide diversity of polymers found in (waste)water. In this context, the development of a simple, cost-effective, and robust procedure capable of providing global concentration values (*e.g.*, mg L^{-1}) is urgently required. Moreover, quantifying MPs by mass rather than particle count is essential for regulatory applicability, as it enables routine monitoring, supports daily process optimization, and facilitates the establishment of standardized thresholds.

In this context, this work explores state-of-the-art analytical methods for MPs quantification, highlighting their strengths and limitations. Building on these insights, we introduce a practical mass-based approach designed to overcome existing challenges by enabling accurate and reproducible MPs quantification in complex water matrices. Developed and validated by our research group in collaboration with Captoplastic S.L., and subsequently patented and commercialized through the spin-off, this method allows rapid and reliable determination of total MPs concentration in (waste)water. To demonstrate its applicability, we present a case study focused on the quantification of MPs in a municipal WWTP, followed by a discussion on future perspectives and the potential role of mass-based approaches for regulatory purposes.

2. Analytical methods for microplastics quantification

The quantification of MPs in aqueous matrices is inherently complex due to their diverse physical and chemical properties and the likely occurrence of co-existing suspended solids of both organic and inorganic origin. Accordingly, a multi-step procedure is required to ensure accurate and reproducible results. These steps, summarized in Fig. 1, comprise the separation of MPs from the aqueous matrix, the removal of interferences and the subsequent quantification and characterization.

2.1. Microplastics separation

Filtration and sieving remain the most widely used techniques for separating MPs from aqueous matrices.[27] The pore size of the selected filter or sieve largely determines the minimum particle size retained. However, very small pore sizes are generally avoided because they are prone to clogging due to suspended solids, which significantly increases processing time. For this reason, fine filters (0.45–100 μm) are typically reserved for relatively clean matrices such as groundwater or drinking water.[28] A cascade filtration strategy is also commonly applied, using sequential filters of decreasing pore size (*e.g.* 5 mm, 500 μm, 200 μm and finally 0.45 μm), which not only facilitates processing but also enables size fractionation of MPs for subsequent analysis.

Despite its widespread use, filtration exhibits important limitations. The process is inherently labor-intensive and time-consuming, particularly because MP analysis typically requires the processing of large sample volumes (ranging from 1 L to over 10 L). Moreover, it demands meticulous handling to minimize particle loss and cross-contamination, often necessitating multiple filters for a single sample, which not only increases operational complexity but also amplifies the risk of analytical errors. There are numerous reports in the literature highlighting substantial inconsistencies in MP quantification using filtration. For instance, studies analyzing table salt (dissolved in water) – a relatively simple and compositionally homogeneous matrix – reported MP concentrations ranging from 0 to over 39 800 MPs $\mathrm{kg_{salt}}^{-1}$, mainly due to the use of filters with varying pore sizes.[29,30] Notably, despite its critical influence on particle retention, the pore size of the filters is often omitted from methodological descriptions, further complicating the interpretation and reproducibility of results. Accordingly, significant deviations have been also observed in real water samples, with reported values differing by a factor of 2–3 in drinking water and up to two orders of magnitude in bottled and treated wastewater.[31–34] This pronounced variability critically undermines the comparability of results across studies and highlights the pressing need for methodological standardization.

More recently, magnetic separation based on the interaction of MPs with magnetic materials has emerged as a promising technique, offering extremely rapid and size-independent recovery. These features represent notable advantages over conventional filtration methods, particularly in terms of operational efficiency and scalability.[35] However, this approach may increase the apparent density of MPs and hinder their subsequent characterization, as the plastic particles become coated with the fine magnetic material used during separation. Furthermore, the application of magnetic nanoparticles (~20 nm) entails inherent limitations, as their reduced magnetic saturation compromises separation kinetics and overall recovery efficiency. These limitations likely account for the comparatively limited adoption of magnetic separation for MP quantification relative to conventional filtration methods so far. Accordingly, the

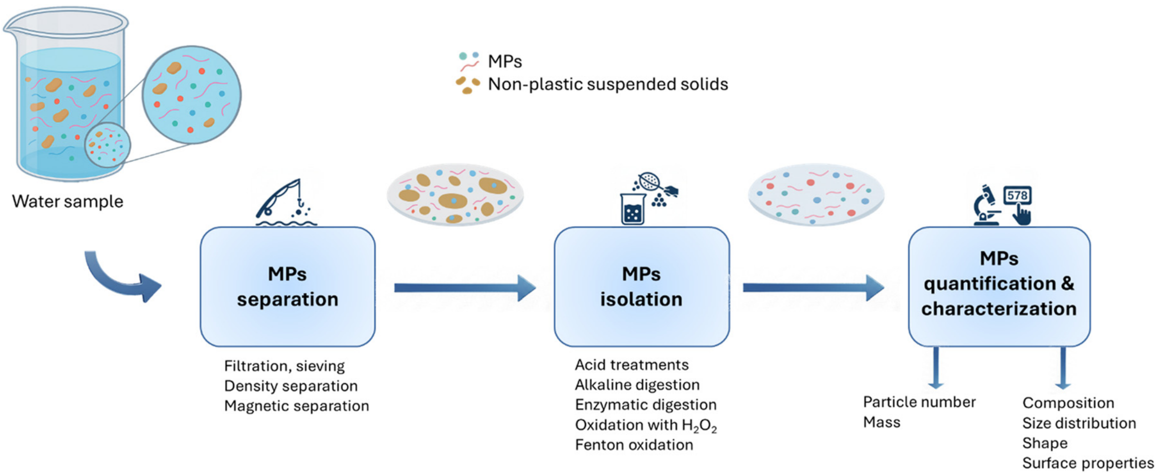


Fig. 1 Schematic representation of the general multi-step procedure required for the quantification and characterization of MPs in aqueous matrices.

effective implementation of magnetic separation for MP quantification still requires comprehensive methodological optimization. This includes improvements in processing speed, cost-efficiency, and operational robustness, as well as the development of reliable strategies for the disaggregation of magnetic aggregates to ensure the recovery of uncoated particles suitable for physicochemical characterization.

Density separation plays a relatively minor role in water analysis for MPs separation, as it is mainly intended for samples with substantial inorganic content. Plastic polymers typically exhibit densities between 0.90 and 1.60 g cm^{-3}, whereas inorganic materials such as sand and rust reach around 2.6 g cm^{-3}.[36] However, in most aqueous matrices, particularly those from WWTPs, organic matter is the prevalent component, limiting the effectiveness of this approach. Accordingly, density separation, based on the difference in particle buoyancy using dense salt solutions (typically NaCl, NaI, or ZnCl) has been mainly applied in the analysis of sediments or solid-rich samples; while its application to water analysis has been mainly explored after MPs isolation from the sample by oxidative digestion.[36]

Alternative approaches, although less common, enable simultaneous separation and quantification of MPs. These include electrostatic separation[37] and flow cytometry,[38] which offer reasonable analytical sensitivity. Electrostatic separation has predominantly been used in solid matrices such as sediments and sands, whereas flow cytometry has shown promise in aqueous environments, albeit necessitating substantial sample pre-concentration to ensure representative quantification and ultimately providing signals corresponding to individual particles, whose identity, size, and morphology cannot be resolved without appropriate reference standards. However, their broader application is limited by the requirement for specialized equipment, prolonged processing times, and elevated costs.

2.2. Microplastics isolation

Following MP separation, the next critical step is to isolate MPs from the retained solids in order to minimize analytical interferences. Aqueous matrices frequently contain suspended solids, predominantly of organic origin, which can interfere with MP analysis and lead to overestimation of concentrations regardless of the separation method employed. Consequently, an additional treatment step is essential to minimize these interferences, making it a critical stage in the analytical workflow. The most common approaches for removing non-plastic organic matter include acid, alkaline, or enzymatic digestion, as well as oxidative processes.

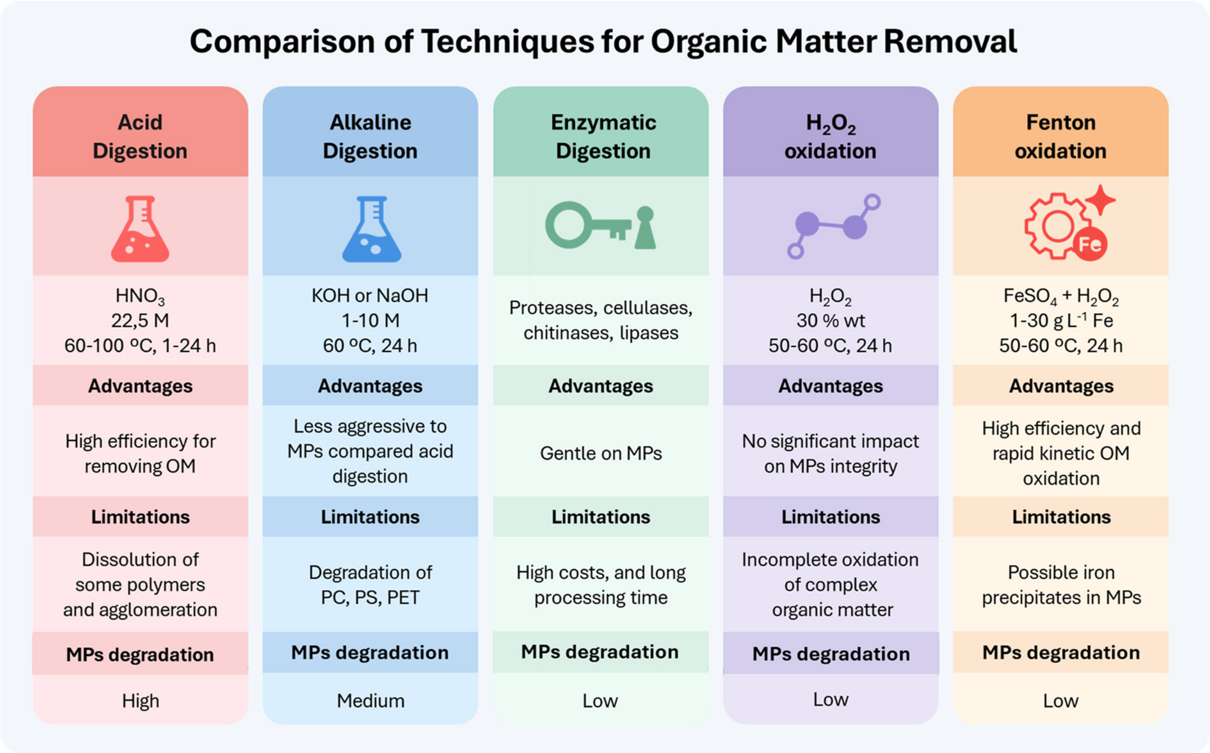
Among acid treatments, nitric acid has shown the highest efficiency.[39] However, this method is highly aggressive, often causing partial dissolution of certain polymers (*e.g.*, polystyrene, polyethylene) and/or particle agglomeration.[40] For this reason, acid digestion is rarely applied in MP analysis of aqueous samples. Alkaline digestion is generally considered less aggressive and thus more suitable, with treatments using KOH (10% v/v) or NaOH (10 M) at 60 °C for 24 h being the most effective.[41] Nevertheless, some polymers such as polycarbonate, polystyrene, and polyethylene terephthalate still exhibit degradation and mass loss under these conditions. Enzymatic digestion has emerged as an attractive alternative due to its milder impact on MPs,[27] but its efficiency strongly depends on the type of organic matter present. In practice, enzymatic protocols often require multiple steps, typically combined with mild oxidation using H_2O_2, and involve long processing times (up to two weeks) and high costs.[42]

In this context, oxidation processes have emerged as a promising alternative to remove interfering organic matter. Among them, treatment with hydrogen peroxide (H_2O_2) is the most widely applied approach in literature. Typically, a concentrated hydrogen peroxide solution (*e.g.* 30% v/v) is added to the sample, thoroughly mixed, and incubated at approximately 50–60 °C for, at least, 24 h.[25,26,43] This process does not affect the MP integrity but the completeness of peroxide digestion of organic material varies based on the composition of the organic content of the sample. In this regard, the Fenton process stands out due to its stronger oxidation efficiency and rapid reaction kinetics. This process relies on the catalytic decomposition of H_2O_2 in the presence of iron salts under acidic conditions, generating hydroxyl radicals (HO·), highly reactive and non-selective species capable of degrading a wide range of organic compounds. Several studies have demonstrated the effectiveness of this approach in eliminating non-plastic organic particles and facilitating MP isolation from diverse aqueous matrices.[41,44–46] However, these studies typically employ high concentrations of H_2O_2 (hundreds of g L^{-1}) and iron (1–30 g L^{-1}). Operating temperatures generally range from ambient to 60 °C, as higher temperatures induce polymer degradation and mass loss.[45,47] Consequently, long reaction times (often up to 24 h) are required to achieve substantial removal of organic matter. Another critical limitation is the deposition of iron precipitates on MP surfaces at such high iron concentrations, which complicates subsequent quantification and characterization. Overall, the effectiveness and limitations of the various methods used to remove organic matter and isolate MPs from aqueous matrices are summarized in Fig. 2.

2.3. Microplastics quantification and characterization

Once the MPs are isolated from the other retained solids in the sample, they can be finally quantified and characterized. The most widespread approach for their identification and quantification is visual flow cytometry inspection, typically followed by chemical characterization using FTIR or Raman spectroscopy, whereas gravimetry and thermoanalytical methods are employed far less frequently.

Visual inspection remains one of the most widely used and accessible methods for detecting plastic particles. Beyond simple detection, this technique enables classification based on size, color, and shape, which can provide valuable insights into their possible sources. Nevertheless, its reliability is limited by



Comparison of Techniques for Organic Matter Removal

Acid Digestion	Alkaline Digestion	Enzymatic Digestion	H_2O_2 oxidation	Fenton oxidation
HNO_3 22,5 M 60-100 °C, 1-24 h	KOH or NaOH 1-10 M 60 °C, 24 h	Proteases, cellulases, chitinases, lipases	H_2O_2 30 % wt 50-60 °C, 24 h	$FeSO_4$ + H_2O_2 1-30 g L^{-1} Fe 50-60 °C, 24 h
Advantages	**Advantages**	**Advantages**	**Advantages**	**Advantages**
High efficiency for removing OM	Less aggressive to MPs compared acid digestion	Gentle on MPs	No significant impact on MPs integrity	High efficiency and rapid kinetic OM oxidation
Limitations	**Limitations**	**Limitations**	**Limitations**	**Limitations**
Dissolution of some polymers and agglomeration	Degradation of PC, PS, PET	High costs, and long processing time	Incomplete oxidation of complex organic matter	Possible iron precipitates in MPs
MPs degradation	**MPs degradation**	**MPs degradation**	**MPs degradation**	**MPs degradation**
High	Medium	Low	Low	Low

Fig. 2 Comparison of methods for isolating MPs by removing organic matter in aqueous matrices.

subjectivity, inter-observer variability, and high misclassification rates.[27] To improve accuracy, several auxiliary approaches, such as melting tests, thermal degradation, and staining protocols, have been proposed. Among these, staining techniques have attracted particular interest, with Nile red standing out due to its rapid application, strong signal intensity, and compatibility with vibrational spectroscopic techniques.[36] Despite these advantages, the identification of certain polymers and fibers remains challenging and as noted in the Introduction section, quantification by visual inspection provides particle counts rather than mass concentrations, which significantly hampers the standardization of analytical procedures.

Plastic particles detected through visual inspection must be chemically characterized to identify their polymer composition and avoid misclassification. FTIR and Raman spectroscopy are the most commonly employed techniques for this purpose, offering non-destructive analysis, high precision, and complementary spectral information. Several FTIR modalities have been employed for MP analysis. Attenuated total reflection FTIR (ATR-FTIR) is particularly useful for irregular, thick, or opaque particles, offering advantages over transmission FTIR. Micro-FTIR (μ-FTIR) enables high-resolution mapping down to 20 μm without the need for prior particle selection. On the other hand, Raman microscopy, allows the characterization of MPs smaller than 20 μm, although its performance may be affected by weak signal intensity and fluorescence interference, which depend on factors such as particle color, biofouling, and degradation state.[27] To date, visual inspection and these spectroscopic imaging techniques have been predominantly used for MPs analysis in WWTPs, focusing on particle-level descriptors such as count, size, morphology, color, and polymer composition. Nevertheless, the abovementioned analytical constraints, combined with the size limitations of spectroscopic techniques, contribute to a systematic underestimation of the total plastic load in WWTP samples.[25]

Thermal analysis encompasses techniques in which samples are heated under controlled conditions to induce structural or compositional changes, including mass loss and the formation of volatile degradation products. When coupled with chromatographic or spectroscopic methods, it enables the identification and quantification of these products. In recent years, thermoanalytical techniques such as TD-GC/MS and py-GC/MS have been introduced for mass-based quantification of MPs. These methods rely on high-temperature and oxygen-free conditions to thermally degrade polymers into characteristic volatile compounds, which are subsequently separated by gas chromatography and detected by mass spectrometry, allowing for the identification and quantification of individual polymer types. Other thermoanalytical techniques such as differential scanning calorimetry (DSC) have also been explored for polymer characterization and semi-quantitative analysis; however, their application to complex environmental matrices remains limited due to sensitivity constraints and matrix interferences.

Despite their analytical potential, thermoanalytical techniques face several challenges that compromise their applicability in complex environmental matrices. Interferences from non-polymeric substances and the aging and/or fouling of plastics can hinder the identification of target compounds.

Moreover, the instrumentation required is costly, the procedures are time-consuming, and their application demands specialized expertise. Perhaps most critically, these methods tend to yield bulk data limited to selected polymers, without capturing the full spectrum of plastic types present in environmental and wastewater samples, which represents an important drawback considering the heterogeneity of polymer compositions in such contexts.

Gravimetry is an approach that estimates MP mass concentration by weighing filters before and after sample filtration and drying. It provides rapid results for relatively large sample volumes and does not require specialized instrumentation or particle-by-particle analysis, unlike microscopy or spectroscopic techniques. Compared with thermoanalytical methods, gravimetry allows straightforward quantification of total plastic content. However, residual non-plastic solids can lead to overestimation if not fully removed in the MPs isolation stage. With careful pre-treatment to eliminate non-plastic material, gravimetry could serve as a simple and effective final quantification step in the analytical workflow.

In the first international comparative study on MP analysis in water, Müller *et al.*[48] assessed six widely used analytical techniques: optical microscopy, μ-FTIR, μ-Raman, Σ-GC/MS, SEM, and particle counters. Five different types of MP reference particles with diameters ranging from 8 μm to 140 μm suspended in ultrapure water were employed. μ-Raman and Σ-GC/MS were the most effective for polymer identification, while polymer mass quantification was only marginally reliable with Σ-GC/MS. μ-FTIR performed best in quantifying particle numbers by polymer type (especially for larger particles), and microscopy yielded the most consistent results for total particle counts. However, the study revealed remarkable disparities between laboratories. For instance, in the large size fraction, the number of PET particles reported ranged from 416 to 1280 depending on the method and lab. Similarly, for PMMA in the small size fraction, reported particle numbers varied dramatically, with a mean of 109 340 and a standard deviation of 82 799. These discrepancies were attributed to differences in sample preparation, blank value handling, extrapolation procedures, and interpretation of protocols. Dealing with polymer mass quantification, only Σ-GC/MS was capable of estimating mass, but results were inconsistent and classified as "questionable." For instance, for PE in the large fraction, some labs reported values close to the theoretical 0.23 mg L^{-1}, while others exceeded 2 mg L^{-1}. Spectroscopic methods such as μ-FTIR and μ-Raman failed to accurately calculate mass based on particle size and density.

Similar conclusions were reached in the EU Joint Research Center inter-laboratory comparison study focused on the current status of the quantification of MPs in water,[49] where PET was selected as target compound. Again, a substantial lack of inter-laboratory reproducibility was found, even for the same analytical technique. Across the 33 participating laboratories, reported concentrations spanned over two orders of magnitude. For number-based determinations, values ranged between approximately 500 and 1100 particles L^{-1} for the indicative range; however, reported data often deviated substantially from this interval. Laboratories employing μ-Raman spectroscopy generally produced higher particle counts than those using μ-FTIR imaging, whereas laser direct infrared (LDIR) spectroscopy consistently yielded significantly lower particle numbers, sometimes less than half of the μ-FTIR mean. In contrast, non-specific optical microscopy results clustered more closely around the indicative mean (670 particles L^{-1}), suggesting that polymer-specific analytical steps introduce additional variability. Even more striking discrepancies were observed in mass-based determinations: for Py-GC/MS, results varied from as low as 19 μg L^{-1} to as high as 680 μg L^{-1}, despite nominally identical analytical protocols and target material. Notably, among all the techniques evaluated, gravimetry gave acceptable results with mean and median within the indicative range, when applied in the absence of other solid organic materials.

All in all, across the existing literature on MP quantification in aqueous matrices, mass-based and particle-based approaches have produced extremely divergent results, both in terms of total MP content and polymer type distribution. A key factor driving these discrepancies is particle size distribution, which significantly influences the relationship between particle count and total mass.[50,51] Large particles disproportionately influence mass-based results measured by thermonalytical techniques while spectroscopic techniques (FTIR, Raman) tend to overrepresent small particles in particle-count data. Primpke *et al.*[50] quantified the concentration of MPs in WWTP effluents by analyzing exactly the same crushed filter membrane by FTIR imaging followed by Py-GC/MS. Remarkably, conversion of FTIR particle counts into mass concentration yielded values up to seven times higher than those determined by Py-GC/MS. In the same lines, contrasting results have been also reported in terms of polymer composition. The same authors reported a particularly high presence of polymethyl methacrylate and polyurethane *via* FTIR analysis while Py-GC/MS detected higher shares of PE and PVC. Recently, Bouzid *et al.*[52] reported comparable findings, while μ-FTIR indicated polypropylene predominance, Py-GC/MS showed polystyrene prevalence in identical samples. Altogether, these systematic discrepancies underscore the urgent need for methodological standardization to achieve reproducible MP quantification across laboratories and analytical platforms. An overview of the analytical methods evaluated in this study for microplastic detection and characterization in aqueous matrices is provided in Table 1.

3. A practical mass-based approach: Captoplastic

The implementation of water quality regulations establishing concentration limits for MPs in WWTPs discharges requires the development of robust analytical methods capable of quantifying MPs in mass concentration units (*e.g.* mg L^{-1}). As previously discussed, particle-counting approaches are labor-intensive, subject to considerable uncertainty, and lack

Table 1 Evaluation of analytical methods for the detection and characterization of microplastics in aqueous matrices

Method	Technique	Scope	Main benefits	Main drawbacks	Ref.
Optical	Visual inspection	Morphological classification (size, color, shape) of retained particles under optical microscope	Simple, inexpensive, and widely available; provides morphological and source information	Subjective; prone to misclassification; no polymer identification; reports counts, not mass	27, 48, 49
	Staining (*e.g.*, Nile red)	Fluorescent labeling of polymer surfaces under epifluorescence microscope	Rapid application; high recovery efficiency; compatible with FTIR/Raman confirmation	Limited polymer selectivity; fluorescence interference; potential overestimation in biofouled samples	36
	FTIR spectroscopy (ATR-, transmission-, μ-FTIR)	Polymer identification based on infrared absorbance spectra. μ-FTIR enables mapping down to ~20 μm	Non-destructive; chemically specific; ATR suitable for irregular or opaque particles	Size limit (~20 μm); interferences from water or organics; time-consuming image analysis	27, 48, 50, 51
	Raman spectroscopy (μ-Raman)	Vibrational spectroscopy for polymer identification, suitable for <20 μm particles	High resolution; complementary to FTIR; particle-level analysis	Fluorescence interference; weak signal in dark or aged MPs; high operator expertise required	27, 48, 49, 52
	LDIR, SEM, particle counters	Advanced optical/imaging tools for automated particle detection and polymer mapping	High throughput; potential automation; improved reproducibility	Expensive; under validation; limited inter-lab reproducibility	48, 49
Thermal	Py-GC/MS (pyrolysis-GC/MS)	Thermal degradation of polymers under inert conditions; volatiles analyzed by GC/MS for polymer ID and quantification	Enables mass-based quantification; polymer-specific fingerprints; unaffected by color or biofouling	Destructive; costly instrumentation; complex calibration; bulk-level data; quantification of only selected polymers	50–52
	TD-GC/MS (thermal desorption-GC/MS)	Controlled heating desorbs polymer volatiles, analyzed by GC/MS	Semi-quantitative; complementary to Py-GC/MS	Sensitive to matrix effects; lower selectivity for some polymers; quantification of only selected polymers	48
Gravimetric	Filter weighing	Filter mass measured before and after drying following MP isolation	Simple; fast; no specialized instrumentation; suitable for large volumes	Non-specific; possible overestimation if non-plastics remain	49

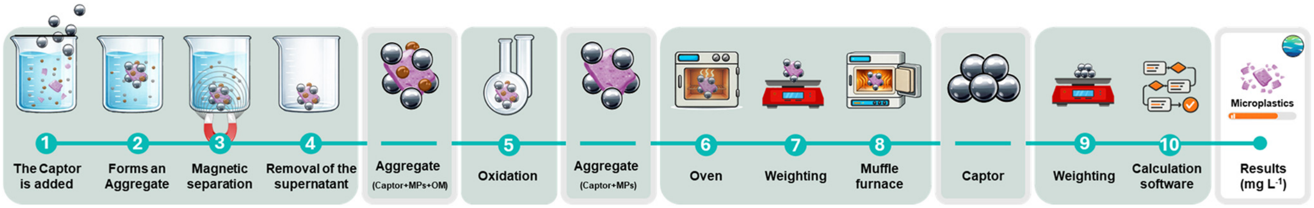


Fig. 3 Schematic representation of the Captoplastic protocol for MPs quantification in water.

intercomparability across studies. Conversely, while thermoanalytical techniques enable the quantification of MPs by mass, they are restricted to specific polymers, demand highly trained personnel, and rely on costly instrumentation. In this context, the patented method developed by Captoplastic in collaboration with our research group at the Universidad Autónoma de Madrid enables reliable, mass-based quantification of MPs, offering a practical and reproducible solution for monitoring under environmentally relevant conditions.

The method is based on a sequence of physical and chemical processes designed to separate, isolate and quantify MPs in aqueous matrices. Fig. 3 summarizes the main steps of the process. Initially, a magnetic captor composed of iron oxide is added to the water sample. These particles, referred to as captor material, almost instantaneously and homogeneously cover the MPs present in the sample, regardless of their size, shape, or composition, although it may also interact with some organic suspended solids. These aggregates, MP-captors, are then rapidly extracted using an external magnetic field, effectively separating MPs from the bulk liquid. Despite the high selectivity of the magnetic captors toward MPs, the recovered solid phase may still contain small amounts of non-plastic organic matter co-aggregated with them. Therefore, this phase is resuspended in water and then selectively oxidized through a microwave-intensified Fenton reaction. This process removes non-plastic organic material while preserving the integrity of MPs, which is critical for accurate quantification. The remaining particles are then filtered, dried, weighed, calcined and weighed again. To ensure reliable mass balance and concentration estimates, the total suspended solids (TSS) content of the original sample, both fixed (ISS – inorganic suspended solids) and volatile fractions (OSS – organic suspended solids), must also be determined. TSS was measured using a modified standard methods protocol with drying at 85–90 °C to preserve MPs. The final MP concentration is subsequently calculated using proprietary software developed by Captoplastic, which integrates all experimental inputs, including the weighed masses and the TSS data, to provide a comprehensive quantification considering the occurrence of suspended solid of both organic and inorganic origin.

The method was validated through a series of experiments using effluent samples collected from the secondary treatment stage of a municipal WWTP in Madrid. It must be noted that strict quality control procedures were applied to minimize contamination and ensure data reliability, including the use of powder-free gloves, pre-combusted glass fiber filters and tools, and the avoidance of plastic materials or synthetic garments during analysis. Procedural blanks prepared with deionized water as the aqueous matrix were systematically included and showed no detectable MP signal. In addition, control assays were performed by introducing organic and inorganic solids in the absence of MPs, and only negligible levels of MPs were observed (<2%), thereby confirming the absence of background contamination and matrix-related interferences. In WWTP effluent samples, MPs were successfully quantified at an average concentration of 0.77 ± 0.06 mg L^{-1}. To assess accuracy and confirm reproducibility, the same effluent was spiked with increasing concentrations (1, 10, and 20 mg) of a reference material,[53] with a particle size distribution of D10 = 91 μm, D50 = 206 μm, and D90 = 311 μm. Across 10 replicates analyzed independently by different technicians, the method achieved an average recovery above 97%, with minimal variability between measurements (see Table S1 in the SI for experimental data). Additional experiments were conducted using MPs obtained by cryogenic milling of PE and PP commercial materials. These assays were designed to evaluate the effect of particle size, covering fractions from <50 μm up to 250 μm. In all cases, recoveries exceeded 90% (see Table S1 in the SI for experimental data). It must be noted that the method enables the quantification of microplastic mass across the entire size range defined in the literature (1 μm to 5 mm), although the experimental validation presented in this study was performed using the size fractions most representative of wastewater treatment systems. These results demonstrate that MP quantification using this approach is independent of polymer type and particle size, highlighting its robustness. Moreover, the combination of fast and efficient MPs recovery, low inter-operator variability, and straightforward analytical workflow confirm the reliability of the method and its suitability for routine monitoring and operational control in real wastewater treatment settings. A comparison with the other analytical techniques currently available for mass-based microplastic quantification is presented in Table 2, where the recovery values obtained with the proposed method are consistent with those reported for TD-GC/MS and Pyr-GC/MS. Other techniques, including LDIR, FTIR and Raman spectroscopy or DSC, are discussed in the manuscript as complementary approaches for microplastics identification and characterization, but are not included in Table 2 as they

Table 2 Comparison of analytical methods used for mass-based quantification of microplastics in wastewater matrices, including polymer types, particle size ranges, concentration ranges, and recovery values

Quantification method	Polymer type	MP size (μm)	MP concentration (mg L^{-1})	Recovery (%)	Ref.
TD-GC/MS	PE, PP, PS, PVC, PET	0.22–5000	5×10^{-3}–70×10^{-3}	89 ± 1–121 ± 5	26
Pyr-GC/MS	PS, PC, PMMA, PP, PET, PE, PVC	>1	2	92 ± 6–112 ± 9	25
Captoplastic	PS (reference material), PE, PP	<50–250*(method valid in 1–5000)	1–20	90 ± 1–106 ± 8	This work

rely on particle-based measurements or indirect mass estimation.

Although the Captoplastic method is inherently destructive, it can also be adapted for MP characterization. In this case, an aliquot of the sample is processed as described above, but the calcination step is omitted. After the removal of organic interferences through the intensified microwave-Fenton oxidation, the magnetic MP-captors aggregates are treated with a concentrated saline solution to release the MPs from the magnetic captor. The recovered MPs can then be characterized by FTIR or Raman spectroscopy, as well as by optical and/or electron microscopy, to identify the polymer types present in the sample and to determine the size distribution and morphology of the MPs. As representative example, Fig. 4 collects the optical and electronic microscopy images and the FTIR spectrum of spiked PS MPs in the WWTP water sample after being submitted to the Captoplastic protocol for MPs isolation, prior to the calcination step and after the subsequent treatment with the saturated saline solution (NaCl, NaH_2PO_4). As observed, MPs are completely covered by the captor once isolated from the sample, but the treatment with the concentrated saline solution completely clean the MP surface, allowing accurate characterization of the polymer by FTIR. Accordingly, the patented procedure is suitable not only for routine monitoring but also allows subsequently in-depth characterization of MP composition in complex aqueous matrices.

4. Case study: analysis of microplastics in WWTP

Once validated, the Captoplastic method was applied to quantify the mass concentration of MPs across the different treatment stages of several municipal WWTPs in Spain, enabling the assessment of their overall removal efficiency. As noted above, WWTPs represent critical interfaces where plastic particles from both domestic and industrial sources accumulate and may be reintroduced into the environment. To the best of our knowledge, studies of this type *i.e.* providing MPs mass concentration across WWTP treatment stages have only recently been conducted using thermoanalytical techniques such as Py-GC/MS[25] and TD-GC/MS.[26] Although these works provided valuable insights, they were limited by the inherent constraints of such techniques, which can only quantify selected polymers and may suffer important deviations in the presence of residual organic interferences or if MPs are aged. Reported MP removal

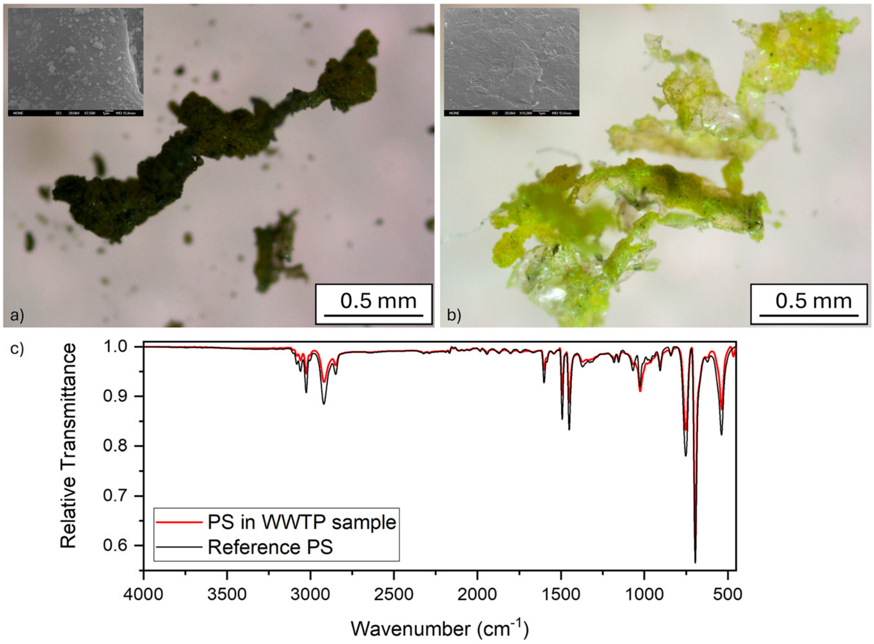


Fig. 4 Optical microscopy images of PS MPs after the intensified-microwave Fenton oxidation (a) and after the subsequent treatment with the concentrated saline solution (b). Insets show the electronic microscopy images of the MPs surface. FTIR spectrum of the clean MP (c).

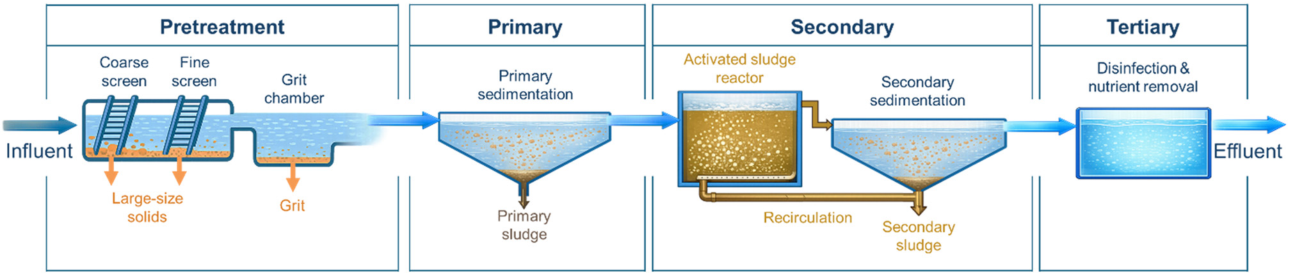


Fig. 5 Typical flow chart of the WWTPs evaluated in this study.

efficiencies, resulting from the combined effect of all treatment stages, were generally high, exceeding 90%, which aligns with previous studies based on MPs quantification by particle count.[8,22–24] Nevertheless, the actual total mass concentration of MPs remained so far largely unclear.

All the WWTPs evaluated in the current work included preliminary and secondary treatment. Primary treatment was implemented in several plants, and tertiary treatment was applied in a subset of facilities. A representative flow chart of the treatment trains evaluated is shown in Fig. 5.

Sampling was conducted during three independent campaigns over a one-month period at key points along the treatment train, depending on the treatment stages present in each plant: after preliminary treatment, and following the primary, secondary, and, when applicable, tertiary steps. Triplicate samples of 1 L were collected at all sampling points, while larger volumes (10 L) were collected from secondary and tertiary effluents to account for their expected lower MP concentrations. The results obtained are summarized in Table 3.

The mass concentration values obtained in this study were notably higher than the previously reported using thermoanalytical techniques, although those works were focused on a limited subset of polymers. For instance, Wu

Table 3 Mass concentration values of MPs, TSS, OSS and ISS in effluents from different treatment stages of the investigated municipal WWTPs (Spain). Values in mg L^{-1} are reported as mean ± standard deviation from three independent sampling campaigns

Zone	Parameter	Pre-treatment	Primary treatment	Secondary treatment	Tertiary treatment
South	MPs	37.88 ± 0.76	8.62 ± 1.29	0.11 ± 0.02	0.05 ± 0.01
	TSS	502.33 ± 23.46	82.33 ± 2.08	0.80 ± 0.00	0.67 ± 0.06
	ISS	261.36 ± 12.70	7.94 ± 0.86	0.06 ± 0.10	0.22 ± 0.10
	OSS	240.97 ± 14.95	74.40 ± 2.04	0.75 ± 0.10	0.45 ± 0.13
Center	MPs	40.23 ± 0.28	3.28 ± 0.38	0.31 ± 0.01	N/A*
	TSS	1247.53 ± 8.62	234.40 ± 9.59	9.93 ± 0.32	
	ISS	146.87 ± 3.37	30.74 ± 4.01	5.51 ± 1.08	
	OSS	1100.66 ± 5.42	203.66 ± 8.07	4.43 ± 0.93	
North	MPs	15.87 ± 0.96	2.4 ± 0.07	0.1 ± 0.04	N/A*
	TSS	218.87 ± 0.99	35.60 ± 0.53	1.80 ± 0.35	
	ISS	49.48 ± 0.66	3.51 ± 1.03	0.11 ± 0.11	
	OSS	178.29 ± 1.38	32.09 ± 0.85	1.69 ± 0.23	
South	MPs	9.67 ± 1.91	ND*	0.2 ± 0.04	N/A*
	TSS	232.67 ± 2.81		5.10 ± 0.61	
	ISS	42.03 ± 3.85		1.22 ± 0.42	
	OSS	190.63 ± 2.61		3.88 ± 0.25	
North	MPs	12.01 ± 1.60	ND*	1.6 ± 0.16	N/A*
	TSS	182.80 ± 3.64		16.70 ± 0.52	
	ISS	25.52 ± 3.92		2.15 ± 0.31	
	OSS	157.281 ± 5.85		14.55 ± 0.54	
South	MPs	14.38 ± 1.16	ND*	0.22 ± 0.05	N/A*
	TSS	315.93 ± 10.98		3.37 ± 0.57	
	ISS	89.72 ± 0.73		0.79 ± 0.13	
	OSS	226.22 ± 10.35		2.58 ± 0.45	
South	MPs	26.58 ± 0.92	N/A*	1.08 ± 0.45	0.61 ± 0.25
	TSS	284.13 ± 12.95		8.33 ± 0.67	3.81 ± 0.13
	ISS	31.77 ± 9.33		1.78 ± 0.23	0.96 ± 0.06
	OSS	252.37 ± 7.48		6.55 ± 0.88	2.85 ± 0.19
North	MPs	8.08 ± 1.39	N/A*	1.01 ± 0.12	ND*
	TSS	226.35 ± 21.20		8.70 ± 0.26	
	ISS	4.081 ± 4.71		1.88 ± 0.05	
	OSS	185.54 ± 19.30		6.59 ± 0.25	

N/A*: treatment not available in the WWTP. ND*: measurement not determined.

et al.[26] who evaluated the mass fate of several MPs in a WWTP by TD-GC/MS, found that polyethylene was the most common polymer detected in the influent (1313.11 ± 336.96 µg L^{-1}) and effluent (25.84 ± 3.75 µg L^{-1}), while PP was the least prevalent in influent (52.47 ± 11.49 µg L^{-1}) and PS in the effluent (2.74 ± 0.26 µg L^{-1}). Similarly, Okoffo *et al.*[25] reported a total plastic content (calculated by the sum of the selected polymers evaluated in the study) in the raw WWTP influent within the range of 840 to 3116 µg L^{-1}, measured by Py-GC/MS. Importantly, these studies rely on target analysis approaches and thus, capture only a restricted fraction of the polymeric spectrum present.

Despite the substantially higher mass concentrations obtained in the present work, the overall MP removal efficiencies observed after the wastewater treatment processes in the WWTP were comparable to those previously reported in the literature with both mass-based and particle-count approaches. For instance, in the abovementioned studies MP removal efficiencies were on average 96% and >99%, respectively. In all cases, the primary treatment stage was identified as the most effective barrier for MP removal,[4,54] which is consistent with its primary role in the removal of suspended solids. In contrast, MPs are not expected to be significantly reduced in the secondary biological process, as they are non-biodegradable, although some degree of association with biomass flocs cannot be excluded.[55] These findings highlight that the total MP mass concentrations in WWTPs may be orders of magnitude higher than those previously reported in the literature. This discrepancy may be explained by the comprehensive nature of the proposed method (Captoplastic) compared to thermoanalytical techniques, as it allows quantifying the total mass of plastics in the sample regardless of their size, shape, or polymeric composition. It is well-known that thermoanalytical techniques are affected by the size, shape and aging of MPs, being particularly less accurate for fibers due to their elongated morphology and low mass, which can lead to incomplete detection and higher uncertainty in identification.[56,57]

As the Captoplastic method accounts for the entire suspended solids fraction, the results reveal that MPs account for roughly 4% to 10% of the OSS in WWTP samples. This observation underscores the significant contribution of MPs to the overall solid load in wastewater and emphasizes the need for mass-based approaches to achieve a more realistic assessment of their occurrence and fate in water treatment systems.

To further contextualize the mass concentrations measured in this study, previously reported estimates of daily MP loads in WWTPs were examined. Okoffo *et al.*[25] evaluated three WWTPs with average flow rates ranging from 3000 to 60 000 m^3 d^{-1} and reported influent MP loads between 2.1 and 196.4 kg d^{-1}, calculated as the sum of the polymers analyzed by Py-GC/MS, while effluent loads ranged from 0.02 to 1.96 kg d^{-1}. Similarly, Wu *et al.*[26] investigated a large WWTP with an average flow rate of approximately 1 000 000 m^3 d^{-1} and estimated influent and effluent MP loads of 2468.3 and 77.3 kg d^{-1}, respectively, based on TD-GC/MS measurements. In the present study, the estimated MP loads obtained using the Captoplastic method ranged from 39.9–1699.7 kg d^{-1} in the influent to 0.9–10.7 kg d^{-1} in the effluent, depending on the WWTP analyzed (flow rates within the range of 1500–117 700 m^3 d^{-1}). The comparison with literature data allows the order of magnitude of MP loads entering and leaving WWTPs to be evaluated and confirms that removal efficiencies remain comparable regardless of the analytical approach used. Consequently, the results provide a more complete estimation of the plastic mass burden in wastewater systems while preserving comparability in terms of treatment performance.

Apart from being quantified in mass, MPs were also fully characterized. For such goal, an aliquot of the sample was submitted to the same analytical protocol used in quantification, except that the calcination step was omitted. The retained solids were treated with a concentrated saline solution, obtaining captor-free MPs. As representative example, Fig. 6 shows the optical microscope images and the FTIR spectra of the most common MPs identified in samples collected along the water treatment train from the WWTP evaluated. PET fibers were by far the predominant type, while fragments of PE and PP were also consistently detected in nearly all samples analyzed. These results are consistent with those previously reported in the literature where MPs were quantified in particle counts,[22–24] and also with the fact that

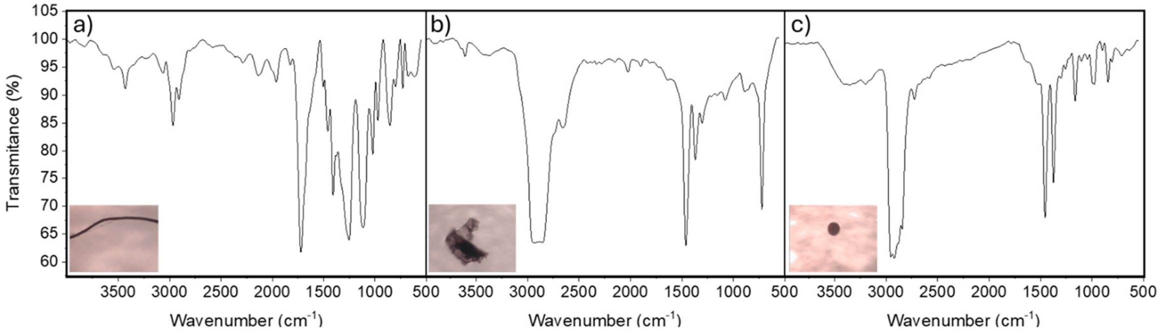


Fig. 6 Representative FTIR spectra of MPs identified in samples collected along the water treatment train of the evaluated WWTP (a: PET, b: PE, c: PP). The inset shows the corresponding particle imaged by micro-FTIR.

PET fibers are one of the most extended MPs found in the environment.[58] On the other hand, the results achieved with thermoanalytical techniques were clearly different in terms of polymer composition. In those studies, PE was identified as the most prevalent kind of polymer, with PET accounting for only a small fraction of the total.[25,26] Again, this fact could be explained by the fact that small MPs, and particularly fibers, could be strongly subestimated in thermoanalytical techniques.

5. Future perspectives and concluding remarks

Despite the growing body of quantitative studies on MPs in aquatic environments, the absence of standardized, reliable and scalable methods continues to limit their applicability in regulatory and risk assessment frameworks. Current monitoring strategies are still largely based on particle count metrics derived from visual and spectroscopic techniques, which are labor intensive, observer dependent and poorly aligned with regulatory and operational requirements.

From a regulatory and engineering perspective, mass-based metrics offer a more meaningful and actionable framework, as they are directly compatible with existing water quality standards, process control strategies and material flow analyses. The mass-based method validated in this work provides a practical and cost-effective tool for comprehensive MP quantification in complex wastewater matrices. Its successful application across multiple full-scale WWTPs highlights its potential to support routine monitoring and future regulatory implementation, contributing to a more robust and comparable assessment of MP emissions to the environment.

Further research is nonetheless required to consolidate mass-based approaches, including broader interlaboratory validation, application across diverse water matrices and the generation of long-term datasets that support method harmonization and regulatory uptake.

Conflicts of interest

There are no conflicts to declare.

Data availability

All experimental data generated during this study are included in the article and the supplementary information (SI). These data are sufficient to reproduce the results reported in this work.

Supplementary information is available. See DOI: https://doi.org/10.1039/d6ew00112b.

Acknowledgements

This research has been supported through the project PID2022-139063OB-I00 funded by MCIN/AEI/10.13039/501100011033 and by ERDF A way of making Europe. D. Rodríguez-Llorente. The authors gratefully acknowledge Captoplastic for providing the data that enabled this study, as well as for their contribution to the development and optimization of the analytical method used for MP quantification.

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