Research article

Organic compounds associated with microplastic pollutants in New Jersey, U.S.A. surface waters

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Abstract: Extensive manufacturing and ubiquitous use in every sector of today's society has resulted in plastics being detected in all terrestrial and aquatic environments examined to date. However, the pervasiveness of small, potentially invisible, microplastics, their associated chemical additives, and organic compounds that absorb to plastic substrates are the topics of recent investigations. These micro- to nano- size plastic particles that are deliberately manufactured or were fragmented from larger plastic products are now ending up in food webs and worldwide environmental systems. Using a pyrolysis GC-MS method, plastic polymer composition was determined in samples obtained from freshwaters in urban New Jersey. Three polymers dominated the samples: polyethylene (43%), polypropylene (33%), and polystyrene (13%). The dominant polymers differed in each river. To identify Persistent Organic Pollutants sorbed to microplastic particles, headspace solid phase micro extraction coupled with gas chromatography/ion trap mass spectrometry was employed. In the majority of upriver sampling locations, Tentatively Identified Compounds were associated with both the microplastic and the water column fractions in roughly equal proportions. However, in the tidal portion of the Passaic River and in samples from Newark and Raritan Bays, the majority of organic compounds were associated with the microplastic fraction only. Based on a search of chemical databases, the possible source/use of 180 of the 223 compounds identified, whose total mass was 1 ng or more, was determined. Forty one percent of the identified compounds were natural substances, thirty five percent were identified as laboratory/research chemicals and seven percent were pharmaceutical or biomedical compounds. Twelve identified compounds are used for industrial purposes, including a plasticizer and

an insecticide. Six compounds are used as cosmetic additives. The findings of this study illustrate the diversity of organic compounds associated with the presence of microplastics in aquatic media.

Keywords: polyethylene; polypropylene; polystyrene; persistent organic contaminants; Raritan River; Passaic River

1. Introduction

Humans residing along urban waterways are important contributors to the global plastic lifecycle. Plastic transport into surface waterways via litter, stormwater runoff, consumer product formulations and point source discharges are well documented [1,2]. Microplastic pollution is present in urban freshwater systems at concentrations equal to or greater than concentrations documented in the world's oceans [3]. Anthropogenic sources of microplastic (5 mm or smaller in size) include discharges from wastewater treatment plants [4,5], the fragmentation of larger plastic debris/litter [6], microfiber releases [7], atmospheric deposition [8], and exfoliating products containing microbeads [9]. The percentages contributed from these various sources to total microplastic concentrations in freshwater aquatic systems has not been well characterized, and likely varies based on land uses, local water management, and population density.

Literature documenting the presence of microplastics in urban waterways is relatively recent, and the full extent of ecological and human impacts associated with freshwater plastic pollution are not well understood [10]. Microplastics have been documented in fin fish [11–13] and shellfish tissues [14–16] along with other plastic-associated contaminants, although there are very few studies examining microplastic impacts on the health of aquatic organisms. Microplastics have also been reported in agricultural fields fertilized with sewage sludge, which are utilized for crops and animal grazing [17], thus allowing portals for microplastics and their associated pollutants to enter into human food supplies.

Potential environmental concerns are also associated with the composition of plastic polymers. These microscopic polymers can act as a substrate for pathogenic and nonpathogenic microbial communities [18]. They also provide an attachment surface for organic compounds present in surface waters [19,20]. In addition to the chemical composition of the more than 30,000 registered plastic polymers [2] and/or compounds resulting from environmental breakdown of plastics, there also exists the potential for persistent organic pollutants (POPs), particularly those that are hydrophobic, to attach themselves to microplastic particles [21]. Transport of POPs, due to microplastic adsorption, is most probably a function of the microplastic composition, aquatic POP concentrations, microbial biofilm formation, mediated by local environmental conditions.

Data describing specific microplastic polymers and/or compounds associated with these polymers is currently insufficient. To address this need for additional data, microplastic and water column samples obtained from freshwater sections of the Raritan and Passaic Rivers and estuarine Raritan and Newark Bays in urban New Jersey, U.S.A. were analyzed. This analysis was undertaken to identify the plastic polymer components and the organic compounds associated with both the recovered microplastic and water column fractions. POP presence in both the water column and in the associated plastic fraction would indicate mobility between the two compartments.

The information gathered from these studies is applicable to other heavily populated urban environments, both in the U.S.A. and worldwide. Adsorption to microplastic particles increases the range of POP transport and/or provides a sink for POPs. Polymer degradation and/or organic

compound desorption into the water column or gastrointestinal track fluids following ingestion could facilitate increased human and environmental exposure potentials.

2. Experimental methods

2.1. Sample collection

Two replicate samples were collected from fourteen locations in the Raritan River (N = 5), Passaic River (N = 7), and Newark and Raritan Bays (N = 2) in urban New Jersey, U.S.A (Figure 1). One replicate was digested with the Fenton Reaction to remove organic material and determine microplastic density. The second replicate was used to produce twenty-eight undigested samples (N = 14 each from water and solid fractions) used for POP analysis. Three of the Passaic River sites (Lyndhurst, Kearny, and Newark below the Dundee Dam impoundment) and two of the Raritan River sites (Piscataway and New Brunswick) are in the tidally influenced portion of these rivers. Briefly, a 330µ mesh manta trawl net was held in a stationary position in the rivers at the water surface for fifteen minutes; the bay samples were collected by pulling the trawl net via boat and the distance calculation was used to determine water volume flow through the net. The collected sample was then filtered to remove organic materials larger than 330µ. A separate 1 L water column grab sample was obtained in a glass bottle from each sampling location at the same time as the microplastic trawl, and all samples were placed on ice for transport back to the laboratory. For complete details of sampling locations, method of sample collection, and analytic methods see Ravit et al. (2017) [6].
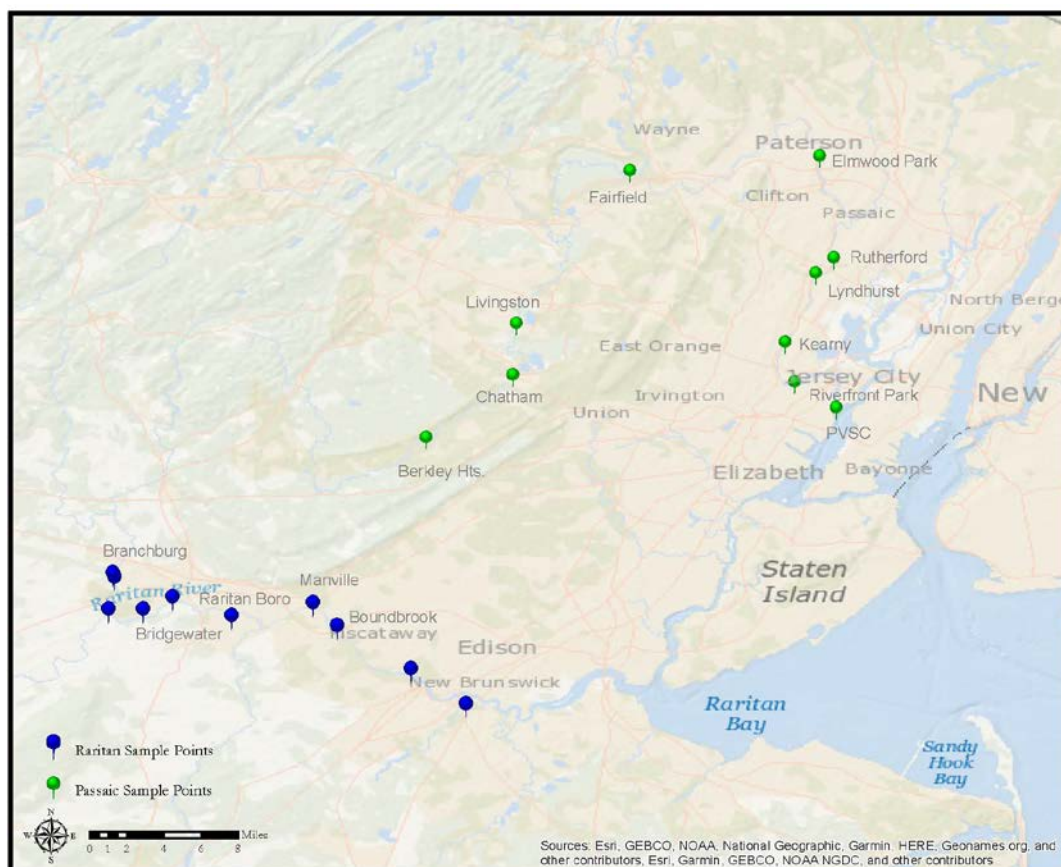

Figure 1. Microplastic sampling locations in the Raritan and Passaic Rivers.

2.2. Polymer analysis

Our previous research used a novel method of pyrolysis GC-MS to determine plastic polymer composition. Briefly, a very small piece of microplastic sample (less than 1 mg) was placed in a quartz tube, which was then placed in a platinum coil and heated to 750 °C. In the pyrolysis process the large polymer chains break down into smaller fragments that are first separated using a DB-5 capillary column and then analyzed by mass spectrometry in the full scan mode. The initial oven temperature of 45 °C was held for 2 min., which was then ramped to 320 °C at a rate of 20 °C/min. The oven was held at 320 °C for 19 min. for a total run time of about 35 min. In-house or literature pyrolysis GC-MS spectra were used for peak matching and polymer identification.

2.3. Organic compound analysis

To identify POPs sorbed to microplastic particles, headspace solid phase micro extraction coupled with gas chromatography/ion trap mass spectrometry (HS-SPME/GC-ITMS) was employed. Briefly, microplastics and site water were analyzed separately using a CTC Analytics Combi PAL system with SPME agitator attachment (Zwingen, Switzerland). For specific details of the Combi PAL HS-SPME and injection program run, chromatographic separation, and ion trap mass spectrometry see Ravit et al. (2017) [6]. Compounds eluted from the GC column were qualitatively identified after background subtraction by comparison of their spectra to the vendor's library and NIST/EPA/NIH 2015 mass spectral library, interpretation of their fragmentation pattern, and their respective molecular ions. Identified VOCs that showed a greater than 70% fit correlation to the library spectrum were designated as Tentatively Identified Compounds (TICs). Analytes were identified/tentatively identified via a library spectral match with the NIST library of MS data. The compound was identified using both MS and MS/MS data. The method has previously been used for unknown identification in raw and finished water samples [22,23]. Fragmentation patterns are often used to improve confidence in an identification assignment.

The general GC/MS method was based on the approach for semi-volatile analysis found in EPA method 525. EPA method 525 is a general-purpose method for determination of organic compounds in finished drinking water, source water, or drinking water in any treatment stage. It uses analyte extraction from the water sample followed by GC/MS analysis and is applicable to a wide range of organic compounds. EPA 525 internal standards (Supelco) were spiked in the sample run as quality control checks for rough quantitation and retention time. Semi-Quantitative values (ng or ppb, ug/L) in the TICs tables (Supplementary data Tables 1 and 2) are calculated by estimating a response factor (calculated from peak areas) from the nearest EPA 525 internal standard, based on GC retention times. Using the CAS number of each TIC an internet search was conducted to try and determine the use/source of the various organic compounds. We did not conduct a search for compounds present at ~1 ng or less.

Table 1. Dry weather versus wet weather microplastic recovery.

Type	# Samples	# Recovered microplastic particles
Dry weather	14	63
Wet weather	5	67
Bay waters	2	13

Table 2. Polymer types recovered from 22 samples.

Type	#	%
Polystyrene	18	13%
Polypropylene	46	33%
Polyethylene	60	43%
Other (6 polymers)	16	11%
Total	140	100%

3. Results

3.1. Microplastic polymers

Polymer identifications were attempted for 143 microplastic fragments from 14 samples collected during dry weather, 5 samples collected after a rainstorm, and 2 samples collected from marine bay waters (Table 1). A very small number of samples could not be analyzed either due to a detection limit issue or a spectral quality issue. The pyrolytic fragmentation pattern was reproducible and unique for a given plastic polymer. Peak pattern distortions, possibly related to weathering, were apparent in a number of samples. However, the marker compounds and qualitative peak patterns were still indicative of the original polymers. Although the microplastics recovered came in multiple shapes/sizes, three polymers dominated the samples (Table 2) collected from these two rivers and produced very identifiable GC-MS chromatograms (Figure 2): polyethylene (43%), polypropylene (33%), and polystyrene (13%). However, the dominant polymers differed in each river. Polyethylene was the most common polymer in the Raritan samples, while polypropylene contributed the highest proportion of the Passaic samples (Figure 3). The Passaic results were dominated by the 55 microplastic fragments recovered from the downriver Lyndhurst wet weather sample event (Figures 4 and 5). The number of microplastic fragments did appear to increase in downstream locations under wet weather conditions, although the Raritan wet weather samples did not exhibit the significantly higher number of microplastics observed in the Passaic Lyndhurst sample (Figure 5).
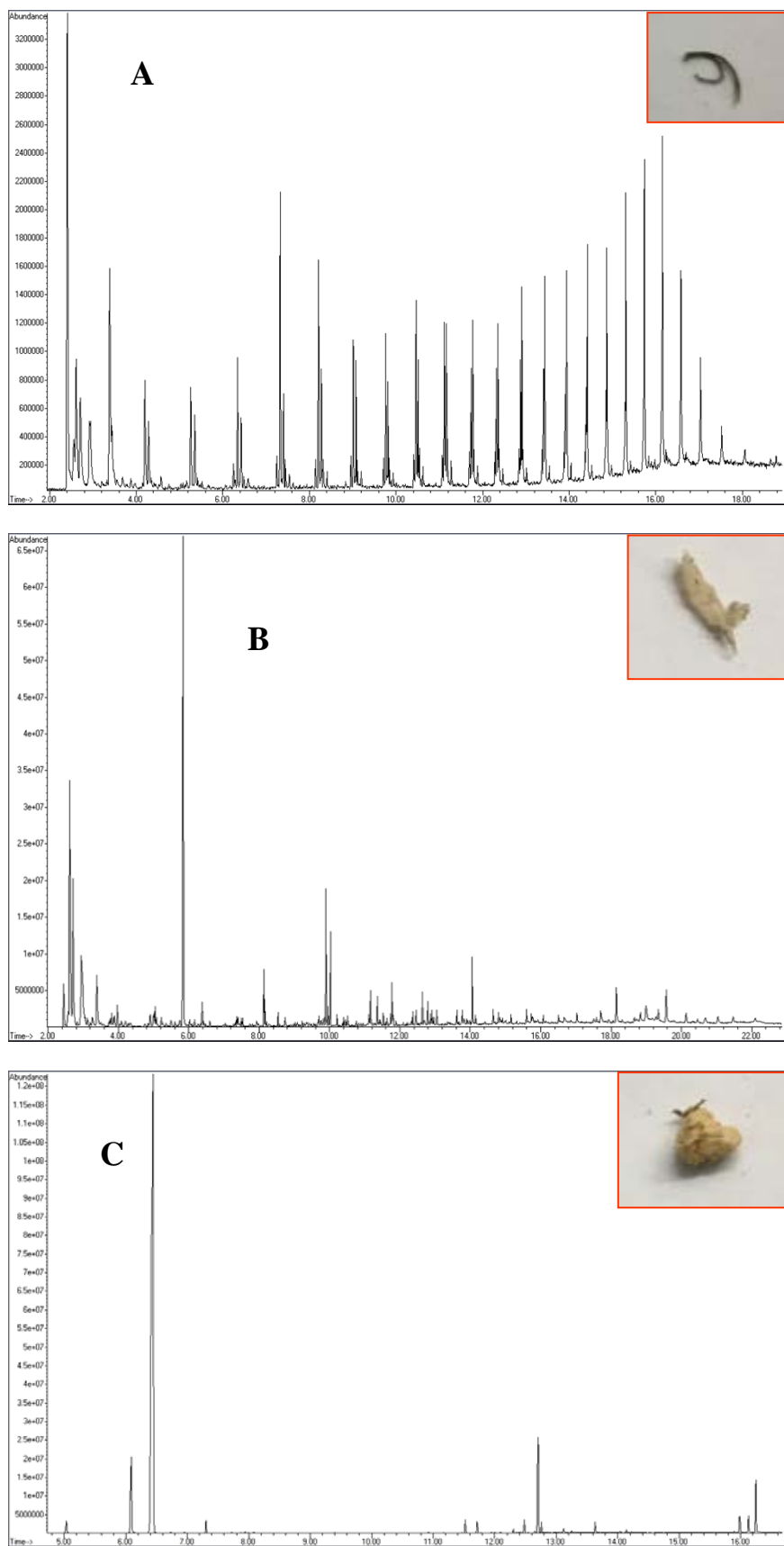
Figure 2. Chromatograms for A) Greenish blue fragment/fiber, Polyethylene; B) Semi-Transparent Fragment, polypropylene; C) Foam, polystyrene.
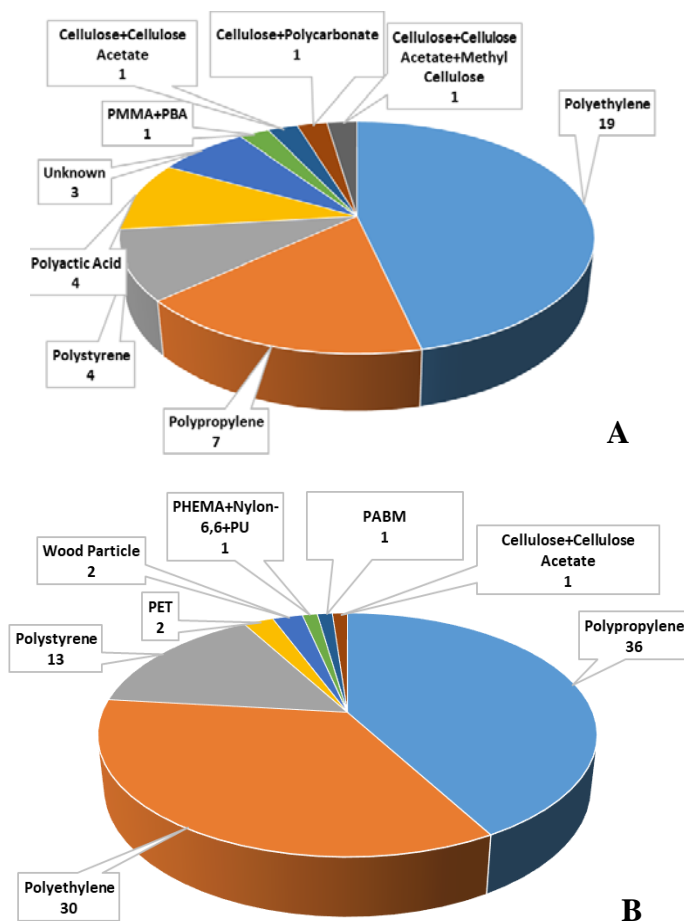
Figure 3. Microplastic fragment polymers recovered from wet and dry weather Raritan and Passaic River samples. Sixty-five percent of the higher number of Passaic microplastics includes 55 wet weather sample fragments from the Lyndhurst sampling location. A) Raritan River (N = 41), Polyethylene >>> Polypropylene >> Polystyrene; B) Passaic River (N = 85), Polypropylene ~ Polyethylene >> Polystyrene.
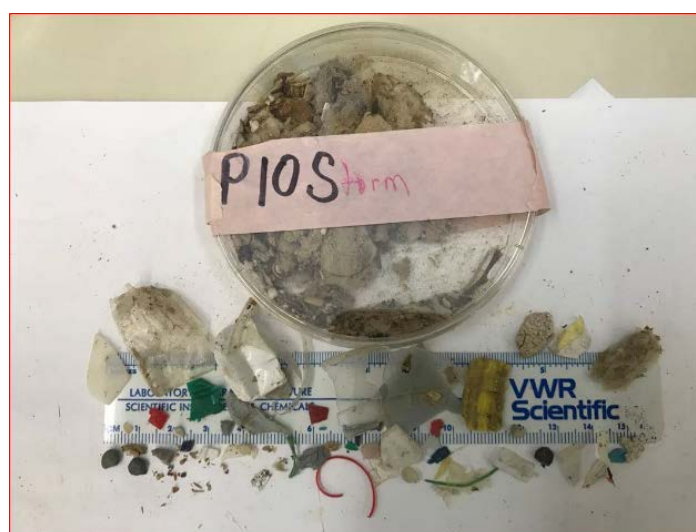

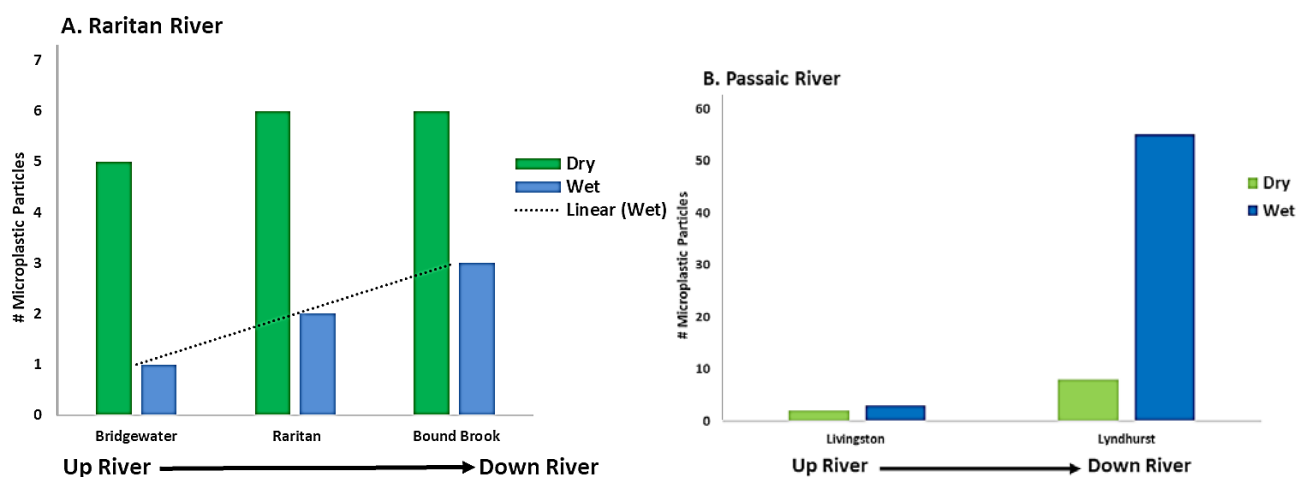**Figure 4.** The wet weather Lyndhurst plastic fragments analyzed by pyrolysis GS-MS.

Figure 5. Number of microplastic fragments observed under wet weather and a dry weather sampling conditions at: A) three Raritan River and B) two Passaic River sampling locations. The Passaic quantity of microplastic fragments at the downriver location is an order of magnitude greater than the number of fragments observed in the other samples.

3.2. Tentatively identified organic compounds

Three hundred and ten organic Tentatively Identified Compounds (TICs) were "identified" based on spectral library match in 28 samples (Table 3; Supplemental Data, Tables 1 and 2). The Passaic River samples contained the highest number of TICs (96% of all compounds identified); 66% of the identified compounds were observed in the Raritan River samples. Only 81 of the identified compounds (26%) were observed in samples from Newark and Raritan Bays. However, this may underrepresent organic compounds in the bay waters because only a single sample was collected from each bay. Although the number of TICs in the Raritan samples was 44 fewer than those found in the Passaic samples, the total Raritan TIC mass was an order of magnitude greater than the total TIC mass observed in the Passaic.

Table 3. Tentatively identified organic compounds (TICs).

Tentatively identified compounds (TICs)	Raritan River		Passaic River		Raritan/Newark Bays	
	#	Total mass	#	Total mass	#	Total mass
Compounds identified	255	1746	299	732	81	197
TICs identified (>10 ng)	16	1443	23	148	5	67
Other identified TICs (<10 ng)	236	303	271	584	74	130

In the majority of upriver sampling locations, TICs were associated with both the microplastic and the water column fractions in roughly equal proportions (Figure. 6). However, in the tidal portion of the Passaic, below the impediment of the Dundee Dam, and in samples from Newark and Raritan Bays, the majority of organic compounds were associated with the microplastic fraction only. A notable exception in both rivers was the high proportion of organics associated with microplastics at the furthest upstream sampling location. This may be due in part to the hydrodynamics in the headwaters of both river systems where low flows and multiple small stream inputs covering large surface areas contribute to concentrating organic inputs.
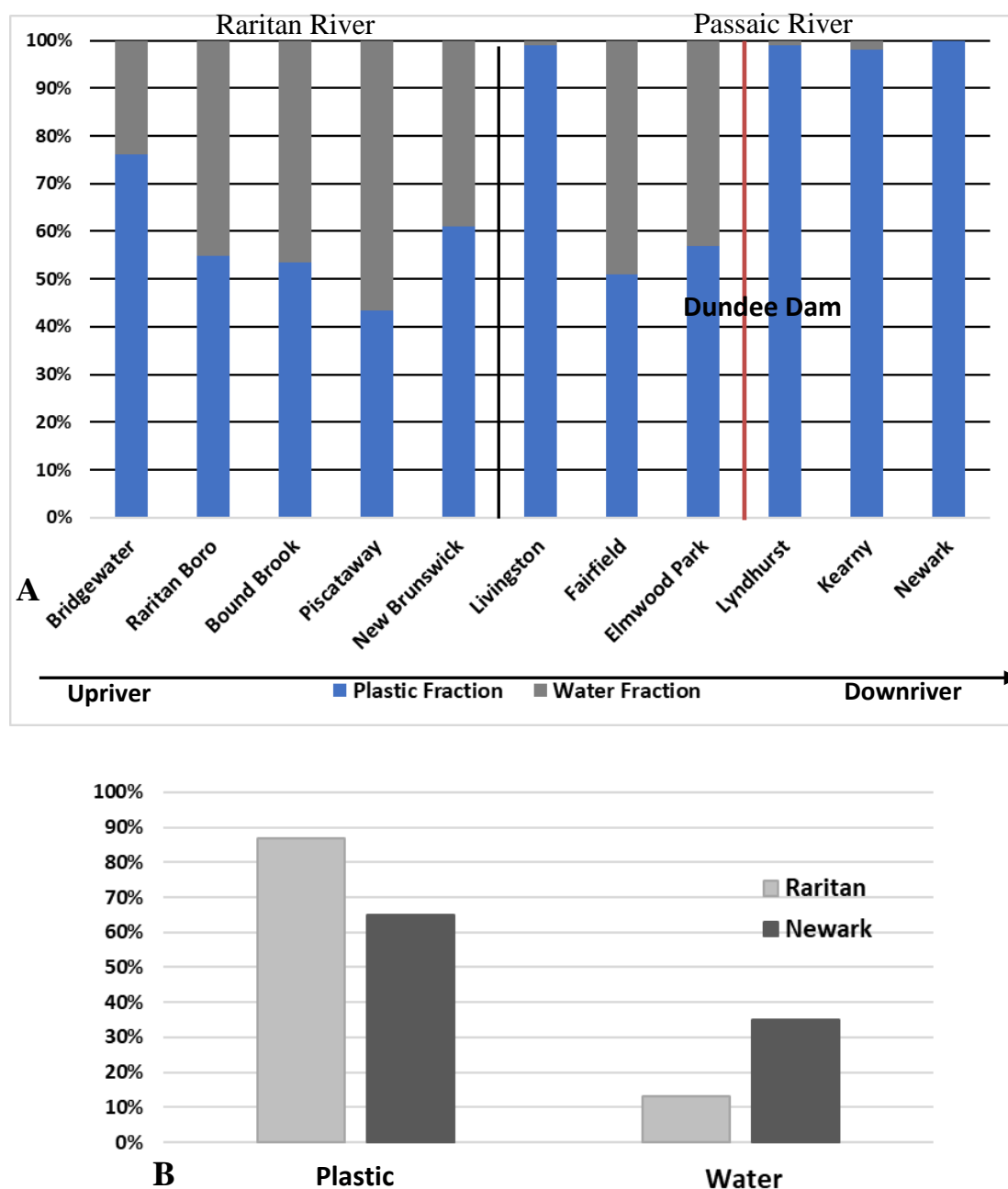
Figure 6. Proportion of organic tentatively identified compounds (TICs) above 10 ng observed in the microplastic versus the water column fractions from the: A) Raritan and Passaic Rivers; and B) Raritan and Newark Bay sampling locations.

Approximately 6–8% of the TICs in both the rivers and bays had an estimated total mass from all samples of >10 ng (Figures 7–9). Based on a search of chemical databases we were able to identify the possible source/use of 180 of the 223 compounds present whose total mass was 1 ng or more (Table 4; Supplemental data, Table 1). Compounds offered for sale without any identified use were included in the category "Research Chemical". However, these compounds may also have industrial uses. We were unable to identify sources/uses for 43 of the identified compounds and we did not conduct a database search for information on 84 compounds whose total mass was less than 1 ng (Supplemental data, Table 2). The Raritan TIC data was dominated by the presence of 16 compounds, all of whose mass exceeded 10 ng.
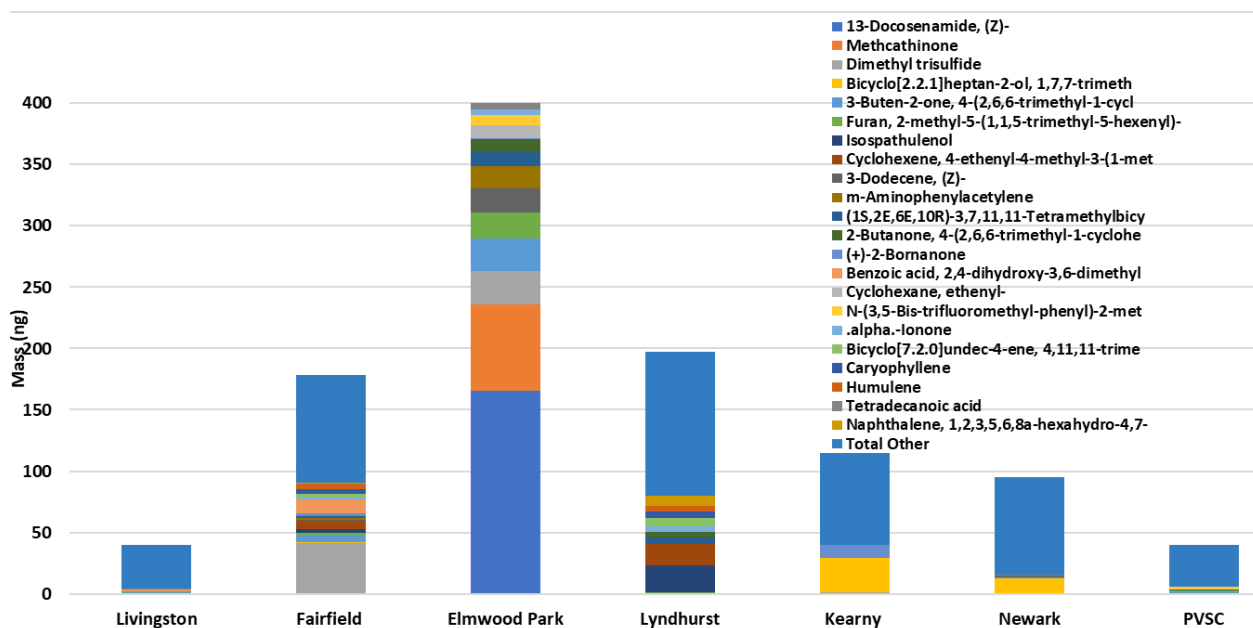
Figure 7. Organic tentatively identified compounds (TICs) above 10 ng and total mass of TICs below 10 ng observed in the Passaic River sampling sites.

A number of naturally occurring compounds had commercial uses, especially as flavoring and fragrance additives. Seventy-three of the identified compounds (41%) were natural substances, of which 53 are also in use as commercial flavoring food additives or fragrance agents. Sixty-three compounds (35%) were identified as laboratory/research chemicals and 13 (7%) were pharmaceutical or biomedical compounds. Twelve identified compounds (7%) are used for industrial purposes, including a plasticizer and an insecticide. Six compounds (3%) are used as cos metic additives.

The compound with the highest mass (608 ng), present at three Raritan River locations (Figure 8), was identified as methcathinone, a monoamine alkaloid and psychoactive stimulant that is used as a recreational drug (Supplemental Data, Table 1). The other five TICs with mass of ~100 ng or greater were 2 natural compounds (dimethyl trisulfide and 13-docosenamide, (Z)-), 1 natural compound also used for fragrance (Indole, 3-methyl-); and 2 laboratory chemicals (pyrolo[3.2-d]pyrimidin-2,4(1H,3H)-dione and m-aminophenylacetylene).
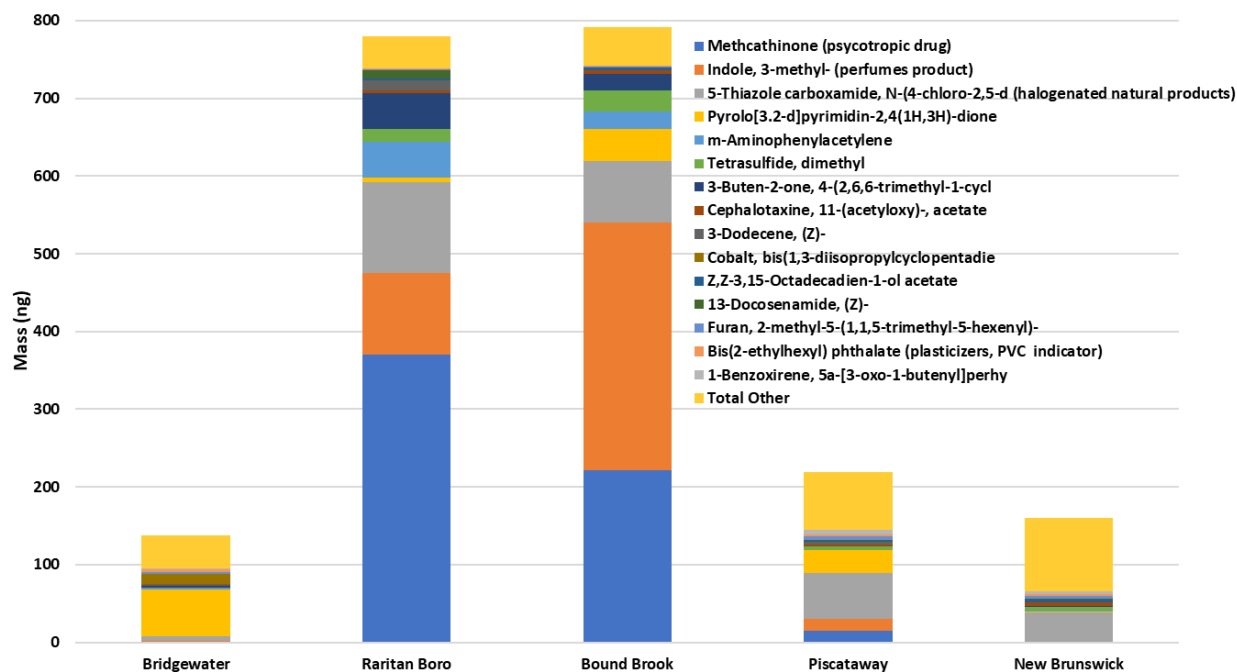
Figure 8. Organic tentatively identified compounds (TICs) above 10 ng and total mass of TICs below 10 ng observed in the Raritan River sampling sites.
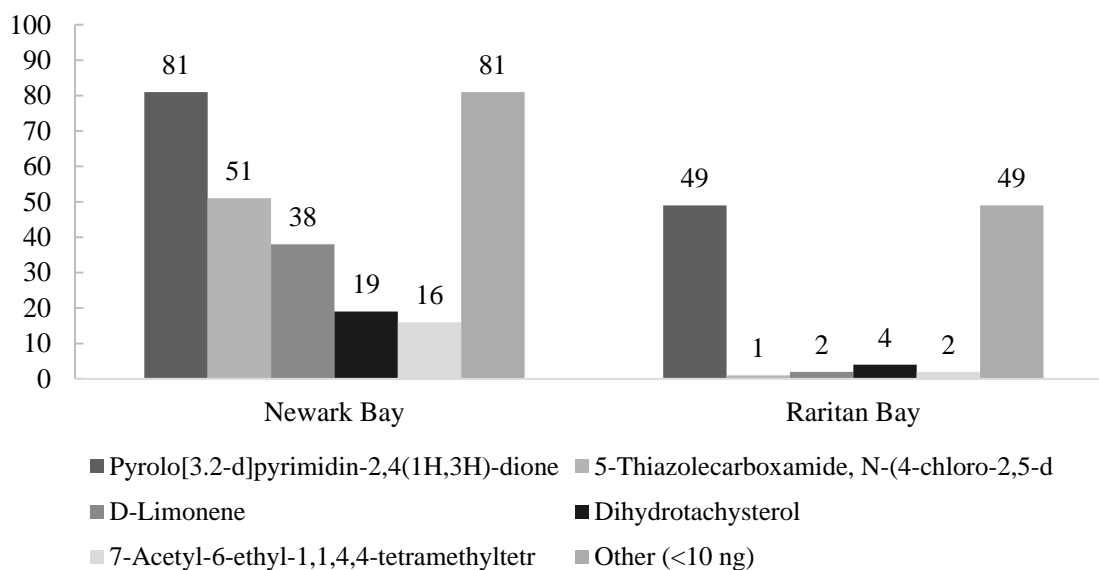

Figure 9. Organic tentatively identified compounds (TICs) above 10 ng and total mass of TICs below 10 ng in the Raritan and Newark Bay sampling locations.

An analysis of specific TICs at each sampling location revealed patterns that differed between both rivers, as well as within each river between the various sampling locations. Raritan TICs were highest at the Raritan Borough and Bound Brook sampling sites and Passaic TICs were highest at the Elmwood Park location (Figure 7). However, Passaic TICs greater than 10 ng were, for the most part, overshadowed by the total mass of TICs present at less than 10 ng (Figure 7). The bay water samples exhibited patterns that were similar to their associated rivers. Newark Bay samples were dominated by

TICs less than 10 ng, while Raritan Bay waters were dominated by specific compounds with mass >10 ng (Figure 9).

4. Discussion

Extensive manufacturing and ubiquitous use in every sector of today's society has resulted in plastics being detected in all terrestrial and aquatic environments examined to date. The current primary public concern over plastics is based on an aesthetic perspective because liter and trash are easily observed on a daily basis. The ongoing news coverage of lethal plastic impacts on sea birds, fish and mammals, and the presence of plastic-created large floating islands, as well as the presence of plastics in the deepest oceans has resulted in increasing public concerns. The public is, however, less knowledgeable about the pervasiveness of small, potentially invisible, microplastics, associated chemical additives, and organic compounds that absorb to plastic substrates that may impact both humans and aquatic animals. These micro- to nano- size plastic particles that are deliberately manufactured or were fragmented from larger plastic products are now ending up in food webs and worldwide environmental systems.

There were two different but related objectives of this research. The first goal was to describe plastic densities along a freshwater to brackish water continuum. The second objective was to identify dominant polymers and the types of POPs that might absorb to these polymers, indicating the potential for these organic compounds to move into aquatic food chains. We also wanted to understand the relationship between POPs sorbed to the plastic fraction and POPs found in the water column. While no clear associations between the type of plastic and class of POP was immediately observable, a larger data set may demonstrate an association in the future. From this study, the greatest link between analytes and samples showed significant overlap between the water fraction and the absorbed fraction in the majority of freshwater samples. What is still lacking is a risk determination for potential effects on aquatic life from these particles and associated compounds [24]. Each compound would need to be assessed for potential adverse or synergistic effects. The findings of this paper illustrate the diversity of organic compounds associated with the presence of microplastics in aquatic media.

The pyrolysis GC-MS analysis was able to differentiate between microplastic polymer compositions using material collected in the manta trawl. The most common plastic polymer present was polyethylene (43%), which is not surprising since the largest volume polymer in commercial use is polyethylene. The second most common polymer was polypropylene (33%). Polystyrene (13%), currently targeted for reduction due to poor recyclability and concerns over toxicity due to styrene ring metabolism to potentially reactive intermediate forms, was the third most common polymer in these samples. Polyethylene and polypropylene are lighter than water. Although polystyrene is heavier than water, when filled with air the expanded polystyrene is also lighter than water. Because the samples were only collected at the water surface, the occurrences of these lighter polymers are not surprising. In several of the 6 other polymers detected, multiple plastic chemical profiles were often observed, which indicates the likelihood of multiple plastic types being present. This is to be expected as new different composite products are being developed and used in commerce.

The HS-SPME/GC-ITMS method identified 310 TICs that were associated with the plastic and/or detected in the water phase. There was not a large difference in the number of TICs recovered from each of the two river systems (delta 44), but the total TIC mass was an order of magnitude greater in the Raritan River. There is no clear explanation for this finding as the Raritan/Passaic basin sizes are relatively similar and the population is 2.5 million for the Passaic River basin and 1.2 for the Raritan

River basin. It is not surprising to have detected the classes of compounds reported in Table 4 due to the large populations and industries present in these watersheds.

In the freshwater reaches of the both rivers the TICs were more equally distributed between the water and the solid plastic phase. However, this was not the case for samples from the brackish waters (lower Passaic River) and the two bays, where the majority of the compounds were associated with the solid microplastic phase. The result from samples taken in the brackish sections of the rivers and the bays is similar to previously described adsorption of POPs associated with marine microplastic samples [25].

Table 4. Identified use categories of tentatively identified compounds (TICs) >1 ng.

Use type	# of Compounds*	% Total
Cosmetic additive	6	3%
Fragrance	6	3%
Industrial (1 plasticizer)	12	7%
Pharmaceutical/Biomedical	13	7%
Food additive	20	11%
Flavor and fragrance	27	15%
Laboratory/research chemical	63	35%
Natural compound*	73	41%

The total mass of individual TICs was dependent on the sample collection site, and ranged from ~100–800 ug for the Raritan and ~50–400 ug for the Passaic River. Of note, different percentages and compounds were detected which appeared to indicate local sources for some of the TICs, which might be used to identify source inputs. The use of the specific TICs associated with certain use categories can also facilitate source identification (Supplemental data). Future studies should compare TICs and levels associated with population-based gradients for better characterizing human inputs.

The open bay water samples had significantly lower plastic particle numbers. This could be due to ocean tidal influences (1–5 ft), change in saltwater density influencing particle buoyancy or other unknown explanations. The density of the particles and fragments will determine in part where the material will ultimately be found. This is one reason why relying solely on the manta net (surface collection) probably underestimates the true plastic load in our rivers and oceans. Baldwin et al. (2016) [26] describes freshwater watershed attributes and hydrology that influence distribution and types of plastic debris in the Great Lakes. In more urbanized areas the impervious and built environments will impact the distribution into the watersheds through direct runoff and inputs from municipal and industrial dischargers. Microplastic particles of different densities will distribute to surface, pelagic and benthic zones, and resuspension or hydrological process influence their distribution.

5. Conclusions

Density of microplastics present in the Raritan and Passaic River basins are comparable to those reported in other freshwater and oceanic systems. The chemicals associated with both the plastic and the the freshwater phase indicates that desorption can occur and that these TICs can be available for uptake through the GI tract or other routes of uptake. Both the pyrolysis GC-MS and the HS-SPME/GC-ITMS methods complement each other in plastic and chemical identification, and should be used in combination when conducting studies of plastic-associated organic compounds. The specific

TICs and use classification can potentially be used to track chemical sources within various riverine systems for source identification and enforcement purposes. Future studies should include monitoring methods that collect samples throughout the water column and the sediments to obtain a better microplastic mass balance.

Acknowledgments

Conflict of interest

The authors declare that they have no conflicts of interest regarding the publication of this paper.

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