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Investigation of microplastics in aquatic environments: An overview of the methods used, from field sampling to laboratory analysis

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ABSTRACT

Microplastics pollution in aquatic ecosystems has aroused increasing global concern, leading to an explosive growth of studies regarding microplastics published in the past few years. To date, there is still a lack of standardized methodologies used for the detection of microplastics within environmental samples, thus hampering comparison of the reported data. This review summarizes the currently used methodologies for sampling, extracting and identifying microplastics in three kinds of aquatic environmental matrices (water, sediment and aquatic biota) and includes a critical discussion of the advantages and limitations of these methodologies. The quality control and quality assurance measures taken to reduce background contamination and validate analytical methods are also discussed. Finally, this review highlights the current challenges and gives suggestions for the future research.

1. Introduction

The sustained growth of the world's production and application of plastic materials has led to a considerable amount of plastic waste released into the terrestrial and aquatic ecosystems [1,2]. Under the combined effects of environmental physicochemical and biotic factors, such as ultraviolet (UV) radiation, mechanical abrasion and microbial action, plastic debris will progressively degrade into a myriad of secondary microplastics (less than 5 mm in size) [1,3]. Microplastics can also be primarily manufactured at a tiny size and eventually end up in the environment [3]. These microscopic plastic particles have been reported to be ubiquitously present in various environmental matrices of aquatic ecosystems across the globe. Waters, sediments and aquatic animals from tropical areas to polar regions of the earth were found to be contaminated with microplastics at varying levels [3–5]. For instance, China's Three Gorges Reservoir was discovered to have a microplastics concentration of up to 1.36×10^7 items per square kilometer of surface water, which is the highest value ever reported in the available literature [6]. The fine size of microplastics makes them easily ingested by a diverse array of aquatic organisms at different trophic levels [7]. Ingestion of microplastics by aquatic organisms can cause a series of negative health impacts, such as mechanical injury, false satiation, low growth rate, increased immune response, energy depletion, blocked enzyme production, decreased fecundity, oxidative stress, and even morbidity [7,8]. In addition, microplastics could concentrate a considerable amount of waterborne toxic pollutants, which may cause toxicological hazards to the aquatic animals once these contaminated microplastics are consumed [7,9].

To attain a better understanding of the environmental effects of microplastics, a rapidly increasing number of studies have focused on monitoring microplastics quantitatively and qualitatively in various aquatic ecosystems around the world [3,9]. Along with the large-scale monitoring programs are a wide variety of operating techniques employed for sampling, processing, identifying and quantifying microplastics from different environmental matrices. The inconsistence of operation protocols is the main problem that impedes spatial and temporal comparisons among the available data. Unfortunately, until recently, there have been no standardized methodological criteria for sampling and subsequent analysis procedures for the microplastics monitoring work. The main objective of this article is to give a comprehensive overview on sampling, handling and instrumental analysis methods currently used for detection of microplastics in water, sediment and biological samples. Advantages and limitations of these methods and the

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quality control and quality assurance approaches throughout the whole sample process are also discussed. By summarizing these, we aim to promote future monitoring programs for microplastics in aquatic environments.

2. Literature review

An extensive literature review was performed using databases such as ISI Web of Knowledge, Science Direct and Google Scholar. The search keywords included “microplastics”, “plastics”, “microlitter”, “microbeads”, “plastic debris”, “plastic fragment”. The database was searched for studies published up to 2018. Reference lists of the retrieved publications were also checked in order to trace back to other relevant literatures. Ultimately, a total of 49 literatures were selected based on the subjects of these literatures regarding environmental matrices (e.g., water, sediment, and biota), sampling strategies, sample handling procedures, and analytical methods for identification and quantification.

3. Sample collection

Microplastics are nearly ubiquitously present in the water surface layer, the water column, beaches, benthic sediments, and various kinds of aquatic organisms [7,9,10]. The selection of sampling method largely depends on the matrices to be sampled and the size limitation of microplastics to be targeted. Generally, there are mainly three sampling strategies utilized to collect microplastic samples from the aquatic environment: selective sampling, bulk sampling and volume-reduced sampling [11].

Selective sampling is applicable in cases where the plastic items are large enough for identification with the naked eye and thus can be extracted directly from environmental matrices. This process is simple and straightforward. The main disadvantages of this sampling strategy are that the size limitation of detectable microplastics is high, and less obvious items are easily overlooked particularly when they are mixed with other debris [12]. Bulk sampling refers to collecting the entire sample without reducing its volume during the sampling process. In theory, all microplastics within the sample, regardless of their size and visibility, can be captured using this method. However, bulk sampling only allows a relatively small amount of a sample to be collected, which may negatively affect the representativeness of the sample [11]. The volume-reduced approach, on the contrary, refers to reducing the entire volume of a bulk sample by fast filtration during sampling and preserving only a small fraction of the sample for subsequent analysis. Therefore, volume-reduced sampling is advantageous for covering large quantities or areas of samples during sampling [12]. The disadvantage of this method is that by fast filtration, the majority of the sample is discarded, which can result in a substantial loss of microplastics, particularly those with a size smaller than the mesh size of sampling tools. For the three sampling strategies, selective method is usually applied in beach sampling [13], bulk method is mainly used to collect sediment samples [14] and occasionally water samples [15], while volume-reduced method seems to be the most popular approach for water samples [16,17]. The bulk and volume-reduced samples require further processing under laboratory conditions.

3.1. Water samples

Water samples can be collected from the water surface or the water column at specific depths. There are a variety of approaches employed for the sampling of microplastics in water, the majority of which are based on the volume-reduced method (Table 1). For surface water sampling, manta trawls and neuston nets are the most commonly used equipment, while for water column sampling, plankton nets, bongo nets, continuous plankton recorders (CPR), multiple opening–closing nets, and near-bottom trawls are the major techniques [12,18]. There are some alternative tools that are occasionally used in surface water or water column sampling for microplastics, such as plankton traps, water collection bottles, or water intake pumps [12]. Mesh size of the sampling tools varies from tens of microns to millimeters, with the most common aperture size being 333 μm. Abundances of microplastics recovered from the water matrix are directly influenced by the mesh size of the sampling tools. It was estimated that an 80 μm mesh could retain up to 250 times higher concentration of plastic fibers than that of a 330 μm mesh [19]. It should be noted that most of the currently used sampling techniques are only applicable to collection of microplastics with certain size ranges. The employment of sampling tools with different mesh sizes makes it difficult to compare the available monitoring data. Units of measurement for abundances of microplastics in water can be present as the number of particles per km^2 [16,20] or m^3 [17,21] of water.

Table 1
Studies on microplastics pollution in water samples.

Location	Sampling	Identification	Abundance	Ref.
Turkish territorial waters of the Mediterranean Sea	Collected by a manta net (333 μm mesh)	Stereomicroscope, FTIR	16339–520213 particles km^{-2}	[16]
Ross Sea (Antarctica)	5 m depth: Collected by the saltwater intake pump system of vessel and then passed through a glass fiber filter (1 μm mesh)	FPA-FTIR	0.17 ± 0.34 particles m^{-3}	[21]
Atlantic Ocean	11 m depth: Collected by a centrifugal pump and then passed through stainless steel sieve (250 μm mesh)	Stereomicroscope, micro-FTIR	1.15 ± 1.45 particles m^{-3}	[68]
Victoria Harbor, Hong Kong	Collected by a plankton net (153 μm mesh)	Stereomicroscope, ATR-FTIR	51–27909 particles m^{-3}	[14]
Lake Winnipeg, Canada	Collected by a manta trawl (333 μm mesh)	Stereomicroscope, SEM-EDS	193420 ± 115567 particles km^{-2}	[69]
Wuhan urban lakes, China	0–20 cm depth: Collected by a Teflon pump and then passed through a steel sieve (50 μm mesh)	Stereomicroscope, FTIR	1660.0 ± 639.1–8925 ± 1591 particles m^{-3}	[17]
Nine rivers in Illinois, USA	Collected by a neuston net (333 μm mesh)	Stereomicroscope, Pyr–GC-MS	15520–4721709 particles day^{-1}	[70]
Xiangxi Bay of Three Gorges Reservoir, China	0–30 cm depth: Collected by a trawl net (112 μm mesh)	Stereomicroscope, Micro-Raman	0.55×10^5–342×10^5 particles km^{-2}	[20]

FTIR: Fourier transform infrared spectroscopy; FPA-FTIR: focal plane array-Fourier transform infrared spectroscopy; micro-FTIR: Fourier transform infrared microspectroscopy; ATR-FTIR: attenuated total reflectance-Fourier transform infrared spectroscopy; SEM-EDS: scanning electron microscopy-energy dispersive X-ray spectroscopy; Pyr-GC-MS: pyrolysis-gas chromatography-mass spectrometry; Micro-Raman: Raman microspectroscopy.

3.2. Sediment samples

Sediments have been considered as the long-term sink for microplastics [10,22]. Sediment samples are generally collected from the beach or water bottom (Table 2). In beach sampling for microplastics, sampling work can be practiced on the whole beach, within several separate zones, or along a transect, such as horizontally along the latest drift line, or vertically from the water edge to the backshore of the beach [12,23]. There is no consistency in the sampling depth for beach samples, but the first 5 cm seems to be the most commonly investigated [23]. Selective method is frequently performed in beach sampling, using tweezers [24], metal shovels [13], or directly by hand [25], to collect larger plastic particles (mostly 1–5 mm in size) that are visually identifiable. Undoubtedly, exclusion of the smaller plastics can cause a considerable underestimation of microplastic abundances being reported. Bulk sampling is an admirable method with which to capture the smaller microplastics in beach samples. However, bulk samples tend to contain large amounts of unwanted materials, thus enormously increasing the handling workload. Subtidal sediment samples can be obtained using metal grabs or box corers [26,27]. Since the distribution of microplastics in subtidal sediments is highly heterogeneous, it is recommended to conduct several replicates in order to obtain a representative sample, especially when utilizing the point-to-point samplers, such as a corer or a grab. Diversity of sampling approaches has caused variations in the quantification units of microplastics for sediment samples. In terms of reporting the results, the abundance of microplastics in sediments can be exhibited as the number of microplastics per units of dry or wet weight (g or kg) [26,28], area (m^2) [29], or volume (mL, L or m^3) [30] of sediment, thereby complicating the comparison between studies.

3.3. Biological samples

There is increasing evidence that microplastics can be ingested by various aquatic animals at different trophic levels. Due to the diversity of the studied organisms and the habits where the organisms are sampled, a large variety of techniques have been employed for sampling the biologically ingested microplastics (Table 3). For instance, zooplankton can be collected with a bongo net [31], fish species can be obtained with the pelagic net, trawl, electrofisher, or from the local fishermen [5,20,32], crustaceans such as shrimp can be captured with bottom trawls, creels, or traps [33,34], and bivalves such as mussels and oysters can be acquired by hand, with a mussel trawl, or directly purchased from a store [35]. There are also several studies that did not specify the sampling method, especially when targeting large aquatic animals such as the whales, sharks, turtles, and seals [36–39]. The currently widely used qualification units of microplastics in aquatic organisms include the number of microplastics by weight of organisms [40], the number of microplastics per individual [41], or the percentage of individuals containing ingested microplastics [20].

4. Sample preparation

After sampling, microplastics contained in the samples must be extracted for subsequent quantification and identification. Techniques involved in extracting microplastics from other unwanted materials mainly include density separation, sieving, digestion, and filtration.

4.1. Density separation

Density separation exploits the density difference between materials of interest and other unwanted materials, using the buoyant force of a liquid with an intermediate density to separate the lighter materials from the denser after a thorough shaking and settling of the mixture materials in the liquid. Density separation is a commonly applied method to isolate microplastics from environmental samples, particularly sediment samples [4,11]. The specific densities for most plastics range from 0.8 to 1.70 g cm^{-3}, while average densities for sand or other deposits are typically 2.65 g cm^{-3}. The most frequently used salt solution for the density separation process is saturated sodium chloride (NaCl) solution (1.202 g cm^{-3}), because NaCl is inexpensive and eco-friendly [10,42]. It is appropriate to use the NaCl solution to extract the low-density microplastics, such as polyethylene (PE, 0.917–0.965 g cm^{-3}), polypropylene (PP, 0.85–0.94 g cm^{-3}) and polystyrene (PS, 1.04–1.1 g cm^{-3}) [23,43]. However, for separation of denser microplastics, such as polyvinylchloride (PVC, 1.3–1.7 g cm^{-3}) and polyethylene terephthalate (PET, 1.4–1.6 g cm^{-3}), the saturated NaCl solution are less efficient, which thus leads to underestimation in qualification of microplastics.

To address this issue, some high-density salt solutions, such as solutions of sodium iodide (NaI, 1.8 g cm^{-3}), zinc chloride ($ZnCl_2$, 1.5–1.7 g cm^{-3}), and sodium polytungstate (SPT, 1.4 g cm^{-3}), have been successfully employed in many studies and considerably

Table 2
Studies on microplastics pollution in sediment samples.

Location	Sampling	Extraction liquid	Identification	Abundance	Ref.
Lake Garda, Italy	Beach (4–6 L): Sediment corer	$ZnCl_2$ solution	Micro-Raman	75 ± 134 particles m^{-2}	[29]
Warnemünde, Germany	Beach (3 dm^3): Metal Plastering trowel	Sodium polytungstate solution	Binocular light microscope, Micro-ATR-FTIR, Micro-Raman	–	[63]
Siling Co basin, China	Beach (0–2 cm depth, 20 cm × 20 cm quadrat): Metal shovel	Potassium formate solution	Stereomicroscope, Micro-Raman	<4–563 ± 1219 particles m^{-2}	[13]
Belgium coastal zone	Subtidal: Van Veen grab	NaCl solution	Binocular light microscope, Micro-FTIR	390 particles kg^{-1} dry weight	[26]
Atlantic Ocean and Mediterranean Sea	Subtidal (25 cm^2): Multicorer	NaI solution	Micro-Raman	0.5 particles 25 cm^{-2}	[27]
Victoria Harbor, Hong Kong	Subtidal (3 kg): Ekman dredge	NaCl solution	Stereomicroscope, ATR-FTIR	49–279 particles kg^{-1} dry weight	[14]
Tributaries of the River Thames, UK	Subtidal (10 cm depth): Stainless steel scoop	$ZnCl_2$ solution	Binocular light microscope, Micro-Raman	66 particles 100 g^{-1}	[28]
Taihu Lake, China	Subtidal (2 kg): Peterson grab	NaCl solution	Stereomicroscope, Micro-FTIR, SEM-EDS	11.0–234.6 particles kg^{-1} dry weight	[51]

Micro-Raman: Raman microspectroscopy; micro-FPA-FTIR: focal plane array-Fourier transform infrared microspectroscopy; micro-FTIR: Fourier transform infrared microspectroscopy; ATR-FTIR: attenuated total reflectance-Fourier transform infrared spectroscopy; SEM-EDS: scanning electron microscopy-energy dispersive X-ray spectroscopy.

Table 3
Studies on microplastics pollution in aquatic biological samples.

Biota	Location	Sampling	Extraction	Identification	Abundance	Ref.
Zooplankton						
Calanoid copepod (*Neocalanus cristatus*) and euphausiid (*Euphausia pacifia*)	Northeast Pacific Ocean	Collected by Bongo nets	$HCl:HNO_3$ (1:1 v:v) and $HCl:H_2O_2$ (1:1 v:v) digestion of the whole body	Stereomicroscope	1 particle per 34 copepods and 1 particle per 17 euphausiids	[31]
Fish						
Brown trout (*Salmo trutta*)	Swedish west coast	Collected by electrofisher	Proteinase-K and then H_2O_2 digestion of the gastrointestinal tract	Stereomicroscope, Raman	68% of analyzed fish contained microplastics	[5]
13 fish species	Xiangxi Bay of Three Gorges Reservoir, China	obtained from local fisherman	10% KOH digestion of gastrointestinal tract	Stereomicroscope, Micro-Raman	25.7% of fish samples contained microplastics	[20]
Crustacean						
Norway lobster (*Nephrops norvegicus*)	Clyde Sea	Collected by a trawl net	The stomach was separate for analysis	Microscope, SEM, Micro-Raman	83% of animals contained plastics	[33]
Brown shrimp (Crangon crangon)	Southern North Sea and Channel area	Collected by a shrimp trawl	$HNO_3:HClO_4$ (4:1 v:v) digestion of the whole body	Stereomicroscope	1.23 ± 0.99 particles per individual	[41]
Bivalve						
Asian clam (Corbicula flumine)	Taihu Lake, China	Collected by a bottom fauna trawl	H_2O_2 digestion of soft tissue	Stereomicroscope, Micro-FTIR, SEM-EDS	0.2–12.5 particles g^{-1} wet weight	[51]
Oyster (*Crassostrea gigas*)	France	Obtained from the supermarket	69% HNO_3 digestion of the whole body	Microscope, Micro-Raman	0.477 ± 0.16 particles g^{-1} wet weight	[40]
Polychaete						
Lugworms (Arenicola marina)	French-Belgian-Dutch North Sea coast	Collected by a bait-pump or shovel	69% HNO_3 digestion of soft tissue	Microscope, Micro-Raman	1.2 ± 2.8 particles g^{-1} wet weight	[71]

Raman: Raman spectroscopy; Micro-Raman: Raman microspectroscopy; SEM: scanning electron microscopy; SEM-EDS: scanning electron microscopy-energy dispersive X-ray spectroscopy.

increased the extraction efficiency for the high-density microplastics (Table 2). Nevertheless, the high-density salts are generally expensive, and some are environmentally hazardous. Repeating the extraction process is another effective way to attain better recovery of microplastics from the sample matrices [44]. For instance, extraction efficiencies of PE microplastics from sediment samples using a NaCl solution can achieve 61%, 83% and 93% for the first, second and third extraction, respectively [43]. Therefore, for the purpose of achieving higher extraction efficiency and minimizing environmental pollution, it is recommended to recycle heavy salt solutions and repeat the extraction process.

4.2. Sieving

Sieving is another frequently used method for isolating microplastics from water and sediment matrices. The sieves are usually made of metal, like stainless steel or copper [23]. The sieve physically captures the solid materials that are larger than the mesh size and allows water and smaller particles to be removed from the sample. The mesh size of sieves mainly depends on the desired size range of microplastics to be collected, with the majority ranging from 0.035 to 4.75 mm [11,27,43]. Water samples can be sieved directly, or may undergo a digestion step prior to sieving in cases when the sample contains large amounts of biological materials [45]. For sediment samples, sieving assists in reducing the sample volume for subsequent extraction [45]. To separate microplastics into several size categories, multi-tier sieving has been successfully employed in numerous studies by using a series of sieves with a decreasing mesh size through which to pass the sample [12]. After sieving, particles with different size ranges are retained on different sieves.

4.3. Digestion

Samples collected from the natural environment inevitably contain dense amounts of naturally occurring organic materials, such as zooplankton, phytoplankton, remnants of aquatic organisms, or biofilms (e.g., brown algae or bacterial film) attached to the surface of plastic particles, which can introduce great challenge to accurate identification and characterization of microplastics. Digestion is a process aiming at removing the interfering organic materials within the environmental samples. Several techniques have been developed for the biomaterial dissolution process, which typically uses oxidizers, acids, or alkaline substances [23,43].

For water and sediment samples, hydrogen peroxide (H_2O_2) has been frequently applied for the digestion of natural organic debris. A treatment of the dried sediment samples, residues on the filters after filtration, or the microplastics themselves using 30% H_2O_2 solution can remove large amounts of the organic impurities [46]. The mixtures of H_2O_2 and other agents such as sulfuric acid (H_2SO_4) or Fe(II) solution can rapidly eliminate the natural organic matters within the samples [47]. The use of mineral acids or alkalis has also proved to be effective in disintegrating the interfering organic fragments [44,48]. In some cases, rinsing with distilled water and ultrasonic cleaning are also used to eliminate the organic or inorganic surface adherents from the microplastic particles [23].

For biotic samples, one of the most commonly used methods to digest biological tissues is acid digestion, using strong oxidizing acids, such as nitric acid (HNO_3), perchloric acid ($HClO_4$), hydrochloric acid (HCl), or a mixture of the above (Table 3). Compared with HCl and H_2O_2, HNO_3 is more efficient in digesting biological tissues, especially when heat is applied [31,48]. The most successful method involves an acid blend, using a mixture (1:4, v:v) of 68% perchloric acid ($HClO_4$) and 65% nitric acid (HNO_3), which can completely remove biological tissues and other natural debris and leave only plastics and silica behind after digestion [35]. Other commonly employed methods for biomaterial digestion typically involve the use of strong bases. For instance, 10% potassium hydroxide (KOH) has been successfully used to isolate microplastics from the digestive tracts of fish species and bivalves [49,50]. In addition, some oxidizing agents such as H_2O_2 and sodium hypochlorite (NaClO) have also been adopted for dissolution of the biological tissues in microplastics research [32,51].

However, caution must be used when employing the chemical digestion approaches, since although these chemical digestants can successfully remove biological materials from samples, they may also have some detrimental effects upon the microplastics themselves, which can cause a significant loss of microplastics of certain shapes or polymer types [43,45]. To avoid possible damage to the microplastics themselves, a promising method involves using the enzymes as an alternative to chemical reagents in the digestion process. An attempt at enzymatic digestion has been made by using the proteolytic enzyme Proteinase-K, which successfully removed 97% of the biomaterials by weight from the sample without destroying any plastic particles [52]. Other technical enzymes such as proteinase, cellulase, amylase, lipase, and chitinase have also exhibited a satisfactory digestion efficacy for biological impurities [53].

4.4. Filtration

Filtration is an effective approach commonly employed to separate microplastic particles from liquids (e.g., bulk water samples or supernatant solutions obtained from the density separation process) by use of a filter medium that allows only liquid to pass through. The media utilized for filtration include glass fibers as the most frequently used filter, and some other filters such as nitrocellulose, polycarbonate membranes, zooplankton filters, or isopore filters [11,23,42]. The pore size of filters generally varies from 0.45 to 20 μm [17,43,54]. Although filtration is a simple process for separating microplastics from liquids, complications often arise because the liquids are full of various kinds of microscopic particulates or debris, which can rapidly clog the filter media and thereby lower its effectiveness [43]. This drawback can be alleviated by several helpful measures, such as reducing the solution volume, settling liquids for a longer time to facilitate the separation of heavier solid particles from the supernatant, performing a prefiltration step using a filter with a larger pore size, or adding some chemicals (e.g., ferrous sulfate) to the liquid to flocculate the solid fraction [43]. To minimize the loss of microplastics due to their adherence to the walls of the laboratory ware, rinsing the walls of glassware on the filter repeatedly during the filtration process is recommended [23].

5. Identification of microplastics

Following field collection and laboratory preparation of samples, target microplastics need to be accurately identified from the remaining matrix. The most commonly used approach for identification of microplastics consists of visual inspection of possible plastics followed by chemical analysis of the polymeric composition, usually involving a combined use of optical and spectroscopic or thermo-analytical techniques (Tables 1–3).

5.1. Optical techniques

Visual identification is the simplest and most commonly used technique in identification of microplastics, which can be achieved by naked-eye observation or with the aid of an optical microscope (typically a stereomicroscope). Shapes and colors are the main basis to determine whether a suspected item is microplastic [55]. To improve the accuracy of identification results, a series of selection criteria are recommended to be strictly followed when visually examining the microplastics: suspected particles or fibers have no visible organic or cellular structures, fibers should have consistent thickness and color along the entire length, particles are clear and uniformly colored, and transparent and white particles should be further confirmed under a high-magnification microscope or a fluorescence microscope [11,23]. Visual identification is an appropriate method for high volume samples, especially in cases where expensive analytical instruments are not available. However, there always exists a potential for bias when identifying microplastics visually and the quality of identification results depends on many factors, such as the subjectivity of the examiner, the sample matrix, the particle shape and size, and the microscope used for inspection [45,56]. In addition, weathered microplastics may have some changes in morphology, making visual identification even more challenging [55]. As the size of the particles being examined decreases, the possibility of misidentification by optical techniques increases considerably [55]. These drawbacks combine to introduce a high error rate to the visual identification results [11,45]. Therefore, utilizing some spectroscopic instruments or other analytical techniques to confirm the identity of suspected microplastics is recommended, especially for the smaller items.

5.2. Scanning electron microscopy (SEM)

Scanning electron microscope (SEM) can provide high-resolution images of a sample by firing a high-intensity electron beam at the sample surface and scanning it in a raster scan pattern. Surface details (<0.5 nm resolution) of the sample are imaged by the electrons at very high magnifications. Potential microplastics can be differentiated from other organic or inorganic impurities by examining the high resolution images of their surface morphology under SEM [55]. SEM can also be used to analyze the weathering progress of microplastics recovered from natural environment by examining the featured surface textures, like cracks and pits, on these plastic particles [13]. The combined use of SEM and energy-dispersive X-ray spectroscopy (SEM-EDS) is able to provide detailed information about the elemental composition of microplastics and the inorganic additives they contain [55,57]. Utilization of SEM-EDS aids in further differentiating natural materials from microplastics via imaging and elemental analysis, which thereby narrows the amount of particles needed for spectroscopic analysis [57]. Although SEM has been successfully used to examine the surface characteristics of microplastics, this technique requires considerable time and effort for sample pre-preparation and thus is not applicable for handling of large number of samples.

5.3. Fourier transform infrared (FTIR) spectroscopy

Fourier transform infrared (FTIR) spectroscopy can provide a unique infrared spectrum for a specific chemical bond. Different materials have different bond compositions, making it possible to identify an unknown substance by comparing its spectrum with the spectra of known materials. Due to its high reliability, FTIR has become one of the most commonly used techniques in chemical characterization of microplastics recovered from environmental samples [18,58]. In monitoring programs for microplastics, FTIR is used mainly in two ways: scanning all the suspected particles [30], or analyzing a set of subsamples to validate the visual identification results [17]. Although there is no doubt that increasing the number of suspected particles to be analyzed using the FTIR can enhance the accuracy of data, limiting factors such time and cost should be taken into account. FTIR can not only accurately identify the polymer types of microplastics, but also provide further information about physiochemical weathering of microplastics by analyzing their oxidation intensity [59]. However, FTIR is only capable of identifying the polymeric composition of microplastics with a size of >10–20 μm and may lose applicability in cases where the target particles are smaller than its aperture size [47]. Furthermore, confirming the identity of suspected microplastic particles using FTIR is a time-consuming work and sometimes requires a highly

experienced operator. Anyhow, FTIR remains a promising technique for chemical identification of environmental microplastics.

The optimized technologies of FTIR, such as FTIR microspectroscopy (micro-FTIR), attenuated total reflectance-FTIR (ATR-FTIR), and focal plane array (FPA)-FTIR spectroscopy (FPA-FTIR), have also been increasingly utilized in microplastics research worldwide. Micro-FTIR facilitates the detection of smaller particles (>10 μm). ATR-FTIR allows large (>500 μm) and irregularly shaped particles to be directly analyzed without a sample preparation step [45]. FPA-FTIR can offer an unbiased high throughput analysis of all plastic particles (>20 μm) by scanning the filter paper with a high degree of lateral resolution [60]. FTIR imaging in transmittance mode as very common FPA-FTIR technique enables chemical and physical characterization of the analyzed particles simultaneously and is thus gaining increasing application in detection and identification of microplastics within environmental samples [55,61]. Compared with FTIR, FPA-FTIR is much faster in detection of microplastics, but the instrumentation is more cost-intensive and requires a high processing power [47].

5.4. Raman spectroscopy

Raman spectroscopy is another frequently used and highly reliable technique for polymer identification of microplastics from various environmental matrices [62]. Identification of microplastics with Raman spectroscopy is undertaken by irradiating monochromatic laser beam onto a suspected sample, which results in a different frequency of the backscattered light due to absorption, scatter or reflection by the sample's specific molecular structure and atomic composition [55]. This so-called Raman shift can produce a unique spectrum for each polymer. Raman spectroscopy enables non-destructive chemical characterization of microplastics, which is highly advantageous in cases where further analysis is needed for the samples [58]. Some of the advantages of FTIR are shared by Raman spectroscopy, such as high reliability, possibility for high throughput screening, low sample amount requirement, and environmental friendless [62]. Relative to FTIR techniques, Raman spectroscopy is advantageous in higher spatial resolution, wider spectral range, narrower spectral bonds, and lower sensitivity to water interference [62,63]. A combination of Raman spectroscopy with microscopy (micro-Raman) makes it possible to identify microplastics down to 1 μm in size, which is extremely challenging for other spectroscopic techniques to achieve [55]. It is practical to obtain spatial chemical images of the whole sample at a spatial resolution of <1 μm by use of micro-Raman spectroscopy coupled with Raman spectral imaging equipment [45]. Raman spectroscopy can also be combined with confocal laser scanning microscopy to locate microplastics within biological tissues [64]. The main drawback of Raman techniques is that it is easily interfered with by the presence of additives, pigments or attached chemicals associated with microplastics, which may negatively affect the identification accuracy [47]. In addition, the signal to noise ratio of Raman spectroscopy is inherently low and thus may increase the difficulty of spectrum analysis [62]. However, this does not prevent Raman spectroscopy from being a powerful analytical technique in microplastic research.

5.5. Pyrolysis–gas chromatography–mass (Pyr-GC-MS) spectrometry

Pyrolysis–gas chromatography–mass (Pyr-GC-MS) spectrometry is a destructive technique that has also been successfully employed in chemical identification of environmental microplastics by analyzing their thermal degradation products [57]. The polymer types of microplastics can be determined by comparing their characteristic pyrograms with reference pyrograms generated by known pure polymers [45]. Pyr-GC-MS allows direct introduction of solid polymer particles with minimal sample pretreatment. In contrast with ATR-FTIR microspectroscopy, a significant advantage of Pyr-GC-MS is the capability of simultaneously providing detailed information about the chemical composition of the polymer and contained organic additives [65]. In addition, Pyr-GC-MS is not sensitive to the shape, size, and associated organic or inorganic contaminants of the analyzed particles [65]. Only a small amount of sample (100–500 μg) is needed for one measurement, indicating that Pyr-GC-MS is applicable for trace analysis [66]. However, this technique requires only one particle to be analyzed per cycle and the time needed for one measurement ranges from 30 to 100 min, which inevitably limits its applicability for analysis of large sample quantities [65]. In view of the fact that potential microplastic particles have to be manually inserted into the pyrolysis tube, only particles that are large enough (>100 μm) to be manually manipulated are suitable to be analyzed by the Pyr-GC-MS [67]. To circumvent these problems, variants of Pyr-GC-MS have been used to develop novel techniques, such as thermal extraction and desorption–gas chromatography–mass spectrometry (TED-GC-MS) [66]. TED-GC-MS combines thermal extraction with thermogravimetric analysis (TGA) and thermal desorption gas chromatography mass spectrometry (TD-GC-MS), making it possible for fast measurement of microplastics of certain polymer types in environmental samples [66]. Compared with spectroscopic methods, the major disadvantages of the thermo-analytical techniques are that they are destructive, only capable of chemical characterization, but fail to provide detailed information about morphological properties of the analyzed microplastics, such as particle size and size distribution [47,65]. In this context, it is recommended that thermo-analytical methods serve as complementary techniques to the spectroscopic methods in order to achieve integrated analysis of microplastics.

6. Quality assurance and quality control (QA/QC)

When conducting monitoring programs on microplastics, it is essential and of vital importance to take rigorous quality assurance and quality control (QA/QC) measures during the whole simple process, in order to improve the quality of data. Barriers involved in the detection of microplastics recovered from the natural environment mainly include contamination and under- or overestimation [42]. In field sampling, to obtain representative samples is crucial for accurate assessment of microplastic abundance in the studied area, which requires an appropriate sampling tool and implementation of a carefully designed sampling strategy [12]. Replicate samples can also help to enhance the reliability of monitoring data.

Background contamination (e.g., airborne fibers) can cause considerable overestimation to the quantitative results of environmental samples [49]. To check the background contamination, a series of procedural blank tests should be conducted during sampling and laboratory handling process [4,18]. For instance, potential airborne contamination can be examined by filtering the air of the workplace through the filter paper for a certain period of time under vacuum condition [17]. Some preventive measures, such as wearing latex gloves and pure cotton clothes, rinsing experimental apparatuses carefully, and keeping the workplace clean, are also helpful to reduce background contamination [4,17]. Recovery of microplastics can be tested with spiked blanks by spiking clean environmental samples (e.g., sediments) with known concentrations of plastic particles and then subjecting the spiked samples to extraction [18]. It is recommended that the spiked plastics comprise similar shapes, size ranges and polymer types to that of

the target microplastics, since these factors can substantially affect the extraction efficiency.

A number of studies have demonstrated that some digestive agents, such as H_2O_2 and strong oxidizing mineral acids (e.g., HNO_3 or $HClO_4$), particularly when at high concentrations or high temperatures, can cause damage or complete dissolution of certain kinds of exposed polymers, thereby obscuring samples or resulting in underestimation [34]. It is therefore imperative to conduct a comprehensive test to determine the potential effects of the applied chemical digestants on plastics prior to using them for sample digestion. Visual examination of microplastics should follow strict selection criteria as mentioned above, in order to reduce false identification. Inclusion of some spectroscopic techniques in chemical characterization of microplastics as much as possible can significantly aid in improving the accuracy of identification results.

7. Conclusions

Microplastics are nearly ubiquitously present in different matrices of the aquatic environment [3,5,9]. This review collated the currently used techniques for monitoring microplastics in three aspects of the aquatic environment: the water, sediment and aquatic biota. The inconsistence of approaches employed in global monitoring programs for microplastics is the main problem that impedes large-scale spatial and temporal comparisons of the exiting data [4,11,45]. Therefore, one basic issue that needs to be urgently addressed is the establishment of standardized methodologies for the operating procedures involved in the cycle of assessing environmental microplastics from field sampling to laboratory analysis.

Although sampling in itself is not challenging, collecting representative samples requires careful design, especially in regard to beach sampling. Future research needs to account for many factors, such as sampling locations, sampling techniques (e.g., sampling tools and mesh sizes), and number of replicates, when conducting a field sampling study. Extraction efficiency of microplastics from the environmental matrices largely depends on the employed extraction solution. It is recommended to reutilize some heavy salts (e.g., $ZnCl_2$) and repeat the density separation procedure for the purpose of raising extraction effectiveness and minimizing environmental hazards. It is necessary to remove the naturally occurring impurities from the plastics before visual and spectroscopic identification. Compared with chemical digestants, the use of enzymes can not only effectively digest the interfering organic matter, but will do little harm to microplastics. Visual counting is a mandatory step for quantitative analysis of microplastics. However, in light of the fact that visual method alone can introduce high misidentification rate as the size of particles decreases, it is therefore essential to conduct subsequent spectroscopic analysis to validate the identification results, for which FTIR and Raman spectroscopies are the most promising techniques. The combined use of spectroscopic techniques and some extended equipment (e.g., FPA-FTIR and TED-GC-MS) can largely increase the efficacy of identification.

Future research should focus on establishing standardized methodologies for sampling and extracting microplastics from environmental matrices and developing highly efficient analytical techniques (e.g., fully or semi-automated analytical technologies) to facilitate rapid and accurate identification and quantification of microplastic particles. In addition, in order to estimate the ecological risks of these microscopic plastic particles, there is an increasing demand to develop reliable and efficient tools and analytical methods capable of adequately detecting and quantifying plastic particles at micron- or even nano-scales in environmental samples. This review seeks to offer a comprehensive understanding of sampling and analytical techniques currently used in the microplastics monitoring programs conducted in aquatic environments and is intended to contribute to establishing standardized methodologies to assess the magnitude of this issue at global level.

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