Environmental Chemistry

Microwave-Assisted Extraction for Quantification of Microplastics Using Pyrolysis–Gas Chromatography/Mass Spectrometry

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Abstract: Microplastics are now recognized as a persistent and global pollutant. To quantitively measure microplastics in environmental matrices, several techniques are used including new methods using pyrolysis–gas chromatography/mass spectrometry (Py–GC/MS). In the present study, a new extraction method using microwave-assisted extraction (MAE) combined with Py–GC/MS was developed to extract and quantify a wide range of plastic polymers, and the method was validated using different environmental matrices. This new extraction method was able to extract polyethylene, polystyrene, polypropylene, poly(methyl-methacrylate) (PMMA), polyvinylchloride (PVC), and polycarbonate in dichloromethane with good recoveries (92.9–119.7%). The limit of detection and limit of quantification (LOQ) of the method ranged from 0.002 to 0.18 µg and from 1.2 to 5.8 µg, respectively. Intra- and interday repeatability values with coefficients of variation less than 25% for all polymers were obtained. Method validation also included a spike and recovery using all polymers from clean water, dirty water, and shrimp and salmon fillet samples, with recoveries of 85 to 111, 87 to 138, 81 to 122, and 50 to 151%, respectively. Finally, the method was tested on unspiked wild mussels and bottled water for proof-of-concept. Both polyethylene and PVC were detected and quantified in mussels, and polycarbonate and polypropylene were detected below the LOQ. For bottled water, polypropylene, polystyrene, and polycarbonate were all detected below the LOQ. We introduce a method combining MAE and Py–GC/MS as a tool for mass quantification of microplastics. This method can be used as a stand-alone, or as a complementary method to spectroscopic techniques. *Environ Toxicol Chem* 2021;00:1–9. © 2021 SETAC

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INTRODUCTION

Contamination of all environmental compartments by plastic debris of various sizes has been documented globally (Horton et al., 2017; Wang et al., 2021). Under natural conditions, debris consisting of larger plastic pieces breaks down (Gerritse et al., 2020; Napper & Thompson, 2019) into smaller pieces called microplastics (less than 5 mm; Arthur et al., 2009). Due to their physical and chemical properties, microplastics are incredibly persistent in the environment. Plastic production has been increasing since the 1950s (Geyer et al., 2017; PlasticsEurope, 2020), and the concentration of microplastics could rise in the oceans if production and use continue in a scenario of business as usual (Lebreton et al., 2019, 2018). In addition to the environment, microplastics contaminate products that are consumed by humans (Cox et al., 2019). This has led to concerns regarding human health (Wright & Kelly, 2017). Consequently, it is important to be able to measure microplastics in the environment to understand exposure and fate—which are key to informing how to prevent their release and to characterize risk to wildlife and humans.

Historically, microplastics were quantified and characterized via microscopy, with no chemical analysis confirming whether a particle was synthetic (Dehaut et al., 2019; Lenz et al., 2015). Today, chemical characterization of particles suspected to be microplastics is strongly advised, and sometimes considered mandatory, in microplastic analysis (Cowger et al., 2020; Dehaut et al., 2019; Hermsen et al., 2018). To date, chemical characterization of microplastics mostly relies on spectroscopic characterization of individual particles (Renner et al., 2019). Spectroscopic methods, namely, Fourier-transform infrared

(FTIR) and Raman spectroscopy are most commonly used today (Dehaut et al., 2019; Rocha-Santos & Duarte, 2015). These techniques are often performed manually, one particle at a time. Automated techniques have been developed (Primpke et al., 2019; Sobhani et al., 2019), but they are not commonly used. Some limitations of the spectroscopic approach are the amount of time it takes to analyze a sample, the detection limit for particle size, the difficulty of getting good spectra with complex particle chemistries, and that results, expressed as a count/volume or mass, do not always compare with toxicological studies using metrics expressed as a mass concentration (Bucci et al., 2020).

Other techniques can be used to detect smaller particles and achieve results expressed as a mass concentration (Dehaut et al., 2020). These include thermoanalytical techniques. One of these techniques is pyrolysis coupled to gas chromatography with mass spectrometry (Py–GC/MS; Dehaut et al., 2020). The Py–GC/MS technique was first used in this field for single particle characterization (Doyen et al., 2019; Fries et al., 2013; Hendrickson et al., 2018; Nuelle et al., 2014), similar to spectroscopy; however, it has been rapidly developed for whole sample mass quantification (Fischer & Scholz-Böttcher, 2017), with a growing interest over the past few years (Funck et al., 2020; Gomiero et al., 2019; Okoffo et al., 2020; Steinmetz et al., 2020).

To complement and implement this method broadly, extraction and sample preparation techniques for Py–GC/MS have been developed. The first technique, developed by Fischer and Scholz-Böttcher (2017, 2019), consisted of an extensive clean-up procedure to reduce organic matter, and is time consuming. Other pretreatment techniques involve dissolving microplastics in solvent (Steinmetz et al., 2020) or using techniques such as pressurized liquid extraction (Dierkes et al., 2019; Okoffo et al., 2020). More recently, La Nasa et al. (2021) proposed a method to only extract polyethylene, polystyrene, and polypropylene from samples using microwave-assisted extraction (MAE). This seems to be a promising technique to extract microplastics from environmental samples in a timely manner for their quantification by Py–GC/MS; however, MAE needs to be applied to a wider range of polymers to complement the study done by La Nasa et al. (2021). The objectives of the present study were to 1) develop an MAE method combined with Py–GC/MS to quantify a wide range of plastic polymers, 2) evaluate the performance of the method, and 3) validate the method using environmental samples.

MATERIALS AND METHODS

Reference plastic and chemicals

Reference plastics were obtained from different sources. Polycarbonate was obtained from Goodfellow; polystyrene, poly(methyl-methacrylate) (PMMA), and polyvinylchloride (PVC) were obtained from plastic products used to create Spectral Libraries of Plastic Particles (Munno et al., 2020). The polypropylene and polyethylene terephthalate (PET) were obtained from Sabic Innovative Plastics and were ground to an

microplastic size using a toll grinder with liquid nitrogen by Custom Processing Services. The polyethylene was obtained from a commercially available product (soap bottle). Each plastic type was first confirmed by Py–GC/MS (see the *Py–GC/MS analysis* section and the Supporting Information Materials and Methods and Figure S1) and μ-Raman spectroscopy before use (Supporting Information Materials and Methods and Figure S2). Dichloromethane (DCM) ACS grade was obtained from Sigma-Aldrich. Polystyrene-d5, used as an internal standard, was obtained from PolymerSource).

MAE

Extraction of the plastic polymers to make standards and to extract plastics from samples was conducted using an MAE Mars6 system (CEM). To dissolve plastic polymers in solution, and thus make standards and extract samples, several parameters were tested. The best method included adding samples to an MAE tube (made of perfluoroalkoxy alkane) with 25 ml of DCM with the following parameters: OneTouch method, 180 °C maintained for 15 minutes. Once the extraction was complete, the DCM containing the standard or sample was transferred to a clean glass tube. For samples, each tube was centrifuged for 5 minutes at 2246 g at 18 °C using a 5810 R centrifuge (Eppendorf), and the supernatant was then transferred to a new clean glass tube. The centrifuge step was not included in the preparation of making a standard solution. For standard solutions and samples, the DCM solution was then evaporated to 2 ml under nitrogen using a TurboVap (Biotage) equipped with a water bath set at 25 °C. The final concentrated sample or standard was then transferred to a clean amber glass GC vial (Agilent) and stored in the dark at 4 to 5 °C until analysis.

Py–GC/MS analysis

The analytical method we used was adapted from Fischer and Scholz-Böttcher (2019). The sample cup containing the standard solution or sample was placed on an AS-1020E autosampler of an EGA/PY3030-D pyrolyzer (Frontier Lab). Samples were pyrolyzed at 590 °C for 12 seconds, and pyrolysis products were injected with a split of 15, on an 8890 GC (Agilent) equipped with an HP-5 ms column (30 m; 0.25 mm; 0.25 μm thickness; Agilent). Pyrolyzer interface and injection port temperatures were set at 300 °C. Helium was used as the carrier gas at a constant flow of 0.8 ml/minute. The oven program was set as follows: 35 °C for 2 minutes, then increased to 310 °C at 3 °C/minutes, and held for 30 minutes. Mass spectra were obtained using an Agilent 5977B MS device. Interface temperature between both instruments was set at 280 °C, source temperature was set at 230 °C, quad temperature was set at 150 °C, ionization voltage was set at 70 eV, and a mass range from 50 to 650 amu was scanned at 2.5 scans/second. Mass spectrum acquisition was started after a solvent delay of 3 minutes. Quantification was completed in MassHunter Qualitative Analysis software (Agilent).

Polymer identification and quantification

Polymer identification and quantification were conducted using characteristic peaks (Supporting Information, Table S1 and Figure S3) published in the literature (Fischer & Scholz-Böttcher, 2019; Hermabessiere et al., 2018; Okoffo et al., 2020; Tsuge et al., 2011). External calibration curves were prepared from standard solutions comprised of a known concentration of each polymer. The solutions were prepped by weighing reference plastic polymers with a precision balance (Explorer 125D; Ohaus) and dissolving them in a measured volume of DCM using the MAE method previously described in the *MAE* section. Calibration curves were comprised of seven levels, which varied slightly depending on the mass of the polymers, obtained by adding 0.5, 1, 1.5, 2, 3.5, 5, and 10 µl of the standard solution to a pyrolysis cup. Solutions were evaporated in the cup for 30 minutes prior to the addition of 5 µl of a polystyrene-d5 solution at 0.122 mg/ml in DCM as an internal standard. Calibration curves had concentrations ranging from 0.5 to 10 µg for polyethylene and PVC, and 0.25 to 5 µg for polypropylene, polystyrene, PMMA, and polycarbonate. Individual calibration curves were produced using the relative response of each polymer by integrating their characteristic peak (Supporting Information, Table S1) and the Styrene-d5 peak (m/z = 109).

Method validation

Plastic recoveries from MAE extraction.

Plastic recoveries by MAE were calculated using the dry weights of each polymer obtained after extraction. For each plastic polymer, recoveries were measured in triplicate. Polymers were extracted individually versus as a mixture. Before extraction, the mass of the plastic ($W_{plastic}$) and empty clean glass tubes (W_{empty}) were measured ($n = 3$; Explorer 125D). Plastic polymers were extracted using the MAE method described in the *MAE* section, and extracts were evaporated to dryness under nitrogen in a water bath at 25 °C using a TurboVap. The mass of the glass tube containing the MAE extract ($W_{extract}$) was then reweighed. Recoveries were calculated as follows:

$$\text{MAE recovery (\%)} = \left(\frac{W_{extract} - W_{empty}}{W_{plastic}} \right) \times 100$$

Precision and limit of detection/limit of quantitation assessments.

Intra- and interday repeatability were evaluated for all polymers using a standard solution with all polymers extracted in a mixture. For intraday repeatability, five replicates of a 2.5-µl solution (polyethylene and PVC: 1 µg/µl and polypropylene, polystyrene, PVC, and polycarbonate: 0.5 µg/µl) were analyzed on the same day. For interday repeatability, one replicate of 2.5 µl/day was analyzed for five consecutive days using the same solution as for intraday repeatability. Precision was evaluated by calculating relative standard deviations (RSD) of peak areas among sample runs.

To calculate the limit of detection (LOD) and limit of quantitation (LOQ), three calibration curves were prepared from standard solutions in the range of 0.5 to 10 µg for polyethylene and PVC and in the range of 0.25 to 5 µg for polypropylene, polystyrene, PMMA, and polycarbonate. Using those calibration curves (Supporting Information, Figure S4), the LOD and LOQ were calculated according to Steinmetz et al. (2020) using the R package "envalysis" (Ver 0.3.3; Steinmetz, 2020).

Plastic recoveries from environmental samples.

To measure plastic polymer recoveries from extracted samples, we performed spike and recoveries using various matrices. To create spike material, different reference polymers were weighed using a precision balance (Explorer 125D; Ohaus). Each polymer was weighed three times to obtain a mean weight. In total, 10 spike mixtures of plastic polymers (polyethylene, polypropylene, polystyrene, PMMA, PVC, and polycarbonate) were prepared and used to spike the following matrices: reverse osmosis water ($n = 3$), dirty reverse osmosis water ($n = 3$), shrimp tissue ($n = 2$), and salmon fillet tissue ($n = 2$). Briefly, for reverse osmosis water and dirty reverse osmosis water, 1 l of water was filtered onto a 25-mm-diameter glass fiber filter (GFF) with a porosity of 1 µm (GF/B; Whatman). For the dirty water sample, some soil sampled outside the facility was added (0.1 g) to the 1 l of reverse osmosis water before filtration. The soil was not characterized and was used to mimic a more complex water sample than reverse osmosis water alone. Shrimp were digested using 40 ml of a 20% (w/v) KOH (Fischer Scientific) solution and were incubated at 60 °C for 24 hours in an oven (Model 6; Thelco) and then filtered onto 25-mm-diameter GF/B filters. For salmon, fillets were digested using 250 ml of 20% (w/v) KOH solution and then incubated at 45 °C for 48 hours in an oven (Model 6; Thelco). The digested solutions were sieved onto a 212-µm sieve, and the fraction less than 212 µm was used. A prepared detergent, 10% Alcojet (w/v; Fischer Scientific), was added to the less than 212-µm digested fraction to a ratio of 1/1 (v/v) to digest the fatty material. The sample was then filtered onto 47-mm-diameter polycarbonate filters with a porosity of 20 µm (Merck Millipore) and 10 µm (Sterlitech). Finally, the fraction less than 10 µm was filtered on 47-mm-diameter GFF with a porosity of 1 µm (GF/B), and these filters were used for analysis. Plastic particles were added manually using tweezers onto the filters once dried. Filters were then added to MAE tubes, and the extraction proceeded as just described. Calibration curves were in the range of 0.5 to 20 µg for polyethylene and PVC and in the range of 0.25 to 10 µg for polypropylene, polystyrene, PMMA, and polycarbonate (Supporting Information, Figure S5). Recoveries were calculated using the formula below:

$$\text{Recovery (\%)} = \frac{\text{Mass recovered}}{\text{Mass spiked}} \times 100$$

Mass recovered is the mass of plastic calculated using the calibration curve (Supporting Information, Figure S5) and mass spiked is the mass of plastic added to the samples (Supporting Information, Table S5).

Method implementation using samples from the environment

The MAE and Py–GC/MS method was implemented on samples obtained from local grocery stores: bottled water and mussels (*Mytilus sp.*). Bottled water (*n* = 5) was filtered (1.5 l) onto 25-mm-diameter GFF with a porosity of 1 μm (GF/B). Filters were stored in clean glass Petri dishes until extraction. Mussels (*n* = 5; Supporting Information, Table S2) were digested using 50 ml of a 20% (w/v) KOH solution and incubated at 60 °C for 24 hours in an oven (Model 6; Thelco). Similar to bottled water, samples were filtered onto GF/B filters (25 mm diameter) and kept in clean glass Petri dish until extraction. For mussels, two filters were used for filtration due to a clogging issue, and both filters were extracted in the same MAE tube. The MAE extraction, Py–GC/MS analysis, and quantification of plastic polymers (Supporting Information, Figure S6) were conducted as described in the previous sections, *MAE*, *Py–GC/MS analysis*, and *Plastic recoveries from environmental samples*.

Quality assurance/quality control

To avoid procedural- and cross-contamination, preventive measures were applied. Cotton laboratory coats and gloves were worn at each step of the analysis. Analysis was done in a laboratory using an air filtration system with HEPA filters, and all surfaces were cleaned with reverse osmosis water before use. All glassware used for microplastic extraction was washed with soap and water, rinsed three times with reverse osmosis water, and baked in a muffle furnace (Thermolyne F30400; Thermo Scientific) at 450 °C for 7 hours. In addition, all GFF used in the present study were also baked at 450 °C for 7 hours before use. For digestion of the organisms, KOH solution and Alcojet solution were filtered before use through a 47-mm-diameter glass fiber filter (GFF) with a porosity of 1 μm (GF/B). Before MAE extraction, empty MAE tubes were cleaned by performing an extraction using the same parameters as samples with 25 ml of clean DCM. For each set of samples, blank samples were run through an MAE tube and processed

as for samples. In addition, for each Py–GC/MS sequence, empty pyrolysis cups were analyzed before and after the sequence to verify that leftover pyrolysis products were not contaminating the system. Also, empty pyrolysis cups were analyzed during the sequence at least every 10 samples to minimize contamination between samples.

RESULTS AND DISCUSSION

Method validation

Plastic recoveries from MAE extraction. Using MAE extraction, we were able to achieve plastic polymer recoveries (Supporting Information, Table S3) in pure DCM 80 and 120% for all polymers except PET (Table 1). Recoveries ranged from a mean of 92.9% for polypropylene to 119.7% for PVC; PET had a recovery of 0% (Table 1). Using the MAE method, we were unable to dissolve PET. Unfortunately, this meant that PET was no longer considered within the present study and is not included in our analysis. This is consistent with another study using pressurized liquid extraction in which PET had low recoveries when spiked in seafood products (Ribeiro et al., 2020). Further technical developments should be tested to extract PET with MAE. In the present study we only tested DCM for the extraction of microplastics, as was used in other studies (La Nasa et al., 2021; Okoffo et al., 2020). Other solvents should also be explored. In addition, increasing temperature and time of extraction might help dissolve PET using MAE. Still, we were able to extract and recover a wider range of polymer types than could be extracted and recovered by La Nasa et al. (2021) using MAE. This difference may be attributed to a difference in MAE parameters. In the present study, a higher volume of DCM was used and the extraction temperature was higher, 180 °C (15 minutes), in comparison with the 80 °C (60 minutes) of La Nasa et al. (2021).

Precision and LOD/LOQ assessments. We measured the precision of our method by measuring the RSD between samples run on the same day and on different days (Supporting Information, Table S4). For intraday repeatability, the RSDs

TABLE 1: Summary of method validation experiments including microwave-assisted extraction recovery, repeatability including intraday and interday, and limit of detection and limit of quantification assessment

Polymer	Characteristic peak	MAE recovery (%)[a]	Intraday RSD (%)	Interday RSD (%)	Calibration range (μg)[b]	Calibration function	R^2	LOD (μg)	LOQ (μg)
PE	C_{10}	103.4 ± 1.6	14.1	14.8	0.51–10.3	$Y = 0.11x - 0.076$	0.99	0.18	2.3
	C_{17}		19.7	21.1		$Y = 0.066x - 0.076$	0.97	0.20	3.2
PP	2,4-Dimethyl-1-heptene	92.9 ± 16.3	21.7	16.1	0.25–4.95	$Y = 0.42x - 0.11$	0.98	0.051	1.2
PS	Styrene trimer	108.3 ± 2.7	21.5	18.2	0.26–5.2	$Y = 0.46x - 0.22$	0.99	0.0024	1.2
PMMA	Methyl-methacrylate	116.9 ± 1.5	9.5	14.3	0.27–5.4	$Y = 1.32x - 0.003$	0.97	0.0047	1.6
PVC	Indene	119.7 ± 1.0	23.6	12.4	0.50–9.95	$Y = 0.088x - 0.003$	0.98	0.058	2.3
PC	4-Isopropenylphenol	116.6 ± 5.8	13.9	15.7	0.28–5.55	$Y = 0.28x + 0.071$	0.89	0.0022	5.8
	Bisphenol A		42	81.7	—[c]	—[c]	—[c]	—[c]	—[c]

[a]Expressed as mean ± standard deviation (*n* = 3).
[b]Expressed as the mass in the pyrolysis cup.
[c]No data taken for bisphenol A because repeatability intraday and interday showed high relative standard deviation.
MAE = microwave-assisted extraction; RSD = relative standard deviation; LOD = limit of detection; LOQ = limit of quantification; PE = polyethylene; PP = polypropylene; PS = polystyrene; PMMA = poly(methyl-methacrylate); PVC = polyvinylchloride; PET = polyethylene terephthalate; PC = polycarbonate.

ranged from 9.5 to 42%. For interday repeatability, the RSDs ranged from 12.4 to 81.7% (Table 1). Bisphenol A, used as the characteristic peak for polycarbonate, had the highest RSD for both intraday and interday repeatability due to strong peak area variability (Supporting Information, Table S3). Consequently, 4-isopropenylphenol was chosen as the polycarbonate characteristic peak because it has intraday and interday repeatability RSD values of 13.9 and 15.7%, respectively (Table 1). By excluding bisphenol A, intraday and interday repeatability ranged from 9.5 to 23.6% and 12.4 to 21.1%, respectively. These measurements of intraday and interday repeatability are in the range of other studies using Py–GC/MS for plastic polymer quantification (Dierkes et al., 2019; Funck et al., 2020; La Nasa et al., 2021; Okoffo et al., 2020; Ribeiro et al., 2020; Steinmetz et al., 2020). Ribeiro et al. (2020) observed an RSD of 17% for the styrene dimer (3-butene-1,3-diyldibenzene), and intraday and interday repeatability between 3.1 and 12% for polyethylene, PMMA, polystyrene, PET, polycarbonate, polypropylene, and PVC (Okoffo et al., 2020). Other studies observed RSDs of less than 15% for repeatability, but only analyzed a handful of polymers (polyethylene, polypropylene, or polystyrene; Dierkes et al., 2019; Funck et al., 2020; La Nasa et al., 2021; Steinmetz et al., 2020). Still other studies did not measure this parameter, and it is not reported in their method development (Gomiero et al., 2019; Kirstein et al., 2021).

The LODs and LOQs calculated for this method are consistent with those in the literature. In the present study, the LODs ranged from 0.002 for polycarbonate to 0.2 μg for polyethylene (alkene C_{17} peak) and the LOQs from 1.2 for polypropylene and polystyrene to 5.8 for polycarbonate (Table 1). In another study using MAE and Py–GC/MS to analyze polyethylene, polypropylene, and polystyrene from sand samples, the LODs were 0.13, 0.26, and 0.05 μg, respectively, and the LOQs were 0.44, 0.85, and 0.20 μg, respectively (La Nasa et al., 2021). In comparison, the LODs we present are lower for polypropylene and polystyrene, and comparable for polyethylene. The LOQs presented by La Nasa et al. (2021) are lower than the LOQs we calculated. La Nasa et al. (2021) do not provide a description of the procedure for calculating LOQs, and thus it is not possible to compare reasons for the discrepancy between results.

Plastic recoveries from environmental samples. Overall, we achieved good recoveries from our spike and recovery trials with different matrices (Supporting Information, Table S5). For reverse osmosis water, the mean plastic polymer recoveries ranged from 85.7% (±5% SD) for polypropylene to 111.4% (±6.3%) for PVC (Table 2). For dirty reverse osmosis water, the mean plastic polymer recoveries ranged from 86.9% (±12.2%) for polypropylene to 138.6% (±33.2%) for PVC (Table 2). For these two matrices, apart from PVC in dirty reverse osmosis water, the recoveries were from a target of 80 to 120%. For shrimp, mean plastic polymer recoveries ranged from 81.9% (±5.6%) for polypropylene to 122.8% (±15.1%) for PMMA (Table 2). The PMMA was the only polymer with a mean recovery outside the range of 80–120%. Finally, for salmon fillets, mean plastic polymer recoveries were more variable, and ranged from 51.1% (±22.6%) for polypropylene to 150.9% (±57.8%) for polycarbonate (Table 2). For salmon, recoveries were less than 70% for PMMA and polystyrene (66 ± 6.5 and 68.9 ± 4.6%, respectively) and fell in the range of 80–120% for polyethylene and PVC (99.8 ± 5.7 and 118.9 ± 8.8%, respectively; Table 2).

It is worth noting that for polyethylene both C_{10} and C_{17} were used to calculate recoveries. Depending on sample complexity, quantification should be done with either one peak or the other. For simpler matrices, like reverse osmosis water, dirty water, or shrimp, using either C_{10} or C_{17} gave similar recoveries (Table 2). Unlike simple matrices, for salmon fillets recoveries for polyethylene were different depending on which peak was used: 225% with C_{10} and 98% with C_{17} (Table 2). In the present study, fatty acids present in the fish fillet increased the alkene C_{10} peak response, leading to recoveries higher than 200%. As stated by Fischer and Scholz-Böttcher (2017), interferences decrease with increasing carbon numbers, which was confirmed in the present study. For more complex matrices such as biota, polyethylene quantification should be done with alkenes that have carbon numbers higher than or equal to C_{16} (Fischer & Scholz-Böttcher, 2017). In the present study, polypropylene had the lowest recoveries across all four matrices, which is opposite to what was reported by Steinmetz et al. (2020), who demonstrated that polypropylene was overestimated due to interferences with polyethylene.

TABLE 2: Plastic polymer recoveries (in %) obtained after microwave-assisted extraction extraction and pyrolysis–gas chromatography/mass spectrometry analysis from reverse osmosis water, dirty reverse osmosis water, shrimp, and salmon fillet expressed as mean ± standard deviation (*n* = 2 or 3)

Polymer	Characteristic peak	Recoveries (%)[a]			
		RO water	Dirty water	Shrimp	Salmon fillet
PE	C10	91.6 ± 12	98.2 ± 14.7	110.8 ± 18.7	225.6 ± 46.9
	C17	103 ± 12.5	100.7 ± 14.7	116.9 ± 20.1	98.8 ± 5.7
PP	2,4-Dimethyl-1-heptene	85.7 ± 5	86.9 ± 12.2	81.9 ± 5.6	51.1 ± 22.6
PS	Styrene trimer	89.5 ± 4	88.5 ± 7.9	92.4 ± 5.1	68.9 ± 4.6
PMMA	Methyl-methacrylate	90.1 ± 19.6	107.6 ± 18.1	122.8 ± 15.1	66 ± 6.5
PVC	Indene	111.4 ± 6.3	138.6 ± 33.2	107.8 ± 28.4	118.9 ± 8.8
PC	4-Isopropenylphenol	102.9 ± 7.1	90.1 ± 9.6[b]	113.6 ± 20.2	150.9 ± 57.8

[a]Recoveries expressed as mean ± standard deviation (*n* = 3) for reverse osmosis water and dirty water and *n* = 2 for shrimp and salmon fillet).
[b]*n* = 2 due to a spiking issue.
Py–GC/MS = pyrolysis–gas chromatography/mass spectrometry; RO = reverse osmosis. For other abbreviations, see Table 1 footnote.

In comparison with other methods developed using Py–GC/MS for microplastic quantification, the present study demonstrates good recoveries across multiple polymers and for different matrices. In other studies, recoveries were 92% for polyethylene, 83% for polypropylene, and 96% for PVC (Gomiero et al., 2019), 114 to 131% for polyethylene, 85 to 95% for polypropylene, and 77 to 118% for polystyrene (Dierkes et al., 2019), 86% for polyethylene (Funck et al., 2020), and between 70 and 128% for polyethylene, polypropylene, and polystyrene (Steinmetz et al., 2020). In the present study, all polymers extractable with MAE were used to perform a spike and recovery test using different samples having different complexities, resulting in recoveries within the range of 85 to 122% for all polymers in reverse osmosis water, dirty water, or shrimp (Table 2). A higher range of recoveries, from 51 to 151%, was obtained for salmon fillet, which is higher than values in the literature (Dierkes et al., 2019; Funck et al., 2020; Gomiero et al., 2019; Steinmetz, 2020) and is probably due to the fatty nature of fish fillets.

Application of the method to environmental samples

We tested our method using MAE for plastic polymer extraction and Py–GC/MS for quantitative analysis on two matrices. We aimed to measure microplastics in real-world samples that represent a diverse level of complexity, that is, bottled water and mussels (Supporting Information, Table S6). In mussels, we detected polyethylene and PVC greater than the LOQ, and polypropylene and polycarbonate greater than the LOD and less than the LOQ (Table 3). We observed measurable concentrations of polyethylene and PVC between 18.0 and 32.9 μg/g of tissue wet weight and 18.8 and 41.6 μg/g of tissue wet weight, respectively (Table 3).

To date, two studies have quantified microplastics in fish and shellfish (Fischer & Scholz-Böttcher, 2017; Ribeiro et al., 2020). Ribeiro et al. (2020) quantified microplastics in another bivalve, the oyster Crassostrea gigas, and found only PVC, with measurable concentrations up to 23.55 μg/g. Interestingly, both the present study and that of Ribeiro et al. (2020) present findings on PVC using Py–GC/MS, which has rarely been reported on in studies using other techniques (Raman and FTIR spectroscopy; Danopoulos et al., 2020), even though numerous studies have reported on microplastics in bivalves globally (Danopoulos et al., 2020). For bottled water, polypropylene, polystyrene, and polycarbonate were detected, but were not quantifiable because all peak areas were less than the LOQ (Table 2). Previous work on microplastics in bottled water suggests that smaller microplastics (less than 5 μm) are the dominant size class (Oßmann et al., 2018; Schymanski et al., 2018). The small mass of these particles may be why we were not able to detect any polymer greater than the LOQ in such a small volume of water (1.5 l). Detection of microplastics in drinking water will require higher volume samples, as recommended by Koelmans et al. (2019). One study using Py–GC/MS filtered 200 to 1100 l of water, reported microplastic concentrations in drinking water samples from a drinking water treatment plant in the range of μg/m^3 (ng/l), and detected and quantified five different polymers (PVC, PA, polyester, polystyrene, and polypropylene; Kirstein et al., 2021). Thus, it is critical that future studies filter large volumes of water to achieve detection limits.

It is worth mentioning that the present extraction method using MAE followed by quantification with Py–GC/MS for microplastics could be used for nanoplastics (particles smaller than 1 μm; Gigault et al., 2018; Hartmann et al., 2019). However, in the present study our filtration methods excluded the nano-sized fraction. The Py–GC/MS method has been used for nanoplastic detection in water samples from the North Atlantic subtropical gyre after concentration of the water with ultrafiltration (Ter Halle et al., 2017). More recently, accelerated solvent extraction, a method similar to MAE, was used to extract and quantify nanoplastics in oysters with Py–GC/MS (Ribeiro et al., 2021). The analysis of nano-sized plastic particles, similar to smaller microplastics, is difficult and likely requires large volumes of samples and preconcentration steps to reach the LOQ. Still, Py–GC/MS is a promising method for nanoplastics and should be explored further.

We have demonstrated the ability to measure microplastic concentrations using MAE extraction with Py–GC/MS. It is worth noting that concentrations using this method will always be as mass concentrations—which does not always align with

TABLE 3: Concentrations of polyethylene, polypropylene, polystyrene, poly(methyl-methacrylate), polyvinylchloride, polyethylene terephthalate, and polycarbonate in mussels (*Mytilus* sp.) and in bottled water expressed as μg/g wet weight and μg/l for mussels and bottled water, respectively

		Concentration (μg/g or μg/L)					
	Sample ID	PE	PP	PS	PMMA	PVC	PC
Mussels (*Mytilus* sp.)	1	32.9	—	—	—	41.6	—
	2	18.5	<LOQ	—	—	<LOQ	—
	3	18.0	—	—	—	36.1	<LOQ
	4	19.7	—	—	—	29.9	—
	5	20.6	—	—	—	18.8	—
Bottled water	1	—	<LOQ	<LOQ	—	—	—
	2	—	<LOQ	<LOQ	—	—	<LOQ
	3	—	<LOQ	<LOQ	—	—	<LOQ
	4	—	<LOQ	<LOQ	—	—	—
	5	—	<LOQ	<LOQ	—	—	<LOQ

— = not detected in the sample; <LOQ = below the limit of quantification; PET = polyethylene terephthalate. For other abbreviations, see the footnote to Table 1.

the reported metrics from environmental samples and toxicity tests in other studies. Thus, for both matrices we tested, it is challenging to compare our results with results published in the literature because most are expressed as a particle count concentration. Some approaches to convert mass concentrations to count concentrations have been developed (Koelmans et al., 2020; Simon et al., 2018), and performing size fractionation during the extraction phase will enable the conversion from mass to count concentration. This may be less essential for nano-sized particles, because to date nanoplastics are generally reported in mass concentration in toxicity testing. Thus, the mass concentration given from Py–GC/MS is similar to what is done for other nanomaterials (Petersen et al., 2021). Thus, for studying both microplastics and nanoplastics, we need to balance the costs and benefits to each approach—taking into consideration the time, effectiveness, applicability, and limitations of each method. As we converge around methods, if both mass and count concentrations are necessary to answer different questions, such methods of conversion can be applied in future research.

CONCLUSIONS

The present study introduces a new method using MAE extraction with Py–GC/MS to extract and quantify microplastics in real-world samples. We demonstrate that MAE with DCM heated at 180 °C for 15 minutes is suitable to extract a wide range of polymer types: polyethylene, polypropylene, polystyrene, PMMA, PVC, and polycarbonate. We show that this method is suitable for various environmental matrices with low matrix interference. Further development may be desired to allow for extraction of PET and better recovery with some complex matrices such as fatty fish fillets. The validation of this method shows that quantification of microplastics using Py–GC/MS is reliable and can achieve low detection limits, to smaller than 1 µg. In addition, Py–GC/MS has the advantage of providing a mass concentration/sample, which is common for other emerging contaminants of interest. We recognize the value of count concentrations and that understanding the size, shape, and color distribution of particles can inform source-reduction and/or particle toxicity.

It is also important to highlight that Py–GC/MS can complement, versus replace, spectroscopic methods as already proposed in previous research (Hermabessiere et al., 2018). The present method using Py–GC/MS can be used as a quantitative tool on its own or as a complement to spectroscopic techniques to assess microplastic contamination in various environmental matrices. The Py–GC/MS technique can also be used as a preliminary tool to achieve an overall quantification, and if concentrations are larger than a level of concern, more time-consuming techniques such as FTIR or Raman spectroscopy can be used to obtain information about size, shape, and color to inform source-reduction and/or mechanisms of toxicity.

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