Analytical Methods

CRITICAL REVIEW

View Article Online
View Journal | View Issue

Cite this: *Anal. Methods*, 2017, **9**, 1369

A review of analytical techniques for quantifying microplastics in sediments

Joanne S. Hanvey,[a] Phoebe J. Lewis,[a] Jennifer L. Lavers,[b] Nicholas D. Crosbie,[c] Karla Pozo[de] and Bradley O. Clarke*[a]

Received 29th September 2016
Accepted 22nd December 2016

DOI: 10.1039/c6ay02707e

www.rsc.org/methods

In this review the analytical techniques for measuring microplastics in sediment have been evaluated. Four primary areas of the analytical process have been identified that include (1) sampling, (2) extraction, (3) quantitation and (4) quality assurance/quality control (QAQC). Each of those sections have their own subject specific challenges and require further method development and harmonisation. The most common approach to extracting microplastics from sediments is density separation. Following extraction, visual counting with an optical microscope is the most common technique for quantifying microplastics; a technique that is labour intensive and prone to human error. Spectroscopy (FTIR; Raman) are the most commonly applied techniques for identifying polymers collected through visual sorting. Improvements and harmonisation on size fractions, sampling approaches, extraction protocols and units for reporting plastic abundance would aid comparison of data generated by different research teams. Further, we advocate the development of strong QAQC procedures to be adopted like other fields of analytical chemistry. Finally, inter-laboratory proficiency testing is recommended to give an indication of the variation and reliability in measurements reported in the scientific literature that may be under- or overestimations of environmental burdens.

1 Introduction

Since the introduction of plastics into western society in the 1950s, deliberate and accidental environmental release has resulted in ubiquitous environmental contamination with plastics debris. As a consequence, plastic pollution has been recovered throughout the planet, in remote regions far from known point sources – such as Antarctica,[1] remote mountain-tops[2] and the deep sea ocean floor[3] – environments that should be pristine, free of human impact. The first reports of marine plastic pollution in the early 1970s[4] were followed by similar reports throughout the world finding widespread, ubiquitous contamination.[5,6] Later analysis of archived samples revealed a dramatic increase of marine plastic debris and microplastics through the late twentieth century.[7] Efforts to understand the extent of widespread environmental contamination and subsequent ecological harm caused by plastics debris are ongoing.[8,9] To develop appropriate management and remediation strategies for this global pollution problem we must have reliable and consistent analytical techniques for measuring plastics in environmental matrices. In this review, we will examine analytical techniques reported in the English language peer-review scientific literature for quantifying microplastics in sediments samples.

There are a wide variety of polymers detected in the environment, with the main types being polyethylene (PE), polypropylene (PP) and polystyrene (PS; see Table 1 for a list of common plastics and their uses). Microplastic debris is broadly classified as either (1) primary microplastics that include production pellet nurdles and microbeads (abrasive particles incorporated into personal care products) or (2) secondary microplastics which are formed by mechanical and chemical breakdown of larger plastic debris and fibres released from synthetic textiles (referred to as microfibers).[10] Environmental plastic litter has been broadly categorised within the literature and it was not until 2009 that 'microplastics' were defined as plastic particles smaller than 5 mm by the National Oceanic and Atmospheric Administration (NOAA) International Research Workshop on the occurrence, effects and fate of microplastic marine debris.[11–13] Initial reports of microplastics in sediments (published in 2004) defined them as being <1 mm (presumably as it was micro (μ) in size)[7] and various authors since have continued to apply this definition.[10] Variations in the collection of size fraction makes the comparison of data between regions

[a]*School of Sciences, Centre for Environmental Sustainability and Remediation, RMIT University, GPO Box 2476, Melbourne, Vic. 3001, Australia. E-mail: bradley.clarke@rmit.edu.au*

[b]*Institute for Marine and Antarctic Studies, University of Tasmania, 20 Castray Esplanade, Battery Point, Tasmania, 7004, Australia*

[c]*Melbourne Water, 990 LaTrobe St, Melbourne, Australia*

[d]*Facultad de Ciencias, Universidad Católica de la Santísima Concepción, Alonso de Ribera 2850, Concepción, Chile*

[e]*RECETOX Research Center for Toxic Compounds in the Environment, Faculty of Science, Masaryk University, Kamenice 753/5, pavilon A29 625 00, Brno, Czech Republic*

Table 1 List of common types of plastics (abbreviation) and their common usage

Type of plastic	Abbreviation	Application of virgin resin	Density (g cm^{-3})
Polyethylene terephthalate	PET/PETE	Disposable beverage bottles, textiles (synthetic fibers), tape, Mylar food packaging and thermal insulation	1.37–1.38
High density polyethylene	HDPE	Plastic bags, plastic lumber, fuel tanks, bottle caps, milk crates	0.93–0.97
Polyvinyl chloride	PVC	Plumbing pipes, door and window frames, garden hoses, electrical cable insulation, inflatable products	1.10–1.47
Low density polyethylene	LDPE	Plastic bags and films six-pack rings, flexible snap-on lids	0.91–0.92
Polypropylene (expanded or non-expanded)	PP	Bottle caps, rope, carpet	0.89–0.92
Polystyrene (expanded or non-expanded)	PS	Disposable cutlery, dinnerware, and take-away food packaging, building insulation, refrigerated bins (*e.g.*, fish boxes)	0.28–1.04
Other resins, such as polycarbonate, nylon, and acrylic	Other	Used for engineering purposes because of their thermal, electrical and chemical properties. *e.g.*, electrical wire insulation	1.15–1.22

and matrices problematic. The elimination of the 1–5 mm or <1 mm size fraction from the study designs will underestimate the amount and type of microplastic present (*e.g.*, production pellet nurdles are typically 2–3 mm while microfibers are often <1 mm) and the overall conclusions drawn.[14] Another further complication is lack of consistency in reporting plastic concentration (defined as amount per medium), units of abundance (or mass) per mass,[15] volume,[10] area[16] and even length.[17] In this review, microplastics have been defined as <5 mm consistent with the NOAA workshop consensus definition. However, as far as the authors are aware the lower limit remains undefined, although most recognise it as 333 μm due to the common use of 333 μm mesh nets used in the field to capture plankton and floating debris.[13] The smallest size fraction reported in environmental samples is 1 μm through the application of this size-filter, however it is very difficult to visually detect plastics <100 μm in size even using a microscope.[18] Below 1 μm scale 'nanoplastics' are the least known area of marine litter, and constitute a very recent area of the environmental science and to date, no studies have successfully accounted for nanoplastics in either freshwater or marine environments.[19] The terminology applied inconsistently through the scientific literature would benefit from harmonisation, aiding consistent study objectives related to size fraction and generation of comparable data sets. A summary of the proposed terminology is listed in Table 2 (please note the size range categories do overlap).

Table 2 Summary of proposed terminology

Size range	Proposed terminology
>20 cm	Macroplastic[29]
5–20 cm	Mesoplastic[29]
1–5 mm	Large microplastic[29]
1–1000 μm	Small microplastic[29]
<1000 nm	Nanoplastic[19]

Analytical approaches applied to measuring plastic debris in environmental samples are not well-developed.[9] Comparison with well-established analytical techniques for quantifying trace level pollution could provide valuable strategies related to microplastics quantitation, but also particularly for quality assurance/quality control (QA/QC). For example, routine practices in other environmental science would require appropriate field and laboratory blanks, laboratory control samples, matrix spikes and recovery standards as part of the analytical process. One key difference between chemical quantification and plastics quantification is diversity of polymers with respect to type, size, colour, and morphology combined with the lack of homogeneity within environmental samples. This causes unique problems (compared to traditional chemical testing) at every stage of the analytical process (sampling, extraction and quantification). Most studies evaluated include a visual counting procedure as part of the analytical process. Human bias has been demonstrated with variable recoveries based upon polymer colour with blue fragments having the highest detection probability, while white fragments had the lowest.[20,21] Furthermore, other factors that impact the quantity reported are the analyst, their experience, as well as other biological material present in the sample that could be confused with plastic.[21] Visual inspection and counting with a microscope is an important technique as it is cheap and non-destructive for sample processing. Visual inspection is an unreliable analytical process, particularly in the lower smaller microplastics (<1 mm) and nanoplastic (<1 μm) range, likely to be generating unreliable and incomparable data.[14]

Currently there are no universally accepted methods for any of these matrices and the methods available all have potential biases.[9] First steps towards addressing the plastics issue should include the promulgation of standard approaches and methods

for collecting, archiving, and reporting data.[22] Studies tend to concentrate on surface sediment debris (*e.g.* beach surveys) or buried debris (*e.g.* core samples), but rarely do they consider both. Without quantifying the debris in both surface sediment and deeper sediment, a true evaluation of the microplastic abundance cannot be achieved. Due to limits in analytical techniques accurately quantifying microplastics in sediments, the percentage of plastic pollution in the microplastic scale is yet to be accurately determined within these environments. To date, the lower size limit used in microplastic assessment studies is highly dependent on the sensitivity of the sampling and extraction technique applied and can potentially lead to reports that underestimate concentrations.[23] Numerous problems associated with the characterisation and quantification of microplastics in sediments can make inter-study comparisons problematic, if not impossible. For example, method inconsistencies between sampling techniques leading to a variety of sampling units (*i.e.*, mass, volume, including variations in the masses and volumes, area and length) reported and also differences in the lower and upper size limit of microplastics implemented.[24] Consistent analytical techniques are important for not only defining the extent of the plastic pollution in the environment but in determining adequate risk mitigation techniques to protect overall freshwater or marine environments.[25]

2 Studies reviewed

In this review, we have examined reports that have quantified microplastics in sediment and collated the reported analytical techniques (Table 3). We have confined this review to English language peer-reviewed scientific studies that have included quantitative measurements of microplastics (<5 mm in diameter) within a certain volume of sediment (marine, freshwater) samples (defined as matter that settles to the bottom of a liquid). Previous reviews in this field have included papers that broadly defined plastics in the environment using terms such as 'marine litter', 'marine debris' or 'plastic fragments'.[23,24,26] We have excluded studies that have reported microplastics on the beach surface sampled with the naked eye rather than within sediment. Further criteria for inclusion in this review were that the authors (i) report microplastic particles within the limits defined by the NOAA workshop (<5 mm); (ii) include information on the sampling, extraction and quantification techniques used to recover microplastics from within sediment matrices and (iii) due to developments within this novel field, were conducted within the past two decades. A total of 43 studies of microplastics in sediments have been evaluated that include data from 18 countries, a global study,[27] plus deep-sea samples, mostly from the Northern Hemisphere (Table 3).

Generally, there are three main aspects of the analytical process for measuring microplastics in sediment samples; they are: (1) sampling, (2) extraction, and (3) quantification. The information provided in each of these sections has been collated and is presented in Table 3 along with a discussion below using these headings. Inclusion of QA/QC practice is missing from most studied evaluated with only 19% including or describing QA/QC approaches. Therefore, we have included a fourth section on this topic. This review provides an overview and comparison of analytical methods applied within the past two decades for quantifying microplastics in sediments. While we do provide a summary of the data reported, it is not our intention or aim to summarise the findings, only the analytical approaches. There is a significant amount of further work required by analytical chemists to harmonise the analytical approaches taken with respect to sampling, extraction, quantification and QA/QC approaches so that reliable and comparable measurements of environmental burdens are reported.

3 Analytical methods

3.1 Sampling

Collection of appropriate samples is the first critical step in quantifying microplastics in the environment. Fig. 1 demonstrates the various sampling locations around the world, as well as differing sample types. All plastic debris that is found in the sediment will have been transported by water movement (rivers, ocean currents[28]) or air from the emission point-source. It is estimated that there are at least 5.25 trillion particles weighing 270 tons at depths between 0.5 and 2 meters below the sea surface.[29] It is likely that land-based emissions that enter rivers are a significant pathway of pollution to the aquatic environment.[30,31] Attention has been focussed on wastewater treatment plants (WWTPs) as a source of pollution through (1) effluent discharges (2) sewage overflow during high rain events[30] (3) runoff from sewage-based fertiliser land application.[32,33] However, evidence is mounting that WWTPs are also a source of small microplastics[34] and microfibers[10] with larger microplastics (*e.g.*, production pellet nurdles) and macroplastics removed during the treatment process. The contribution of rivers to aquatic pollution could be significant. For example, the Danube river in Germany is estimated to release 4.2 tonnes of plastic debris per day into the Black Sea.[35] In Paris, novel data on atmospheric deposition rates for microplastics combined with estimates for sewage effluent[36] highlighted the significant mass release of plastics, primarily microfibers, associated with an urban environment (3–10 tons per day) into freshwater ecosystems.[37] Proximity to urban centres and onshore ocean currents is thought increase the reported concentrations of marine plastics debris and have an important impact on litter abundances on coastal beaches;[38,39] this is indicative of the percentage of studies that sampled beach sediment (58%) to determine the extent of contamination (Fig. 1c). Therefore, the selection of sampling site will have a significant impact of the abundance and type of plastic reported (see Fig. 1). For example, a South Korean north-side beach contained a 100-fold lower abundance than two south-side beaches that faced southerly wind and currents that were prevalent through the study season.[28] Typically, most studies have reported polyethylene as the major polymer type detected in beach sediment since the 1970s.[40,41] However, this is not always the case and polymer type can vary significantly based upon location and point-source.[28] For example, 90% of the plastics debris recovered from sediments in a South Korean study were styrofoam.[28] Once in the aquatic environment, it is hypothesized that while initially

Table 3 Summary of articles from the English language peer reviewed scientific literature reporting a quantitative measurement of microplastics particles (<5000 μm) in aquatic sediment

Country	Habitat	Size range	Abundance (mean; range)	Sampling	Extraction	Quantification	QA/QC	Reference
Japan, Russia	Beach ($n = 26$)	>300 μm	Japan (2610 pieces per m^2), Russia (32 pieces per m^2)	3 sample locations; 8 L sand; quadrat (40 cm × 40 cm); depth (0–5 cm)	Field density separation (seawater); filtered (0.3 mm mesh net)	Visually counted/sorted (optical microscope); gravimetric		Kusui *et al.*, 2003 (ref. 16)
USA	Beach (9 locations; 2 sites per location)	1–15 mm	NA	Wrack line & berm; quadrat 61 cm × 61 cm; 5.5 cm depth; field sieved (1.15 mm)	Density separation (freshwater) sieved (1, 2.8, 4.75 mm); dried	Visually counted/ sorted; gravimetric		McDermid *et al.*, 2004 (ref. 50)
UK	Estuarine/ subtidal sediment; beach ($n = 17$)	>1.6 μm	NA	Strandline, subtidal; $n = 5$ at each location	Density separation (NaCl 1.2 kg L^{-1}); stirred; filtered (Whatman GF/A 1.6 μm)	Visually counted/ sorted; polymer identification (FTIR)		Thompson *et al.*, 2004 (ref. 7)
Singapore	Beach ($n = 8$)	>1.6 μm	NA	1 cm surface ($n = 4$); sub-surface (10–11 cm, $n = 4$)	Density separation (hypersaturated solution, 1.2 kg L^{-1}); filtered (1.6 μm)	Polymer identification (FTIR)	LCS (3.45 μm PE/ PP spiked into sediment), no data recovery reported	Ng *et al.*, 2006 (ref. 51)
India	Marine sediment (10 locations)	>1.6 μm	81 mg plastics per kg sediment	10 sampling locations; 5 (between high tide a low water mark) × 10 kg samples (each location); depth (0–5 cm); sieved	Density separation (30% NaCl); filtered (1.6 μm)	Visually counted/sorted (optical microscope); polymer identification (FTIR); surface characterisation (SEM)		Reddy *et al.*, 2006 (ref. 15)
USA (Hawaii)	Beach (18 locations)	NA	NA	Strandline; parallel; 150–190 g sampled; additional samples (at 1 beach) perpendicular to shoreline; 1 cm and 10 cm depth; 6 m transect	Density separation (sodium polytungstate 1.4 g mL^{-1}); particles then immersed in 1.2 g mL^{-1} sodium polytungstate & ethanol : water mixtures of various densities, including 1 g mL^{-1}, 0.9549 g mL^{-1}, 0.9408 g mL^{-1}, and 0.911 g mL^{-1}	Visually counted/sorted (stereomicroscope); polymer identification (FESEM; FTIR; micro-ATR)		Corcoran *et al.*, 2009 (ref. 79)
Brazil	Beaches (11 locations)	1–20 mm	NA	Strandline; quadrats (900 cm × 900 cm); 2 cm depth	Oven dried (100 °C); sieved (1 mm); sorted 2–5, <10, <15, <20 mm (small items) <25, <30, <50, <100 mm (medium items); density separation; seawater; debris <1 mm not considered	Visually counted/sorted		Ivar do Sul *et al.*, 2009 (ref. 80)

Table 3 (*Contd.*)

Country	Habitat	Size range	Abundance (mean; range)	Sampling	Extraction	Quantification	QA/QC	Reference
England	Estuary (3 locations, 2 sites)	<1000 μm, 1–10 mm	NA	Strandline; randomly placed quadrates (5 replicates × 50 cm × 50 cm); depth (0–3 cm)	Density separation (saturated NaCl)	Polymer identification (FTIR)		Browne *et al.*, 2010 (ref. 81)
USA (Hawaii)	Beach ($n = 5$)	NA	NA	Transect line 40 m parallel to shoreline; swaths at 10 m intervals perpendicular to shoreline; visible plastic + 5 g surrounding sediment	NA	Polymer identification (FTIR microscopy); polymer characterisation (SEM)		Cooper *et al.*, 2010 (ref. 69)
Brazil	Beach (1 location)	>500–1000 μm	NA	Strandline; 100 m transect; depth (0–2 cm); 9 replicates; quadrat (988 cm × 988 cm); wire cloths field sieving (0.5, 1 mm)	Density separation (filtered seawater)	Visually counted/sorted		Costa *et al.*, 2010 (ref. 82)
Global	Beach sediment (18 locations)	<1000 μm	8–124 pieces per L	Strandline; depth (0–1 cm)	Density separation, 50 mL sediment, saturated NaCl, 3 extractions	Polymer identification (FTIR)		Browne *et al.*, 2011 (ref. 10)
USA (Hawaii)	Beach sediment (5 locations; 3 sites)	<250–5000 μm	1.34% w/w	3 sampling points per location (strandline, 1 m seaward, 2 m past strandline); core sampling (5 cm diameter, 25 depth)	Density separation (NaCl 1.2 g cm^{-3}); sieved (0.25, 0.5, 1, 2, 4 mm)	Visually counted/ sorted; polymer identification (FTIR)		Carson *et al.*, 2011 (ref. 40)
Belgium	Marine sediment (three harbours, 3–4 sites at each)	>63 μm	170 (49–390) pieces per kg	3 sampling points (strandline, intertidal, subtidal zone; parallel); sediment cores (2–7 cm)	Density separation, 1 kg sediment, 3 L conc saline solution; stirred; settle 1 h; sieved (38 μm)	Visually counted/sorted (binocular microscope); polymer identification (FTIR)	LCS; size range 'similar to what was found in field'; 68.8–97.5% recovery	Claessens *et al.*, 2011 (ref. 45)
Portugal	Beach (5 locations)	>1 μm	190 (29–393) pieces per m^2	Strandline; two quadrats (50 cm × 50 cm; 2 m × 2 m) in duplicate; depth (0–2 cm); samples sieved *in situ* (2.5–3.5 mm)	Density separation, concentrated NaCl 140 g L^{-1}; stirred vigorously; filtered (Whatman GF/C 1 μm)	Polymer identification (μ-FTIR)		Martins *et al.*, 2011 (ref. 83)
Canada	Freshwater sediment	<5 mm	NA	Parallel to shoreline; 60 m transect; visible debris sampled (within 1 m); two replicates (2 m × 2 m)	Separation into 3 categories (<5 mm, >5 mm and PS); ultrasonicated in DI water for 4 min	Polymer identification (FTIR); surface characterisation (SEM)		Zbyszewski *et al.*, 2011 (ref. 67)

Table 3 (*Contd.*)

Country	Habitat	Size range	Abundance (mean; range)	Sampling	Extraction	Quantification	QA/QC	Reference
USA	Beaches ($n = 8$)	2–5 mm	NA	Visual sample collection (macroplastic < 50 mm); in-field sifted (2 mm); 18 replicates; quadrats (1 m × 1 m); surface (1 inch)	Density separation, concentrated NaCl 140 g L^{-1}; stirred vigorously; filtered (Whatman GF/C 1 μm)	Visually counted/sorted		Van *et al.*, 2012 (ref. 84)
Portugal	Beach ($n = 10$)	>3 mm	2400/m^{-2}	Strandline line; 2 × 2 m; 3–5 replicates; 2 cm depth; field sieved (3 mm)	Density separation, concentrated NaCl 140 g L^{-1}; stirred vigorously; filtered (Whatman GF/C 1 μm)	Visual counting sorting		Antunes *et al.*, 2013 (ref. 85)
Germany	Beach ($n = 2$)	>0.45 μm		2 random samples; depth (0–2 cm)	Density separation, NaCl (1.2 g cm^{-3}); manual shaking; filtered (0.45 μm nitrocellulose)	Stereomicroscope; SEM; Pyr-GC combination with mass spectrometry (MS)	Laboratory control sample (black PE pellets, 1 × 0.2 × 0.2 m; 10 pieces added to 175 g sediment; recovery 80–100%)	Fries *et al.*, 2013 (ref. 52)
South Korea	Beach ($n = 10$)	>2 mm	980 pieces per m^2	Strandline line; 50 m transect; 10 replicates; quadrat (50 cm × 50 cm); depth (0–5 cm)	Field sieved (2 mm)	Visual identified and sorting		Heo *et al.*, 2013 (ref. 56)
Italy	Subalpine lake sediment ($n = 2$)	NA	NA	Random grid sampling	Density separation, no further details provided	Visual counting (microscope); polymer identification (Raman)		Imhof *et al.*, 2013 (ref. 86)
India	Beach ($n = 4$)	1–5 mm	69 (12–960) pieces per m^2	Strandline; quadrats (50 × 50 cm); depth (0–2 cm); sieved (1 mm)	Density separation, NaCl, 140 g L^{-1}; floating plastic recovered; washed; dried	Visual counting and sorting		Jayasiri *et al.*, 2013 (ref. 87)
Italy	Marine sediment (10 locations)	>0.7 μm	670–2200 pieces per kg	Superficial sediment (0–5 cm depth)	Density separation, conc NaCl, 120 g L^{-1}; shaken; sieved 32 μm steel wire; resuspended filtered (GF/F 0.7 μm)	Polymer identification (μ-FTIR)	Method blank; ambient microplastics contamination from lab and equipment	Vianello *et al.*, 2013 (ref. 41)
Germany	Beach (3 locations; 6 samples at each)	100–1000 μm	NA	Upper and lower drift line; random of upper, lower perpendicular to those; 6 accumulations and 6 'bare' sediments sampled; 0.25 × 0.25 m	Sieved (1 mm mesh); density separation (two step; saturated NaCl; NaI)	Visual counted (>1 mm fraction by naked eye; <1 mm microscope); polymer identification (TD-Pyr-GC/MS)		Dekiff *et al.*, 2014 (ref. 20)

Table 3 (*Contd.*)

Country	Habitat	Size range	Abundance (mean; range)	Sampling	Extraction	Quantification	QA/QC	Reference
Belgium	Beach, river	>250 μm	1.5 pieces per m^2	Parallel to shoreline; 50 m transect; random; 3 replicates; quadrats (25 cm × 25 cm)	Density separation, NaCl, (360 g L^{-1}); shaking 2 minutes × 2; sieved (250 μm)	Visual identification and counting (light microscope)		Laglbauer *et al.*, 2014 (ref. 88)
Singapore	Intertidal mangroves (n = 7)	<20 to >5000 μm	37 (12–63) pieces per kg	'Undisturbed areas'; quadrats (1.5 m × 1.5 m); 2–3 m apart; depth (3–4 cm)	Density separation, conc saline solution, (1.18 g L^{-1}); shaking (mechanical shaker 2 min 200 rpm); settle 6 h; sieved 0.1 cm (remove debris); filtered (1.7 μm GF/A)	Visual sorting and counting (optical microscope); polymer identification (FTIR)	LCS 500–600 μm PE spheres into sediment (n = 2; recovery 55–72%)	Mohamed Nor *et al.*, 2014 (ref. 89)
Germany	Beach (n = 3)	>1 μm	NA	'Recreation zone'; random samples 14 km beach; max 80 m apart; depth (0–3 cm)	Sieved 1 mm mesh; fluidisation (saturated NaCl); density separation (NaI 1.8 g cm^{-3}); shaken 10 s; filtered (nitrocellulose 0.45 μm); matrix removal (30% H_2O_2 treatment)	Visual counting/sorting (microscope); polymer identification (Pyr-GC/MS)	Laboratory control sample (1 mm size PE, PP, PET, PS, PVC, EPS, PUR; recovery 68–99%); procedural blank	Nuelle *et al.*, 2014 (ref. 53)
Brazil	Beach (n = 15)	>1 mm	5400 pieces per m^3 (surface layer reported)	100 × 1 m^2 trenches; 10 cm depth sampled; 10 samples at 0.1 m depth intervals until depth (1.0 m)	Density separation (seawater); sieve 1 mm mesh	Polymer identification (Raman)		Turra *et al.*, 2014 (ref. 46)
NA (deep-sea)	Deep-sea sediment (n = 12)	>32 μm	1.4–40 pieces per 50 mL	Cores; 1000–3500 km depth; megacorers or boxcorer; 5.7, 7.4 or 10 cm diameter	Method 1-centrifuge; density separation, NaCl or Ludox-TM 40; sieve (32 μm) Method 2-density separation; conc NaCl solution; filtration (Whatman GF/A 1.6 μm)	Visual counting/sorting (binocular microscope); polymer identification (FTIR)		Woodall *et al.*, 2014 (ref. 43)
Canada	Freshwater lake (3 lakes; 26 locations)	<10 cm	NA	Parallel to shoreline; 60 m transect; 10 m interval; visible <10 cm particles collected	Cleaned (ultrasonic bath, DI water); separated by hand into 4 categories	Visual counting/sorting; polymer identification (FTIR); surface characterisation (SEM)		Zbyszewski *et al.*, 2014 (ref. 90)

Table 3 (*Contd.*)

Country	Habitat	Size range	Abundance (mean; range)	Sampling	Extraction	Quantification	QA/QC	Reference
Canada	Freshwater lake sediment (2 cores)	500–5000 μm		Bottom sediment; 2 boxcore samples (7 cm diameter); 15 × 2 cm samples (depth 30 cm, 150 samples); sieved (0.5, 0.71, 0.85, 1 mm)	Density separation (two step; distilled water floating particles removed); sediment dried (60 °C), density separated (sodium polytungstate, 1.5 g cm^{-3}); floating particles removed	Visually counted and sorted (microscope); polymer identification (FTIR)		Corcoran, 2015 (ref. 73)
Switzerland	Freshwater lake sediment (3 beaches, $n = 67$)	300–5000 μm	1300 (20–7200) pieces per m^2	Strandline; 4 locations; quadrats (30 cm × 30 cm)	Density separation; 250 mL sediment with 7 L saline solution, 320 g NaCl per L; air flow; sieve (300, 1000, 5000 μm); over dried at 60 °C; matrix removal (35% H_2O_2 with 0.05 M Fe catalyst)	Visually counted (stereomicroscope); polymer identification (FTIR)		Faure *et al.*, 2015 (ref. 91)
NA (Pacific ocean)	Deep-sea sediment ($n = 12$)	0.3–200 mm	60–2000 pieces per m^2	Deep sea; boxcorer; 0.25 m^2; subsamples 0–2 cm, 2–20 cm	Sieve (300, 500, 1000 μm)	Visually counted with some aid of stereomicroscope		Fischer *et al.*, 2015 (ref. 47)
Hong Kong	Beach ($n = 25$)	315–5000 μm	5595 (106–15 554) pieces per m^2	Tide line; parallel; 30 m transect; depth (0–4 cm); quadrat 50 cm × 50 cm	In field sieve (315 μm); density separation (tap water, sonication); wet sieved (315 μm)	Visually counted and sorted (microscope); gravimetrically		Fok *et al.*, 2015 (ref. 92)
South Korea	Beach sediment (3 locations; $n = 21$)	50–5000 μm	46 000 (56–290 000) particles per m^2	Tide line; divided into districts (every 30 m); quadrat (50 cm × 50 cm); depth (0–2 cm); on-site sieved (1000, 5000 μm)	Density separation, NaCl, 2.16 g cm^{-3}; sieved 4000, 2800, 2000, and 1000 μm; supernatant of 1000 μm sieved (300, 500 μm) and filtered (0.75 μm)	Visually counted (microscope); polymer identification (FTIR; particles >300 μm)		Kim *et al.*, 2015 (ref. 28)
Germany	River sediment ($n = 8$)	63–5000 μm	4000 particles per kg	Between wrack line and water line; 3–4 kg composite sample comprising 35–40 random samples over 10–15 m range; quadrat (30 cm × 30 cm); depth (2–3 cm)	Oven dried; sieved (63, 200, 630 μm); 630 fraction visually separated; density separation, NaCl 365 g L^{-1}; settle overnight; filtered (glass fibre); matrix removal (H_2O_2/H_2SO_4 1 : 3 ratio)	Visually counted (binocular microscope); gravimetrically; classified by shape; polymer identification (FTIR)		Klein *et al.*, 2015 (ref. 63)
South Africa	Beach sediment (21 sites)	65–5000 μm	670 particles per m^2	Strandline; triplicate composite samples; depth (0–5 cm); 1200 mL subsample taken	Density separation; saturated saline solution; repeated five times	Visually counted (dissecting microscope); visual		Nel *et al.*, 2015 (ref. 77)

Table 3 (*Contd.*)

Country	Habitat	Size range	Abundance (mean; range)	Sampling	Extraction	Quantification	QA/QC	Reference
						sorting (fragments & fibre, colour)		
Korea	Beach sediment (6 beaches; 10 sites)	>1 μm		Strandline; 6 locations; quadrat (50 × 50 cm); depth (0–5 cm)	In-field sieved (1 mm); density separation, 50 mL sand with 33 mL saturated NaCl; filtered (1.2 μm); oven dried (60 °C)	Visual counting (naked eye > 1 mm; microscope for <1 mm); polymer identification (FTIR)	Blank analysis (distilled water, $n = 3$)	Song *et al.*, 2015 (ref. 72)
Germany	Beach sediment (5 locations; 14 sites)	>55.5 μm		Strandline; 500 mL; depth (1–2 cm)	Density separation; $CaCl_2$ (1.30–1.35 g mL^{-1}); air venting; settle overnight; 55 μm zooplankton filter; H_2O_2 digestion for matrix removal	Visually counted (stereomicroscope with camera)	LCS (200 PE 100–1000 μm particles spiked in cleaned sediment; recovery ranged varied based on colour; ~55% recovery)	Stolte *et al.*, 2015 (ref. 64)
UK	Atlantic ocean marine sediment ($n = 2$)	>32 μm	NA	Deep sea; corers; 0–2 cm and >5 cm subsamples	Centrifuge; 1.16 spec gravity, 8 spin cycle (4000 rpm, 5 min); 32 μm sieve	Visually counted (stereomicroscope)	Background microfiber levels tested	Woodall *et al.*, 2015 (ref. 93)
Spain	Marine sediment ($n = 3$)	63 to >2100 μm	0.90 pieces per g	Core tubes 30 cm × 3.5 cm; site replicates 1.5 m apart; 8–10 m depth	Manual sieve (≥0.063, 0.125, 0.25, 0.5, 1, ≥2 mm); density separation; distilled water; mixed 15 m; filter details not provided	Visually counted (stereomicroscope with camera)	Petri dishes left in work space and checked for particle contamination	Alomar *et al.*, 2016 (ref. 78)
Taiwan	Beach intertidal ($n = 4$)	≥38 to ≥4000 μm	32–42 560 pieces per m^3	Middle of inter-tidal zone; quadrat (50 cm × 50 cm); depth (0–5 cm, 5–10 cm), dried 60 °C for 2 days	Manual sieve (≥0.038, 0.125, 1, 2, ≥4 mm); density separation, (1.17 g NaCl per cm^3); vigorous shaking 30 s	Visually counted (stereomicroscope); polymer identification (ATR-FTIR; SR-FTIR)		Kunz *et al.*, 2016 (ref. 58)
India	Beach surface sediments ($n = 9$)	2–5 mm	24–145 pieces per m^2	Tide line parallel; quadrats (1 m × 1 m)	Density separation; 1.2 kg NaCl per L; stirred; filtered (Whatman GF/A 1.6 μm)	Visually counted (stereomicroscope with digital camera); polymer identification (FTIR)		Veerasingam *et al.*, 2016 (ref. 94)
USA	Estuary, intertidal sandy sediment ($n = 7$)	200–5000 μm	51 (5–117) pieces per m^2	Tide line parallel; 12 replicates; quadrats (25 cm × 25 cm); depth (top 3–6 cm)	On-site manual sieve (0.5–5 mm sieve); density separation (elutriation; aeration 200 μm)	Visually counted; identification (FTIR)	LCS recovery 97.25% (unknown polymer or size)	Wessel *et al.*, 2016 (ref. 59)

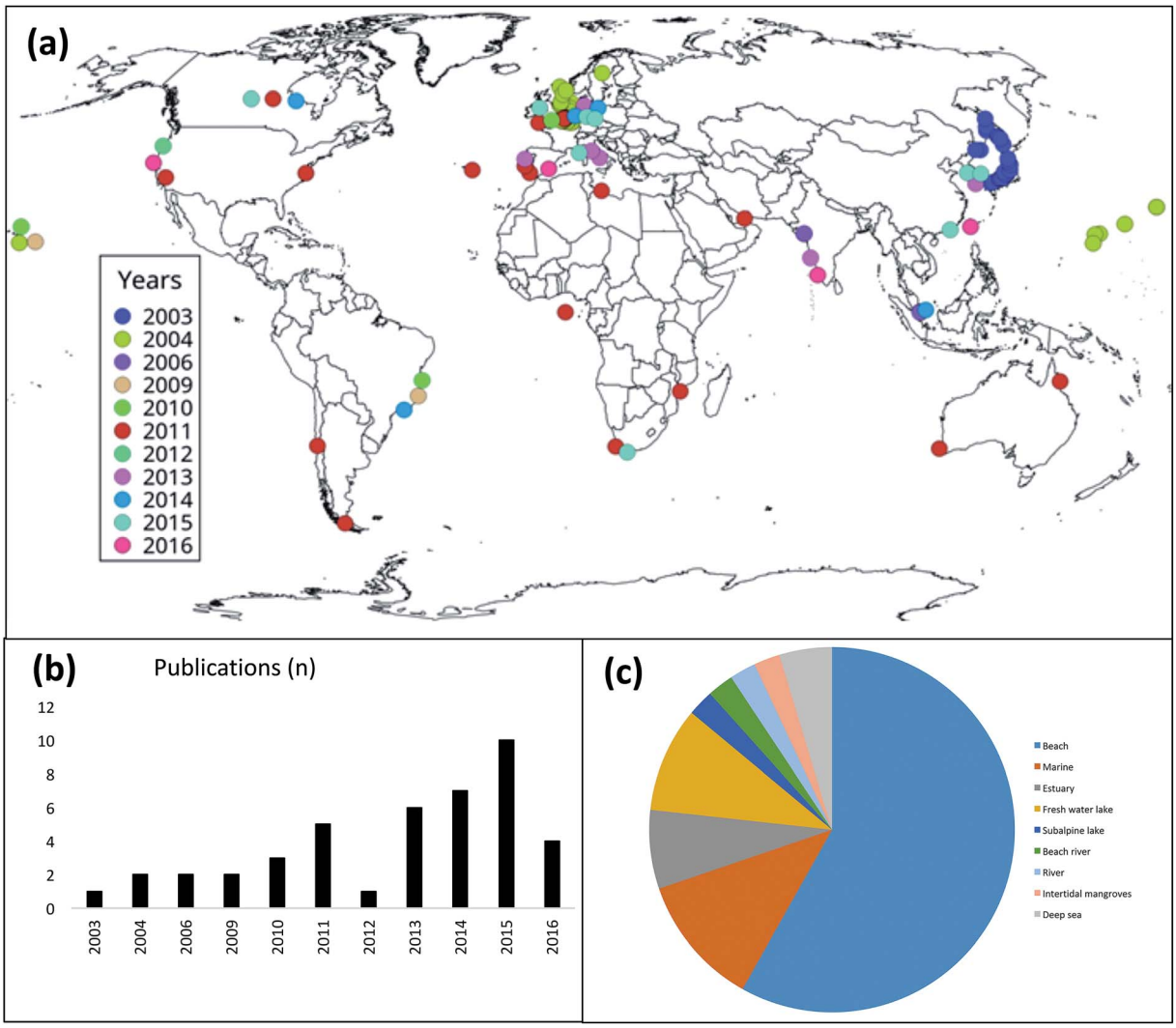

Fig. 1 (a) A world view of the locations and year of publications where microplastics have been recorded within sediment samples, (b) the total number (*n*) of publications per year, and (c) percentage of studies based upon type of sediment and location.

buoyant, in time plastics will biofoul[42] or following ingesting, will sink to the aquatic sediment where it will persist for decades. Due to long-range environmental movement, plastic microfibers have been detected in deep-sea sediments (1000–3500 m depth) ranging from 1.4 to 40 pieces per 50 mL (mean ± s.e.: 13.4 ± 3.5).[43] The proximity to city, ocean currents, type of sediment sampled must be considered when choosing an appropriate sampling site.

Once the study location is decided upon, the sampling approach on-site is applied. Each study considered in our review has applied unique methodology for the sampling approach and in many instances the methodology is incomplete to allow the reader to fully understand the technique applied which has implications for reproducibility. Transects are a common approach when conducting a beach survey (14%), with quadrats of various sizes utilised (51%), potentially the top surface layer of variable depth and then either collected or sieved on site. The variation in results from each of these decisions will strongly influence the results and the comparability of the findings. It is well known that the strandline (tide-line) will contain an abundance of marine debris, including microplastics, and so this area is often targeted to be part of the transect (56% of studies). This has the strong potential to bias the results, as the researchers are aiming to find debris rather than conducting a systematic measurement of the environment.

The sediment depth is another aspect of sampling that is likely to influence the reported concentrations and apparent environmental burden of plastics. The majority of studies will underestimate the levels of plastics by only focusing on the surface layer and not accounting for buried litter.[44] Further there are contradictory reports drawing conclusions about the abundance of plastic with depth. Studying the stratification of sediment cores to a depth of 25 cm in Hawaii indicates that 50% of microplastic fragments were contained in the topmost 5 cm of each core, and that the top 15 cm hosted 95% of all detected plastic particles.[40] Similar results were reported at the high-water line at a Belgian beach, where the top 16 cm layer contained 65% of all microplastics, with 40% detected in the top 8

cm layer alone.[45] While another study demonstrated that the surface layer accounted for <10% of the total abundance in the sediment column in their sample location.[46]

General sampling strategies are defined as bulk or volume-reduced, depending on the type of microplastic accumulation being investigated. For beach sediments and shorelines, bulk sampling – taking a defined volume of sediment,[24] usually from a defined quadrat area and depth – was the most common method for sampling beach sediments ($n = 22$). Marine sediments ($n = 6$) were collected by bulk sampling, with a box corer or sediment grab.[47,48] Volume-reduced sampling, used by 30% of the studies reviewed, involved processing sand *in situ* with 1–5 mm sieves.

Further method inconsistencies between these sampling techniques have led to a variety of sampling units reported.[23] Reporting units are generally in microplastic abundance per surface area (m^2),[49] however some quadrats are also sampled to depth, reporting volume in cubic metres (m^3),[46] mL or L (ref. 7 and 50) or weight (g to kg).[51] Conversion between measurements requires density estimates, leading to assumptions and further inconsistencies comparing microplastic abundance between littoral zones.[23] The top 15 cm of sediment contains 95% of microplastic particles, with 50% found in the topmost 5 cm.[40] Depths sampled across the studies reviewed range from 1 to 5 cm, collected using metal spoons or spatulas, while a rotating drum sampler has been used for sediment subsurface bulk sampling (10–11 cm depth).[51] Analysis of pellet distribution below the subsurface involved a number of 1 m^2 trenches dug using a shovel, however how sediment was sampled remained unspecified.[46] Across all bulk and volume-reduced sampling methods, sediment samples are either stored in PET drinking bottles[52] or glass bottles.[20,53]

The current lack of standardisation between sampling methods hinders inter-study comparison on the reported abundances of microplastics in sediments worldwide.[23,26] Differences between sampling design could be overcome by rigorous reporting methods on the complete set of sampling details including depth, weight or volume, density and water content details of sediments sampled.[24] Simple improvements for standardisation could include reporting using SI Units for sediment mass (g) rather than volume and reporting dry weight values.

Differences in sampling design hinder spatiotemporal comparisons between studies, as microplastic abundance reported within a number of studies is due to deposition over an unknown time scale.[54] Shoreline and beach studies have defined quadrat sizes at random or regular intervals along a transect, usually running parallel to the 'wrack line' or 'strandline', referring to the most recent flotsam deposited at the high tide line.[10] It is clear that microplastics are not homogeneously distributed across the shoreline and results can vary widely even within a close geographic distance, however most studies assume small-scale variations within beaches are insignificant.[55] To date, no clear relationship has been found between the amount of microplastic litter and general survey method, except methods that included the total length of the vegetation line (or 'berm') as well as 'random' sampling tends to have a higher mean.[54] A single study detailed using a random number generator for quadrat sampling.[56]

3.2 Extraction

3.2.1 Physical separation. Once the samples have been collected the microplastics of varying polymer, size, colour, morphology must be removed and isolated from what can be a complicated matrix. The most commonly applied technique for isolating microplastics is with on-site sieving which has been successfully applied for the large microplastics (>1 mm). Depending upon the nature of the study, the plastics are simply removed and placed in a collection bag for visual processing at a later stage. However, this approach is not adequate for small microplastics (<1 mm) and further laboratory extraction techniques must be applied.

3.2.2 Density separation. To extract the small microplastics (<1 mm) from a sediment sample, the most commonly reported technique is density separation combined with filtration. This method was applied in 84% of studies examined (36/43; Table 3). The density separation extraction technique generally involves four main steps; (1) introduction of aqueous solvent of specific density, (2) mixing for defined periods of time, (3) a settling equilibration time and (4) filtering to specific size fractions.

The density separation approach to isolate microplastics from sediment is possible by exploiting the difference in density of polymers. For example, PE/PP have a density lower than water and would be expected to float on the water surface without density modification (Table 1; LDPE 0.91–0.92 g mL^{-1}; HDPE 0.93–0.97 g mL^{-1}; PP 0.89–0.92 g mL^{-1}). By increasing the density of the solvent, it is possible to create a solution where higher density polymers float (Table 1; PS 0.28–1.04 g mL^{-1}; PVC 1.10–1.47 g mL^{-1}). Common environmental samples (*i.e.* soil, sand) have higher densities (approximately 2.55 g mL^{-1} and >1.44 g mL^{-1} respectively) than these polymers making separation a practical technique for separation.[24] The separating solution is most commonly a concentrated salt solution such as NaCl of varying densities ($n = 19/43$), the most common being 1.2 g mL^{-1} (50%). Other solutions used include zinc chloride ($ZnCl_2$) ($n = 1$), calcium chloride ($CaCl_2$) ($n = 1$), sodium iodide (NaI) ($n = 1$), sodium polytungstate ($n = 2$), filtered seawater ($n = 4$) and distilled water ($n = 2$). This leaves a total of 17 out of the 40 density separation techniques that do not specify a separation fluid and 15 that also lack a corresponding density. The variation in separating fluids used in addition to these being unknown in 43% of the density separation studies highlights the need for a standardised method and appropriate reporting of these methods.

Shaking or stirring following the introduction of a separating fluid is important to ensure polymers can adequately detach from the matrix. Shaking of the sample is either done mechanically with a centrifuge[57] or mechanical shaker,[51] or by vigorous manual shaking.[58] Stirring was the other preferred method of mixing, done either manually or mechanically (magnetic). Time frames were rarely indicated for stirring, which introduces a subjective element in terms of deciding when the polymer has sufficiently separated from the matrix. Aeration,[59–61] and inversion[48] were other techniques used for this step.

Settling times after mixing varied between each study, with some left to settle for as little as five minutes,[62] and as long as 'overnight' (assumed to be upwards of 12 hours).[63,64] The timing of settling was only specified in seven studies, highlighting the necessity for better reporting of methods.

3.2.3 Filtration. The most common filtration techniques among studies are sieving and vacuum filtration (generally dry sorting and wet sorting, respectively). Dry sorting by sieving can be done before the density separation method to reduce the bulk of a sample or separate them into size fractions, or instead of the density separation technique. Wet sorting is generally conducted after the density separation method to isolate the floating plastic particles, and is carried out by vacuum filtration. These methods are consistent throughout all studies, with either one of these methods occurring to isolate polymers. The differing factor between these is the filter type and size.

Seventeen studies reported filtering their samples during the extraction process. Filter types used for wet sorting include most commonly glass fibre filters (Whatman® GF/A, GF/C or GF/F; $n = 11$), but also include nitrocellulose filters ($n = 1$), isopore filters ($n = 1$), and zooplankton filters ($n = 1$). Twenty studies reported using sieving as their method of isolating polymers during their extraction step, 14 of which used it as their only sorting step within their extraction process (sans density separation). This leaves the remaining 6 studies combining sieving and filtration during their extraction process. Regardless of the method or technique used, the size and type of the filter paper is a large inconsistency between all studies, with no particular size fractions being used to determine the quantity of 'microplastics'. It is this that emphasises the need to clearly define the size ranges of microplastics in the environment, with our recommendations listed in Table 1.

The typical size categorisation of microplastics diameter use here is <5 mm in diameter here, we endorse the use of large-microplastic, small-microplastic, and nanoplastic as being three separate categories within this range. With the implementation of these three size fractions, more structured research can be achieved that can be compared between researchers, sites, and nations. Another inconsistent analytical approach is the pore size of the filter paper used. These range from 0.3 μm (ref. 65) to 5 mm,[66] but do not necessarily correspond to the size ranges reported in their findings. For example, one study report using the Whatman® GF/F filter with pore size 0.7 μm, and that their smallest plastic particle detected was 15 μm.[41] This infers that anything between 0.7 μm and 15 μm is still being captured. This becomes problematic when weighing is introduced as a quantification method, but also begs the question as to whether all plastic particles were counted and measured correctly.[32]

3.2.4 Matrix removal. Matrix removal is a process that can be applied to the destructive removal of interfering organic matter present in the sample. The difficult task of separating polymers from their matrix is aided using solutions to digest organic matter; making filtration and quantification more accurate and less time consuming later. Studies have included chemical matrix removal as well as physical matrix removal steps and reported higher filtration rates and a need to remove material (organic matter, calcium carbonate, *etc.*) from the plastic particles.[53,63,64]

Hydrogen peroxide (30% H_2O_2) was found to leave polymer size, shape and resulting spectra unchanged,[53] and was determined to be the best solution to digest samples with higher organic matter, compared to 20% H_2O_2, 37% hydrochloric acid (HCl), and various concentrations of sodium hydroxide (NaOH).[53] Mixtures of H_2O_2 and sulphuric acid (H_2SO_4) (1 : 3) have been used to aid in the digestion of organic matter,[63] with bleach being utilised to destroy natural debris in another study.[34] Mechanical matrix removal is also seen in studies where samples were ultrasonicated to remove excess matrix from the samples.[67–69]

The matrix removal step is only present in 12% (5/43) of studies, despite it being an important step in the quantification of microplastics from sediment samples. Its importance is based on the content of organic matter that is likely to be ubiquitous throughout all sediment samples, making it necessary to ensure all matter is digested before analysis of the plastics. The inconsistencies between studies indicate that the matrix removal step needs to be validated and included in all sediment extraction processes going forward. Gravimetric techniques are likely to overestimate concentration without matrix removal. A complimentary approach for measuring the total mass of plastics is through gravimetric analysis. The approaches vary and can include measuring the mass of individual microplastics particles or measuring the filter paper as a whole.[16,50]

3.3 Quantification and identification

3.3.1 Manual counting – optical microscope. The most common quantification technique for microplastics is visual counting and sorting into various categories, typically based upon polymer type, colour, size and morphology. Visual sorting and identification of microplastics under a dissection microscope has been a common method reported in terms of quantification ($n = 27$), but has many limitations in terms of accuracy. Visual counting can lead to either extreme over[18] or underestimations of plastic content,[70] based on the extent of the size ranges of plastics in the environment, as well as the risk of counting non-plastic particles as plastic. For example, approximately 20% particles initially identified as microplastics by visual observation, were later identified as aluminium silicate from coal ash using scanning electron microscope (SEM).[32]

The application of spectroscopy in the confirmation of the presence of plastic is extremely important, and can increase the accuracy of visual counting. Studies have used a combination of microscopy and spectroscopy (*i.e.* microscope and Fourier transform infrared spectroscopy (FTIR)) to first count the potential microplastic particles, followed by either confirming or denying that they are plastics.[65]

Recovery of visual counting methods is highly unreliable and quantitation using quality control samples demonstrate that the colour of microplastics with 70–100% recovery for blue, violet, green hues and 0–40% recover for yellow, orange, pink and orange microplastics with transparent microplastics recovered about 45–63%.[64]

3.3.2 Polymer identification (FTIR; Raman; scanning electron microscope). Spectroscopy is required to confirm the identification of plastics, and their synthesis polymer for particles <1 mm in size.[9] Identification methods used to classify microplastics from environmental samples include Raman spectroscopy, Fourier transform infrared spectroscopy (including micro-FTIR and ATR-FTIR), and scanning electron microscopy (SEM). These techniques assist in identifying colour, shape, morphology, chemical composition and structure. Each of these identifying features is important in determining the types of plastic pollution that is found in the environment.

FTIR is used to obtain infrared spectrum of emission or absorption spectra as well as to collect high spectral resolution data, which facilitates the determination of the structure of molecules.[71] FTIR is the most common method in the identification of microplastics from sediment and water samples reported (23/43; 53%; Table 3). A combination of FTIR and optical microscopy has also been utilised ($n = 4$) (micro-FTIR) in the identification of polymers from sediment samples.[72,73] The FTIR analytical method has many advantages in terms of plastic identification. Based on the high concentration of studies relying on FTIR spectroscopy we are confident that this is an appropriate measure of polymer type within sediment samples. One advantage of FTIR reflectance spectroscopy is its non-invasive nature, as it allows for samples to be analysed without destroying the sample.

Raman spectroscopy is a spectroscopic technique based on the interaction of a sample and the consequent changes of photons in monochromatic light and has the ability to provide structural information about plastics which can then be investigated to identify their polymer type.[74] Raman spectroscopy was only used in two studies, where it was combined to both identify and quantify plastics in sediment using Raman micro-spectroscopy. This allows for a non-destructive identification tool, where particles in the μm range can be quantified.

SEM gives detailed information about the size and shape of a particle. SEM can assist in identifying inorganic plastic additives, and gives a clear image of the size and topography of the particle. Seven studies employed SEM to obtain identification information about plastic particles from sediment samples. Combining SEM with other identification techniques such as FTIR can be advantageous, where certain plastic types could be particular shapes. A study showed that most of their polyethylene samples were rounded, and most of their polypropylene samples were fragments.[67] This information can help to give an indication of the source of the plastics sampled, *i.e.* broken down fragments from large plastic, or primary nurdles.[15,67]

3.3.3 Emerging techniques. Three studies used pyrolysis gas chromatography/mass spectrometry (GC/MS) to identify the polymer found in environmental samples. Pyrolysis is a technique where a sample is burnt in the absence of oxygen and it typically applied for thermogravimetric analysis. The decomposition of the polymer related to temperature provide a specific signature relative to specific polymers. If combustion is fed into a gas chromatography for separation of chemical constituents being determining the chemical identity through mass spectrometry (MS). This is combined with Pry-GC obtains structural information about macromolecules by GC/MS analysis and is a powerful tools for identification of polymer.[53] The major disadvantages are that you determine the mass of polymer per sample and do not get any information on the number, type and morphology of the plastics present in the sample.

A new technique for quantifying the amount of plastics in solid samples has been reported using pressurised fluid extraction (PFE).[75] Whole polymers (melting, destructive) are appropriate for measuring the total amount of plastics present in a sample but they will fail to capture the morphological characteristics, that currently can only be captured through the labour intensive visual counting.

3.4 QA/QC

Analytical approaches applied to measuring plastic debris in environmental samples are not well-developed.[9] The quantification of microplastics from environmental samples has not been optimised, and involves many hurdles such as contamination, overestimation and underestimation.[53] These hurdles become apparent at the extraction process, where validation studies and blanks should be included as part of the analytical process.

Only seven studies of the 43-analysed conducted some sort of laboratory control sample (LCS) or validation trials. Validation studies retrieved recovery rates of up to 100% (ref. 61) and as little as 54.9%.[76] The size range of spiked plastics varied greatly and higher recoveries were reported for the larger spiked plastics compared to the smaller size fraction.

Laboratory blanks were used in three of the studies evaluated, which determined whether contamination from the laboratory or clothing of scientists was effecting results.[57,77,78] Determining whether contamination is occurring in the laboratory environment is imperative to ensure plastic content is not overestimated by counting or weighing said contamination.[77] The combination of validation studies and blanks are essential in reporting findings of a study, without them the it cannot be concluded that the method used was reliable or valid.

4 Conclusion

In this review, we collated the analytical approach for measuring microplastics in sediment. The four primary areas of the analytical process that have been summarised; (1) sampling, (2) extraction, (3) quantitation and (4) QA/QC. Each of those sections have their own subject specific challenges and require further method development and harmonisation. Specifically, improvements and harmonisation on size fractions, sampling approaches, extraction protocols and units for reporting plastic abundance would be improve the comparability of data in this research area. The issues facing scientists measuring microplastics in sediment samples are like other matrices and biological samples. Further, we would advocate for the development of strong QA/QC procedures to be adopted like other fields of analytical chemistry. Finally, inter-laboratory proficiency testing

is recommended to give an indication of the variation in measurements reported in the scientific literature to this point. While visual counting has been an important technique for beginning our understanding of microplastics in environmental samples, it is labour intensive and prone to human error, and analytical techniques must move forward.

Acknowledgements

The authors thank projects Fondecyt 1161673 (K. Pozo), the Czech Ministry of Education, Youth and Sports projects (LO1214 and LM2015051) and the anonymous reviewers for providing comments on earlier drafts of this manuscript.

References

1 P. Convey, D. Barnes and A. Morton, *Polar Biol.*, 2002, **25**, 612–617.
2 C. M. Free, O. P. Jensen, S. A. Mason, M. Eriksen, N. J. Williamson and B. Boldgiv, *Mar. Pollut. Bull.*, 2014, **85**, 156–163.
3 F. Galgani, A. Souplet and Y. Cadiou, *Mar. Ecol.: Prog. Ser.*, 1996, **142**, 225–234.
4 E. J. Carpenter, S. J. Anderson, G. R. Harvey, H. P. Miklas and B. B. Peck, *Science*, 1972, **178**, 749–750.
5 M. R. Gregory, *Mar. Pollut. Bull.*, 1977, **8**, 82–84.
6 J. G. Shiber, *Mar. Pollut. Bull.*, 1979, **10**, 28–30.
7 R. C. Thompson, Y. Olsen, R. P. Mitchell, A. Davis, S. J. Rowland, A. W. G. John, D. McGonigle and A. E. Russell, *Science*, 2004, **304**, 838.
8 S. C. Gall and R. C. Thompson, *Mar. Pollut. Bull.*, 2015, **92**, 170–179.
9 GESAMP, *Sources, fate and effects of microplastics in the marine environment: a global assessment*, ed. P. J. Kershaw, IMO/FAO/UNESCO-IOC/UNIDO/WMO/IAEA/UN/UNEP/UNDP Joint Group of Experts on the Scientific Aspects of Marine Environmental Protection, Rep. Stud. GESAMP No. 90, 2015, p. 96.
10 M. A. Browne, P. Crump, S. J. Niven, E. Teuten, A. Tonkin, T. Galloway and R. Thompson, *Environ. Sci. Technol.*, 2011, **45**, 9175–9179.
11 A. L. Andrady, *Mar. Pollut. Bull.*, 2011, **62**, 1596–1605.
12 K. Betts, *Environ. Sci. Technol.*, 2008, **42**, 8995.
13 C. Arthur, J. Baker and H. Bamford, *Proceedings of the International Research Workshop on the Occurrence, Effects, and Fate of Microplastic Marine Debris (Sept 9–11, 2008)*, National Oceanic and Atmospheric Administration (NOAA), Technical Memorandum NOS-OR&R-30, 2009.
14 J. L. Lavers, S. Oppel and A. L. Bond, *Mar. Environ. Res.*, 2016, **119**, 245–251.
15 M. S. Reddy, B. Shaik, S. Adimurthy and G. Ramachandraiah, *Estuarine, Coastal Shelf Sci.*, 2006, **68**, 656–660.
16 T. Kusui and M. Noda, *Mar. Pollut. Bull.*, 2003, **47**, 175–179.
17 M. R. Gregory, *Mar. Environ. Res.*, 1983, **10**, 73–92.
18 R. Lenz, K. Enders, C. A. Stedmon, D. M. A. Mackenzie and T. G. Nielsen, *Mar. Pollut. Bull.*, 2015, **100**, 82–91.
19 A. A. Koelmans, E. Besseling and W. J. Shim, in *Marine Anthropogenic Litter*, ed. M. Bergmann, L. Gutow and M. Klages, Springer International Publishing, Cham, 2015, pp. 325–340, DOI: 10.1007/978-3-319-16510-3_12.
20 J. H. Dekiff, D. Remy, J. Klasmeier and E. Fries, *Environ. Pollut.*, 2014, **186**, 248–256.
21 J. L. Lavers and A. L. Bond, *Mar. Environ. Res.*, 2016, **113**, 1–6.
22 A. Vegter, M. Barletta, C. Beck, J. Borrero, H. Burton, M. Campbell, M. Costa, M. Eriksen, C. Eriksson, A. Estrades, K. Gilardi, B. Hardesty, J. Ivar do Sul, J. Lavers, B. Lazar, L. Lebreton, W. Nichols, C. Ribic, P. Ryan, Q. Schuyler, S. Smith, H. Takada, K. Townsend, C. Wabnitz, C. Wilcox, L. Young and M. Hamann, *Endangered Species Research*, 2014, vol. 25, pp. 225–247.
23 L. Van Cauwenberghe, L. Devriese, F. Galgani, J. Robbens and C. R. Janssen, *Mar. Environ. Res.*, 2015, **111**, 5–17.
24 V. Hidalgo-Ruz, L. Gutow, R. C. Thompson and M. Thiel, *Environ. Sci. Technol.*, 2012, **46**, 3060–3075.
25 C. M. Rochman, M. A. Browne, A. J. Underwood, J. A. van Franeker, R. C. Thompson and L. A. Amaral-Zettler, *Ecology*, 2016, **97**, 302–312.
26 T. Rocha-Santos and A. C. Duarte, *TrAC, Trends Anal. Chem.*, 2015, **65**, 47–53.
27 M. A. Browne, T. S. Galloway and R. C. Thompson, *Environ. Sci. Technol.*, 2010, **44**, 3404–3409.
28 I.-S. Kim, D.-H. Chae, S.-K. Kim, S. Choi and S.-B. Woo, *Arch. Environ. Contam. Toxicol.*, 2015, **69**, 299–309.
29 M. Eriksen, L. C. M. Lebreton, H. S. Carson, M. Thiel, C. J. Moore, J. C. Borerro, F. Galgani, P. G. Ryan and J. Reisser, *PLoS One*, 2014, **9**, e111913.
30 D. Morritt, P. V. Stefanoudis, D. Pearce, O. A. Crimmen and P. F. Clark, *Mar. Pollut. Bull.*, 2014, **78**, 196–200.
31 J. R. Jambeck, R. Geyer, C. Wilcox, T. R. Siegler, M. Perryman, A. Andrady, R. Narayan and K. L. Law, *Science*, 2015, **347**, 768–771.
32 M. Eriksen, S. Mason, S. Wilson, C. Box, A. Zellers, W. Edwards, H. Farley and S. Amato, *Mar. Pollut. Bull.*, 2013, **77**, 177–182.
33 K. A. V. Zubris and B. K. Richards, *Environ. Pollut.*, 2005, **138**, 201–211.
34 S. A. Carr, J. Liu and A. G. Tesoro, *Water Res.*, 2016, **91**, 174–182.
35 A. Lechner, H. Keckeis, F. Lumesberger-Loisl, B. Zens, R. Krusch, M. Tritthart, M. Glas and E. Schludermann, *Environ. Pollut.*, 2014, **188**, 177–181.
36 R. Dris, J. Gasperi, V. Rocher, M. Saad, N. Renault and B. Tassin, *Environ. Chem.*, 2015, **12**, 592–599.
37 R. Dris, J. Gasperi, M. Saad, C. Mirande and B. Tassin, *Mar. Pollut. Bull.*, 2016, **104**, 290–293.
38 S. Rech, V. Macaya-Caquilpán, J. F. Pantoja, M. M. Rivadeneira, D. Jofre Madariaga and M. Thiel, *Mar. Pollut. Bull.*, 2014, **82**, 66–75.
39 Y. C. Jang, J. Lee, S. Hong, J. Y. Mok, K. S. Kim, Y. J. Lee, H.-W. Choi, H. Kang and S. Lee, *Mar. Pollut. Bull.*, 2014, **86**, 505–511.
40 H. S. Carson, S. L. Colbert, M. J. Kaylor and K. J. McDermid, *Mar. Pollut. Bull.*, 2011, **62**, 1708–1713.
41 A. Vianello, A. Boldrin, P. Guerriero, V. Moschino, R. Rella, A. Sturaro and L. Da Ros, *Estuarine, Coastal Shelf Sci.*, 2013, **130**, 54–61.

42 N. Kowalski, A. M. Reichardt and J. J. Waniek, *Mar. Pollut. Bull.*, 2016, **109**, 310–319.
43 L. C. Woodall, A. Sanchez-Vidal, M. Canals, G. L. J. Paterson, R. Coppock, V. Sleight, A. Calafat, A. D. Rogers, B. E. Narayanaswamy and R. C. Thompson, *R. Soc. Open Sci.*, 2014, **1**, 140317.
44 A. T. Williams and D. T. Tudor, *Mar. Pollut. Bull.*, 2001, **42**, 1031–1039.
45 M. Claessens, S. De Meester, L. Van Landuyt, K. De Clerck and C. R. Janssen, *Mar. Pollut. Bull.*, 2011, **62**, 2199–2204.
46 A. Turra, A. B. Manzano, R. J. S. Dias, M. M. Mahiques, L. Barbosa, D. Balthazar-Silva and F. T. Moreira, *Sci. Rep.*, 2014, **4**, 4435.
47 V. Fischer, N. O. Elsner, N. Brenke, E. Schwabe and A. Brandt, *Deep Sea Res., Part II*, 2015, **111**, 399–405.
48 J. P. Harrison, J. J. Ojeda and M. E. Romero-González, *Sci. Total Environ.*, 2012, **416**, 455–463.
49 J. A. Ivar do Sul and M. F. Costa, *Environ. Pollut.*, 2014, **185**, 352–364.
50 K. J. McDermid and T. L. McMullen, *Mar. Pollut. Bull.*, 2004, **48**, 790–794.
51 K. L. Ng and J. P. Obbard, *Mar. Pollut. Bull.*, 2006, **52**, 761–767.
52 E. Fries, J. H. Dekiff, J. Willmeyer, M.-T. Nuelle, M. Ebert and D. Remy, *Environ. Sci.: Processes Impacts*, 2013, **15**, 1949–1956.
53 M.-T. Nuelle, J. H. Dekiff, D. Remy and E. Fries, *Environ. Pollut.*, 2014, **184**, 161–169.
54 K. Velander and M. Mocogni, *Mar. Pollut. Bull.*, 1999, **38**, 1134–1140.
55 L. Thornton and N. L. Jackson, *Mar. Pollut. Bull.*, 1998, **36**, 705–711.
56 N. W. Heo, S. H. Hong, G. M. Han, S. Hong, J. Lee, Y. K. Song, M. Jang and W. J. Shim, *Ocean Sci. J.*, 2013, **48**, 225–233.
57 L. C. Woodall, A. Sanchez-Vidal, M. Canals, G. L. J. Paterson, R. Coppock, V. Sleight, A. Calafat, A. D. Rogers, B. E. Narayanaswamy and R. C. Thompson, *The deep sea is a major sink for microplastic debris*, 2014.
58 A. Kunz, B. A. Walther, L. Löwemark and Y.-C. Lee, *Mar. Pollut. Bull.*, 2016, **111**, 126–135.
59 C. C. Wessel, G. R. Lockridge, D. Battiste and J. Cebrian, *Mar. Pollut. Bull.*, 2016, **109**, 178–183.
60 X. Zhu, *Mar. Pollut. Bull.*, 2015, **92**, 69–72.
61 M. Claessens, L. Van Cauwenberghe, M. B. Vandegehuchte and C. R. Janssen, *Mar. Pollut. Bull.*, 2013, **70**, 227–233.
62 P. L. Corcoran, T. Norris, T. Ceccanese, M. J. Walzak, P. A. Helm and C. H. Marvin, *Environ. Pollut.*, 2015, **204**, 17–25.
63 S. Klein, E. Worch and T. P. Knepper, *Environ. Sci. Technol.*, 2015, **49**, 6070–6076.
64 A. Stolte, S. Forster, G. Gerdts and H. Schubert, *Mar. Pollut. Bull.*, 2015, **99**, 216–229.
65 H. K. Imhof, J. Schmid, R. Niessner, N. P. Ivleva and C. Laforsch, *Limnol. Oceanogr.: Methods*, 2012, **10**, 524–537.
66 F. Faure, M. Corbaz, H. Baecher and L. de Alencastro, *Archives of Science*, 2012, **65**, 157–164.
67 M. Zbyszewski and P. L. Corcoran, *Water, Air, Soil Pollut.*, 2011, **220**, 365–372.
68 K. Ashton, L. Holmes and A. Turner, *Mar. Pollut. Bull.*, 2010, **60**, 2050–2055.
69 D. A. Cooper and P. L. Corcoran, *Mar. Pollut. Bull.*, 2010, **60**, 650–654.
70 M. G. J. Löder, M. Kuczera, S. Mintenig, C. Lorenz and G. Gerdts, *Environ. Chem.*, 2015, **12**, 563–581.
71 A. L. Lusher, M. McHugh and R. C. Thompson, *Mar. Pollut. Bull.*, 2013, **67**, 94–99.
72 Y. K. Song, S. H. Hong, M. Jang, G. M. Han, M. Rani, J. Lee and W. J. Shim, *Mar. Pollut. Bull.*, 2015, **93**, 202–209.
73 P. L. Corcoran, *Environ. Sci.: Processes Impacts*, 2015, **17**, 1363–1369.
74 V. Allen, J. H. Kalivas and R. G. Rodriguez, *Appl. Spectrosc.*, 1999, **53**, 672–681.
75 S. Fuller and A. Gautam, *Environ. Sci. Technol.*, 2016, **50**, 5774–5780.
76 N. H. M. Nor and J. P. Obbard, *Mar. Pollut. Bull.*, 2014, **79**, 278–283.
77 H. A. Nel and P. W. Froneman, *Mar. Pollut. Bull.*, 2015, **101**, 274–279.
78 C. Alomar, F. Estarellas and S. Deudero, *Mar. Environ. Res.*, 2016, **115**, 1–10.
79 P. L. Corcoran, M. C. Biesinger and M. Grifi, *Mar. Pollut. Bull.*, 2009, **58**, 80–84.
80 J. A. Ivar do Sul, Â. Spengler and M. F. Costa, *Mar. Pollut. Bull.*, 2009, **58**, 1236–1238.
81 M. A. Browne, T. S. Galloway and R. C. Thompson, *Environ. Sci. Technol.*, 2010, **44**, 3404–3409.
82 M. F. Costa, J. A. Ivar do Sul, J. S. Silva-Cavalcanti, M. C. Araujo, A. Spengler and P. S. Tourinho, *Environ. Monit. Assess.*, 2010, **168**, 299–304.
83 J. Martins and P. Sobral, *Mar. Pollut. Bull.*, 2011, **62**, 2649–2653.
84 A. Van, C. M. Rochman, E. M. Flores, K. L. Hill, E. Vargas, S. A. Vargas and E. Hoh, *Chemosphere*, 2012, **86**, 258–263.
85 J. C. Antunes, J. G. L. Frias, A. C. Micaelo and P. Sobral, *Estuarine, Coastal Shelf Sci.*, 2013, **130**, 62–69.
86 H. K. Imhof, N. P. Ivleva, J. Schmid, R. Niessner and C. Laforsch, *Curr. Biol.*, 2013, **23**, R867–R868.
87 H. B. Jayasiri, C. S. Purushothaman and A. Vennila, *Mar. Pollut. Bull.*, 2013, **77**, 107–112.
88 B. J. L. Laglbauer, R. M. Franco-Santos, M. Andreu-Cazenave, L. Brunelli, M. Papadatou, A. Palatinus, M. Grego and T. Deprez, *Mar. Pollut. Bull.*, 2014, **89**, 356–366.
89 N. H. Mohamed Nor and J. P. Obbard, *Mar. Pollut. Bull.*, 2014, **79**, 278–283.
90 M. Zbyszewski, P. L. Corcoran and A. Hockin, *J. Great Lakes Res.*, 2014, **40**, 288–299.
91 F. Faure, C. Demars, O. Wieser, M. Kunz and L. F. de Alencastro, *Environ. Chem.*, 2015, **12**, 582–591.
92 L. Fok and P. K. Cheung, *Mar. Pollut. Bull.*, 2015, **99**, 112–118.
93 L. C. Woodall, C. Gwinnett, M. Packer, R. C. Thompson, L. F. Robinson and G. L. J. Paterson, *Mar. Pollut. Bull.*, 2015, **95**, 40–46.
94 S. Veerasingam, M. Mugilarasan, R. Venkatachalapathy and P. Vethamony, *Mar. Pollut. Bull.*, 2016, **109**, 196–204.