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Editorial: Analytical methodologies for the analysis and monitoring of nano/microplastics pollution

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Editorial on the Research Topic
Analytical methodologies for the analysis and monitoring of nano/microplastics pollution

The evaluation of the environmental impact of nano- and microplastics is one of the biggest environmental challenges nowadays. Indeed, the massive consumption of plastics is leading to the occurrence of micronic and submicronic plastic particles in almost all environmental compartments (water, air, soils, food, interfaces…). From an environmental risk assessment point of view, the development of new analytical strategies able to detect, identify and quantify nano- and microplastics at low concentration in a wide range of environmental matrices is required. However, the analysis and monitoring of nano/microplastics pollution is specially challenging due to several reasons. First of all, dedicated and complex sample preparation procedures must be developed, since the use of analytical techniques commonly used for the analysis of inorganic nanoparticles is not straightforward. Secondly, there exist large Research Topic between plastic particles on the physical-chemical properties that regulate the particles fate, e.g., density, porosity, composition. Attachment of microorganisms and biofilm growth on plastic surfaces further complicate the environmental fate and reactivity of plastic particles, necessitating complex sample treatment prior to analysis. Therefore, upgrading existing or developing new methods and analytical strategies for quantifying the numbers and physical-chemical properties of nano- and microplastics in the environment is essential.

In this context, the current Research Topic "*Analytical methodologies for the nalysis and monitoring of nano/microplastic pollution*" was focused on providing a global overview of the most recent analytical strategies developed to fill the gap of the analysis of nano/microplastics in different environmental compartments. The Research Topic includes 3 Original Research and one Methods article, which are summarized below:

In the first Original Research article, Goedecke et al. investigated the occurrence and the mass fractions of microplastics in a municipal wastewater treatment plant (WWTP) effluent for several days in winter and summer. For this purpose, authors applied a fractionated filtration of the effluent by using three different mesh sizes (500, 100, and 50 µm). This approach allowed to obtain representative sample volumes of 1 m^3 of various types of waste water. For detection purposes, the microplastics mass fractions of the dried suspended particulate matter were determined by thermal extraction desorption–gas chromatography/mass spectrometry (TED-GC/MS). For the first time, TED-GC/MS was used for the determination of microplastics mass fraction without the previously required additional sample pretreatment. In addition, the identification of microplastics was done by screening for specific markers in the GC/MS data. Polyethylene (PE), polystyrene (PS), and polypropylene (PP) were identified in effluent samples with polymer masses varying significantly between 5 and 50 mg m^{-3} depending on the sampling day, the season and the size class. For instance, PE, PS, and PP were detected on summer days, whereas on winter days only PE and PS were detected. This method can be applied in routine for a fast determination the microplastics release into the environment by WWTP.

The second Original Research article described a new method for microplastics ingested by copepods with a new size limit of detection of 1 µm, developed by Thery et al. Authors used epifluorescence microscopy and Raman microspectroscopy to identify small microplastics (<10 µm) in the copepod *Eurytemora affinis* collected in the Seine estuary (France). Firstly, epifluorescence microscopy was sued to confirm the ingestion of three type of microplastics (Polylactic acid (PAL), PS and, PP) labelled with Nile Red. Afterward, the authors tried to pair this method with Raman microspectroscopy. To do that, the first critical point was to develop an digestion protocol able to extract the intact polymers while degrading the organisms. For this purpose, they developed an enzymatic digestion based on the use of proteinase K coupled with ultra-sonication. Since the use of black polycarbonate (PC) membrane filters induced a significant background fluorescence during Raman identification of microplastics dyed with Nile Red, the authors proposed an alternative approach. Using aluminum oxide filters replacing the staining method with a classical observation with stereomicroscopic magnifier allowed reaching a size limit for microplastics detection in copepods down to 1 µm. Finally, the developed method was applied to copepods collected in the natural environment, where an average of 0.28 microplastics per copepod were identified.

The second Original Research article Gondikas et al. explored sampling, sample treatment, and analytical techniques for measuring boat paint nano- and microplastics in seawater, within marinas on the Swedish coast. Taking into account that particle numbers are expected to increase exponentially with decreasing particle size, the authors used a combination of sampling techniques to cover a wide range of sizes. Manta trawling was used for particles >300 µm, *in situ* vacuum filtration was used for capturing particles >10 µm on a filter membrane, while the filtrate was also collected to account for particles <10 µm. Automated microscopy procedures were used to avoid operator bias. Light microscopy with criteria on shape and color produced a fast screening and categorization of the larger size fraction. Automated light and scanning electron microscopy procedures were applied to the medium size fraction (10–300 µm), while single particle ICPMS was used to measure the finer size fraction (<10 µm). A comparison of the various identification criteria used in combination with the analytical methods is also presented. When color is used to identify boat paint microplastics, an increasing trend of particle number to number of boats is observed, however, the analysis is limited to colors standing out from natural colors (e.g., bright red and blue). Using metal content as the identification criterion, narrows the analysis focus to antifouling paint particles, which pose a higher environmental risk. However, additional criteria need to be applied to eliminate other anthropogenic or natural sources. The study concludes that a correlative microscopy approach incorporating both visual appearance and metal content offers a promising way forward.

Finally, in the Methods article a new microplastic extraction strategy is described by Li et al. The authors present a novel application able to i) efficiently break down the stable organic tissue of banana prawns (Penaeus merguiensis), and ii) isolate microplastics from the digestive tract without altering their properties. Five treatments (acid, alkaline, oxidant, enzyme, and microwave-assisted oxidant digestion) and seven reference polymers (polyamide (PA), PE, polyester (PES), PP, PS, polyvinyl chloride (PVC), and rayon) were tested. Microwave-assisted oxidant digestion showed the best microplastic extraction and quantification results. In addition, the method achieved a high digestion efficiency (>90% recovery rate of spiked microplastics) and satisfactory visual assessment, with minimal size, shape, and Fourier transform infrared (FTIR) spectral changes for all polymers except rayon. Finally, it was successfully applied to disintegrate the lining of P. merguiensis digestive tracts.

Author contributions

All authors listed have made a substantial, direct, and intellectual contribution to the work and approved it for publication.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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