



A critical overview of the analytical approaches to the occurrence, the fate and the behavior of microplastics in the environment



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ABSTRACT

Plastics can be found in food packaging, shopping bags, and household items, such as toothbrushes and pens, and facial cleansers. Due to the high disposability and low recovery of discharged materials, plastics materials have become debris accumulating in the environment. Microplastics have a dimension <5 mm and possess physico-chemical properties (e.g., size, density, color and chemical composition) that are key contributors to their bioavailability to organisms. This review addresses the analytical approaches to characterization and quantification of microplastics in the environment and discusses recent studies on their occurrence, fate, and behavior. This critical overview includes a general assessment of sampling and sample handling, and compares methods for morphological and physical classification, and methodologies for chemical characterization and quantification of the microplastics. Finally, this review addresses the advantages and the disadvantages of these techniques, and comments on future applications and potential research interest within this field.

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1. Introduction

The global annual production of plastics is around 280 million tons, the vast majority being for disposable use [1,2]. Due to the high disposability, low recovery of such waste materials, high durability, accidental release, and indiscriminate discards, plastics have become debris accumulating at an uncontrolled rate in the environment, where they are subjected to erosion by wind and rain, and to river-driven transport, mostly reaching coastal areas [1,3]. Generally, these items accumulate as debris in the environment, including the seas and regulated landfill sites [1]. The polymers most widely

produced as plastics and encountered as plastics debris are polypropylene, polyethylene, polystyrene, polyethylene terephthalate, and polyvinylchloride [3,4].

Research on environmental impacts of plastics debris has gained a new dimension due to the study of microplastics and their impacts on terrestrial ecosystems and marine environment [1,5]. The National Oceanic and Atmospheric Administration (NOAA) defined microplastics as particles less than 5 mm in size [3]. Since a positive relationship between microplastics abundance and human population density has been established, the increase in human population would probably lead to an increase in the prevalence of microplastics in the environment [6,7]. In order to understand the potential impacts of microplastics in the environment, their identification and characterization have been attempted in relation to seawater, sandy and muddy sediments, plankton samples,

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marine-sewage disposal, sewage effluent, washing-machine effluent, facial cleansers, and vertebrate and invertebrate ingestion [8–10]. The physico-chemical properties of microplastics, such as size, shape, density, color and chemical composition, greatly affect their transport in the environment and their bioavailability [3]. The toxicological effects on marine organisms are also influenced by the physico-chemical characteristics of microplastics [3].

Taking this into consideration, the complete characterization of microplastics in terms of abundance, distribution and chemical composition is of paramount importance in order to characterize their environmental impacts and to enable large-scale spatial and temporal comparisons. This critical overview therefore covers the different analytical approaches used for characterization and quantification of microplastics in the environment and discusses the most recent studies on their occurrence, fate and behavior.

2. Sampling and sample handling

Microplastics can directly enter the environment, whereas large items are continuously fragmenting until they attain micrometric dimensions in size [8]. They can reach the soil and aquatic systems via the effluents of wastewater-treatment plants but they can also be resuspended by wind from landfills and become airborne until the atmospheric conditions allow entry into the ecosystems by deposition. There is also a pattern of accumulation along the shorelines, mostly due to port activities and discharges from river mouths [9]. Based on research on the Web of Knowledge, microplastics have been studied more intensively in the marine environment and on shorelines. Concerning the inland environment, there are no studies in soils or atmospheric aerosols. Sewage effluents, washing-machine effluents and facial cleansers have been studied as examples of microplastics spreading into the environment [9,10].

Sampling microplastics in the water column can be achieved by:

- (1) using a trawl along a transect, such as manta net and Neuston net for surface waters [11]
- (2) using bongo nets for mid-water levels [5];
- (3) benthic trawls for seabed [5]; and,
- (4) vessels for surface and mid-water levels [12,13].

Typically, 333–335- μm mesh apertures are the most used for the net [11,14] and, when a different size aperture is used, it can produce large variations in the quantity of microplastics collected [5].

Sampling sediments can allow the benthic material from beaches, estuaries and seafloor to be assessed for the presence of microplastics [5,15]. Using stainless-steel spoons or spatula [16,17] and box-corer [18] usually collects superficial sediments or sand from the beaches and estuaries, while seabed sediments are collected using core [19] and bottom trawl [20]. There is a general lack of specific sampling protocols for collection of microplastics in sediments and water.

Biological sampling involves examining microplastics in marine organisms through dissection of marine animals, and investigation of regurgitation in seabirds and analysis for the presence of microplastics in their gut contents [5].

Separation of microplastics from samples has been done by density flotation, filtration and sieving [9,10,12,13,15–29], as shown in Table 1. The specific density of plastics particles varies considerably, since it depends on the type of polymer and the manufacturing process, in the range 0.8–1.4 g cm^{-3} [29]. These values do not take into account the effect of adding several additives that might be incorporated into the production process, so they refer only to the virgin resin, biofouling and weathering [29].

Since sand or sediments have densities around 2.65 g cm^{-3} , the difference in density can be used to separate the lighter microplastics from the heavier sand or sediments. A salt-saturated solution (usually

Table 1
Methods for the separation of microplastics [9,10,12,13,15–29]

Method	Mechanism	Size range	Complementary technique
Filtration	Size fractionation	Down to 2 μm	Optical microscopy
Density flotation	Salt addition (usually NaCl or NaI) and floatation	^a	
Sieving	Size-exclusion	0.038–4.75 μm	

^a Not applied.

NaCl or NaI) is added to sand or sediments and mixed by shaking or using a vortex. After mixing, the sediment will settle to the bottom, while the microplastics will remain in suspension or float to the surface of the solution. The supernatant is then extracted for further processing of the microplastics particles.

The Marine Strategy Framework Directive (MSFD) Technical Subgroup on Marine Litter recommended the use of NaCl for the separation of microplastics by density flotation [23,30], since it is an inexpensive, eco-friendly salt. However, the use of saturated solution of NaCl (1.2 g cm^{-3}) or tap water may lead to underestimation of the microplastics content in sediments because the solution density is too low to enable the flotation of all polymers, principally those containing additives [23]. Instead, an NaI-saturated solution density (1.6 g cm^{-3}) is enough to separate the polymers containing additives, so it is preferable to use NaI.

Cauwenberghe et al. [19], in an attempt to investigate the presence of microplastics in deep sea, collected sediments in a surface area of 25 m^2 . After recovery, cores were cut into horizontal slices by extruding them and slicing the sediment with a metal plate. The cores were cut into 1-cm-thick slices and the top centimeter of the sediment cores was wet sieved, first on a 1-mm mesh sieve and subsequently on a 35- μm mesh sieve. The fraction remaining on the 35- μm mesh sieve was used for further separation based on density flotation by adding a solution of NaI. The solids were then transferred to a centrifuge tube followed by shaking and centrifugation. After centrifugation the top layer containing the microplastics was vacuum filtered. As in this example, the microplastics are usually separated from the supernatant, resulting from the density flotation by filtration, either vacuum assisted or under normal pressure.

Microplastics can also be optically identified in filter cakes using techniques, such as binocular microscopy [24,25]. Finally, particles that are optically identified as microplastics can be separated and singled out using tweezers.

Microplastics can also be separated from samples using sieves of several mesh sizes, which allow classification into different size categories. The sieves used are usually made of stainless steel [12] and copper [13]. Desforges et al. [13], aboard a research cruise vessel, collected seawater at 4.5 m below the surface using the seawater-intake system of the vessel. A flow meter measured the volume of water pumped for 10–20 min from the seawater intake and the readings were converted to cubic meters of water filtered. Water was first filtered through a 5-mm filter to remove large debris and organisms before entering the intake system and then running through a series of copper sieves of diminishing pore size: 250 μm , 125 μm and 62.5 μm . The material on each sieve was rinsed with seawater into glass vials and stored.

In order to prevent the contamination of samples with airborne microplastics during the extraction process, Cauwenberghe et al. [19] used materials previously rinsed with filtered deionized water and in a clean fume hood. Materials, such as containers, must be rinsed onto the filter or sieve, since microplastics may stick to the walls, so losing a proportion of the analyte. During sample collection, cotton clothing should be worn rather than synthetic fabrics to avoid contamination by plastics fibers [9]. Quality-control samples must be used to check whether there has been sample

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