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Supplementary information

Mass Spectroscopy Based Identification for MP

Thermally Transformation Combined with GC/MS methods involve breaking down polymers (usually by pyrolysis, i.e. heating in the absence of oxygen) and analyzing the resulting thermal degradation products. After gas chromatographic separation, the chromatogram of the pyrolysis products – often called a *pyrogram* – serves as a fingerprint of the original polymer. Using GC/MS, the volatile breakdown products can be characterised and understood at the molecular level. By targeting characteristic pyrolysis products, one can determine the mass of each polymer present; in this way, real-time identification and quantification of polymers in complex samples is possible. Such polymer-specific mass data are indispensable for constructing mass balances and modeling the distribution and fate of plastics, and they will be essential for future regulatory efforts. Furthermore, thermal analysis can detect not only polymers but also plastic-associated additives and certain degradation byproducts, thus providing data that are important for assessing the environmental and health risks of microplastics. It should be noted, however, that the results of these mass-based methods represent bulk values for each polymer type (e.g., total mass of polystyrene), without revealing whether that mass came from pure polymer items or components of copolymers, and without giving any information on particle counts, sizes, or shapes [1, 2].

Pyrolysis-GC/MS has long been applied in polymer analysis; as early as 1966 it was used to detect tire wear debris on roadways and by 1986 it enabled the first identification of polystyrene as an anthropogenic pollutant in sediments and soils [3, 4]. Later, it was employed to detect polymers such as PS, PVC, PVA, PB, ABS, and SBR in coastal sediments [5-7]. Since then, Py-GC/MS has been widely used to analyze microplastics across diverse matrices, including marine and freshwater sediments [8-14], waters [15-20], biota [1, 17, 21, 22], sewage sludge [11, 23], airborne particulates from household dryer lint [24], soil [25, 26], commercial sea salt [27], and drinking water [28]. More recently, its applicability has extended to nanoplastic detection in both model and environmental samples [29-32]

In terms of instrumentation, there are two main types of pyrolysis setups commonly coupled to GC/MS: (i) a conventional pyrolyser coupled to GC/MS (often simply called Py-GC/MS), and (ii) a TED-GC/MS system, where TED stands for thermal extraction and desorption. The following sections describe the modes of operation and configurations of these systems. In a Py-GC/MS system, several modes of operation can be used [33], e.g., *single-shot* or *double-shot* (or multi-shot) analysis, evolved gas analysis (EGA-MS), and reactive pyrolysis (also known as thermochemolysis). In the single-shot mode, the sample is rapidly heated (typically to a pyrolysis temperature above 500 °C in a matter of milliseconds) and held at that temperature to completely decompose the macromolecules. The decomposition products are immediately carried into the GC column for separation and then identified by MS [2, 13, 33]. This one-step approach is straightforward and is used to detect polymer signatures (and, to some extent, certain additives) present in the sample.

The double-shot mode (also called multi-shot or *thermodesorption (TD) Py-GC/MS*) involves at least two sequential heating steps, allowing different classes of compounds to be analyzed separately. In the first step (a lower-temperature *thermal desorption*), volatile and semi-volatile compounds – for example, monomers, oligomers, plastic additives, and any sorbed environmental contaminants – are released from the sample without decomposing the polymer backbone. These volatiles can be analyzed by GC/MS to identify additives or pollutants [34, 35]. In the second step, the temperature is raised to pyrolyze the remaining nonvolatile polymer chains, and the resulting fragments are analyzed, revealing the identity of the polymer(s). This double-shot approach has proven useful for characterizing both the additives (volatile fraction) and the polymers (pyrolysis fraction) in microplastic samples [9, 12]. Moreover, performing a controlled TD step at moderate temperature can remove or reduce interfering organic matter in a complex sample before the pyrolysis step, thereby improving the identification and quantification of the polymer degradation products in samples with high organic matrix content [36, 37].

In EGA-MS (evolved gas analysis mass spectrometry), the sample is heated through a continuous temperature ramp (instead of a single set-point), and the evolving gases are sent directly into a mass detector without full chromatographic separation. Practically, this is achieved by replacing the analytical GC column with a short, inert capillary (e.g., ~2.5 m × 0.15 mm i.d.) that simply transfers volatiles from the GC injector to the MS [33]. The MS then records a continuous thermogram (total ion current vs. temperature) showing at what temperatures different decomposition products are released. This approach sacrifices compound separation, but the temperature profile can be a quick indicator of polymer types present (each polymer tends to decompose in a characteristic temperature range).

Thermochemolysis (pyrolysis in the presence of a derivatization agent) involves adding a reagent such as tetramethylammonium hydroxide (TMAH) to the sample before pyrolysis. The reagent induces cleavage of certain functional groups (especially esters and ethers) and simultaneously methylates them, which can significantly enhance the detection of polymers that produce polar fragments. For instance, adding TMAH greatly improves the sensitivity for detecting polymers like PET or polycarbonate via Py-GC/MS, by producing distinctive methylated monomers upon heating [1, 2, 33].

For polymer identification by Py-GC/MS, one can either analyze a *single* small particle or fiber, or a representative sub-sample on the order of a few micrograms (if the sample contains many particles). The material is placed in a small sample holder (also called a *pyrolysis boat* or cup) which is then dropped into or adjacent to the pyrolyzer furnace. Several types of pyrolyzers and sample holder designs exist (e.g., filament, curie point, microfurnace, etc.), differing in geometry and sample capacity [2]. In a filament pyrolyzer, the sample is typically held in an open or semi-closed quartz tube (dimensions can vary by system, with inner diameters of ~0.2–1.3 cm and various lengths [9, 12, 14, 38] that is rapidly heated by an electrically resistive platinum coil. In a CP pyrolyzer, the sample is placed on a small piece of ferromagnetic metal foil (often ~2 mm × 8 mm) that is heated by induction to its Curie temperature (the point at which the metal's magnetic properties abruptly change, which fixes the maximum temperature achieved). CP pyrolyzers provide very precise temperature control and often operate with the foil enclosed to some degree, which can be advantageous for reproducibility. Microfurnace pyrolyzers use a tiny electrically heated furnace (often a narrow ceramic tube) into which the sample (in a small cup or on a wire) is introduced; these can offer very rapid heating rates and good control as well. Each type has its own advantages in terms of sample size capacity, heating speed, and maintenance considerations, but all serve the same purpose of thermally fragmenting the polymers for analysis.

In the thermo-extraction/desorption GC/MS approach, a thermogravimetric analyzer (TGA) is typically used to heat the sample in a controlled manner under an inert gas (usually nitrogen), and the gases released at specific temperature intervals are collected and transferred to a GC/MS for analysis. In practice, the TGA can be interfaced with the GC/MS by trapping evolved volatiles on an adsorbent, followed by thermal desorption into the GC. The TED-GC/MS technique effectively combines a quantitative TGA measurement with the compound identification power of GC/MS. It allows larger sample masses (tens of milligrams) to be analyzed in one run, improving detection limits for low plastic content samples. TED-GC/MS has shown particular promise for complex matrices like environmental dissolved organic matter (DOM) or atmospheric fallout, where detecting trace levels of polymers is challenging. For example, using a TED-PTR-MS system (thermal desorption coupled to proton-transfer-reaction MS), researchers have detected positive polymer signatures from as little as 10 ng of polystyrene mixed in DOM (in Arctic snow samples), and identified PET, PVC, and polypropylene carbonate in aerosol deposition by analyzing melted snow filters [39, 40]. Although care must be taken to account for low recoveries (in one case only ~15% of PS was recovered [39]and to avoid interference from even minor impurities [40], the TD-PTR/MS and related TED-GC/MS methods appear to offer very high sensitivity for NMP analysis. Polymers in complex matrices can be quantified using TGA-MS, as demonstrated by a method for detecting PET in soil without extensive pretreatment, achieving an LOD of 0.07 wt% and LOQ of 1.72 wt% [41]. Alternatively, evolved gases from TGA can be analyzed by FTIR (TGA-FTIR), which has also been applied to plastics analysis [42, 43].

Other Mass-based Identification

TGA–DSC is another thermoanalytical method for quantifying MPs in complex samples, relying on polymer melting transitions for identification [44, 45]. It is best suited for crystalline polymers such as PE, PP, PA, and PET, while amorphous polymers like PS cannot be directly analyzed. Majewsky et al. applied this method to LDPE, PP, PET, PES, and PA, showing distinct melting points for LDPE (101 °C), PP (164 °C), and PA (216 °C), but overlapping peaks for PET and PES (250–261 °C) [46]. Due to this overlap, they focused on quantifying PE and PP, achieving detection limits of 2.5 wt% and 5 wt%, respectively. In wastewater effluent, PE accounted for 17–34% of solids (81–257 mg/m^3), while PP was not detected [46].

Differential Scanning Calorimetry (DSC) has also been applied to mixtures of common polymers (LDPE, HDPE, PP, PET), with studies showing that particle size can strongly influence the signal quality. [47]. They prepared size-classified polymer samples (fractions of 23–256 μm, 256–645 μm, and 645–1000 μm obtained by sieving) and showed that using a 10 °C/min heating rate under N_2, the melting peaks of the four polymers could be distinguished for the larger particle sizes. However, both the ability to identify polymers and the accuracy of mass quantification by DSC were significantly influenced by particle size. Smaller particles tended to broaden and shift the thermal peaks. The authors therefore recommended pre-sieving samples to a narrow size range when using DSC for MP analysis, to improve consistency. They demonstrated this approach on seawater samples spiked with known polymers, successfully identifying the polymers after isolating a specific size fraction [47]. An extended DSC protocol has been used to quantify semi-crystalline MPs in industrial wastewater, applying a three-step heating–cooling cycle under N_2 to H_20_2-treated samples in the 10–5000 µm range [106]. PE and PP were most abundant, with smaller amounts of PA and PET detected. MP concentrations in effluents were low (0.5–35.5 µg/L), similar to organic micropollutants, and one industrial WWTP was shown to remove >99.99% of MPs [44].

Matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-ToF MS) is another technology that permits the "soft" ionization of polymers and detection of high-mass ions, and it has recently been recognized as a promising tool for microplastic analysis. In MALDI-ToF, polymer molecules can be ionized with minimal fragmentation, producing characteristic mass spectral patterns (for instance, series of peaks corresponding to polymer repeat units or end-group mass differences). This can allow both identification of the polymer type and estimation of its molecular weight distribution. Researchers have applied MALDI-ToF MS to identify microplastics by their unique mass spectral signatures and even to quantify them by comparing signal intensities to those of known polymer standards [48-50]. One advantage of MALDI is that it can analyze extracts or even small particles directly (after mixing with a suitable matrix compound), offering rapid analysis and the potential for high throughput screening of samples for common polymer types. However, the technique may be biased toward more readily ionizable polymers and could struggle with complex mixtures without prior separation. Initial studies are nonetheless encouraging, showing that MALDI-ToF can detect polymers like PE, PP, PET, PS, and polyolefin copolymers in environmental extracts and that it holds potential for estimating the amounts of each polymer present[48-50].

An alternative approach to quantifying microplastics focuses on detecting and measuring specific elemental constituents of the polymers. Inductively coupled plasma (ICP) spectrometry techniques – either ICP coupled to optical emission spectroscopy (ICP-OES) or to mass spectrometry (ICP-MS) – can be used to target elements that are uniquely associated with certain plastics. For example, some plastics contain inorganic filler or additive elements (Titanium from TiO_2 pigment in paints and plastics, antimony used as a catalyst in PET, bromine in brominated flame retardants, etc.). By measuring the concentration of such tracer elements in a digested sample, one can infer the quantity of the corresponding plastic, provided the element-to-polymer ratio is known. Single-particle ICP-MS (SP-ICP-MS) is another variant, in which the instrument is operated in a mode to detect individual particles as discrete bursts of ion counts (this has been used mainly for metallic and metal-oxide nanoparticles, but conceptually could be applied to metal-containing plastic particles as well). Several studies have explored ICP-based quantification of microplastics using elements like Ti, Zn, or others as markers [51-53]. While these methods can be highly sensitive (ICP-MS can detect trace metals at sub-ppb levels) and useful for specific polymers, they are obviously limited to plastics that contain a suitable elemental tag. Moreover, care must be taken to distinguish target elements originating from microplastics versus those from natural sources or other anthropogenic particles in the sample. Overall, ICP techniques may serve as a complementary quantitative tool, especially in situations where direct polymer detection is difficult, but they do not provide molecular identification of the polymer itself.

Particle-based Quantification Methods for MP

Vibrational spectroscopic techniques – primarily FTIR (Fourier-transform infrared) and Raman spectroscopy – are the workhorses for nondestructive chemical identification of individual microplastic particles. These methods detect the characteristic vibrational fingerprints of polymers, allowing confident identification of the polymer type of even very small particles. Modern instrumental developments, such as focal plane array detectors and automated microscope stages, have enabled micro-FTIR imaging and micro-Raman imaging that can scan filters containing thousands of particles and automatically recognize polymer spectra.

Micro-FTIR spectroscopy (in the mid-infrared range) can identify plastic particles down to roughly 10–20 µm in size on a filter using transmission or reflection modes, and even smaller down to ~5–10 µm using specialized infrared microscopes with MCT (mercury cadmium telluride) or FPA (focal plane array) detectors. The typical approach for bulk samples is to filter the sample to collect solid particles, then analyze the entire filter either by point-by-point mapping or full-field imaging. Using an FPA detector (which captures an image of many pixels simultaneously, each with an IR spectrum), one can achieve high-throughput analysis of microplastics – for example, tens of thousands of particles on a filter can be analyzed in a few hours. The trade-off is that infrared wavelengths are relatively long, so spatial resolution is diffraction-limited to on the order of the wavelength (~10 µm for mid-IR), meaning particles smaller than a few micrometers are difficult to resolve. Common filter materials like mixed cellulose ester or polycarbonate have their own IR signatures, so filters such as aluminum oxide (Anodisc) that have minimal IR absorption are often used for micro-FTIR imaging in transmission [54, 55]. Micro-FTIR spectroscopy can provide rich “chemical images” of a sample filter, indicating the number of MP particles, their sizes, shapes, and polymer identities. It is often considered the method of choice for efficiently analyzing the smaller microplastics (down to ~10 µm) in environmental samples.

Table 1S Qualitative cost rating and availability of analytical techniques for micro- and nanoplastic (MNP) analysis.

Category	Technique	Approx. Instrument Cost*	Operational Cost / Maintenance	Availability	Remarks / Typical Setting
Mass-Based Methods	Py-GC/MS	★★★ (High, > USD 150 k)	Medium – consumables & training	Widely available in advanced analytical labs	Gold-standard for polymer quantification; requires trained operators
	TED-GC/MS	★★★★ (Very High)	High – special interface & calibration	Limited to specialised research labs	Excellent for low-polymer samples; complex setup
	MALDI-ToF MS	★★★★ (Very High)	Medium	Limited to mass spectrometry facilities	Rapid, high-resolution polymer identification
	TD-PTR MS	★★★★★ (Extremely High)	High	Rare – few research centres	Ultra-sensitive; used in atmospheric or trace studies
	qNMR	★★★ (High)	Medium	Moderate availability in chemical labs	Quantitative, non-destructive polymer fingerprinting
	HPLC	★★ (Moderate)	Low	Very common worldwide	Mainly for additive analysis
	DSC / TGA-DSC	★★ (Moderate)	Low	Widely available in polymer labs	Rapid screening for semi-crystalline polymers
Particle-Based Methods	μ-FTIR	★★★ (High)	Medium	Common in environmental & materials labs	Automated imaging systems available
	μ-Raman	★★★★ (Very High)	Medium–High	Moderate – in research institutes	High resolution; fluorescence can limit use
	ATR-FTIR	★★ (Moderate)	Low	Common instrument	Rapid point analysis of opaque samples
	ToF-SIMS	★★★★★ (Extremely High)	Very High	Rare – national facilities only	Ultra-surface sensitive; requires expert operator
	CARS /	★★★★	Very High	Very rare –	Fluorescence-free

	SRS	★ (Extremely High)		laser specialist labs	imaging; still research-stage
	NIR	★★ (Moderate)	Low	Widely commercial	Useful for bulk plastic sorting & field screening
Fractionation Methods	HDC-SEC	★★★ (High)	Medium	Moderate availability in polymer labs	Requires polymer-compatible solvents
	AF4-MALS / AF4-Raman	★★★★ (Very High)	High	Limited – few specialised centres	Ideal for nanoplastics separation; slow analysis
Characterisation Methods	SEM/EDX	★★★ (High)	Medium	Common in materials labs	High-resolution imaging of surface morphology
	SEM-Raman	★★★★★ (Extremely High)	High	Rare – integrated systems limited	Combines morphology and chemical data
	AFM-IR	★★★★★ (Extremely High)	High	Rare – few research facilities	Nanoscale chemical mapping (10–100 nm)
	Nano-FTIR	★★★★★ (Extremely High)	Very High	Very limited – synchrotron or national lab use	Ultimate nanoscale resolution (<50 nm)

* **Cost scale:** ★ Low (< USD 50 k), ★★ Moderate (50–100 k), ★★★ High (100–200 k), ★★★★ Very High (200–400 k), ★★★★★ Extremely High (> 400 k)

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