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Distribution, abundance, and diversity of microplastics in the upper St. Lawrence River☆

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ABSTRACT

Microplastics are pervasive pollutants in fresh waters, but their distribution, abundance, and diversity in fluvial environments remain poorly documented. Previous research indicated that large polyethylene microbeads were abundant in the freshwater sediments of the St. Lawrence River. Here we extend this work by quantifying the abundance of a broad range of sizes and types of microplastics in sediments and surface water samples, and we relate these metrics to environmental variables. We sampled 21 sites for sediments that spanned a land use gradient, and 10 surface water stations above and below wastewater effluent sites, along the fluvial corridor of the St. Lawrence River between Montreal and Quebec City from July to August 2017. Microplastics were removed from sediments using an oil extraction protocol and enumerated under fluorescent microscopy. We tested predictions that environmental filters and known point sources affect microplastic concentrations in the river. The mean concentration of microplastics across all sediment sampling sites was 832 (±150 SE) plastics per kg dry weight (range 65–7562 plastics per kg dry weight), which is among the highest recorded (in the top 25%) for the world's freshwater and marine systems. Microplastic concentrations in the sediments were significantly related to a suite of environmental variables including land use and sediment particle characteristics. Particle characteristics, proximity to point sources (urban land use), and environmental filters (sediment compositional variables, % organic carbon, % inorganic carbon and distance from shore) each explained a significant fraction of variation in the microplastic composition in the sediment, with environmental filters having the greatest influence. We present a protocol that could be used to efficiently and accurately detect a broad range of microplastics until a standardized protocol is established for large-scale monitoring.

1. Introduction

Over the past 50 years, annual plastic production has grown 20-fold from 15 million tonnes to nearly 300 million tonnes (Plastics Europe, 2018). Plastic debris is accumulating rapidly in the natural environment (Corcoran et al., 2014) and is now recognized as a global contaminant with potential impacts on ecosystems, food security and human health (Derraik, 2002; Thompson et al., 2009; Law, 2017). In particular, there is growing public and scientific concern surrounding the pervasiveness and effects of microplastics: synthetic polymer particles ranging from 1 μm to 5 mm in size, which typically occur in the form of beads, fragments, fibres and foams. They are manufactured for use in industrial and biotechnological applications, and as components in pharmaceuticals, cosmetics and personal care products, but are also produced from the breakdown of larger plastic debris (Law & Thompson, 2014).

Diffusing from terrestrial sources mainly through flowing waters (Lebreton et al., 2017), microplastics are ubiquitous within aquatic environments. They have been discovered on virtually every beach sampled worldwide (Andrady, 2011; Cole et al., 2011; Corcoran et al., 2015; Nelms et al., 2017), in ocean surface waters (Eriksen et al., 2014), in deep sea sediments (Van Cauwenberghe et al., 2013; Woodall et al., 2014), and within Arctic sea ice (Obbard et al., 2014). In rivers, they accumulate in sediments (Castañeda et al., 2014; Klein et al., 2015) and can outnumber fish larvae within the water column (Lechner et al., 2014). The

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ecological implications of the widespread influx of microplastics have begun to emerge. Researchers have documented the consumption of microplastics by over a hundred species of invertebrates and vertebrates (GESAMP, 2016; Auta et al., 2017) – probably a miniscule fraction of the actual number of organisms that ingest the material. Heavy ingestion of microplastics carries potential energetic and toxicological costs (Wright et al., 2013; Rochman et al., 2013), especially as plastic surfaces readily adsorb contaminants, including toxic metals (Ashton et al., 2010; Holmes et al., 2012; Nakashima et al., 2012) and persistent organic pollutants (Frias et al., 2010; Rochman et al., 2013). Through trophic interactions, these particles and their contaminant burdens can be transferred to higher levels of the food web (Setälä et al., 2014). Accurate risk assessment of exposure to this burgeoning form of pollution requires a comprehensive understanding of the types, diversity, distribution, and sources of microplastics in the aquatic environment.

The presence of microplastics in marine systems has been well recognized for over a decade (e.g. Andrady, 2011; Cole et al., 2011; Do Sul and Costa, 2014; Auta et al., 2017), but the vast majority of studies on freshwater systems have emerged just within the past few years (Hurley et al., 2018). The fate of microplastic particles in riverine environments is likely to become a fertile area of research, as rivers are governed by unique physical, chemical, and biological processes whose roles in dispersing plastic particles remain largely unstudied. The few studies that have examined patterns of microplastic distribution and abundance within river systems have related these patterns to sources of microplastics, such as proximity to industrial areas, areas with high urban density, and wastewater effluents (e.g. Mani et al., 2015; Baldwin et al., 2016; Leslie et al., 2017; Rodrigues et al., 2018; Peng et al., 2018). However, high concentrations of microplastics have been discovered in the sediments of rivers in remote areas that lack such sources (Klein et al., 2015). Little attention has yet been given to the role of environmental filters in governing the distribution, abundance, and diversity of microplastics in rivers. Early evidence from modelling studies suggested that fate of microplastics are controlled by particle characteristics and hydrodynamics (Nizzetto et al., 2016; Besseling et al., 2017), but the results are equivocal when examined empirically. For example, Nel et al. (2018) found that water flow, substrate type and sediment organic matter may determine microplastic distribution within the sediments of a South African river; conversely, Vermaire et al. (2017) found that sediment organic matter and substrate grain size were not significant predictors of microplastic abundance in a Canadian river. A better understanding of which environmental variables best predict the where, how many, and what types of microplastics are found in rivers is crucial for adapting environmental monitoring and risk assessment of this form of pollution.

The St. Lawrence River is a large urbanized watershed that offers important information regarding the extent to which such ecosystems are contaminated by microplastics and the factors that govern their distribution and local abundance. The river is likely a major conduit for the transport of plastic from urban centers in Lake Ontario and the Island of Montreal downstream to the marine environment. To date, one study has reported on the abundance of microplastics in the St. Lawrence River (Castañeda et al., 2014) and found large polyethylene microbeads (400–2000 μm diameter) widely distributed in sediments across the river bed, in concentrations of thousands of microbeads per square meter at one site – a magnitude that rivals some of the world's most contaminated sediments in both freshwater and marine systems (Hurley et al., 2018). However, Castañeda et al. (2014) focused exclusively on microbeads, thus the extent to which the St. Lawrence River is contaminated with microplastics of various types remains to be determined.

Here, we tested factors affecting the distribution, abundance, and diversity of microplastics within the sediments and surface waters of the upper St. Lawrence River. We hypothesized that local microplastic concentrations within the sediments are correlated with site characteristics (sources and filters) such that depositional areas and areas with high urban land use will yield the highest concentrations.

2. Materials & methods

2.1. Selection of sediment sampling sites

Accounting for land use types, a GIS-based site selection technique was employed to choose sediment sampling sites *a priori* across the length of the river using ArcGIS 10.5. To do this, a 30-m resolution raster land cover map that contained 130 distinct land cover classification types was used (MDDELCC, 2016). Using the 'reclass' tool in ArcGIS, these land cover types were reclassified into 8 groups: urban areas, agricultural areas, natural areas, industrial areas, grassland areas, bare ground areas, other (snow & clouds), and water. Once reclassified, 100 random sampling points were placed on the St. Lawrence River and a 5 km buffer was placed around each point. Using the 'extract by mask' tool, each buffer zone extracted the land cover in the area. The exact sampling location was then determined by placing the point at the most downstream portion of the buffer zone to encapsulate the land cover 10 km directly upstream from the sampling location. From these 100 random points, 24 sampling locations were chosen that best represented the diversity of land cover on the river and were divided into six land cover types: urbanized areas (>50% urban land cover), agricultural areas (>50% agriculture land cover), forested areas (>50% forested land cover), agricultural & urban mix (>30% agricultural and urban land cover), agriculture and forested mix (>30% agricultural and forested land cover) and a mixed area (>20% urban, >20% agriculture, >20% forested land cover). In addition to the 24 sediment sites determined by land cover, one site (Gentilly-2 power plant) was added to the study because it contained the highest density of microbeads found in the study conducted in 2014 (Castañeda et al., 2014), resulting in 25 total sediment sample locations (Fig. 1).

2.2. Sediment sampling

The fluvial section of the upper St. Lawrence River from Ile de Salaberry to Quebec City was sampled during July and August 2017. At each location three petite Ponar grabs (15 cm × 15 cm area, 2.4 L volume) were taken and a series of physical and limnological variables were measured, at sites ~100 m apart. Each sediment grab sample was transferred into a clean capped bucket (opened only at the time of sampling) and transported back to the lab for analysis. The samples were left to rest for 12–24 h to allow for the sediment to settle to the bottom of the bucket. Once settled, the excess water was siphoned from the bucket. The total volume of the sample was measured, the sample was mixed, and a 250 mL subsample was taken for the analysis of microplastics. The remaining sediment was transferred into WhirlPak bags for storage in a refrigerator at 4 °C.

2.3. Laboratory protocol for the extraction of microplastics from sediment

Sediment samples were wet sieved into 8 different sediment size fractions according to the Wentworth scale (Wentworth,

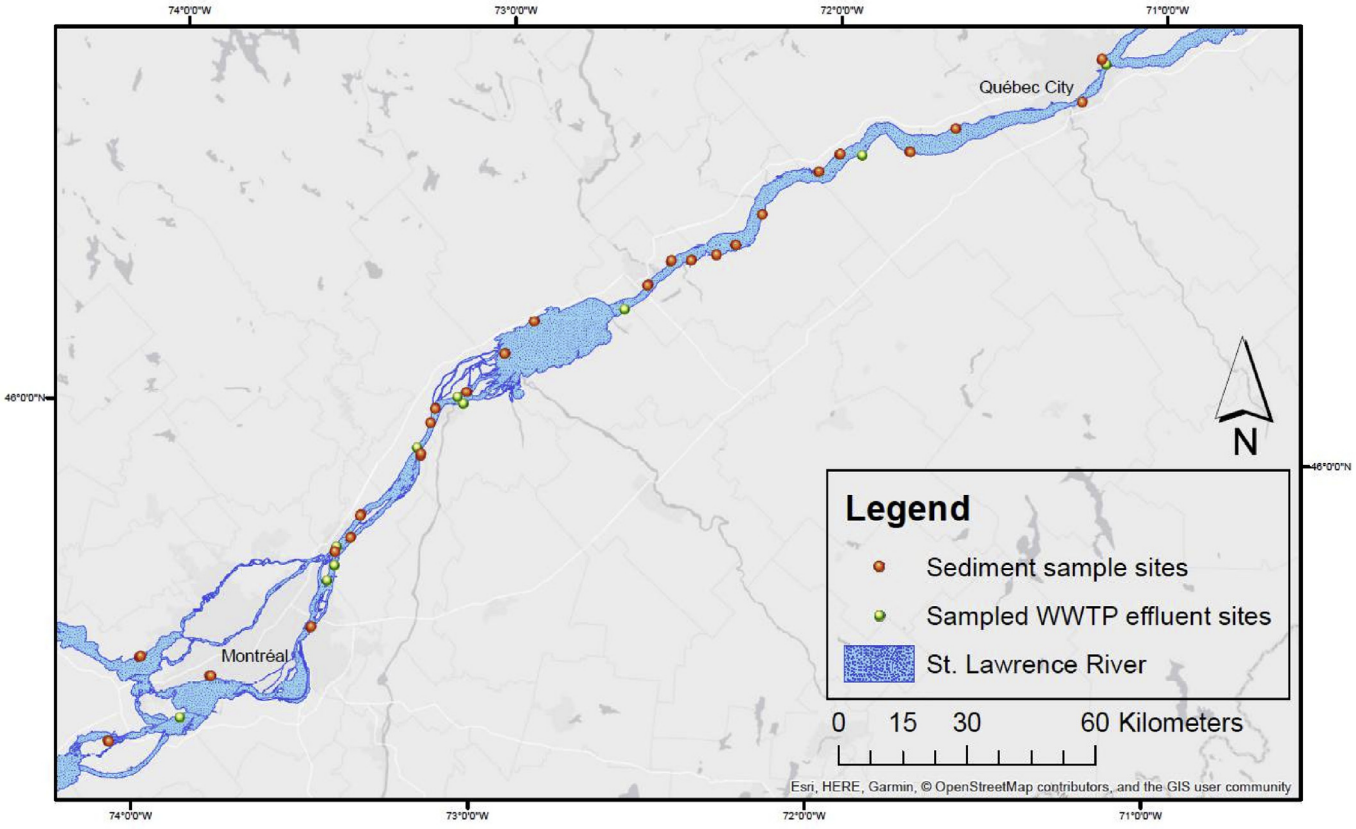

Fig. 1. Map of the upper St. Lawrence River indicating locations where sediment samples and surface water samples were collected.

1922): clay (<4 μm), silt (4–63 μm), very fine sand (63–125 μm), fine sand (125–250 μm), medium sand (250–500 μm), coarse sand (500–1000 μm), very coarse sand (1–2 mm), and very fine gravel (2–4 mm) and fine gravel (>4 mm). The clay and silt fractions were combined as clay/silt (1–63 μm). Once sieved, each size fraction was transferred into aluminum trays and dried in a drying oven at 55 °C. All dried material for each retained size fraction was weighed to determine the dry weight of the sediment and the proportion of sediment in each grain size category. Each size fraction was then transferred into WhirlPak bags to be stored in the laboratory before undergoing microplastic extraction.

2.4. Microplastic extraction

Microplastics were obtained from each size fraction of sediment using a canola oil extraction method following Crichton et al. (2017); this method was chosen because it is independent of plastic density, had the shortest processing time, and resulted in a lower cost per sample compared to other extraction methods. Since each sediment sample contained eight different size fractions, it was determined that the six lower size fractions would undergo this extraction, and the two remaining size fractions (fine gravel, very fine gravel) would be examined for microplastics under a light stereoscope (Leica MS5).

The sediment size fractions chosen for extraction were transferred from the WhirlPak bags into a 250 mL Erlenmeyer flask. Since the protocol outlined by Crichton et al. (2017) is designed for a maximum of 50 g dry sediment, size fractions were split if they were greater than 50 g, with a maximum of 6 extractions for one size fraction (up to 300 g). If a size fraction was larger than 300 g the sample was not included in the analysis (n = 1). Following extraction, all retained material within the oil layer was filtered through a borosilicate filter (1 μm pore size) using a vacuum pump. Once the oil had passed through, 30 ml of methanol was added to the sample and filtered to remove any remaining oil residue. The retained material was then backwashed and transferred into a 20 mL scintillation vial, which was subsequently placed in a drying oven at 55 °C to evaporate the excess water added during the backwashing step.

2.5. Surface water sampling and processing

Ten wastewater effluent sites within the upper St. Lawrence River were sampled (Fig. 1). For water collection, the bottle sampling method developed by Vermaire et al. (2017) was applied in which 4 L acid-washed plastic jugs were used to collect water at a depth of 0–5 cm a total of 25 times to filter 100 L of water through a new piece of 100 μm nylon mesh. This procedure was repeated three times at each location, including one located 500 m upstream and another 500 m downstream of the effluent. After each 100 L sample of water was filtered, the filters were carefully removed from the plastic cylinder, placed directly into a Whirl-Pak bag, and sealed. Samples were then transported to McGill University where they were kept at −20 °C until processed in the laboratory. Sampling blank samples were collected to account for any plastic particles added during the sampling procedure (see procedure verification and contamination). Microplastics were extracted from water samples using the same extraction protocol applied to the sediment samples (Crichton et al., 2017).

2.6. Sample digestion and staining

Owing to the large size of our samples, organic digestion was applied after oil extraction, thus digesting only the material retained on the filters. The same organic digestion process was applied for both water and sediment samples. Each vial received 10 ml of 30% hydrogen peroxide, and then the samples were left to digest for 24 h at room temperature before undergoing an 8-h heat treatment at 55 °C. Once digested, the samples were transferred back onto a borosilicate filter (pore size 1 μm) by vacuum filtration and prepared for analysis.

To quantify the microplastic particles retained on each filter, Nile Red staining and fluorescent microscopy was applied – a technique that has been deemed to accurately quantify microplastics from environmental samples owing to its ability to differentiate between microplastic and natural particles (such as silica fragments or natural fibres) and has been validated to be similar in accuracy to FT-IR and Raman Spectroscopy (Shim et al., 2016; Maes et al., 2017; Erni-Cassola et al., 2017; Catarino et al., 2018). The staining method followed the protocol of Erni-Cassola et al. (2017) where the filters are dyed with a few drops of Nile Red dissolved in 95% methanol (at 1 μg/mL), and then filters were covered with a glass cover slip to protect samples from airborne contamination. Following staining, microplastics were analyzed under a fluorescent compound microscope (Olympus BX43), coupled with a GFP filter (excitation max at 490 nm and emission max at 525 nm) at 100 × magnification.

2.7. Microplastic identification & enumeration

Microplastic identification followed a protocol that identifies plastic particles depending on the intensity of fluorescence (high, moderate, none), colour and plastic type. High-fluorescent particles are those that surpass a pixel brightness threshold in ImageJ outlined by Erni-Cassola et al. (2017) and represent four plastic polymers: polyethylene, polypropylene, polystyrene and Nylon-6. However, not all plastic particles fluoresce or are able to surpass the pixel brightness threshold (Erni-Cassola et al., 2017). As such, for moderately and non-fluorescent particles, identification trees for individual plastic forms (fibres, fragments and microbeads) were developed to be used in combination with the fluorescence microscopy to differentiate plastic particles from other hydrophobic particles (Figs. S1–S4). The identification trees were built using characteristics described in the literature (Norén, 2007; Nor & Obbard, 2014; Eerkes-Medrano et al., 2015; Cheung et al., 2016; Catarino et al., 2018) to aid in differentiating a plastic particle or fibre from those that are naturally occurring or are semi-synthetic (e.g. silk, cotton). This resulted in plastics being quantified into three groups: those that are highly fluorescent (i.e. surpass the pixel brightness threshold), those that were moderately fluorescent but identified as plastic owing to their physical characteristics, and non-fluorescent particles that were identified owing to their physical characteristics.

Microplastics of each size fraction were pooled by plastic type (fibre, microbead, fragment, film, foam) and level of fluorescence (high, moderate and none). Very small (<10 μm) particles that could not be identified were classified as unidentifiable fragments. Microplastic counts were then adjusted by the retention rate of the spiked plastics within the sample to give the estimated count for each sample.

2.8. Procedure verification and contamination

Verification of the laboratory procedure was assessed by spiking the samples with five different plastic types to determine the retention rate. Samples were spiked with known microplastics of varying densities and included PVC fragments (1.3–1.45 g/cm^3), nylon fibres (1.11–1.18 g/cm^3), polyester fibres (1.39–1.44 g/cm^3), polyethylene microbeads (0.91–0.94 g/cm^3) and polypropylene fragments (0.91 g/cm^3). All spiked microplastics were virgin and easily distinguished from environmental microplastics owing to their bright colour and/or lack of weathering.

A variety of steps were taken to mitigate possible contamination of samples. All microplastic extractions were completed under a laminar flow hood. We used only reverse osmosis water during sample extractions and washed all glassware and tools between samples with soap and water, and then rinsed thoroughly to ensure all soap was removed. In addition, work surfaces were regularly wiped with distilled H_2O and then 70% ethanol. Cotton lab coats were worn at all times, and only glass or stainless-steel tools were used during sample processing in the lab. All reagents and solutions were filtered through a borosilicate filter (1 μm pore size) and stored in clean glass containers prior to use. Samples were kept covered with aluminum foil. To quantify potential contamination of samples from microplastics present within the laboratory or between samples, both procedural and contamination blanks underwent the full laboratory procedure. For both the water and sediment samples, procedural blanks were run in parallel after every 7–8 samples (10%) to account for potential cross contamination during the extraction procedure and provided a measure of any contamination from reagents and equipment. The contamination blanks, which quantified environmental microplastics present within the lab, were obtained by placing wet borosilicate filters near all work not undertaken under the laminar flow hood. Plastic totals quantified in environmental samples were adjusted by removing the number of plastics observed in the contamination blanks.

2.9. Statistical analysis

All statistical analysis of the data was undertaken in R version 3.5.1 (R Core Team, 2018), with the exception of our calculation of median Phi scores for grain size analysis. We used the GRADISTAT particle size analysis software (Blott et al., 2001) to calculate median particle size statistics for sieved samples.

For each sampling site, the concentration of microplastics in surface water and sediment was calculated by dividing the number of identified microplastic particles by the total volume of water (L-1) measured and the dry weight (kg^{-1} dry weight) of the sediment sample, respectively. Of the 25 sediment sites sampled, we excluded four sites (bringing our total number to 21) because the sample volume was too large (n = 1), we had poor (<10%) retention rates (n = 2), or the sample was lost during sample processing (n = 1). The retention rate for sediment samples varied among samples and microplastic types; the mean retention rate was 67% ± 2.3 (SE) for fibres, 63% ± 3.5 (SE) for microbeads, and 61% ± 2.2 (SE) for fragments. For all data analyses, results are expressed as corrected microplastic concentrations, such that we re-scaled data to account for variation in retention rates.

Microplastic particles were sorted into five categories (fibres, films, foams microbeads and fragments) for which separate concentrations were determined. Variation in these concentrations were related to a suite of environmental variables using multivariate canonical ordination (redundancy analysis, RDA) using the rda function from the {vegan} package in R (Oksanen et al., 2017). Before applying the RDA, concentration data were Hellinger transformed (Legendre and Gallagher, 2001), and all environmental variables were Box-Cox transformed (Sakia, 1992) to eliminate the influence of extreme values on ordination scores and normalize the

Table 1
Table outlining the categories of predictors and rationale for their inclusion in this study. Bold environmental predictors were chosen to be analyzed in the global model (passed assumptions) and the asterisk represent predictors that were chosen through forward selection.

Predictor group	Environmental predictors	Rationale (point source or environmental filter)
Land use	Land use classes: **Urban***, **Agricultural, Natural, Industry, Grassland,** Bare ground, Other, Water	Point source of plastic: Urban, industry and agricultural land use. Background reference sites: grasslands and natural areas.
Spatial variables	**Asymmetic Eigenvector Maps (AEM)**	Used to examine a unidirectional effect (increase in MP with distance downstream).
Sediment variables	**% Fine Gravel,** % Very Fine Gravel, %Very Course Sand, **% Course Sand**, **% Medium Sand***, **% Fine Sand, % Very Fine Sand, % Silt & Clay***, **Median Phi score***	Environmental filter: Dynamic environments characterized by higher % of gravel and coarse sand. Depositional environments: characterized by higher % of silt and clay.
Organic content	**% Organic content***, **% Inorganic content***	Environmental filter: Depositional environment are typically characterized by high organic content. Organic content could aid in depositing microplastics. Flat river-bottom: typically characterized by limestone bedrock which contains high inorganic content; could facilitate organic production. Moreover carbonates are heavy and biofilms could promote calcite precipitation on microplastics causing them to settle.
Physical variables	**Depth, Secchi Depth, Distance to shore***, **Distance to WWTP**	Environmental filter: Hydrological variables Point source: Distance to WWTP
Riverine variables	**Avg. velocity 1m (m/s),** avg. velocity 2 m (m/s), avg. velocity 3 m (m/s), **min. velocity 1m (m/s),** min. velocity 2 m (m/s), min. velocity 3 m (m/s), max velocity 1 m (m/s), max velocity 2 m (m/s), max velocity 3 m (m/s), Specific conductivity (s/m) (top), **Specific conductivity (s/m) (bottom)**	Environmental filter: Hydrological variables; environmental chemistry

environmental variables.

To determine the factors that best explained microplastic abundance, diversity and distribution, 35 environmental variables were chosen; these represented potential sources, hydrological filters, and limnological filters (Table 1). Environmental variables that were not normally distributed or with high correlation coefficients ($r > 0.75$) were excluded in the final RDA analysis, leaving 22 variables to be analyzed in the global model. Removal of colinear variables was done to ensure that the model was stable (Borcard et al., 2018). To determine the most parsimonious set of predictors for each dataset, forward selection of environmental variables was applied using the Blanchet et al. (2008) double stopping criterion. When more than four environmental variables were selected, they were grouped based on the type of proxy they represent (e.g., environmental filter, point source; see Table 1) and analyzed in variance partitioning analysis (Borcard et al., 1992).

To determine if the concentrations of microplastics in water are different between sampling sites located, respectively, 500 m upstream and 500 downstream of wastewater effluents, we conducted paired t-tests for each individual effluent station. Additionally, a linear mixed effect model was developed to assess whether microplastic concentrations can be predicted by relative distance from the effluent location (i.e. upstream or downstream):

$$MPconcentration \sim sampling\ location + (1 + sampling\ location \mid effluent\ location) + \varepsilon$$

In this model, sampling location (either upstream or downstream) is the fixed effect, whereas effluent location in the river is the random effect, allowing us to examine if the intercepts and slopes of the microplastic concentration and sampling location relationship varied by effluent location.

3. Results

3.1. Sediment samples

Microplastics were found in all sediment samples. After adjusting for particles found in the contamination blanks (Supplementary data, Table S1), the mean and median values (±SE) across all sediment sampling sites were 832 ± 150 and 429 ± 188 particles per kg dry weight, respectively. Across all sites, concentrations ranged from 65 to 7562 plastics per kg dry weight. Microbeads were the most abundant type (489 ± 120 microplastics per kg dry weight), followed by fragments (220 ± 81) and fibres (122 ± 18). Small microbeads (<400 μm) constitute ~95% of identified microbead types. Microplastic abundance was very heterogenous across the sediments with concentrations varying in magnitude even within the same sampling site (Fig. 2). The highest concentration of microplastics was found in a site just downstream of the Island of Montréal largely dominated by unidentifiable (<10 μm) highly fluorescent fragments and small moderately fluorescent microbeads (Fig. 2).

The complete suite of environmental variables explained 34.3% of the variance in plastic composition (RDA, $p = 0.002$). Seven of the 22 variables that were chosen through forward selection (Table 2) accounted for over three-quarters of this explained variance (26.7%; $p = 0.001$) (Fig. 3). Fibres were strongly associated with variables characterizing depositional environments (e.g. % organic carbon, % silt & clay), whereas fragments were associated with areas of high urban land use, and microbeads were associated with greater proportions of medium-sized sands (250–500 μm). After dividing variables into environmental filters and microplastic sources, variance partitioning analysis showed that the former explained 22.6% ($p = 0.021$) whereas the microplastic sources (urban land cover) explained 15% ($p = 0.037$) of variance (Fig. 4).

3.2. Water samples

After the removal of extremely high measurements (one upstream and one downstream, but not from the same effluent site), the mean and median values (±SE) of microplastic particles across all sampling sites were 0.12 ± 0.01 and 0.12 ± 0.01 microplastics per litre upstream and 0.16 ± 0.02 and 0.16 ± 0.02 microplastics per litre downstream of wastewater effluents, respectively (Fig. 5). In only one case out of the ten wastewater effluents was the average microplastic concentration significantly higher downstream compared to upstream of the effluent site ($p = 0.024$); otherwise (Fig. 6), there were marginal differences in mean concentrations of microplastics when comparing all upstream and downstream concentrations ($p = 0.06$; Table 3). With linear mixed effect modelling, we found that the model treating the upstream vs

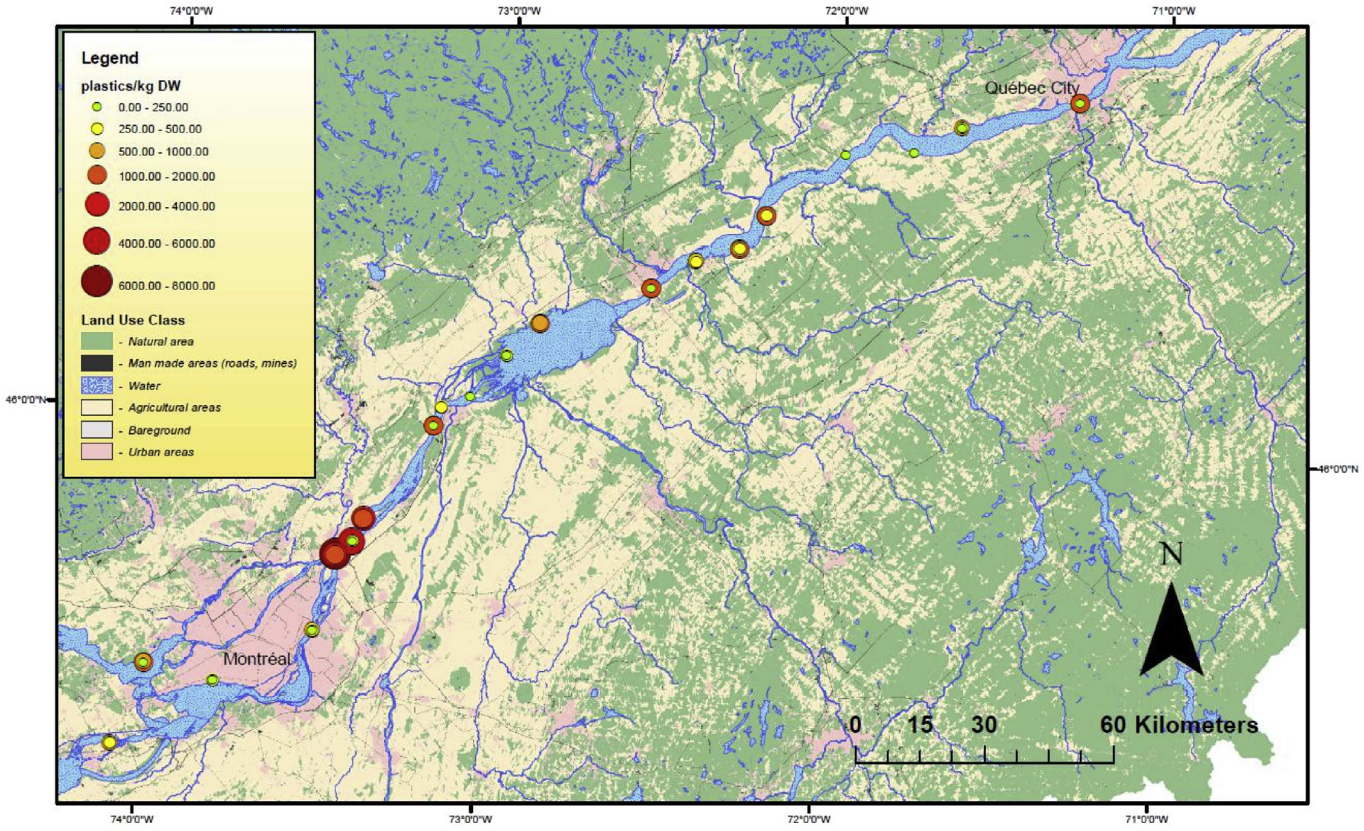

Fig. 2. Density of microplastics (plastics/kg DW) in the sediments collected across our network of sites. At each site, three samples were taken; circle size and colour indicate the concentration of microplastics in each of these samples. All density measurements have been adjusted based on laboratory retention rates. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Table 2
Seven environmental variables were identified with forward selection when examining microplastic composition. Significant values are bolded.

Variable	Adjusted R2	Adjusted R2 Cumulative	F-value	p-value
% Inorganic Carbon	0.05	0.05	3.916	**0.041***
% Urban land use	0.04	0.09	3.444	**0.048***
% Medium sand	0.03	0.12	3.167	**0.045***
%Organic Carbon	0.04	0.16	3.801	**0.045***
Distance to shore	0.04	0.20	3.583	**0.044***
Phi score	0.04	0.24	4.330	**0.026***
% Silt & Clay	0.03	0.27	3.066	**0.047***

downstream location identity of the sample as a fixed effect and wastewater effluent location as a random effect had higher predictive power than the linear model; however, the slope from the linear mixed effect model was not significantly different from zero at the 0.05 level (Fig. S5).

4. Discussion

4.1. Concentration of microplastics in the sediments

In comparison to other studies using similar metrics, the mean concentration of microplastics in the St. Lawrence River was among the highest recorded for freshwater and marine systems globally (Fig. S6). The mean concentration is of the same order of magnitude as those measured in highly contaminated rivers and lakes near densely populated cities in China (Peng et al., 2018; Wen et al., 2018). Within the eastern Canadian region, concentrations in the

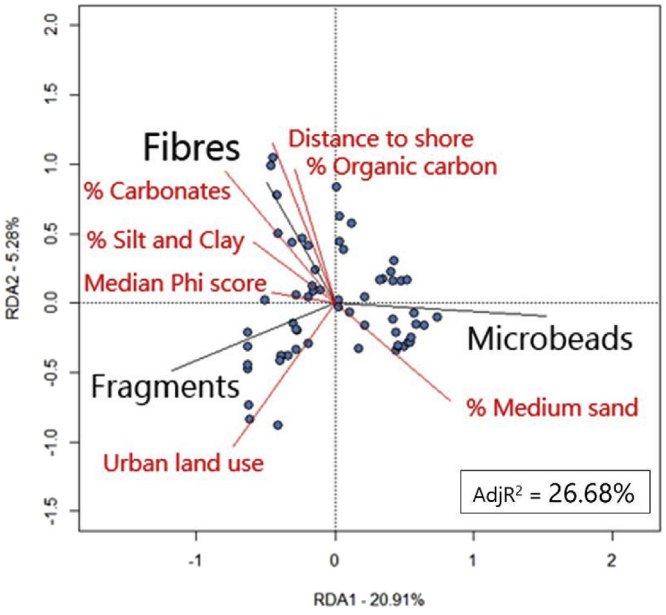

Fig. 3. An ordination biplot of environmental variables and microplastic taxa obtained by RDA (black arrows depict plastic types and red arrows depict environmental predictors). Fibres were found to be associated with variables commonly linked with depositional environments, whereas fragments were found in areas of high urban land use. Microbeads were associated with medium sized sand (250–500 μm), typical of dynamic areas. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

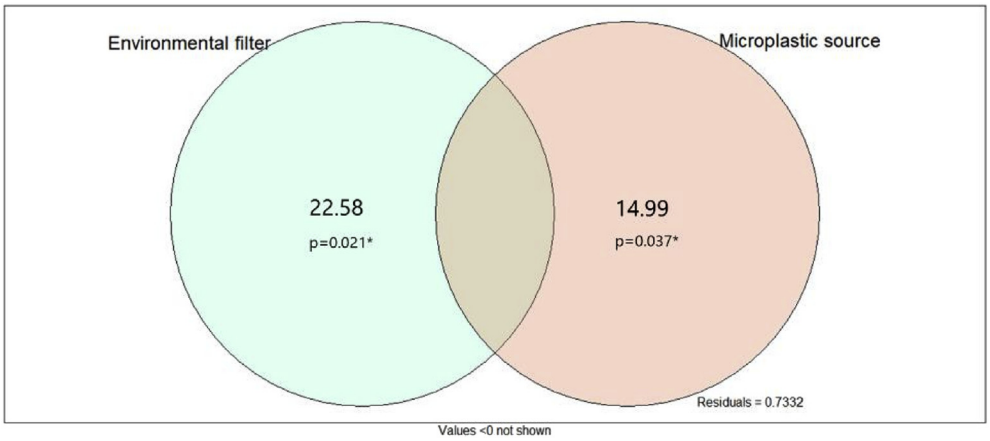

Fig. 4. Variance partitioned into two groups; environmental filter (Phi score, % medium sand, % silt and clay, % carbon, % inorganic carbon and distance from shore) and microplastic source (% urban land use). Both groups explained a significant portion of the variance ($p < 0.05$).

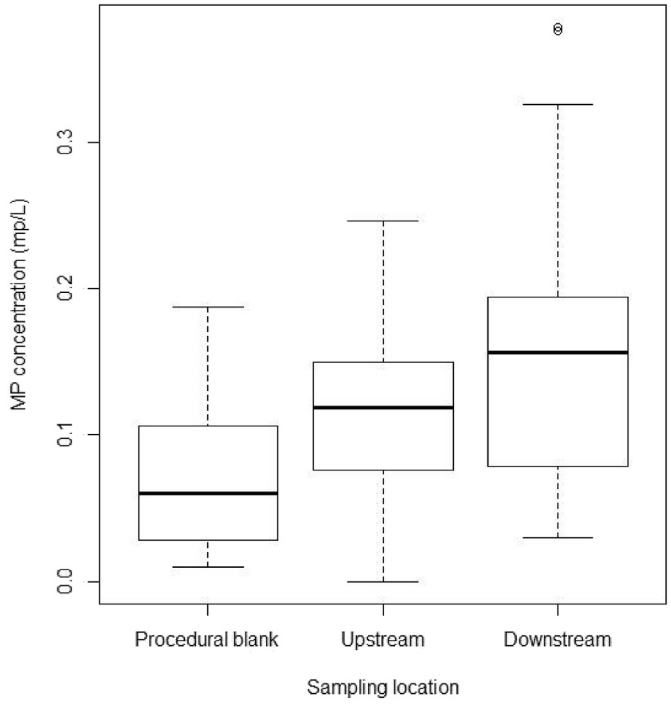

Fig. 5. Boxplots displaying concentration of microplastics in procedural blank, upstream and downstream samples. Procedural blank samples contained a mean and median concentration of 0.075 ± 0.02 and 0.06 ± 0.03 microplastics per litre.

St. Lawrence River are slightly higher than those measured in the Lake Ontario tributaries (610 items·kg^{-1}) (Ballent et al., 2016) and nearly four times higher than in the Ottawa River (220 items·kg^{-1}) (Vermaire et al., 2017).

Concentrations of microplastics measured in this study were generally higher than those previously recorded in 2013 by Castañeda et al. (2014), who found large microbeads (>400 μm) along the upper St. Lawrence River at a median concentration of 52 microbeads·m^{-2}, whereas our median concentration was 2240 microbeads·m^{-2}. Castañeda et al. (2014) also found an extreme local concentration of 1.4×10^5 microbeads·m^{-2} at one site near the Gentilly-2 powerplant. In our study, concentrations of microplastics varied from 1554 to 2.66×10^5 items·m^{-2} with mean and median concentrations of 2.2×10^4 items·m^{-2} and 8280 items·m^{-2}, respectively. However, it is worth noting that our methodology allowed us to identify a broader variety of microplastics including those present in the organic-rich fraction of the sediment. For example, 95% of the microbeads in our study were smaller than the lower size limit of 400 μm used by Castañeda et al. (2014). We did not find the same level of abundance of microbeads at the Gentilly-2 site as did Castañeda et al. (2014). To test whether this difference was due to the organic digestion step in our methodology (cf. Munno et al., 2018), we applied the same method used in Castañeda et al. (2014) on untreated sediment samples collected in 2017 from the Gentilly-2 site and confirmed a high concentration of larger microbeads at this site (216 microbeads·kg^{-1} dry weight or 1.85×10^4 microbeads·m^{-2}). Presuming that this large fraction of microbeads was excluded from our results across sampling sites owing to our procedures, the concentrations reported here should be considered conservative. Further investigations are required to determine the effect of different methodological procedures on the estimated abundances of microplastics.

As has been observed recently in other lotic systems (Ballent et al., 2016; Peng et al., 2018; Wen et al., 2018), St. Lawrence River sediments contained more fragments and microbeads than fibres. Given that fibres are often comprised of polymers that are denser than water, they remain in suspension in turbulent mixing systems but will sink in calm open waters (Cable et al., 2017), and thus tend to dominate lake and marine bottom sediments (Fischer et al., 2016; Pagter et al., 2018; Abidli et al., 2018; Mu et al., 2019). Indeed, microplastics of fibrous shapes exhibit a slower settling velocity than fragments and spheres (Khatmullina and Isachenko (2017).

4.2. Factors that govern microplastic abundance and distributions

To assess the risk of exposure to biota and to determine the efficacy of policy interventions, biomonitoring programs need to 1) focus their sampling effort on habitats where microplastic abundance is maximal, and 2) consider factors that govern temporal and spatial variability of microplastics in aquatic environments (Syberg et al., 2015; Koelmans et al., 2017). Previous research suggested that particle distribution is governed by proximity to point sources (Lechner et al., 2014; Mani et al., 2015; Ballent et al., 2016; Leslie et al., 2017) as well as by particle characteristics, topography, water flow, water depth, organic content and substrate type (Castañeda et al., 2014; Eerkes-Medrano et al., 2015; Nel et al., 2018). In our study, over a quarter of the variance in microplastic

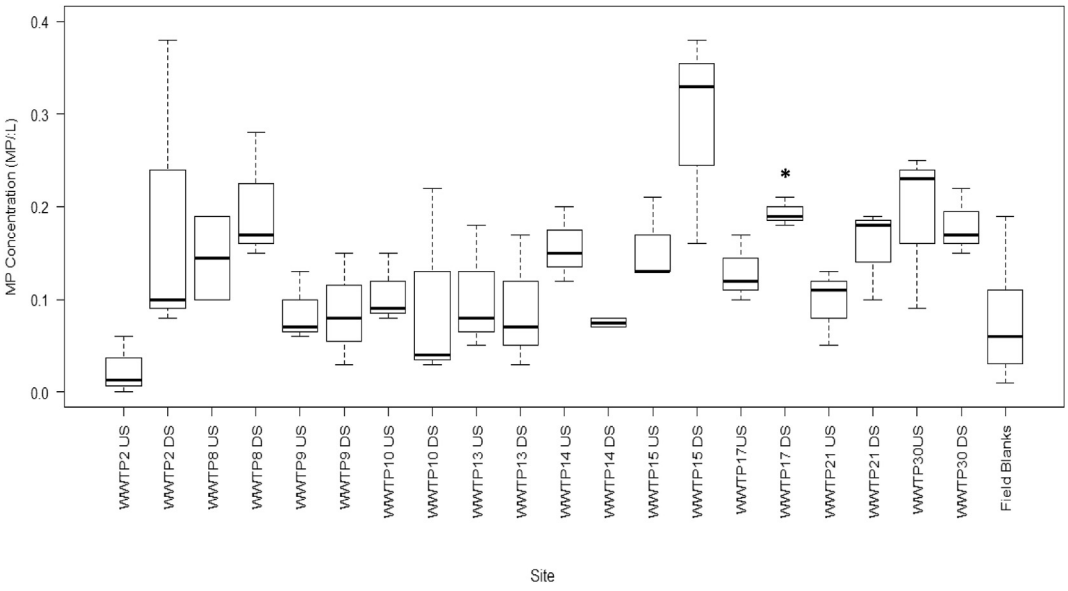

Fig. 6. Concentration of microplastics found in the surface water samples across each wastewater effluent site. US represents upstream samples whereas DS represents downstream samples. The thick black line represents the mean concentration found at each site. For WWTP8 US and WWTP14 DS, one concentration was removed as they were outliers and not included in the linear mixed effect model. The asterix represents the pair that is significantly different U/S vs D/S.

Table 3
P-values arising from paired t-tests looking at the mean difference in microplastic concentrations upstream and downstream of wastewater effluents. Effluent sites are defined by their location and treatment type. Marginally significant p-values are shown in italics and significant values **are bolded.**

Site ID	Effluent location	Type of **Treatment**	Paired *t*-test at	Paired *t*-test all
			Individual effluents (p-value)	**upstream vs.downstream effluents (p-value)**
WWTP2	Notre-Dame-de-l'Île-Perrot, Québec	Aerated ponds	0.1788	
WWTP8	Montréal, Québec	Physico-chemical	0.4506	
WWTP9	Varennes, Québec	Aerated ponds	0.9806	
WWTP10	Repentigny, Québec	Physico-chemical	0.9012	
WWTP13	Lanoraie, Québec	Aerated ponds	0.8335	*0.0552*
WWTP14	Sorel Tracy, Québec	Aerated ponds	0.4732	
WWTP15	Saint-Ignace-de-Loyola, Québec	Aerated ponds	0.2872	
WWTP17	Nicolet, Québec	Aerated ponds	**0.0241***	
WWTP21	Lotbinière, Québec	Aerated ponds	0.3504	
WWTP30	Quebec City, Québec	Biofiltration	0.8541	

concentration in river sediments was explained by seven variables and different microplastic forms were correlated with different predictor groups (Fig. 3; Table 1). Higher relative abundances of fibres were correlated with characteristics associated with depositional environments (% organic content, % silt & clay, phi score), consistent with previous studies (Fischer et al., 2016; Nel et al., 2018). Higher fragment relative abundances were correlated with urban land use (see also Mani et al., 2015; Peng et al., 2018). Fragments occurred most often as small (<10 μm) particles that were highly fluorescent; such a size fraction is significant for risk assessment as the severity of toxicity increases with decreasing particle size (Anbumani & Kakkar, 2018).

In contrast, higher relative abundances of microbeads were correlated with the presence of medium-sized sand, which settles to lotic sediments more frequently than does finer material. There was also a weak inverse correlation with sediment size (median phi score). Although we suspect that such microplastic-sediment relationships are ubiquitous, none to our knowledge has been previously reported for riverine systems. In a shallow coastal marine environment, Alomar et al. (2016) found microplastics consistently present in coarse sands but observed no trend with sediment grain size.

Contrary to expectation, proximity to wastewater effluents (an assumed point source of microplastics into rivers; Murphy et al., 2016; Mason et al., 2016) was not a significant predictor of microplastic abundances in the sediments. We also only found a significant increase in microplastic concentration downstream of the wastewater effluent in water samples at only one of ten effluent sites (Table 3). A linear mixed effect model examining microplastic concentration as a function of sampling location was also non-significant (Tables S5–S7; Fig. S5). Similarly, Hoellein et al. (2017) found that microplastic concentrations in surface water did not correlate with distance downstream from wastewater effluent location. Effluents are often strategically placed in areas of the river that are fast flowing, which could enable the rapid dilution and downstream transport of microplastics.

4.3. A continued call for standardization

There are growing calls to develop standardized protocols for the sampling, extraction, separation and identification of microplastics (e.g. Twiss, 2016; Prata et al., 2018). Each of these steps is

often an expensive and time-consuming process. Many factors contribute to the varying efficiency of identification methods, thereby further challenging large-scale monitoring programs (Yu et al., 2018). To support evidence-based policy and increase comparability of disparate studies, researchers must adopt cost-effective standardized protocols that enable efficient and accurate enumeration of microplastics in a broad range of environmental contexts. Here, we adapted a protocol that uses methods that are inexpensive, require a short processing time, and which can be applied to a broad scope of microplastic types in both water and sediment samples.

Many techniques have been developed to extract microplastics from environmental media. The most common extraction method is density separation (Van Cauwenberghe et al., 2015; Prata et al., 2018), in which low-density particles such as microplastics float and can thus recovered from the supernatant. Other methods include combining density separations or various solvents with a mechanical apparatus to stimulate separation (Imhof et al., 2012; Fuller and Gautam, 2016). These solutions and solvents are often expensive, inefficient at extracting a broad range of microplastics, and require special handling because of their toxicity (Li et al., 2018).

Conversely, the oil extraction protocol used in this study requires safe, low-cost materials that are readily purchased (e.g. canola oil). Furthermore, this protocol is density independent, as it manipulates the oleophilic properties of plastic, thereby allowing for all plastic types to be separated. Additionally, the processing time for oil extraction is much shorter (a few minutes) compared with density separation (a few hours). Our retention rate was lower than expected for this method, likely as a result of conducting organic digestion post-extraction (Crichton et al., 2017). To improve retention rates, we recommend that sediment samples be digested prior to microplastic extraction or that alternatives for the digestion step (e.g. KOH) be employed.

Many studies have relied upon visual identification coupled with spectroscopic testing (FT-IR, Raman) or chromatographic methods (pyrolysis GC/MS, liquid chromatography) to quantify plastic particles within a study. Such studies can be limited in their ability to detect small microplastics (<300 μm; the 'lost microplastic fraction') which could be easily identified using the Nile red tagging method (Shim et al., 2016; Erni-Cassola et al., 2017). The costs of Nile red and a fluorescent microscope are lower than that of obtaining and using spectroscopic or chromatographic equipment, and both are nearly as accurate in terms of quantification (Shim et al., 2016). The principal limitation of the Nile red tagging method is that it reveals no information on the chemical composition of the sample.

Finally, a standardized identification protocol is needed. Generally, microplastics are identified using physical factors and visual factors (Norén, 2007; Cheung et al., 2016). However, visual sorting is strongly affected by variation in researcher identification, microscopy quality and the sample matrix (Li et al., 2018). Furthermore, some studies do not discriminate the fibres within their analysis, including those of cellulosic or semi-synthetic origin, resulting in an over-estimation of microplastics in their samples (Lusher et al., 2014). In this study we applied identification trees that, in combination with Nile red tagging, should aid in more accurate identification microplastic particles. Our identification trees apply commonly used visual characteristics for plastic particles to identify cellulosic or semi-synthetic fibres and they build in criteria to avoid overestimation of plastic from other fluorescent particles such as organic debris that might be stained by the dye. These identification trees conservatively excluded white or translucent fragments and fibres, because these could be bleached natural particles that did not fully digest in peroxide (Erni-Cassola et al., 2017; Li et al., 2018). Indeed, our identification trees could potentially result in an underestimation of microplastics, as some previous studies have found that white and translucent plastics comprise a significant portion of microplastics (Peng et al., 2018; Lin et al., 2018; Zhu et al., 2018).

5. Conclusions

This study provided the first comprehensive quantification of the diversity of microplastics in the sediments of the St. Lawrence River, and revealed a mean concentration of microplastics that is among the highest recorded globally. The local composition of microplastics was explained, in part, by point sources and environmental filters (e.g., sediment compositional variables, distance to shore and urban land use) that can be used to guide monitoring programs regarding which areas are most vulnerable to microplastic accumulation and, consequently, which biotic communities are subject to maximal exposure. The results could also inform the design of more environmentally relevant experiments seeking to test the impacts of microplastics exposure on aquatic biota. Differences in methodology accounted for different recorded abundances of large microbeads in our study compared to Castañeda et al. (2014), highlighting the need for a standardized protocol. Future research in large rivers should assess the mass balance of microplastic loads and their fate during transport to the ocean, to determine whether river sediments are best viewed as permanent or transient sinks for microplastic pollution.

Main Finding

Variation in the distribution, abundance and diversity of microplastics across a large river is related to environmental filters, point sources, and sediment characteristics.

CRediT authorship contribution statement

Alex Crew: Conceptualization, Methodology, Investigation, Formal analysis, Writing - original draft, Writing - review & editing. **Irene Gregory-Eaves:** Supervision, Conceptualization, Writing - original draft, Writing - review & editing, Resources. **Anthony Ricciardi:** Supervision, Conceptualization, Writing - original draft, Writing - review & editing, Resources, Funding acquisition.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envpol.2020.113994.

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