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A review of microplastics in sediments: Spatial and temporal occurrences, biological effects, and analytic methods

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ABSTRACT

The persistence and prevalence of microplastics in the environment have raised concerns among scientists, the general public, and environmental regulatory agencies, and a large number of studies on microplastics in sediments have been published. We reviewed those studies and focused on understanding the spatial and temporal variations, biological effects, and analytic methods of microplastics in sediments. Microplastics are widely distributed in marine and freshwater sediments. The distribution of microplastics is mainly influenced by anthropogenic input and environmental processes that determine the transportation, transformation and accumulation. The appearance of microplastics in sedimentary records has been proposed as a stratigraphic marker for the onset of the Anthropocene Epoch. Our current understanding of biological effects of microplastics is based mainly on field research and laboratory-scale experiments using unrealistic concentration and limited forms of microplastics. Future studies should consider environmentally relevant concentration, the diverse composition and form of microplastics presenting in the natural environment. Density separation method for separation of microplastics in sediments is the most commonly used, and component identification by optical analysis (Fourier-transform infrared spectroscopy spectra, Raman spectra etc.) are important analytic methods. Yet the separation method remains to be standardized across laboratories. Other separation methods include electrostatics separation, organic solvent extraction and magnet separation. We recommend the combination of density separation, electrostatic separation and organic solvent separation to improve the separation efficiency of microplastics and to standardize the analytic process for sediment microplastics in future studies.

1. Introduction

An estimated 5–13 million tons of plastics are exported to the ocean each year, yet those floating on the ocean surface are only around 0.3 million tons/year, suggesting that a significant fraction of plastics are assimilated by marine organisms or have accumulated in sediments (Matsuguma et al., 2017). The widespread distribution of plastic polymers has drawn the attention of scientists (Shahul Hamid et al., 2018). Over the past decade, a large number of studies have been conducted to understand the environmental distributions and consequences of microplastics (Browne et al., 2011; Eriksen et al., 2014; Lehner et al., 2019; Marka et al., 2010). The distribution and effects of microplastics were studied by many researchers in the world. Relative to numerous studies focusing on microplastics contamination in water columns, less research attention has been given to microplastics in sediments. However, sediments can act as both a source and sink for microplastics and play an important role in regulating the distribution of microplastics within an aquatic environment (Jambeck et al., 2015; Woodall et al., 2014).

Van Cauwenberghe et al. (2015b) summarized the occurrence, effects and techniques of microplastics in sediments and pointed out deficiencies of previous researches including no clear definition of microplastics, environmentally unrealistic experiments in the effect assessments of microplastics and no standard separation technique. At present, the spatial distribution of microplastics in other sediments including river sediments, lake sediments and cropped soils were also

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widely studied to understand its production and influencing factors (Wang et al., 2017a; Xiong et al., 2018; Zhang and Liu, 2018). For the analysis techniques, the most common approach is to extract plastic particles from the sediment by using a density separation, and analyze the features of microplastics including morphology, particle size and color etc. (Van Cauwenberghe et al. (2015b). While, microplastic concentration provides an importation parameter for characterizing its level of contamination and a referring value for cultural experiments to access the biological effects (Lenza et al., 2016). A standard and improved technique is also beneficial to the comparison of spatial and temporal variations between different studies, and then discuss the mechanism of production, transportation and sedimentation process for microplastics.

Thus this paper reviews and summarizes the available literature on studies of microplastics in all kinds of sediments all around the world mainly after 2015. Due to the exclusively anthropogenic origin and resistance to environmental degradation of plastics, microplastic particles have been becoming a new dating tool for recent young sediments, with the rapid increase in microplastic abundance marking the decade of the 1950s (Wang et al., 2018a). Therefore, it is important and necessary to evaluate whether the temporal trend of plastics accumulation in marine sediments reflects the temporal trend of human production and consumptions of plastics on land. In addition to describing spatial and temporal variations for microplastic, this review also summarizes the latest studies in biological effects, separation and identification methods of microplastics in sediments. The main objectives of this review were to: (1) obtain a more accurate and clear understanding of the distributions and features (types and morphology) of microplastics in all kinds of sediments more than marine sediment, spatially and temporally, (2) make an further assessment of progress and deficiency of studies in microplastics biological effects, and (3) summarize the new methods of extracting microplastics and identifying the components, and propose a combined method to extract microplastics and concerning of small microplastics in the future.

2. Brief research history and definitions of microplastics

Scientific research on plastics began in the 1970s. Carpenter and Smith (1972) first reported the concentration, morphology, and attachment of plastic particles in the surface waters of the Sargasso Sea. Wong et al. (1974) determined the content of plastics at the surface of the Pacific Ocean and showed that possible factors regulating the distribution were marine oil transport, prevailing wind transport and surface circulation. However, these two studies did not draw enough attention from the scientific community at the time of the publication. It was not until much later when Thompson et al. (2004) proposed the concept of microplastics that plastic pollution has received wide attention from researchers and the public.

The number of published scientific, peer-reviewed articles on microplastics have increased rapidly since 2011, following an exponential pattern (Fig. 1a). The majority of studies were published after 2015, and the number of articles over the last 3 years (2016–2018) exceeded the total number of articles published 2006–2015. The articles of microplastics related to sediments also increased exponentially, accounting for approximately one-third of total number of articles on microplastics (Fig. 1b).

Despite the increasing research attention on microplastics, there is still much ambiguity in the definition of microplastics. For instance, microplastics may be defined as plastic particle sizes ≤ 5 mm (Erni-Cassola et al., 2017; Machado et al., 2018) or ≤ 1 mm (Browne et al., 2011; Marka et al., 2010). Hartmann et al. (2019) defined plastics, on the basis of the polymer composition, solid state and solubility, as “objects which consist of synthetic or heavily modified natural polymers as an essential ingredient that, when present in natural environments without fulfilling an intended function, are solid and insoluble at 20 °C″, and they further classified the plastics into four types based on the particle sizes (Table 1): 1 to < 1000 nm for nanoplastics, 1 to < 1000 μm for microplastics, 1 to < 10 mm for mesoplastics, and ≥ 1 cm for macroplastics. The origin of microplastics is commonly divided into primary microplastics and secondary microplastics (UNEP, 2016). Primary microplastics are intentionally manufactured to a specific size, such as plastic microspheres in cosmetics (Lei et al., 2017), and secondary microplastics are produced during the use or weathering of plastics (Browne et al., 2011; Jahnke et al., 2017).

3. Spatial variation of microplastics in sediments

Microplastics abundance shows high spatial variability, varying as a function of anthropogenic influences (Hitchcock and Mitrovic, 2019; Veerasingam et al., 2016) and natural conditions that influence the transport and accumulation of microplastics such as river flow velocity, wind speed, wind direction, and tides (Zhang, 2017). Overall, microplastics tend to accumulate in human-impacted rivers and coastal oceans with weak hydrodynamics (Hitchcock and Mitrovic, 2019; Zhang et al., 2019b). Biofilms and sizes of microplastics also influence the spatial variation in microplastics. For example, microplastics less than 300 μm or covered by biofilms are preferentially preserved in sediments (Michels et al., 2018; Wang et al., 2018c).

3.1. Microplastics in Europe

Microplastics abundance in sediments exhibits a large variability across European countries. Among a range of studies reporting the concentration of microplastics in surface sediments (Table 2), the highest concentration of microplastics was reported in sediments from the Mediterranean Sea in Spain (Alomar et al., 2016). Relatively low concentrations were found in the Algarve coastal sediments in Portugal (Frias et al., 2016) and the Baltic Sea near Russia (Zobkov and Esiukova, 2017). Microplastics in sediments contained a variety of components, including polypropylene, polystyrene, polyvinyl chloride, polyester, poly (ethylene-propylene), poly (vinyl acetate), polyethylene terephthalate, polyamide and rayon. The most common components found were polypropylene and polyethylene. The morphology of microplastics included fragments, fibers, films and granules, with fragments and fibers being most prevalent.

3.2. Microplastics in China

China is a large producer and consumer of plastics. Resin and polypropylene fiber production in China accounts for 28% and 68% of the world's total, respectively (Geyer et al., 2017). In 2017, packaging wastes generated by food delivery in China's mega cities reached 1.5 million metric tons (Song et al., 2018). The rate of plastic recycling is low—only 25% of plastics were recycled and the remainder of plastics were discarded to the environment in 2014 (Geyer et al., 2017). Most studies on microplastics in China started after 2010, generating some preliminary assessments of microplastics distribution in China (Sun, 2016; Wang et al., 2017b; Yang et al., 2018; Zhu et al., 2018a). The Chinese government has increased funding support to the relevant research areas, including a key special theme on monitoring marine microplastics and assessing the associated environmental and ecological consequences.

Most studies on microplastics in sediments in China were published in over the last three years (Table 3), and the study areas are mainly focused on the Yangtze River, the Yellow Sea and the Bohai Sea. Overall, the concentrations of microplastics in sediments were higher in China than in Europe (Table 2 and Table 3). The Chai river valley influenced by agricultural activities had the highest concentration, up to 42960 items/kg dry sediment (Zhang and Liu, 2018). Notably, remote lakes in the Tibet Plateau exhibited a high level of microplastics, demonstrating that microplastics can be transported to remote areas and the microplastics may be transported to the lakes by rivers (Zhang et al.,

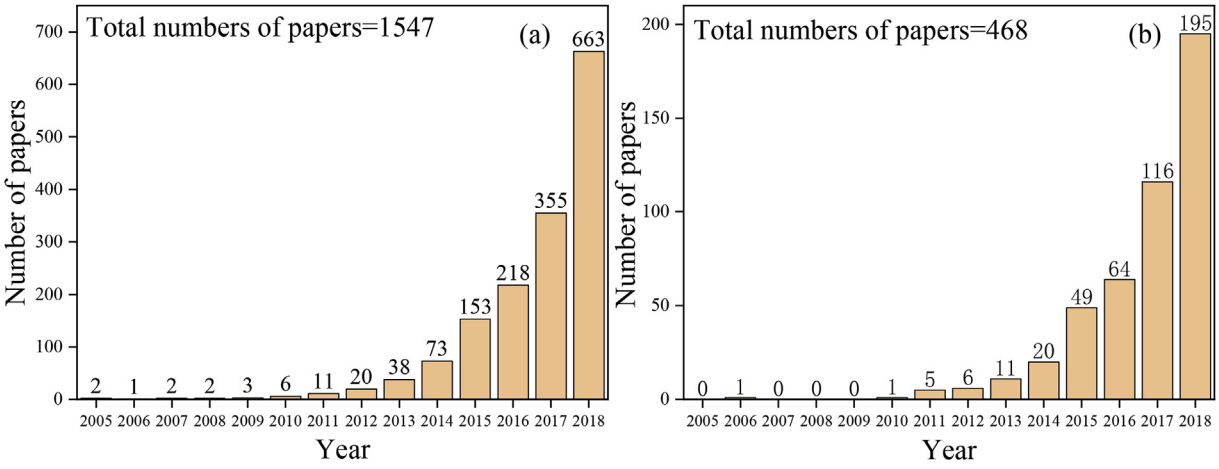

Fig. 1. (a) the number of published papers (science technology) on microplastics from year 2005–2018; (b) the number of published papers (science technology) on microplastics in sediments from year 2005–2018 (searching in Web of Science with TS = microplastics and TS = microplastics sediments, respectively).

Table 1
Categories of plastics based on size (Hartmann et al., 2019).

Name	Size
Nanoplastics	1 to < 1000 nm
Microplastics	1 to < 1000 μm
Mesoplastics	1 to < 10 mm
Macroplastics	≥1 cm

2016). Similar to Europe, the main components of microplastics are dominated by polypropylene and polyethylene. The morphology of microplastics is also diverse, including fibers, films, granules, fragments and foams, with fibers being the most prevalent morphology.

3.3. Microplastics in others countries and areas

The concentrations of microplastics in nearshore sediments of Lake Ontario was up to 27,830 items/kg dry sediment (Table 4), which is the highest reported value among nearshore sediments around the world (Ballent et al., 2016). Sediments from the Complex Lagoon-Channel of Bizerte (Tunisia) and the Atoyac Rivers basin, Mexico, also showed high microplastics concentrations, more than 3000 items/kg dry sediment (Abidli et al., 2017; Shruti et al., 2018). The concentrations of microplastics in the study areas of Iran (Naji et al., 2017) and the United States (Wessel et al., 2016) were lower than those in the Yellow Sea (Wang et al., 2019b) and the Bohai Sea (Yu et al., 2016). Microplastics were reported in remote regions. Peng et al. (2018b) found that concentrations of microplastics varied from 200 to 2200 pieces per liter in the hadal sediments of the Mariana Trench and Mu et al. (2019) detected microplastics in polar regions.

Comparing microplastic concentrations across different regions around the world (Tables 2–4), Europe had the lowest concentrations. However, large variability was also reported within a study area, such as the Complex Lagoon-Channel of Bizerte in Tunisia and the Chai river valley in China (Table 3). Although most studies have been performed on marine sediments, inland lake sediments were also heavily polluted by microplastics (e.g., Lake Ontario in Canada). Overall, polypropylene and polyethylene were the most common component of microplastics in the world sediments and the most common microplastic morphology consisted of fibers and fragments. While, it also can be noticed that the units of concentrations in sediments were not uniform and that the most methods were half-quantified. This created difficulties for comparing different studies. A standard and improved method is needed to measure concentrations of microplastics in sediments, while also helping to better understand the pattern of production, transportation and distributions of microplastics in sediments.

4. Chronological changes in microplastics in sediments

In comparison to numerous studies focusing on the spatial variation of microplastics in surface sediments, a limited number of studies have evaluated chronological changes that require dating sediment cores and analyzing microplastics of different sediment horizons. Substantial increases in the concentrations of different forms of microplastics have been observed in sediments from different geographic regions (Claessens et al., 2011; Matsuguma et al., 2017), mirroring the rapid growth of plastic production around the world. For example, in the

Table 2
Abundance of microplastics in sediments across Europe.

Country	Location	Concentration (items/kg dry)	Major composition	Major morphology	References
France	Gulf of Biscay	67 ± 76	PP, PE, PS, PVC, PEST	Fragments	Phuong et al. (2018)
Italy	Pianosa Island	1.09 g/m^2	Nylon, PP and PE	Filaments	Mistri et al. (2018)
Russia	Baltic Sea	34 ± 10	–	Fragments, Fibers, films	Zobkov and Esiukova (2017)
Portuguese	Algarve coastal waters	10 ± 1	PP, RY	Fragments, fibers	Frias et al. (2016)
Switzerland	Floodplain	5 mg/kg	PE	–	Scheurer and Bigalke (2018)
Spain	Mediterranean Sea	100–900	–	Fragments, fibers	Alomar et al. (2016)
Poland	Southern Baltic Sea	25–53	PEST, EPM, PVA	fibers	Graca et al. (2017)
Portugal	Antuã River	18–629	PP, PE, PS, PET	Fragments, Granules	Rodrigues et al. (2018)
Ireland	Irish Continental Shelf	1.42–11.24 items/per station	PA, PET	fibers, Fragments	Martin et al. (2017)
UK	The upper River Tame catchment	165 particles/kg	PE, PVC and polymethyl methacrylate	Fragments and fibers	Tibbetts et al. (2018)

PP: polypropylene; PE: polyethylene; PS: polystyrene; PVC: polyvinyl chloride; PEST: polyester; EPM: poly (ethylene-propylene); PVA: poly (vinyl acetate); PET: polyethylene terephthalate; PA: polyamide; RY: rayon; "-": not documented.

Table 3
Abundance of microplastics in sediments across China.

Location	Concentration (items/kg dry)	Major composition	Major morphology	References
Xiangshan Bay	1739 ± 2153	PE, Synthetic cellulose fibers	–	Chen et al. (2018)
South Yellow Sea	560–4205	PP, PE, PS, Nylon	Fibers	Wang et al. (2019b)
Poyang Lake	54–506	PP, PE	Fibers	Yuan et al. (2019)
North Yellow Sea	37.1 ± 42.7	PP	Films, fibers	Zhu et al. (2018b)
Bohai Sea and Yellow Sea	171.8[a], 123.6[b],72.0[c]	RY, PE, PET	Fibers	Zhao et al. (2018)
Shanghai	802	PP	Granules	Peng et al. (2018a)
Yellow Sea and East China Sea	60–240	PE, PET, PEST, Acrylic, cellulose, cellophane	Fibers	Zhang et al. (2019a)
Dongting Lake	200–1150	PET, PP, PE, PS, PVC	Fibers	Jiang et al. (2018)
Changjiang Estuary	212 ± 9	RY, PEST, Acrylic	Fibers	Peng et al. (2017)
Three Gorges Reservoir	25–300	PS, PP, PE	Fibers	Di and Wang (2018)
Taihu Lake	11–234.6	cellophane	Fibers	Su et al. (2016)
Guangzhou	80–9597	PP, PE	Fibers	Lin et al. (2018)
Bohai Sea	102.9–163.3	PEVA, LDPE, PS	Fragments, films	Yu et al. (2016)
Qinghai Lake	50-1292 items/m^2	PP, PE	Fibers, films	Xiong et al. (2018)
Bohai Sea and Yellow Sea	1.3–14712.5	PP, PE	Films, foams	Zhou et al. (2018a)
Chai river valley	7100–42960	–-	Fibers	Zhang and Liu (2018)
Tibet plateau	8 ± 14–-563 ± 1219 items/m^2	PE, PP	Films, fibers, foams, fragments	Zhang et al. (2016)
South Yellow Sea	560-4205 n/g wet weight	PP, PE, nylon and PS	Fibers	Wang et al. (2019b)
Hong Kong	49–279	PP, HDPE and LDPE	Fragments and pellets	Tsang et al. (2017)
Beijiang River littoral zone	178 ± 69–-544 ± 107	PP and PE	Fragments and films	Wang et al. (2017a)
Hong Kong	169 ± 48–-221 ± 45 items/kg wet weight	PP and PET	Fibers and films	Cheang et al. (2018)
Changsha	270.17 ± 48.23–866.59 ± 37.96	PS	Fragments	Wen et al. (2018)
Mao Sea in Guangxi Province	520 ± 8–-2310 ± 29	PP, PE and PS	–-	Li et al. (2019)
Wushan county of Gansu Province	360–1320	PE, PVC and PS	Fibers	Ding et al. (2019)
Poyang Lake	54–506	PP and PE	Fibers	Yuan et al. (2019)

PP: polypropylene; PE: polyethylene; HDPE: high-density polyethylene; LDPE: light density polyethylene; PS: polystyrene; PVC: polyvinyl chloride; PEST: polyester; PET: polyethylene terephthalate; RY: rayon; PEVA: polyethylene vinyl acetate; a: Bohai Sea; b: North Yellow Sea; c: South Yellow Sea; "-": not documented.

sediment cores collected from Belgian beaches, Claessens et al. (2011) found that the concentrations of microplastics tripled over the last approximately 16 years (from 1993 to 1996 to 2005–2008). In the continental shelf sediments off western Ireland, Martin et al. (2017) analyzed microplastics from 11 stations of various proximities to urban and industrialized areas. They found that most microplastics occurred in sediments post-dating the onset of plastic production in the 1940s and attributed those before the onset to post-depositional transport. Willis et al. (2017) analyzed the microplastics from two coastal sediments cores (150 m and 1000 m offshore, respectively) in Australia and observed the highest concentrations in the upper layers (15 cm and above) dated 1976 or younger. They further compared the abundance of microfibers in the sediment cores through time with the predicted accumulation based on the global plastic production values and found the two independent datasets exhibited consistent temporal trends. Matsuguma et al. (2017) analyzed coastal sediments in Asia (Japan, Thailand, Malaysia) and Africa (South Africa), and they observed that microplastics concentrations in sediments increased towards the surface at all countries. However, the years of appearance and rapid rise varied among the countries (e.g., the detection of microplastics began in the 1950s for Japan and in the 1960s for Thailand), which may reflect regional differences in the timing of commercial production and consumption of plastics. To date, all studies have demonstrated that coastal sediments serve as an overall reliable archive of plastic pollution on land, yet the spatial scale of the contributing geographic regions remains unclear and the temporal tends (e.g., the ages of the beginning and rise of microplastics in sediments) vary regionally.

5. Biological effects of microplastics

As the probability of biological exposure to microplastics increases in the environment, so does the associated ecological damage (Bour et al., 2018; Li et al., 2018; Su et al., 2018). Many studies were conducted to evaluate the effects of microplastics on aquatic organisms (Desforges et al., 2015; Jabeen et al., 2017; Li et al., 2016; Steer et al., 2017; Van Cauwenberghe et al., 2015a). Field studies indicated that organisms in sediments assimilated microplastics from surrounding environments (e.g., Fang et al., 2018; Su et al., 2018; Wang et al., 2019b). Benthic organisms contained mean concentrations of microplastics ranging from 0.02 to 0.46 items/g wet weight or (i.e., 0.04–1.67 items/individual) in the shelf of the Bering and Chukchi Sea (Fang et al., 2018). In the South Yellow Sea, microplastics in the tissues of benthic organism were also detected, with the concentrations varying from 1.7 to 47.0 n/g wet weight (Wang et al., 2019b). Su et al.

Table 4
Abundance of microplastics in sediments across world except for Europe and China.

Country/Region	Location	Concentration (items/kg dry)	Major composition	Major morphology	References
Tunisia	Complex Lagoon-Channel of Bizerte	3000–18000	–	Fibers	Abidli et al. (2017)
America	Gulf of Mexico estuaries	50.6 ± 9.96[a],13.2 ± 2.96[b]	PP, PE	Fragments, Fibers, Foam	Wessel et al. (2016)
Arctic	Chukchi Sea and Chunchi Basin	5.30–68.88	PP, PET and RY	Fibers	Mu et al. (2019)
Canada	Lake Ontario nearshore	20–27830	PE, PS	Fibers, Fragments	Ballent et al. (2016)
Iran	Persian Gulf	61 ± 49	PE, PET, Nylon	Fibers, Films	Naji et al. (2017)
Mexico	Atoyac Rivers basin	4500 ± 702.23	–	Films, Fragments	Shruti et al. (2018)

PP: polypropylene; PE: polyethylene; PS: polystyrene; PET: polyethylene terephthalate; a: The more marine-influenced locations; b: The more freshwater-dominated locations; "-": not documented.

(2018) measured the concentrations of microplastics in clams, water, and sediments in the middle to lower Yangtze River Basin, and they found that the concentrations of microplastics in clams were dependent on those in water and sediments, suggesting clams could be used to evaluate microplastic pollution in freshwater systems.

The adverse effects of microplastics in sediments on organisms have been demonstrated by many experiments. Lei et al. (2018) assessed the effects of five types of pristine microplastics polyamides, polyethylene, polypropylene, polyvinyl chloride and polystyrene particles on nematode *Caenorhabditis elegans* (benthic) by exposing the organisms for two days. The concentrations of suspended microplastics were 0.5, 1.0, 5.0, and 10.0 mg/m^2 and the particle sizes included 0.1 μm, 1.0 μm and 5.0 μm. Their results showed all five types of microplastics could cause death, body length reduction, decreases in intestinal calcium levels, and increased expression of glutathione S-transferase 4 enzyme and 1.0 μm polystyrene particles could cause significant toxicity on *Caenorhabditis elegans* among three sizes. Bour et al. (2018) used three concentrations (1, 10, and 25 mg/kg of sediment) and three size classes (4–6, 20–25 and 125–500 μm) of polyethylene microparticles to understand their effects on two sediment-dwelling bivalve species, *Ennucula tenuis* and *Abra nitida*. Their results showed that the effects were significantly depended on the concentration and size of polyethylene, and the most severe effects after four weeks' exposure were related to the largest size and elevated concentrations of particles. van Weert et al. (2019) examined the effects on sediment-rooted macrophytes *Myriophyllum spicatum* and *Elodea* sp. utilizing six doses of polystyrene nanoplastics (50–190 nm, up to 3% sediment dry weight) and polystyrene microplastics (20–50 μm, up to 10% dry weight) over three-week incubations, and their results showed the shoot to root ratio of both macrophytes decreased due to nanoplastics exposure that hindered nutrient accumulation. Deng et al. (2017) exposed mice to fluorescent and virgin polystyrene microplastics of two sizes (5 and 20 μm) and found that microplastics accumulated in liver, kidney and gut of mice, negatively impacting lipid metabolism and oxidative stress of mice, and the accumulation pattern strongly dependent on the size of microplastics. Notably, humans can be exposed to microplastics through air and food webs (i.e. (Su et al., 2019),) with potentially negative impacts (Cai et al., 2017; Smith et al., 2018; Wang et al., 2019c). Kim et al. (2018) reported that commercial food-grade salts contained microplastics, with higher concentrations in sea salt than in lake salt and rock salt.

In the previous review by Van Cauwenberghe et al. (2015b), it was noted that incubation experiments typically use only one type or size of plastics at concentrations much higher than the environmentally relevant level. However, Ziajahromi et al. (2018) found the size of microplastics is a key factor mediating the biological impacts of microplastics through an incubation experiment where sediment-dwelling invertebrates *Chironomus tepperi* were exposed to polyethylene microplastics of four different sizes (1–4, 10–27, 43–54 and 100–126 μm) at the level of environmentally relevant concentrations (500 particles/kg sediment). Polyethylene microplastics were demonstrated to reduce the survival, growth, and emergence of *C. tepperi*, with the most significant effects observed for the size range of 10–27 μm (Ziajahromi et al., 2018).

We further noted that the incubation experiments have not evaluated the effects of parameters that influenced the size, form, and chemical compositions of microplastics, including biotic and abiotic weathering, sorption and the presence of biofilms (Acosta-Coley et al., 2019; Cai et al., 2018; Lobelle and Cunliffe, 2011; Wang et al., 2019a). Based on Fourier-transform infrared spectroscopy (FTIR) and Raman spectra analysis, the compositions of polyethylene, polypropylene and polystyrene plastic pellets changed after three months of UV irradiation in three difference environments including artificial seawater, ultrapure water and air. As the time of irradiation increased, changes in FTIR spectra became more pronounced, with increasing observations of granular oxidation, cracks and flakes (Cai et al., 2018). The degree of weathering exerts an important control on the sorption capacity of plastics debris. Acosta-Coley et al. (2019) found that degraded pellets were more enriched with Ba, Cr, Rb, Sr, Ce, Zr, Ni and Pb element than new pellets because the degraded pellets had a larger surface area for sorption. Similarly, Wang et al. (2018b) suggested that the sorption of plastic fibers of phenanthrene may be enhanced by a small size and rough surface. It is commonly observed in nature that microplastics are covered by biofilms, where diverse bacterial communities colonized the surface of microplastics, yet the influences of biofilms on the fate of microplastics remain unclear (Curren and Leong, 2019; Michels et al., 2018).

6. Analytical methods of microplastics in sediment

Current sampling and separation methods used for analyzing microplastics in sediments vary across studies. Van Cauwenberghe et al. (2015b) summarized sampling and separation methods employed in studies prior to 2015, where they noted inconsistencies from three different aspects: size range, recovery rate and quantification measures (e.g., varied units of reported microplastics concentration). Here, we summarized the progress of analytical methods after 2015.

Separating microplastics from sediments with low recovery will lead to underestimations of microplastic concentrations (Erni-Cassola et al., 2017). Density separation is the most common methods to extract microplastics in sediments (Table 5). Zhou et al. (2016) improved the density separation method by including a continuous flow-air floatation separation device comprising liquid storage, air floating overflow, and filtration, which achieved a recovery rate of 97% for the size of 0.2–5 mm. The device was convenient to operate and can handle a large volume of sediment samples. Hurley et al. (2018) included an additional step of density separation, using filtered reverse osmosis (RO) water (ρ = 1 g/cm^3) for low-density microplastics and NaI solution (ρ = 1.8 g/cm^3) for high-density microplastics, and they recommended that each separation step should be performed twice to ensure a recovery. They reported high rates of recovery for large polyethylene microbeads (850–1000 μm), small polyethylene microbeads

Table 5
Extraction methods of microplastics.

Pretreatment	Size	Separation method		References
		Reagents	Digestion	
65 °C dried and 1 mm sieve	0.5–5 mm	NaCl 1.2 g/cm^3	65% HNO_3	Scheurer and Bigalke (2018)
70 °C dried	Mainly < 2 mm	$Na_6H_2W_{12}O_{40}$ 1.5 g/cm^3	–	Ballent et al. (2016)
50 °C dried	1.33 ± 1.69 mm	$ZnCl_2$ 1.75 g/cm^3	–	Chen et al. (2018)
60 °C dried	0.05–5 mm	NaI 1.8 g/cm^3	–	Wang et al. (2019b)
70 °C dried	Mainly < 0.5 mm	NaI 1.6 g/cm^3	30% H_2O_2	Zhu et al. (2018b)
sieved with 1 mm mesh size sieves	0.3–5 mm	HCO_2K 1.5 g/cm^3	–	Zhang et al. (2017)
80 °C and sieved with 1 mm stainless steel metal	0.04–2 mm	demineralized water	–	Phuong et al. (2018)

"-": not documented.

(425–500 μm) and polyethylene terephthalate fibers (PET, 322–395 μm), which were nearly 100%, 92%–98% and 79%–86%, respectively. Density separation does not effectively recover small microplastic particles. For example, the recovery of nanoplastics beads (0.05 μm) and small microbeads (2.6 μm and 4.8 μm) ranged from 5 to 16% with using $ZnCl_2$ solution ($\rho = 1.6\ g/cm^3$) in samples of biosolids and soils (Wang et al., 2018d).

New analytical approaches for separating microplastics from sediments began to emerge. For example, the electrostatic separation method takes advantage of differences in electrostatic behaviors between microplastics and sediment matrix (Felsing et al., 2018). Samples were charged at a high voltage, and microplastics were separated out via passing through a rotating metal drum as the least conductive material. The recovery rate was more than 99% with sizes ranging from 63 μm to 5 mm (Felsing et al., 2018). Grbic et al. (2019) used magnets for the separation of microplastics. These authors thoroughly mixed 100 g sediment with 200 mL RO water to create a homogeneous mixture and then added approximately 2 mg of modified Fe nanoparticles and microplastics plastics (200 μm-1mm). The mixture was thoroughly mixed again before a N52-grade neodymium magnet was immersed in the sample. The mean recovery rate of polypropylene, polyvinyl chloride, polyurethane, polystyrene and polyethylene were 49% ± 12%, 85% ± 27%, 79% ± 21%, 87% ± 26% and 90% ± 47%, respectively, and the lower recovery was attributed to possible impeding effects of soil particles on the interactions between nanoplastics and microplastics (Grbic et al., 2019).

Digestion can further improve the separation of microplastics from sediments by removing natural organic matter and biofilms, which interfere with the identification of microplastic components (Loder et al., 2017). The digestion reagents included acids (Zobkov and Esiukova, 2017), alkalis (Dehaut et al., 2016), oxidants (Bergmann et al., 2017), and enzymes (Loder et al., 2017). The temperature of digestion should be less than 60 °C due to the loss of microplastics at higher temperatures (Munno et al. (2018). Hurley et al. (2018) compared the regents for removing interfering organics, including 30% (v/v) H_2O_2 (60 °C and 70 °C), Fenton's reagent, NaOH (1 M and 10 M at 60 °C), and KOH (10%, 60 °C), and they found that Fenton's reagent was the most efficient in reducing natural organic components while preserving microplastics and NaOH solution was unsuitable for this purpose. Fenton's reagent consisted of 30% (v/v) H_2O_2 and an iron catalyst that was a mixture of 20 g iron(II) sulfate heptahydrate and 1 L filtered RO water, and it was recommended to use below 40 °C t at pH 3 for 2 h to digest soil and sludge samples.

Another separation method that recently appeared is the organic solvent separation method. This method extracts microplastics based on their compatibility with organic solvents. Fuller and Gautam (2016) used methanol and dichloromethane as solvents to extract microplastics using the pressurized fluid extraction method, yielding a recovery rate 80%-120%. Moreover, this method can not only extract plastics but also remove other organic matter (e.g., biofilms) and thus is convenient using infrared spectroscopy or other method to identify microplastics. However, organic solvents can change the morphological characteristics of microplastics that hinders accurate source identifications.

A variety of methods have been used to identify the components of microplastics, and the most commonly used ones are Raman Spectroscopy (Di and Wang, 2018; Zhang et al., 2016; Zhao et al., 2017) (Fig. 2a), Fourier Transform Infrared Spectroscopy (FTIR) (Fuller and Gautam, 2016; Lourenco et al., 2017) (Fig. 2b) and Thermal Desorption Pyrolysis Gas Chromatography/Mass Spectrometry (TD-Pyr -GC/MS) (Dekiff et al., 2014). The minimum particle size of microplastics that can be identified by these three methods is 1 μm (Imhof et al., 2013), 20 μm (Mintenig et al., 2017), and 100 μm (Dekiff et al., 2014), respectively. The identification of microplastics in environmental samples is usually achieved by comparing the sample to a reference standard material. However, microplastics in environmental samples have been modified by mechanical crushing and chemical and biological

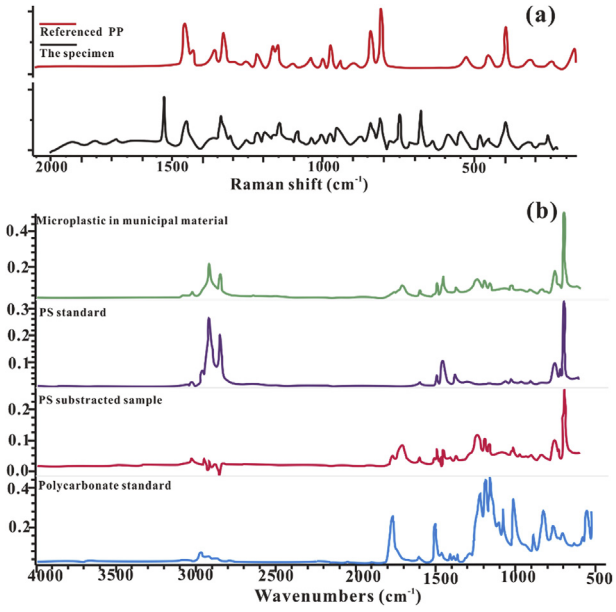

Fig. 2. (a) Raman spectra of microplastics from marine snow (Zhao et al., 2017); (b) Infrared spectra of microplastics from a composted waste (e.g., municipal waste samples) (Fuller and Gautam, 2016).

weathering to various degrees and additives (e.g., pigments) are often added (Jahnke et al., 2017; Zhao et al., 2017; Zhou et al., 2018b). Therefore, the sample is usually considered as the same with the reference material when the spectral similarity is more than 80% (Lin et al., 2018).

Improvements in the analytical methods of microplastics in sediments are needed to enhance the accuracy and across-study comparisons. For the separation of microplastics from sediments, it is important to establish standard procedures with high recovery rates and inexpensive, environmentally safe chemicals.

7. Future research

Current research on microplastics mainly focuses on determining the spatial distributions of microplastics in sediments and their biological effects. Small-sized microplastics, e.g., nanoplastics remain poorly understood due to the limitations of existing analytical methods. Developing an effective extraction and identification method standardized across laboratories is a must to accurately determine the pollution and ecological risks of microplastics. We recommend the combination of density separation, electrostatic separation and organic solvent separation to improve the separation efficiency of microplastics and to standardize the analytic process for sediment microplastics in future studies. In addition, understanding the source, sink, migration, and accumulation of microplastics in sediments will lay the foundation for the environmental regulatory agencies to establish polices to manage and reduce microplastic pollution.

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