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Comparison of Microplastic Detection Methods in Wastewater Treatment Plants †

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Abstract: A plastic can be turned into millions of fragments of microplastic particles by anthropogenic activities and environmental events (such as wind, UV light, and the water wave action). Due to their surface hydrophobicity, absorbance of persistent organic pollutants, potential to transport contaminants and persistent properties, microplastics have the potential to become widely dispersed in the water environment via hydrodynamic processes and water currents. Plastic materials are durable and, rather than decomposing, they break down into small plastic particles over time. These small particles of less than 5 mm are usually defined as microplastics. As a consequence of the large plastic production rates, plastic waste accumulation in the natural environment has rapidly increased worldwide. However, the effects of plastic wastes in different ecosystems are still largely unknown. Water and wastewater treatment plants are important facilities to estimate plastic waste release to or retention amounts in the environment. Sampling, analysis and standardization of measurements in microplastic analysis is still an ongoing issue. Since wastewater has a mixed matrix, very few microplastic measurements have been made so far. Furthermore, the lack of a standard and viable method to identify microplastics has limited the correct assessment of microplastics and may lead to an incorrect estimation. In this study, microplastic sampling techniques, extraction methods and identification methods of microplastics in wastewater were compared. It was concluded that studies were mostly performed with grap-type sampling, wet peroxite oxidation and identification methods with a microscope and Fourier Transform Infrared Spectrophotometer (FTIR). In the FTIR analysis to determine the polymer structure of microplastics, the most common type of polymers were found to be polypropylene (PP) and polyethylene (PE).

Keywords: microplastics; wastewater; FT-IR; quality assurance/quality control (QA/QC); polypropylene (PP); polyethylene (PE)

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1. Introduction

Intensive production of plastics began in the 1950s. Plastic production has exceeded 348 million tons to date and is expected to double by 2035 [1,2]. Since the full mineralization of the polymers in the plastics will take hundreds of years, plastics cause serious pollution due to their cumulative and permanent properties. [3]. Plastics are produced with various combinations of more than 5000 various polymers and other chemicals. [1]. Generally, MPs are divided into categories of either primary or secondary MPs. Primary MPs are manufactured as such and are used either as resin pellets to produce larger items, directly in cosmetic products such as facial scrubs and toothpastes, or in abrasive blasting (e.g., to remove lacquers). Compared to this deliberate use, secondary MPs are formed from the disintegration of larger plastic debris [4]. Primary microplastics mostly originate from drugs, personal care products, and pellets used for the production of plastic consumer products [5,6]. These microplastics are transported through the sewage system to wastewater treatment plants and then to the receiving water environments [7] and run-off or mismanagement from the industries producing or storing the pellets, which are used in various products, including personal care products [8]. Wastewater treatment plants (WWTPs) are considered to be the main recipients of terrestrial microplastics before they enter natural aquatic systems [9], which convert primary microplastics into secondary microplastics. Daily human activities are the main source of microplastics found in domestic wastewater. This paper thus focuses on reviewing the sampling, separation and recognition methods used for microplastics' analysis in wastewater treatment plants. The aims' are to: (1) compare the methodologies used for the analysis of MPs in wastewater; (2) identify the research gaps and limitations of current techniques; (3) develop a classification system to estimate the information provided by the current study and by future studies.

2. Materials and Methods

2.1. Sampling and Processing Methods

The studies included in this review identified four types of sample collection technique: grab samples, composite samples, extraction pumps, and Neuston nets. The advantages and disadvantages of these methods are summarized in Table 1.

Table 1. Comparison of MPs Sampling Techniques [10].

Processing Method	Advantages	Disadvantages
NOAA Method	Organic matter is dissolved, resulting in clean MPs Mps used by several studies	Might need more than one digestion step, increasing the time required Different solutions were used to facilitate separation based on density through flotation; $ZnCl_2$ and NaI had higher extraction efficiency than NaCl, but both are more expensive than NaCI
Simple Filtration	Easy, time-saving, and low cost	Difficulties in separating plastic particles from other organic or nonorganic particles.
Centrifugation	Easy and simple to use	Fractioning and deformation of plastic particles, resulting in misrepresentations of quantity, shape, and size
Staining Method	Easy and low cost	False affirmation of some MP polymers

After sampling, MPs were purified from other particles, such as organic and inorganic colloids. Table 2 presents a summary of the studies, as well as the advantages and limitations of each of the processing techniques described below.

Table 2. Comparison of the Sample Processing Techniques [10].

Processing Method	Advantages	Disadvantages
NOAA Method	Organic matter is dissolved, resulting in clean MPs Mps used by several studies	Might need more than one digestion step, increasing the time required Different solutions were used to facilitate separation based on density through flotation; $ZnCl_2$ and NaI had higher extraction efficiency than NaCl, but both are more expensive than NaCI

Table 2. *Cont.*

Processing Method	Advantages	Disadvantages
Simple Filtration	Easy, time-saving, and low cost	Difficulties in separating plastic particles from other organic or nonorganic particles.
Centrifugation	Easy and simple to use	Fractioning and deformation of plastic particles, resulting in misrepresentation quantity, shape, and size
Staining Method	Easy and low cost	False affirmation of some MP polymers

2.2. Polymer Characterization

After processing, MPs identification should be done. MPs can have various colors, shapes, sizes, and composition; consequently, several categorization methods can be applied. The most reported identification methods were visual inspection using an optical microscope, Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy, and scanning electron microscopy (SEM). The advantages and disadvantages of each of the identification methods are summarizes in Table 3.

Table 3. Advantages and Limitations of the Methods Used for Identifying MPs [10].

Techniques	Advantages	Disadvantages
Microscope	Fast and easy Identifies shape, size, and colors	Lack of information on the plastic Composition; not confirmative to plastic nature of the particle
FT-IR	Identifies the composition of the polymer Confirmation of the composition of the MP Able to detect small plastic particles (~20 μm)	Expensive Tedious work and time-consuming to analyze all of the particles retained on the filter Wavelength radiation can be a limiting detection factor
Raman	Identification of the composition of the polymer; confirmation of the composition of the MP; detection of small microplastics (1 μm) and nanoplastic (<1 μm)	Expensive instrumentation Time-consuming Interference with pigments and contaminants
SEM/EDS	Clear and high-resolution images of particles Facilitates differentiation between plastic and mineral particles due to the dominant inorganic elements (Si, Ca)	Nonaffirmative results in plastic particles; Lack of information on the type of polymer

3. Results

Plastic and microplastic pollution, with respect to their size, their invisibility to the naked eye and their durability in the environment has attracted attention in the last decade. The structural properties of plastics is very convenient for manufacturers; their production continues in significant amounts, and they are widely used in personal care products. Their direct effects on organisms in aquatic environments via feeding and their indirect effect of releasing the additives that are toxic to organisms and present at different levels in their structure are widely studied in the literature. Their potential to carry hydrophobic chemicals and antibiotics by sorption and desorption, as well as their potential to carry microorganisms over long distances, were evaluated. The microorganism layer attached to the microplastic particles and the transport of chemicals and antibiotics with this layer have also recently been mentioned recently. Therefore, microplastics are an important pollutant for investigation in the environment. Wastewater treatment plants are major potential receivers of primary microplastics such as beads in personal care products, fibers from washing clothes in high amounts and secondary microplastics from combined sewage systems due to stormwater runoffs. Therefore, they must be investigated to prevent any damage to the environment. Wastewater treatment plants were generally reported to be successful for the removal of the microplastics. However, they still discharge a considerable amount of microplastics into the environment and are considered an important point source. Regarding the structure of microplastics and the contents of wastewaters such as organic material, various microorganisms, potential chemicals from the fugitive discharge of industrial plants and from chemicals used in cleaning and antibiotics, used by people; questions arise regarding the potential hazards. Therefore, the assessment of microplastics in wastewater streams is crucial both for their direct hazards and indirect hazards to the organisms that they interact with. Studies considering the determination of microplastics in wastewater treatment plants differentiate in terms of data and sample collection and processing, report the MP concentrations in different units and use different compositional segmentations due to the different methods of analyzing. The variability in influent loads, temporal conditions and plant operational conditions make the assessments of microplastics difficult. The reviewed studies showed that microplastics in smaller sizes are most likely to be released into environments and detection of these microplastics would lead to a more realistic approach to the microplastics problem, with wastewater treatment plants being a point source. The removal of MP may vary depending upon the various units and operations used in wastewater treatment plants.

As a result of the study, it was observed that there was no consistency between studies in terms of sampling, sample processing, characterization, identification techniques, quality assurance procedures and reported results of MPs. Studies on these topics, standardized procedures for all steps in the evaluation of MPs, will increase the accuracy of results, reduce the effort and time required, and help make meaningful interpretations and comparisons between studies.

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