Paper Presentation

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Assessing the Mass Concentration of Microplastics and Nanoplastics in Wastewater Treatment Plants by Pyrolysis Gas Chromatography−Mass Spectrometry

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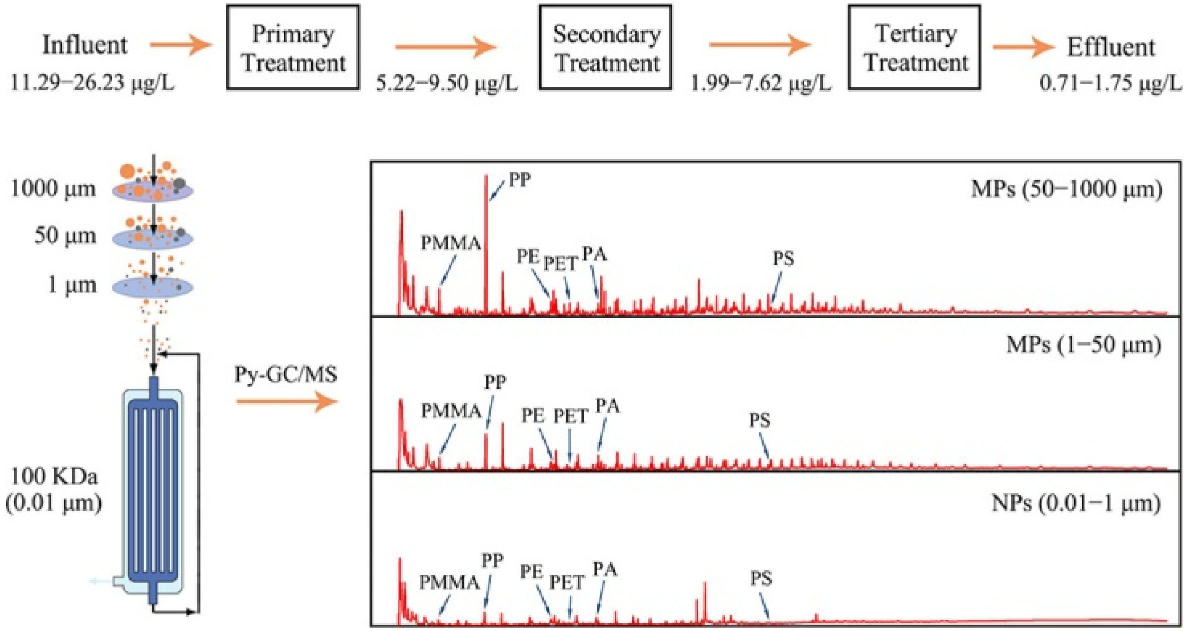

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AIM OF STUDY

The objective of this study is to investigate the **mass concentration** of microplastics(MPs) and nanoplastics(NPs) in Wastewater Treatment Plants (WWTPs) by Pyrolysis-GC/MS. An ultrafiltration-based method was further developed to concentrate and detect trace NPs in WWTPs.

BACKGROUND

RESEARCH GROUP

ELSEVIER

Water Research

Volume 142, 1 October 2018, Pages 1-9

Quantification of microplastic mass and removal rates at wastewater treatment plants applying Focal Plane Array (FPA)-based Fourier Transform Infrared (FT-IR) imaging

Márta Simon, Nikki van Alst, Jes Vollertsen

frontiers in Environmental Chemistry

ORIGINAL RESEARCH
published: 22 March 2022
doi: 10.3389/fenvc.2022.844633

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Development of a Routine Screening Method for the Microplastic Mass Content in a Wastewater Treatment Plant Effluent

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ELSEVIER

Water Research

Volume 152, 1 April 2019, Pages 21-37

Review

Microplastics in wastewater treatment plants: Detection, occurrence and removal

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INTRODUCTION

1. **Microplastics:** Microplastics are fragments of plastic that are less than 5 mm in length.

2. **Nanoplastics:** Nanoplastics are solid plastic particles that are between 1 nanometer and 1 micrometer in size.

3. **Pyrolysis Gas Chromatography- Mass Spectrometry (GC-MS):** It is a chemical analysis method that breaks down samples into smaller fragments through controlled thermal degradation. The sample is heated to high temperatures, which decomposes it into smaller molecules. These molecules are then separated by gas chromatography and identified by mass spectrometry

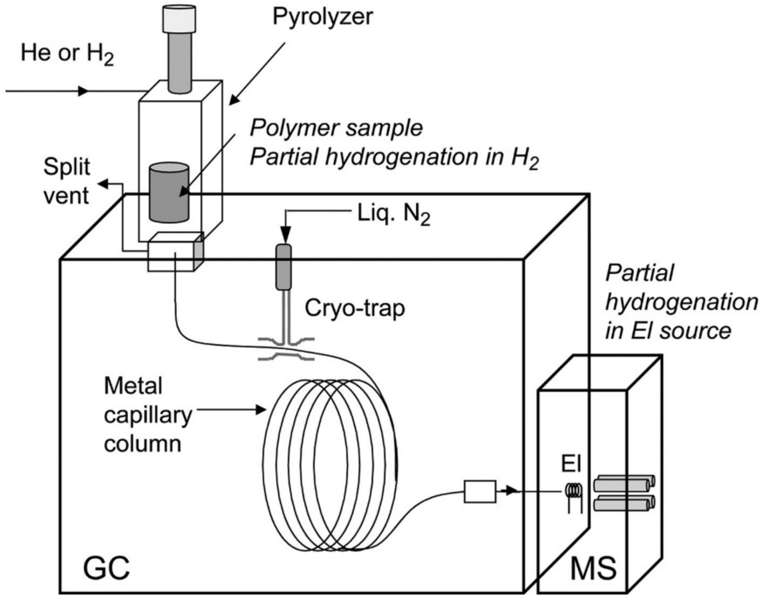


Fig: Schematic showing pyrolysis Gas Chromatography- Mass Spectrometry

Watanabe, A., Watanabe, C., Freeman, R. R., Teramae, N. & Ohtani, H. Hydrogenation Reactions during Pyrolysis-Gas Chromatography/Mass Spectrometry Analysis of Polymer Samples Using Hydrogen Carrier Gas. *Anal. Chem.* **88**, 5462–5468 (2016).

SAMPLE PREPARATION

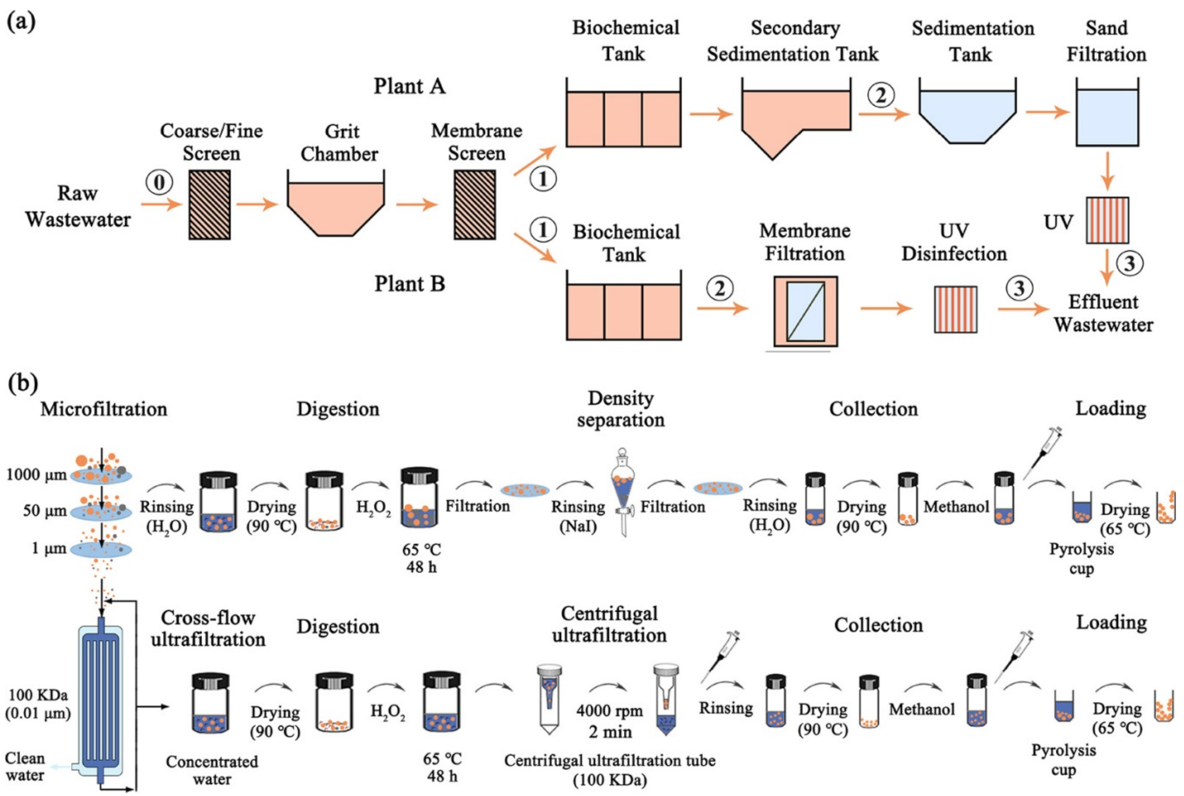


Fig: (a) Flowchart of the treatment processes and sampling sites in two WWTPs
(b) And pretreatment procedures of wastewater samples for MP and NP.
0, 1, 2, 3 means raw wastewater, water treated after the primary, secondary, and tertiary treatment

METHOD

- Six polymer types including polymethyl methacrylate (PMMA), polypropylene (PP), polystyrene (PS), polyethylene (PE), polyethylene terephthalate (PET), and polyamide (PA) that are widely found in WWTPs were selected.
- The indicator ions for these polymers were selected and their selectivity was tested by analyzing selected organic compounds.

  - PMMA → Methyl methacrylate (m/z =100)
  - PP → 2, 4-Dimethyl-1- heptene (m/z =126)
  - PS → 5-hexene-1, 3, 5-triyltribenzene (m/z =312)
  - PE → 1,12-tridecadiene (m/z =180)
  - PET → vinyl benzoate (m/z =148)
  - PA → ε-caprolactam (m/z =113)

- Extraction and recovery efficiency of MPs and NPs were tested by the formula:

$$\text{recovery (\%)} = \frac{C_2 - C_0}{C_1} \times 100$$

where C_0 (μg/L) is mass concentration of MPs in the control samples without spiked and C_2 (μg/L) is mass concentration of MPs detected in samples spiked with a known concentration C_1 (μg/L).
The MPs and NPs showed a recovery percentage of 60-70% which is matching with the previously reported data.

RESULTS AND DISCUSSIONS

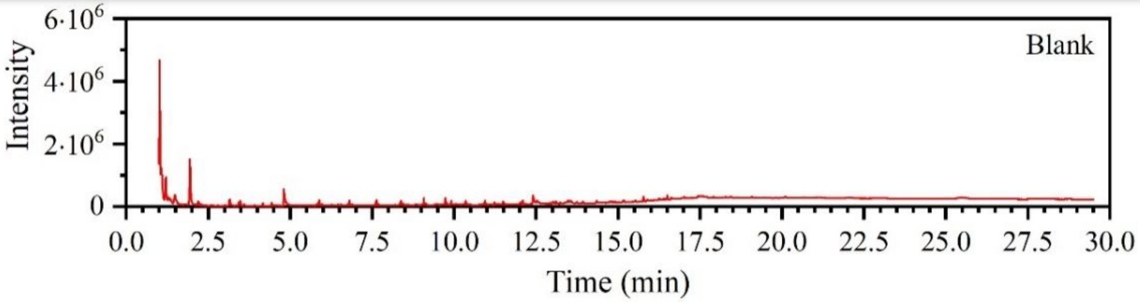


Fig: Chromatograms from blank samples prepared using the same steps as sample treatment

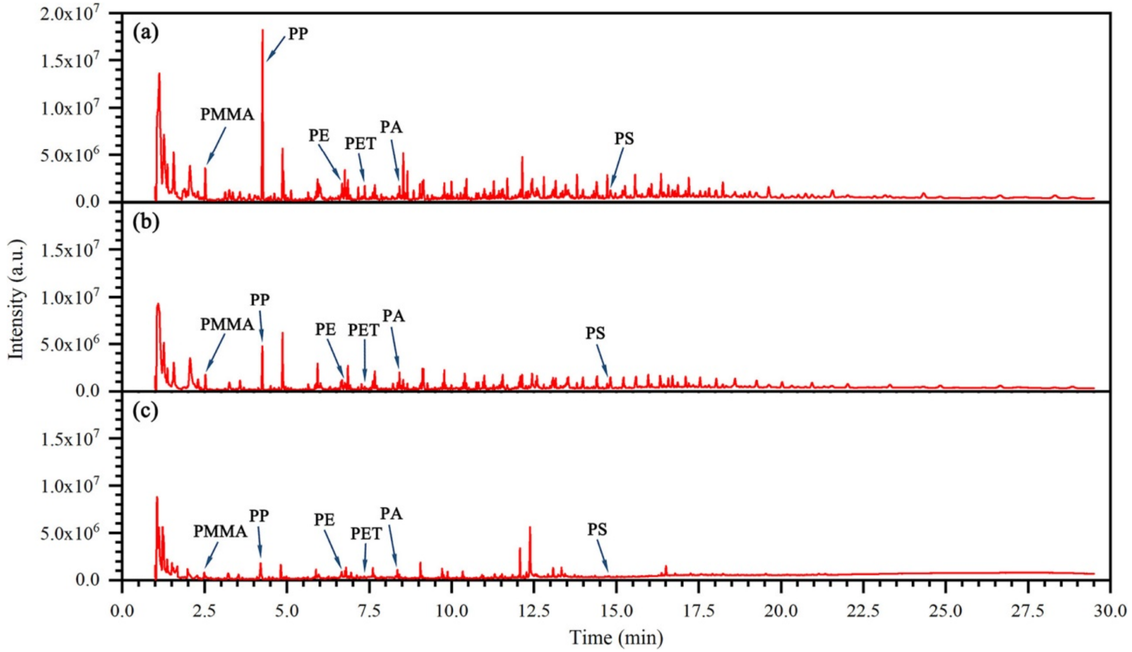


Fig: Chromatograms from representative samples of MPs with the size ranges of 50−1000 μm (a), 1−50 μm (b), and 0.01−1 μm (c).

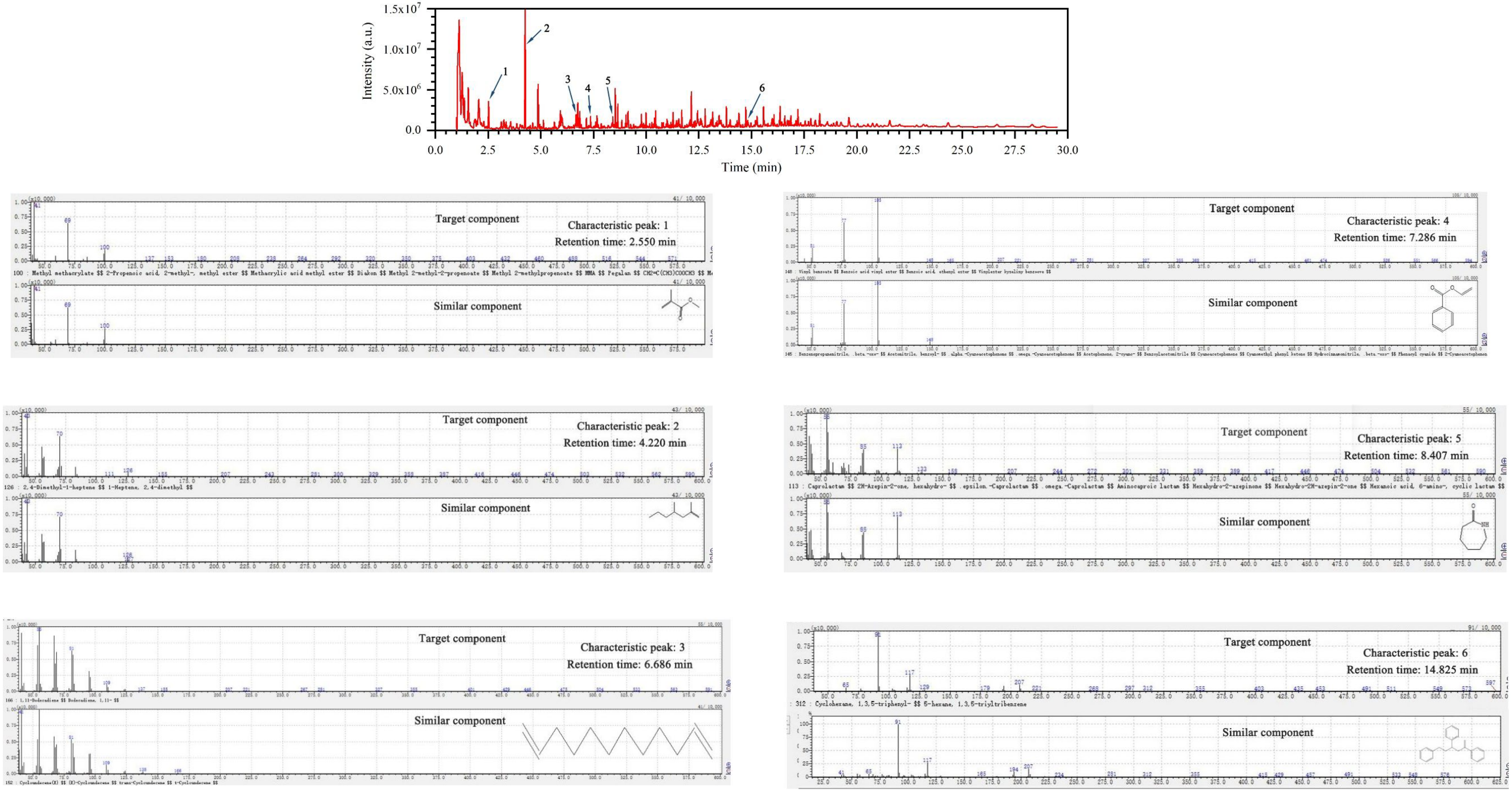


Fig: Results of similarity analysis the characteristic peaks of a representative sample

External calibration curves were obtained by analyzing different amounts of the standard plastics (0.1−10 μg for PMMA, PA, and PS and 0.1−200 μg for PP, PE, and PET).

Table S4. Characteristic components and calibration functions of six plastics.

Plastic type	Characteristic components	Linear range	Calibration functions	Linearity (R^2)	RSD (%)
PMMA	Methyl methacrylate	0.1−10 μg	y = 413336x - 42490	0.99	6.9−15.2
PP	2, 4-Dimethyl-1-heptene	0.1−10 μg	y = 112249x + 15378	0.99	11.3−19.0
		10−200 μg	y = 64822x + 633030	0.98	4.2−12.6
PS	5-hexene1, 3, 5-triyltribenzene	0.1−10 μg	y = 140908x + 13673	0.99	9.2−13.2
PE	1,12-tridecadiene	0.1−10 μg	y = 26634x + 23504	0.99	3.9−16.6
		10−200 μg	y = 15838x + 179704	0.99	10.6−16.9
PET	Vinyl benzoate	0.1−10 μg	y = 76077x + 16800	0.99	9.4−17.8
		10−200 μg	y = 53151x - 32925	0.98	4.4−17.8
PA	ε-caprolactam	0.1−10 μg	y = 269819x - 5550	0.99	5.9−16.9

Table: Mass Concentration (μg/L) and Removal Efficiency (%) of Total MPs in Plants A and B

WWTP	total MPs	0 (influent)	1 (primary effluent)	2 (secondary effluent)	3 (tertiary effluent)
		sampling site			
plant A	mass concentration (μg/L)	26.23 ± 7.71	9.50 ± 1.22	7.62 ± 0.67	1.75 ± 0.02
	removal efficiency (%)	0	63.8	70.9	93.3
plant B	mass concentration (μg/L)	11.29 ± 0.71	5.22 ± 0.06	1.99 ± 0.52	0.71 ± 0.12
	removal efficiency (%)	0	53.8	82.4	93.7

[a]Locations 0, 1, 2, and 3 mean raw wastewater and treated water after primary, secondary, and tertiary treatment, respectively.

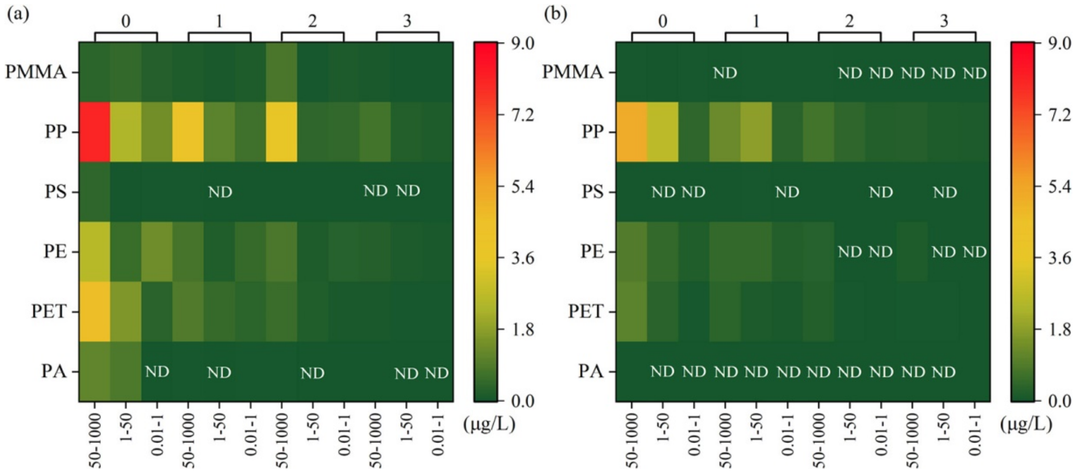


Fig: Heatmaps representing the average concentration of different types with different size ranges at the whole treatment processes of plants A (a) and B (b).

Only polypropylene(PP), polyethylene(PE), polyethylene terepthalate(PET) was considered for further analysis.

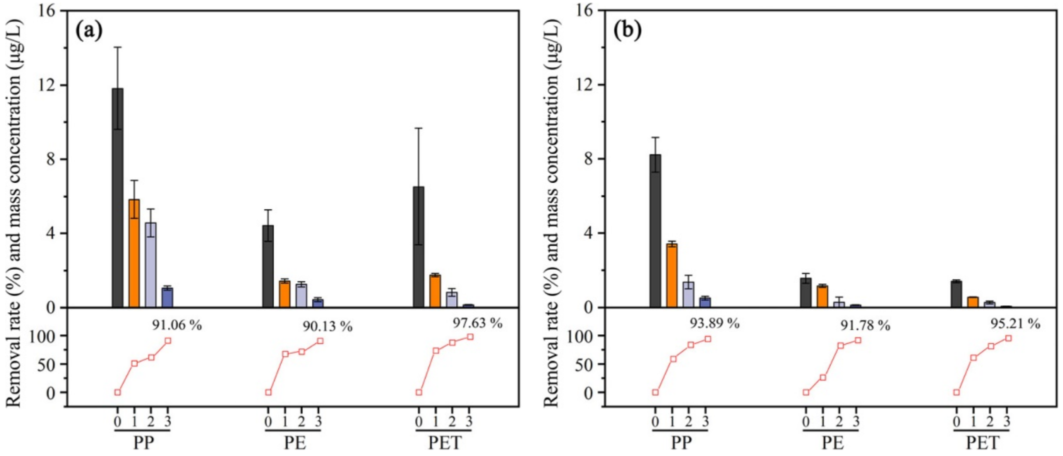
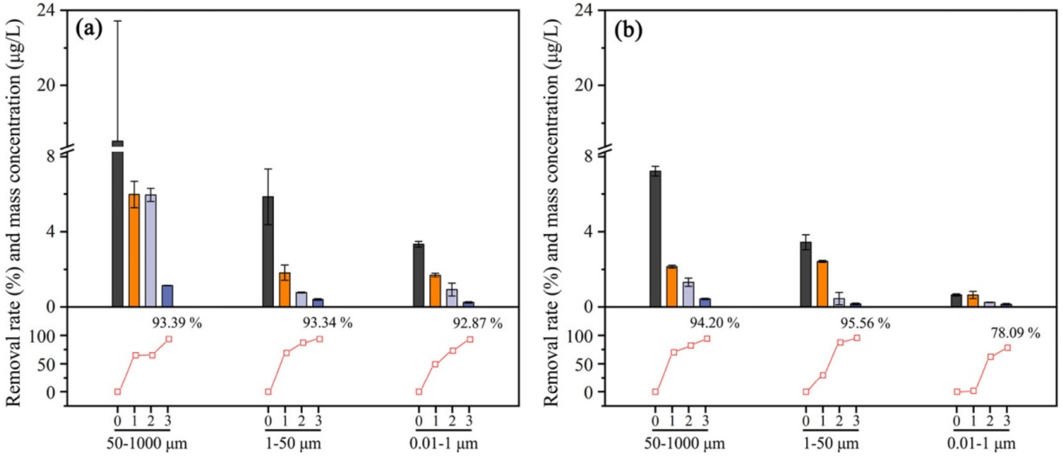


Fig: Mass concentration and removal efficiency of the main MPs and NPs (PP, PE, and PET) over the wastewater treatment process in plants A (a) and B (b).

Fig: Mass concentration and removal efficiency of MPs with different size ranges in the whole wastewater treatment process in plants A (a) and B (b).



CONCLUSIONS

- In this study, the concentration of MPs was quantified by mass rather than the particle number as investigated in previous studies.
- The mass investigated in this study is important to evaluate the relationship between the quantity and size of MPs, especially when there is no suitable method to quantify sub-MPs and NPs in WWTPs.
- Nearly 90% of the MPs and NPs are removed by the treatment plants.
- There are different removal efficiencies of WWTPs towards different types of MPs and NPs.
- The amount of MPs and NPs released into the aquatic environment are relatively low.
- The emissions of NPs could be negligible. But treatment is essential as the photodegradation of MPs to NPs in aquatic environment.

Thank you