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Justin L. Wilkens, Andrew D. McQueen, Joshua J. LeMonte,
and Burton C. Suedel

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Preface

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Initial Survey of Microplastics in Bottom Sediments from United States Waterways

Abstract
Given the reported extent of microplastics in the aquatic environment, environmentally relevant exposure information for sediments dredged by the US Army Corps of Engineers will lend context to the risks posed by this contaminant dur-ing dredging. We measured the occurrence, abundance, and polymer composition of microplastics in sediments collected from nine dredged waterways and two non-dredged reference areas. The number of particles in sediment samples ranged from 162 to 6110 particles/kg dry wt., with a mean of 1636 particles/kg dry wt. Fragments were the most prevalent shape observed among the 11 study sites (100% frequency of occurrence), followed by fibers (81%), spheres (75%), foams (38%) and films (34%). Based on analyses of chemical composition of the particles using Fourier transform infrared spectroscopy, polyethylene:propylene was the most common polymer type observed. Consistent with results presented by other investiga-tors, microplastic concentrations and polymer types in bottom sediments in this study were also aligned with the most widely used plastics worldwide.

There is increasing global awareness of the extent of plastic debris in the aquatic environment (UNEP 2016), with particular focus on microplastics as an emerging contaminant due to the potential adverse effects to freshwater and marine species (Thompson et al. 2004; Wagner and Lambert 2018). Microplastics include particles ranging in size from 1 µm to 5 mm and include any synthetic particle or polymeric matrix with regular and irregular shape originating from primary or secondary manufacturing origin (Frias and Nash 2019).

In North America, studies reporting microplastic abundance have predominantly focused on surface waters and beach sediments due in part to ease of access and relatively low sample collection costs (Eriksen et al. 2013; Driedger et al. 2015). Less is known regarding the extent of microplastics in bottom sediments (i.e., subtidal zone or littoral zone and deeper; non-beach sediments). In marine sites, Graham and Thompson (2009) surveyed three shallow subtidal sediments sites on the Florida and Maine coast and detected microplastics in all sample sites. In freshwater, several surveys have occurred in Lake Ontario and Canadian rivers where microplastics were detected in nearly all sediment samples collected (Castañeda et al. 2014; Corcoran et al. 2015; Ballent et al. 2016; Vermaire et al. 2017). With the exception of these studies, there is an overall lack of knowledge about the occurrence and characteristics of microplastics in bottom sediments in the United States (US). In the US, the US Army Corps of Engineers (USACE) is responsible for maintaining and improving nearly 25,000 miles of inland and intracoastal waterways, coastal channels, ports, harbors, and turning basins. Given the reported extent of microplastics in the aquatic environment, exposure information (e.g., concentration, spatiotemporal extent, plastic type, shape, etc.) is needed for sediments to be dredged from these waterways to lend context to the ecological risks posed by this emerging contaminant during dredging. To address this concern, the objective of this research was to measure the concentration and polymer composition of microplastics (< 5 mm) in sediments collected from waterways where dredging commonly occurs and to compare those results with those reported by other investigators.

Materials and Methods

Sediments collected from harbors and channels routinely dredged in the Great Lakes, Gulf of Mexico, Long Island Sound, Atlantic Ocean, and the Mississippi River were examined (Table 1). Samples used for this study were obtained opportunistically from sediments collected for dredged sediment evaluations using sample collection criteria described in the Inland Testing Manual and Ocean Testing Manual (U.S. Environmental Protection Agency/U.S. Army Corps of Engineers (USEPA/USACE) 1991, 1998). This approach provided the opportunity to leverage existing sampling programs aimed at assessing the potential for contaminant-related impacts associated with sediment placement in aquatic environments from sediments excavated from coastal and inland navigational waterways. Sediments were collected using standardized sample handling, preservation, and storage protocols to minimize any changes in the composition of the sample and avoid chemical, physical, or biological contamination (U.S. Environmental Protection Agency/U.S. Army Corps of Engineers (USEPA/USACE) 1991, 1998). Sediments were collected using a combination of coring and surface grab methods (e.g., stainless steel split spoon sampler or Ekman dredge) to obtain samples of surficial (recently shoaled) bottom sediments in the navigation channel (Table 1).

From each sampled site, up to 132 L of sediment was sealed in RO rinsed, one-time use, 19 L high density polyethylene (HDPE) containers or in two cases (Horseshoe Lake and Port Everglades) was stored in 1 L wide-mouth glass jars with metal lids in temperature controlled conditions (4°C ± 1°C). Due to the unavoidable use of HDPE containers at most sampling sites (due to the sampling protocols and sediment volumes collected), the potential bias was evaluated using multiple lines of evidence to inform the acceptability of these samples to meet the study objectives. The evaluation including visual inspection of the original sample containers and visual comparisons of the container plastic type (via particles manually abraded) to the particles observed in the sediment samples (e.g., color, shape, weathered condition). In all cases there was no evidence of abrasion on the sidewalls and bottom of the sample containers. Additionally, the manually abraded plastic type and condition (i.e., unweathered) did not visually match the type and condition of microplastics observed in the sediment samples. Based on the lines of evidence, the authors concluded that any influence of storage conditions from this study were likely minimal, and do not meaningfully influence the results of this study. Sediments used for this study were subsampled (n = 3) from the original containers and composited, yielding one sample per site (n = 11). Selected reference sites included non-dredged marine and freshwater sites: Corpus Christi Bay marine reference ("Ref A") and Horseshoe Lake

Table 1 Bottom sediments in United States waterways subjected to microplastic analysis

Geographic region and sub-region	Sample site[a] (site designation)	Particle size (%)			
		Gravel	Sand	Silt	Clay
Marine					
Gulf of Mexico					
Corpus Christi Bay	Corpus Christi, TX (Ref A)[b]	0	59	21	20
	Corpus Christi Harbor, TX (CCH)[b]	2	75	8	15
Mouth of Mississippi River	Southwest Pass, LA (SWP)[c]	0	27	47	26
Atlantic Ocean					
Long Island Sound	Mianus River Cos Cob Harbor, CT (MR)[c]	0	21	49	30
	Milford Harbor, CT (MH)[c]	0	13	45	42
	Westport, CT (WP)[c]	3	31	66[d]	
Miami Terrace	Kings Bay, FL, GA (KB)[c]	0	71	15	14
	Port Everglades, FL (PE)[c]	1	48	27	24
Freshwater					
Great Lakes					
Lake Erie	Cleveland Harbor, OH (CH)[c]	3	29	54	14
	Lorain/Toledo Harbor, OH (LTH)[c]	1	16	43	40
Mississippi River					
Mississippi Alluvial Valley	Horseshoe Lake, MS (Ref B)[c]	1	5	54	40

[a]Reference sites A (Ref A) and B (Ref B) have no known history of dredging activities

[b]Collected via split spoon

[c]Collected via Ekman dredge

[d]Silt and clay fractions combined

("Ref B"), a freshwater oxbow lake situated along the Mississippi River.

The extraction of microplastics was performed via density separation as described by Thompson et al. (2004) with modifications according to Hurley et al. (2018) and Coppock et al. (2017) to isolate microplastics between > 30 µm and 5 mm. Homogenized sediment from each location (n = 11 sites) was subsampled three times (50 g wet wt. each; n = 32 [minus a single subsample for Kings Bay]) and wet sieved (2 mm No. 10 sieve) to separate larger (i.e., 2 to 5 mm; only two microplastic particles were encountered large enough to be retained on the 2 mm sieve) from smaller particles (< 2 mm). Density separation was performed on the 50 g (wet wt.) sieved sediment by the addition of 250 mL of supersaturated solution of zinc chloride (1.4 g/cm^3) stirred with a stainless steel spatula for 1 min and allowed to settle for 24 h. After settling, the supernatant was decanted and vacuum filtered (0.45 µm). To increase recovery of microplastics, each sediment was subject to a second density separation extraction using the methods described above. After vacuum filtering, the filter paper was treated with Fenton's reagent (a mixture of H_2O_2 and iron (II) sulfate heptahydrate in reverse osmosis [RO] water) to remove readily oxidizable organic material (Hurley et al. 2018). Following the oxidation step, each sample was rinsed with RO water, vacuum filtered, and placed into a petri dish covered with aluminum foil and allowed to air dry prior to visual analysis.

Extraction efficiency was quantified following the same procedure by using sediment spiked with either polystyrene or polypropylene granules (n = 20–30) of irregular shapes and sizes (< 1 mm) and a translucent plastic microbead (n = 10; 100 µm ± 1.5 µm) reference standard (4000 series monosized particles, 4310A, ThermoFisher Scientific). The mean (n = 3) extraction efficiency of spiked sediment was 73% (± 7%) for the first extract and 24% (± 13%) for the second extract (i.e., 97% ± 6% particles recovered). To determine the potential influence of laboratory contamination (e.g., airborne), method blanks (n = 3) were conducted using the same separation procedure in the absence of sediment (i.e., zinc chloride only). Method blanks did not have appreciable occurrences of microplastics, with only two fibers and four fragments occurring across all six replicates (i.e., < 2% of average particle counts among sediment sites observed in this study).

For visual analysis of microplastics, a Leica stereomicroscope with up to × 40 magnification was used. Microplastic particles were categorized into five different types based on physical features: (1) spheres – round shaped; (2) fibers – length > diameter; (3) fragments – rigid, sharp crooked edges, irregular shape; (4) films – irregular shape, usually thin, flexible and transparent; and (5) foams – often white to yellow colored, soft, irregular shaped. To aid visual observations, suspected microplastics were tested with a cold and then flame-heated needle (plastic particles should melt upon contact with a hot needle). To identify the plastic polymer type and to confirm visual observations, a randomly selected subset of up to 15 microplastic particles of each shape category from each sediment location (n = 11) were analyzed using a Thermo Nicolet iS50 bench Fourier transform infrared (FTIR) spectrometer coupled to a Nicolet Continuum Infrared Microscope. In total, microplastic particles randomly selected from the 11 sediment samples for FTIR analysis consisted of 49 fibers, 5 films, 5 foams, 70 fragments, and 36 spheres, and ranged from 50 to 1400 µm in size. Sample analysis, data analysis, and microplastic FTIR spectra fitting was performed with Thermo OMNIC v8.1 series software. The chemical composition of the microplastics was determined via comparisons to the Hummel polymer sample library as provided in the Thermo OMNIC software and reported as percent match to a reference polymer.

Microplastic abundance was reported as the number of microplastic particles per kilogram of sediment (dry weight) for each sample site. Using R software (R Core Team 2018), microplastic abundance data were analyzed by parametric statistics after validating homogeneity of variances between the numbers of microplastic particles at each sample site determined by Levene's test. To compare statistical differences of microplastic numbers between sample sites, one-way analysis of variance (ANOVA) and Dunnett's post hoc test were used to compare each sample site (n = 9) with the two reference sites: Corpus Christi Reference marine site (Ref A); and Horseshoe Lake freshwater site (Ref B).

Results and Discussion

Microplastics were observed in all sediments examined including the freshwater and marine reference sites. Microplastics concentrations ranged from 162 to 6110 particles/kg dry wt. (mean = 1636 particles/kg dry wt.), with the highest concentration observed at Cleveland Harbor (mean = 5019 particles/kg dry wt.; s.d. = 966) and the lowest concentration observed at Southwest Pass (mean = 217 particles/kg dry wt.; s.d. = 94) (Fig. 1). Cleveland Harbor, Corpus Christi Harbor, Milford Harbor, Port Everglades, and Westport had a significantly greater mean number of microplastics ($p < 0.05$) as compared to the Corpus Christi Reference site (Ref A). Additionally, Cleveland Harbor and Corpus Christi Harbor had a significantly greater mean number of microplastics ($p < 0.001$) as compared to Horseshoe Lake (Ref B). A statistically significant difference as compared to a control does not imply a cause-and-effect relationship between sediments and the presence of microplastics, as there are limited data regarding the spatial and temporal extent of microplastics and varying waste management practices near each

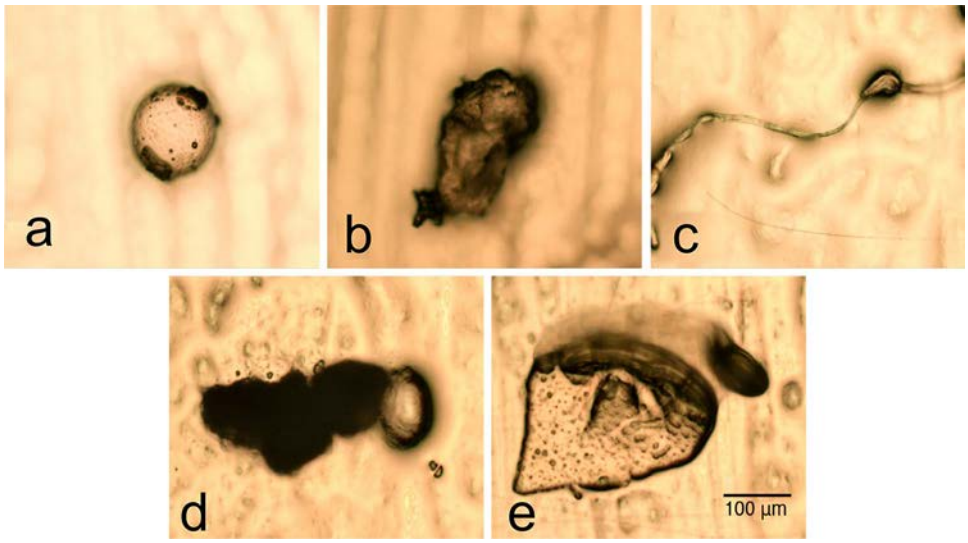

Fig. 1 Images of microplastic shapes observed in bottom sediments: sphere (**a**), fragment (**b**), fiber (**c**), foam (**d**), and film (**e**)

site. Additional study is required to infer the relationship between microplastics in sediments and their source.

Among all sites, five microplastic particle shapes (fibers, films, foams, fragments, spheres) were detected. Many of the particle shapes examined were less than 1.0 mm in size (Fig. 1). The vast majority of microplastic particles observed in these samples were visually highly weathered (based on coloration and shape), consistent with the images presented in the figure.

Fragments were the most frequently occurring particle observed in sediment subsamples (n = 32) examined (Fig. 2). Fragments occurred in 100% of samples followed by fibers (81%), spheres (75%), foams (38%) and films (34%). Based on percent composition among all subsamples, microplastic shapes were primarily fragments (54%) followed by spheres (22%), fibers (19%), foams (3%), and films (1%).

The average percent match of the microplastic spectral signature with the Hummel polymer sample library was 64% (s.d. 21%). The spectroscopic analysis detected the presence of 14 polymer types: aromatic hydrocarbon resin, polyacrylonitrile:MMA, polyethylene (PE), polyethylene:propylene (PEP), polyethylene:propylene:diene, polymethyl methacrylate, polystyrene (PS), polystyrene:4-vinylpyridine (PS type), polystyrene:acrylonitrile:MMA (PS type), polystyrene:vinylidene chloride (PS type), polyvinyl chloride, polyvinyltoluene:butadiene, polyester, and urethane. The majority of microplastics in study samples were identified as PEP and occurred at every site (n = 11 sites, 113 of 165 particles, mean 68%, range 40%–100%) followed by PS and PS types (n = 9 sites, 28 particles, mean 11%, range 7%–20%). Fibers (92%), films (100%), foams (100%), and fragments (74%) were mainly composed of PEP, while

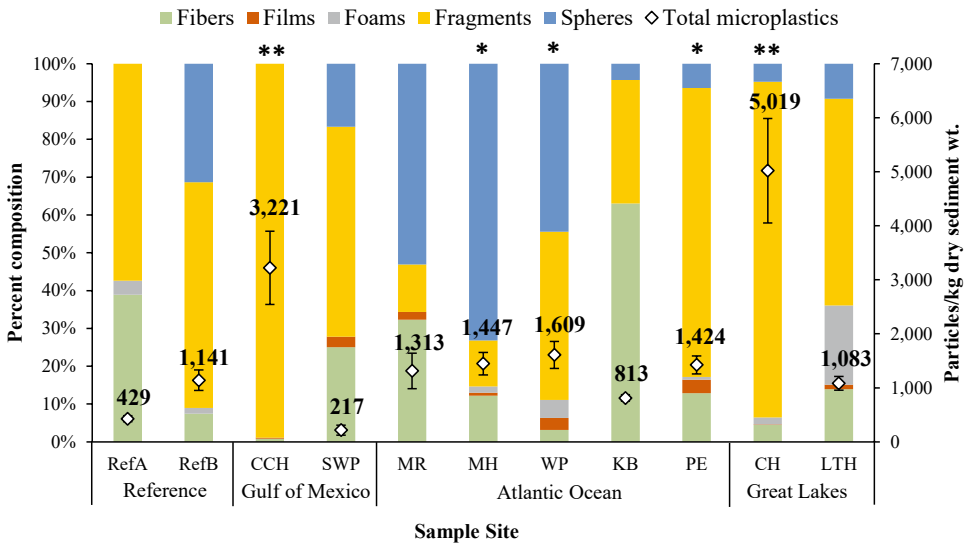

Fig. 2 Mean number of microplastic particles (particle/kg dry wt.) and percent composition of microplastic shapes (fibers, films, foams, fragments, spheres) for each sediment. Error bars indicate standard deviations. Sample sites with one asterisk (*) were significantly different ($p < 0.05$, $\alpha = 0.05$) from Ref A (marine) while sample sites with two asterisks were significantly different from both Ref A and Ref B

spheres were composed of mainly PS and PS types (58%) followed by PEP (22%). These FTIR results are comparable to those reported for microplastics in bottom sediments from similar studies in marine and freshwater environments and are consistent with the most widely produced plastics worldwide (e.g., polyethylene, polyvinyl chloride, polypropylene, polystyrene, and polycarbonate) (Andrady 2003).

Inherently, comparisons across studies reporting microplastics should be done with caution, as currently there is no standardized extraction and identification method. To simplify our comparison of microplastic abundances, we focused on comparing studies that used density separation as the primary extraction method and sediments collected in subtidal zones and inland waterways (e.g., bottom sediments that may be subject to relocation via dredging). In coastal areas around the world, microplastics are abundant in bottom sediments occurring in harbors, canals, estuaries, lagoons, and other nearshore areas. Studies in a lagoon in Tunisia (Abidli et al. 2017) and a nearshore area along the North Sea coast (Leslie et al. 2017) reported quantities ranging from 3305 to 7960 particles/kg dry wt., respectively, similar to those found at Corpus Christi Harbor, but were otherwise 3–7 times higher than concentrations of microplastics observed in the other sediments examined herein. Studies conducted in Durban Bay, South Africa (Naidoo et al. 2015) and Tokyo Bay, Japan (Matsuguma et al. 2017) reported quantities (1165 and 1845 particles/kg dry wt., respectively) similar to current study results. In contrast, studies in Tolo Harbor, Japan (Tsang et al. 2017), and harbors in Belgium (Claessens et al. 2011), reported quantities of 121 and 167 particles/kg dry wt., respectively, 10–15 times lower than those observed in this study. Few if any studies were found reporting miscroplastic concentrations in bottom sediments in US waters. Graham and Thompson (2009) reported an average of 113 particles/kg dry wt. for bottom sediments collected off the coasts of Florida and Maine (converted from mL to kg/dry wt.). Limited information that was found in freshwater environments also showed the presence of microplastics in bottom sediments in both lacustrine and riverine environments worldwide. In Canada, microplastic concentrations in bottom sediments from Lake Ontario (Corcoran et al. 2015; Ballent et al. 2016) and the Ottawa River (Vermaire et al. 2017) ranged from 220 to 921 particles/kg dry wt. Castañeda et al. (2014) reported an average of 13,759 particles/m^2 in St. Lawrence River sediments. The available data reported for bottom sediments indicate that microplastics are ubiquitous in both marine and freshwater environments.

The sediments collected in this study were from inland and intracoastal waterways, coastal channels, ports, harbors, and turning basins, which encompass a diverse array of highly modified depositional environments. These areas are part of major waterborne transit routes for commodities and support many industrial activities commonly associated with higher population densities, all of which influence the presence of microplastics in bottom sediments. Consistent with results presented by other investigators from the US and other regions, microplastics were present at similar concentrations in all bottom sediments in this study and the polymer composition was aligned with the most widely used plastics worldwide. These data provide additional evidence as to the ubiquitous nature of microplastics in bottom sediments; bottom sediments in waterways targeted for dredging show similar concentrations.

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14. ABSTRACT

Given the reported extent of microplastics in the aquatic environment, environmentally relevant exposure information for sediments dredged by the US Army Corps of Engineers will lend context to the risks posed by this contaminant during dredging. We measured the occurrence, abundance, and polymer composition of microplastics in sediments collected from nine dredged waterways and two non-dredged reference areas. The number of particles in sediment samples ranged from 162 to 6110 particles/kg dry wt., with a mean of 1636 particles/kg dry wt. Fragments were the most prevalent shape observed among the 11 study sites (100% frequency of occurrence), followed by fibers (81%), spheres (75%), foams (38%) and films (34%). Based on analyses of chemical composition of the particles using Fourier transform infrared spectroscopy, polyethylene:propylene was the most common polymer type observed. Consistent with results presented by other investigators microplastic concentrations and polymer types in bottom sediments in this study were also aligned with the most widely used plastics worldwide.

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