Spatial and temporal variation of microplastics in the Ottawa River watershed with citizen science as a complementary sampling methodology.

By

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Abstract

Microplastic concentrations were investigated in water and sediment in the Ottawa River watershed in Quebec and Ontario, Canada. Microplastic concentrations were measured temporally during precipitation events in an urban creek in the City of Ottawa and on the main channel of the Ottawa River within the Ottawa/Gatineau urban area. The temporal events sampled included heavy rainfall, snowfall and snow melt with microplastic concentrations measured from these events compared to concentrations during non-precipitation events. The results showed sewage overflows contribute to large short-term inputs of microplastics to the Ottawa River, however, spring snowmelt presented the highest increase of microplastic concentration for both the urban creek and main channel of the river. Additionally, the research examined the spatial distribution of microplastics in river water throughout the Ottawa River at 105 sample points on the main channel and tributaries. An ANCOVA analysis demonstrated only two significant spatial factors related to microplastic concentration, with distance downstream from the river source and an increase of microplastics at boat launch locations. However, these were both only weak relationships.

The research incorporated two citizen science projects to investigate microplastic concentration in water and sediment in the Ottawa River watershed, while evaluating the potential of citizen science as a complementary sampling tool for microplastic research. With robust project design and implementation, citizen science is an excellent complementary tool for examining microplastic concentration in freshwater environments as it can reduce research costs, while increasing spatial scope of microplastic projects.

Additionally, increasing citizen science capacity in microplastic research and monitoring is a useful tool to engage volunteers and involve them in environmental education while contributing to advancing the understanding of microplastic pollution.

Preface

Integrated manuscripts

Chapters 2-6 of this thesis are drawn directly from manuscripts either published, submitted or in preparation for submission as a journal article.

Formatting was adjusted to meet the thesis formatting guidelines, but the original content of the journal articles was not altered. Specific adaptations of the journal articles and submissions to the thesis include 1) the removal of abstracts and keywords 2) re-formatting, including font and margins, using Times New Roman 12 point font for all text in the thesis 3) adaptation of the table of contents to reflect the integration of the journal articles and submissions into the thesis including re-numbering headings, figures and tables to reflect the integration into the thesis format 4) reformatting and re-numbering of figures, tables and captions and 5) merging the journal article references into the reference section of the thesis. The original citations for the material presented in these chapters are:

Chapter 2: Forrest, S. A., Bourdages, M. P. T., & Vermaire, J. C. (2020). Microplastics in Freshwater Ecosystems. In T. Rocha-Santos, M. Costa, & C. Mouneyrac (Eds.), Handbook of Microplastics in the Environment (pp. 1–19). Springer International Publishing. https://doi.org/10.1007/978-3-030-10618-8_2-1.

Chapter 3: Forrest, S. A., Holman, L., Murphy, M., & Vermaire, J. C. (2019). Citizen science sampling programs as a technique for monitoring microplastic pollution:

Results, lessons learned and recommendations for working with volunteers for monitoring plastic pollution in freshwater ecosystems. Environmental Monitoring and Assessment, 191(3), 172. https://doi.org/10.1007/s10661-019-7297-3.

Chapter 4: Evaluating community science sampling for microplastics in shore sediments of large river watersheds. FACETS (accepted and in print, DOI: 10.1139/facets-2022-0104.).

Chapter 6: Forrest, S. A., McMahon, D., Adams, W. A., & Vermaire, J. C. (2022). Change in microplastic concentration during various temporal events downstream of a combined sewage overflow and in an urban stormwater creek. Frontiers in Water, 4, 958130. https://doi.org/10.3389/frwa.2022.958130

Statement of co-authorship

Chapters 2-3 and 6 in this thesis are drawn from published articles. The authors of each article have read and signed statements outlining the role of Shaun Allan Forrest as principal investigator and main author for each published article.

Copyrighted material

Citing material in this thesis

Material from chapters 2-3 and 6 should be referenced from the original journal publication where possible, otherwise, the thesis may be cited as:

Forrest, S.A. (2022), Spatial and temporal variation of microplastics in the Ottawa River watershed with citizen science as a complementary sampling methodology. Ph.D. Thesis, Carleton University, Ottawa, Canada.

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List of abbreviations and symbols

$CaCl_2$	Calcium chloride
CDN	Canadian dollars
CSO	Combined sewage overflow
ºC	Degrees Celsius
cm^3	Cubic centimetres
FTIR	Fourier transform infrared spectroscopy
g	Grams
g/m^3	Grams per cubic centimetre
HOCs	Hydrophobic organic contaminants
IPW	International Pellet Watch
kg	kilograms
km	Kilometres
µm	Micron
m^3	Cubic metres
mm	Millimetres
ml	Millilitres
MP	Microplastics
mp/m^3	Microplastics per cubic metre
NaCl	Sodium chloride
Nal	Sodium iodide
OEP	Oil extraction protocol
PAH	polycyclic aromatic hydrocarbons
PCB	Polychlorinated biphenyls
POPs	Persistent organic compounds
p/m^3	Particles per cubic metre
%	Percent
QA/QC	Quality assurance/quality control
WWTP	Wastewater treatment plant
$ZnBr_2$	Zinc bromide

1. Introduction

This thesis focuses on the spatial and temporal distribution of microplastics in water and sediment in the Ottawa River watershed in Ontario and Quebec, Canada. Additionally, the research aims to identify and verify the major sources and/or conduits of microplastics into the watershed. The research is an investigation of how microplastics are dispersed throughout the watershed, in the main channel of the Ottawa River and its major tributaries, while identifying major point sources of microplastics. The research evaluates the potential role of citizen science[1] as a complementary research tool for examining microplastics in water and sediment throughout the watershed.

The majority of microplastic research has focused on marine environments, with only around 4% of published research focusing on freshwater environments (Wagner & Lambert, 2018). Furthermore, rivers are established as a major contributor of plastic pollution to the world's oceans, with estimations of between 1.15 and 2.41 million tonnes of plastic waste entering oceans via rivers annually (Lebreton et al., 2017). Previous research identifies microplastics can interact negatively with numerous organisms, including freshwater species (Hurley et al., 2017; Guilhermino et al., 2018; Windsor et al., 2019). Additionally, inadvertent human consumption is another potential adverse effect microplastics pose (Wright & Kelly, 2017; Karbalaei et al., 2018; Cox et al., 2019).

[1] At the time of publishing, the majority of published research where volunteers collected samples in microplastic research, used the term *citizen science*. The term *community science* was also an accepted description for these projects, however, the most common term at the time of writing this thesis was citizen science, thus, this was the adapted term for the majority of the thesis. However, I acknowledge there may be a shift towards using the term community science rather than citizen science in future research and this includes chapter 4, the most recent submitted manuscript, where community science was adopted.

There is limited research on the spatial and temporal distribution of microplastics in Canadian riverine environments, thus it is important to understand not only global freshwater environments in greater detail, but specifically Canadian watersheds. The Ottawa River watershed is the chosen study zone as it represents a large watershed, approximately 140,000 square kilometres (Figure 1.1), while in itself a tributary of the Saint Lawrence drainage basin, which has the highest average water discharge in North America (Benke & Cushing, 2005). It has already been established that the St. Lawrence contains a significant amount of microplastic pollution in both water and sediment (Castañeda et al., 2014; Crew et al., 2020). Therefore, as the major tributary of the Saint Lawrence, it is important to understand the spatial distribution in relation to potential inputs (how microplastics enter the river), throughputs (how microplastics move through river systems), sinks (how many microplastics are caught, temporarily or permanently in river systems) and outputs (how many microplastics exit the river system) of microplastics, as it provides an important perspective for Canadian rivers. Because the Ottawa Watershed is large, it contains many different landscapes and population bases of varying sizes from densely populated regions, namely the cities of Ottawa/Gatineau urban area, to numerous towns and small settlements along the main trunk and tributaries. Additionally, the watershed has sparsely populated regions with minimal or no human activity. This combined with the varying landscapes through which the Ottawa River and its tributaries flow, presents an array of differing environments to examine microplastic concentration.

1.1 Research objectives

The research presented in this thesis is organised around the following primary objectives:

The first objective is to evaluate the overall spatial distribution of microplastics in water and sediment across the Ottawa River watershed, including examining the main channel of the Ottawa River in addition to the predominant tributaries of the watershed. The research will evaluate the contribution of major sources and/or conduits of microplastics to the watershed highlighted in previous research in freshwater environments, in addition to evaluating additional sources, conduits, or sinks of microplastics to the watershed, for example the contribution of snowmelt.

The second objective of the research is to evaluate the role of citizen science as a complementary research tool in a large watershed. The research will evaluate the role of citizen science as a tool for spatial analysis of microplastics in water and sediment, while evaluating the scientific integrity of the process, thus, evaluating the potential to use volunteers in spatial analysis of microplastics in Canadian freshwater environments.

The third objective is to evaluate the temporal change of microplastics during previously identified high-load events, namely during combined sewage overflow and high stormwater flow events in addition to evaluating other potential load events like snowmelt. The aim is to measure microplastic concentrations during these events to establish the change of microplastics as they enter the Ottawa River from important point sources.

1.2 Placing the thesis in the context of geography

Geography is a broad term encompassing many facets of the physical and human aspects of the globe. This thesis specifically highlights the interaction of humans within a particular physical aspect of the environment, namely a watershed. Human activity is currently polluting watersheds through indiscriminate disposal of plastic waste, and this research examines how the pollutant varies spatially and temporally throughout the physical aspects of a large watershed.

1.3 Thesis structure

Four primary research papers compromise the majority of this thesis, with a fifth published review paper representing the introduction/literature review of the thesis. Three published journal articles, one journal article accepted at the proofing stage and one journal article prepared for submission cover the three main research objectives and are represented in chapters 2-6. The first publication is presented in Chapter 2 as a literature review of freshwater microplastics, which outlines the important background information and previous research in freshwater microplastics. Chapters 3 and 4 analyse the effectiveness of two citizen science projects. Chapter 3 and 6 are published journal articles, with chapter 4 accepted and will be published in an upcoming journal series. Chapter 5 has been prepared for journal submission. Chapter 5 is a watershed scale analysis of the spatial distribution of microplastics in river water. Chapter 6 presents the temporal analysis of stormwater and combined sewage overflows as major conduits of microplastics to the Ottawa River and freshwater Canadian environments.

The specific methodologies used in the research are not presented in this chapter, as each of the integrated chapters discuss their respective methods of research in detail. Chapter 7 is the thesis Conclusion Chapter and follows the integrated chapters and summarises the key findings, implications and discusses the future direction of microplastic research in Canadian freshwater environments.

2. Background

2.1 Introduction

Plastic waste has become a worldwide problem, with discarded plastics evident throughout global aquatic environments. Global production of plastics has soared to 460 million tonnes in 2019, with only 9% of this production being recycled (OCED, 2022). Plastic waste is consistently found in aquatic environments throughout the globe including the ocean surface (Cozar et al., 2014), deep- sea sediments (Van Cauwenberghe et al., 2013), lagoons (Vianello et al., 2013), beaches (Wessel et al., 2016), freshwater lakes (Eriksen et al., 2013; Eerkes-Medrano et al., 2015) and rivers (Vermaire et al., 2017). As most plastics are not biodegradable (Geyer et al., 2017), they can continue to break-up into smaller and smaller fragments without completely disappearing, in fact it is estimated that most plastics will take thousands of years to completely degrade (Crawford & Quinn, 2017). As a consequence, tiny plastics, or microplastics (plastic pieces <5 mm in size; Masura et al., 2015), are now present in soils (Nizzetto et al., 2016; Huang et al., 2020; Li et al., 2020), remote lakes (Free et al., 2014; K. Zhang et al., 2016), remote rivers (Jiang et al., 2019), polar regions (Obbard, 2018) and all environmental compartments in all continents, including aquatic environments (McCormick et al., 2014; Horton et al., 2017; Mahon et al., 2017). Within the marine environment, microplastics are now considered the primary constituents of marine debris (Moore, 2008; Barnes et al., 2009; Cole et al., 2011).

The term ‘microplastics’ was not formally adopted until after 2004 (Thompson, 2005) even though small plastics have been observed in the marine environment since the 1970s (Buchanan, 1971; Carpenter & Smith, 1972). Microplastics can be classed as either primary or secondary microplastics. Primary microplastics are specifically manufactured to be plastics of microscopic size. Secondary microplastics are the result of a breakup or fragmentation of larger plastics. The plastic breakup can be due to a combination of physical, chemical or biological processes reducing the integrity of the plastic, leading it to fragment (Cole et al., 2011). Another example of secondary microplastics is microfibres that are shed from synthetic clothing. Currently, microfibres are one of the most common forms of microplastic pollution encountered in freshwater systems, including lakes and river systems (Browne et al., 2011; McCormick et al., 2014; Vermaire et al., 2017; Wang et al., 2017).

Freshwater microplastic research has only recently gained momentum, with only about 4% of microplastic research related to freshwater (Wagner & Lambert, 2018). Freshwater environments can act as a conduit of microplastics to global marine areas through rivers or can also act as a sink for microplastics, for example in isolated freshwater lakes or lake and river sediments (Wagner and Lambert, 2018). Additionally, freshwaters can play a role as transformers of plastic pollution modifying the shape, size, and texture of plastic pollution through physical, chemical, and biological processes. Once microplastics are released into freshwater systems their fate is poorly understood, however, a portion would be transported to ocean environments by rivers, with some, retained in sinks such as sediments (Browne et al., 2011; Free et al., 2014).

2.2 Microplastics in river systems

Rivers have been identified as a major source of plastic pollution reaching the world's oceans (Lebreton et al., 2017), however, research on microplastic contamination in rivers is still gaining momentum with only a limited understanding to the mechanisms that controls transport, dispersion and fate of microplastics in river systems (P. J. Anderson et al., 2017; Vermaire et al., 2017; Wagner & Lambert, 2018). What is clear is that microplastic contamination is prevalent in many river systems around the world, including in remote or rural regions of river systems (Forrest et al., 2019). Plastics and microplastics originating from terrestrial or atmospheric sources released into freshwater environments may be transported to the oceans via rivers but could also be retained in sinks within freshwater environments, including river sediments or isolated lakes (Browne et al., 2011; Free et al., 2014; Jiang et al., 2019). Microplastic entrainment and deposition in rivers can be attributed to factors including (but not limited to) upstream land use (Mani et al., 2016), urban runoff (Nizzetto et al., 2016), effluent discharge (Moore et al., 2011), river hydraulics and river morphology (Besseling et al., 2015), population density (Yonkos et al., 2014) and the amount of urbanisation and industrialisation surrounding a river (Lechner et al., 2014). In addition, barriers such as dams and weirs also influence the concentration, composition, and transport of microplastics in freshwater ecosystems (K. Zhang et al., 2015). Other important factors to consider are related to the polymer itself, for example, size, shape, density and if it has any organic and/or inorganic materials, including biofouling attached to the plastics (Figure 2.2). Environmental factors that could affect microplastics distribution in rivers include winds, currents, waves, and water density (Prata et al., 2019). In short, microplastic concentration in river systems can be highly variable both spatially and temporally (e.g., high flows vs low flows) and researchers are at the early stages of understanding these mechanisms.

Research has suggested that microfibres do not tend to settle in river sediments as often as fragments (Leslie et al., 2017), but are rather entrained in the water column and more likely to be transported downstream. Nonetheless, some research has indicated the presence of microfibres in river sediments (Vermaire et al., 2017; Hurley & Nizzetto, 2018), that have potentially precipitated out of the water column in lower energy environments and/or as part of a conglomerate of material that has become more dense than water. River sediments also have been shown to contain a substantial amount of primary microplastics in more industrialized river systems (Castañeda et al., 2014). However, floods, or high-flow events such as spring freshets can dislodge microplastics from sediments (Hurley and Nizzetto, 2018) and entrain them in the water column, sending microplastics further downstream to settle again, or to be transported to marine environments. In order to understand the occurrence, transport and fate of microplastics in river ecosystems there is a growing need to increase both the spatial and temporal resolution of sampling in these systems.

Figure 2-1Example of microplastics extracted from the Trent-Severn waterway in Ontario, Canada.

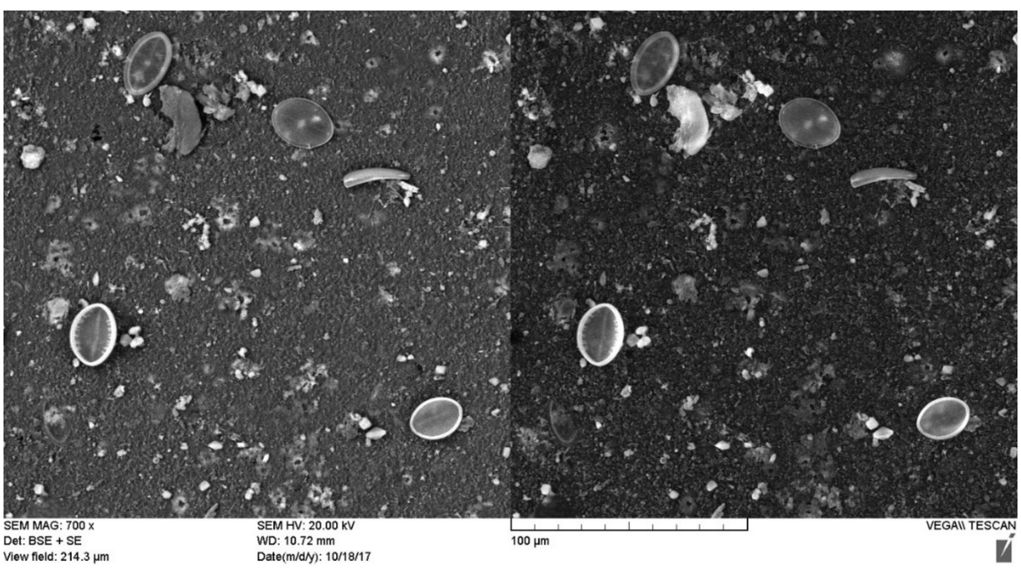

Figure 2-2 SEM image highlighting microplastics from the Ottawa river containing biofilms with diatoms growing on them.

2.3 Microplastics in Lakes

Microplastics were first reported in freshwater lakes only in 2013 (Eriksen et al., 2013) highlighting the recent concern and focus of microplastic contamination in these systems. Research has focused on microplastic contamination in lakes of different sizes, dimensions and proximities to urban areas, with all demonstrating striking amounts of microplastic pollution (Eriksen et al., 2013; Free et al., 2014; Driedger et al., 2015; J. C. Anderson et al., 2016). Lakes have also been described as a 'semi-closed' system for microplastics (Fischer et al., 2016) whereby microplastics may be contained in sinks in lakes (i.e. sediments), and/or circulated within the open water system, or enter tributaries and exit the lake environment. The movement of microplastics within lake systems will depend on wind strength, wind direction, lake morphology, prevailing currents and storm events. Additionally, a lake's geographic position (latitude and longitude) can determine microplastic transport and deposition, as seasonal temperatures and density stratification in the water can cause varying mixing and stagnation phases of microplastics in the water column (Boehrer & Schultze, 2008).

The fate of microplastics in the surface waters of lakes depends on the polymer type, size, shape and additional factors such as biofouling which could affect the buoyancy of the plastics. Additionally, as freshwater is not as dense as saltwater, the critical density of a plastic to sink is slightly lower than in marine systems. For example, plastics prone to float in marine environments, namely polyethylene, polypropylene and polystyrene, have an increased chance to sink in freshwater environments (Ballent et al., 2016), especially if there are any biofouling mechanisms.

Similar to river systems, lakes can present large shorelines and thus exhibit some similarities to marine environments, as lake and river shores can provide the opportunity for the breakdown of larger plastics through mechanical weathering and photodegradation. In this way freshwater systems can also act of transformers of plastic pollution before it reaches marine systems. There are numerous studies examining the integration of plastics and microplastics in marine beaches, but little research has focused on microplastics on lake or river shores (Zbyszewski & Corcoran, 2011). Though similarities have been drawn to marine beaches, weathering on lake or river shores can still differ due to factors such as the varying weathering rates in different water chemistries, i.e. salt versus freshwater (Biesinger et al., 2011). Microplastics have also been identified in bottom sediments of lakes (Corcoran et al., 2015), further indicating that lakes are sinks for microplastic pollution.

Similar to river systems microfibres are also the dominant microplastic in lake environments (Anderson et al., 2017), highlighting the potential influence of urban areas and wastewater treatment effluent. However, research has suggested even remote lakes away from urban areas and anthropogenic influence exhibit microplastic contamination (Free et al., 2014; Biginagwa et al., 2016; K. Zhang et al., 2016; Y. Zhang et al., 2019). These findings suggest the role of atmospheric transport of microplastics (Dris et al., 2017; Dehghani et al., 2017; Cai et al., 2017) to remote aquatic systems, or potentially trophic transfer by animals (Hurley & Nizzetto, 2018; Provencher et al., 2018). Though research into microplastics in lakes and freshwater environments is somewhat limited, there is growing evidence of interaction (for example microplastic ingestion and adhesion to organisms) with benthic environments in lake (Driedger et al., 2015) and river systems

(Windsor et al., 2019), suggesting bioaccumulation is also a potential mechanism of microplastic pollution in both marine and freshwater systems.

2.4 Sources of microplastics to freshwater ecosystems

To further understand microplastic contamination in freshwater ecosystems, it is imperative to understand potential inputs of microplastics to freshwater environments. There are multiple pathways for microplastics to enter freshwater ecosystems including wastewater and storm water, atmospheric deposition, and the breakup of larger plastics within rivers, lakes, and shorelines (Figure 2.3). In particular, previous research has identified wastewater treatment plants and stormwater flow (including overland runoff) as a potentially important source of microplastics to freshwater environments (Estahbanati & Fahrenfeld, 2016; Horton et al., 2017; Vermaire et al., 2017; Windsor et al., 2019; Grbić et al., 2020). This is not to disregard the potential of other inputs, for example atmospheric fallout; however, to date, very little research exists on the influence of atmospheric deposition on microplastic concentration or composition in freshwater ecosystems.

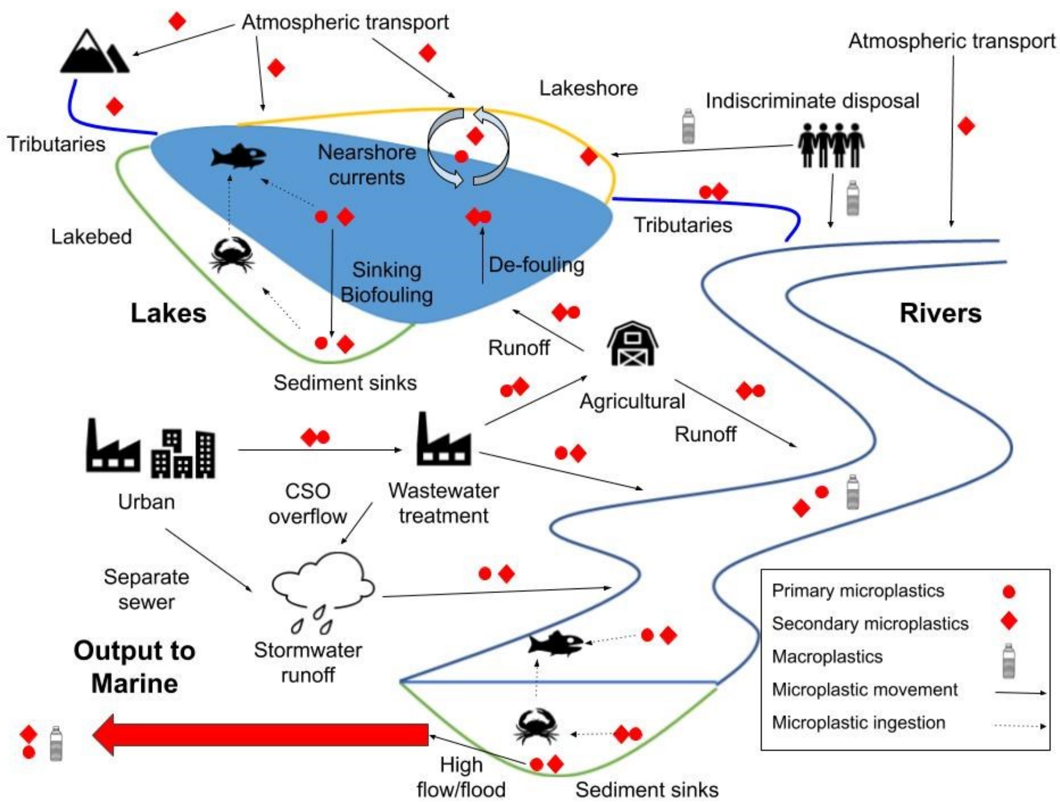

Figure 2-3 A diagram depicting the movement of microplastics in freshwater environments, including sources, dispersion and fate.

2.4.1 Wastewater treatment plants

One of the main conduits of microplastics to freshwater environments has been identified as wastewater treatment plants (WWTP). This is especially true in urban rivers, where effluent disposal can expel primary and secondary microplastics into river environments (Estahbanati & Fahrenfeld, 2016; Leslie et al., 2017; Talvitie et al., 2017; Ziajahromi et al., 2017) including a high amount of textile fibres in wastewater from laundry (Belzagui et al., 2019) Additionally, wastewater sludge captures a substantial amount of microplastics and in many cases is returned to the land as soil conditioner (Zubris & Richards, 2005; Corradini et al., 2019), creating the potential for the microplastics within the sludge to enter freshwater environments through runoff. For example, the European Union has estimated between 63,000 and 430,000 metric tonnes of microplastics within sewage sludge are deposited annually on European agricultural lands (Nizzetto et al., 2016).

The wastewater treatment process is very efficient in removing microplastics received from industrial and domestic wastewater. Removal efficiency is as high as 99% in tertiary treated effluent (Talvitie et al., 2017), however, globally many municipalities only have primary or secondary wastewater treatment (or none at all), in which case microplastic removal efficiency will be greatly reduced (Murphy et al., 2016; Mintenig et al., 2017; Ziajahromi et al., 2017). However, given the amount of water a WWTP processes and the huge amounts of plastic pollution entering our wastewater systems, even high efficiency rates can still amount to a substantial deposition of microplastics to receiving waters (Murphy et al., 2016; Leslie et al., 2017; Talvitie et al., 2017; Ziajahromi et al., 2017). For example, in the USA, 17 WWTP effluent streams were tested (Mason et al., 2016) noting 0.05 (+/- 0.024) microplastics per litre of effluent. Another study in San

Francisco noted a slightly higher 0.086 microplastics per litre of effluent (Sutton et al., 2016). These figures may appear low, however, these concentrations are orders of magnitude above the concentration of microplastics in most lakes and rivers and most treatment facilities process millions of litres of wastewater a day resulting in estimated microplastic discharges from WWTPs in the order of millions of microplastic particles per day (Mason et al., 2016).

It is also noted the level of treatment can affect the amount of microplastics released to the aquatic environment, for example, tertiary or advanced wastewater treatments are typically more efficient in microplastic removal. Additionally, the presence of combined sewer overflows can significantly load lake environments of microplastics during storm or overflow events, similar to river systems.

Microplastic conveyance through WWTP can also depend on the adoption of a combined or separate sewer system. A combined sewer system conveys both sewage and stormwater to a WWTP. During normal conditions, all of this influent gets treated to a set standard and the effluent is discharged to a receiving water body. However, during high flow or storm events, a combined sewage overflow may redirect excess flow away from the WWTP directly to a receiving body of water. The typical 99% removal rate (in the case of most tertiary treatments) is now bypassed potentially resulting to a substantial microplastic load into a receiving body of water. The other construction option adopted by urban municipalities is the implementation of a separate system, where sewage and stormwater are separated, and stormwater flows directly to an aquatic environment. From a microplastic pollution perspective, the separate sewer system may also not be ideal, as any stormwater flow is not processed for microplastics, thus any microplastic load that may have been removed by a WWTP within a combined system, is now conveyed directly into a river, lake or ocean. Stormwater runoff entering aquatic environments directly can contain microplastics from tires (Dehghani et al., 2017) and can collect plastic debris where these macroplastics can then begin to breakdown, depending on the environment it ends in. Further research on microplastic loading to lakes and rivers following large rainfall events will help quantify the role of storm events in loading microplastic pollution to freshwater ecosystems.

2.4.2 Stormwater

Stormwater is an additional identified conduit for microplastics into rivers. The types of microplastics in stormwater can vary to a greater extent than WWTPs, due to an increase in potential microplastic sources, for example vehicle abrasion from tire treads (Dehghani et al., 2017; Horton et al., 2017). Stormwater does add an important temporal analysis component to research with outflow occurring in rain and/or melt events. For example, a Los Angeles study suggested that up to three times the plastic concentrations in rivers were measured after wet events, leading to runoff as a major contributor (in combination with potential terrestrial inputs) in transporting plastics to river systems (Moore et al., 2011). Additionally, urban creeks or waterways, can be a collection point for stormwater runoff during precipitation events.

2.4.3 Atmospheric deposition

Remote locations, away from any anthropogenic influences, are also prone to microplastic contamination (Browne et al., 2011; Free et al., 2014; Jiang et al., 2019). There are suggestions that microplastics, more specifically microfibres, are capable of atmospheric transport (Dris et al., 2016; Cai et al., 2017; Dehghani et al., 2017; Dris et al., 2017; Gasperi et al., 2018). Microfibres can potentially travel great distances before settling in remote areas through processes such as atmospheric fallout. However, some research suggests other microplastic shapes besides microfibres could also be transported by the atmosphere including fragments (Cai et al., 2017) and even fragments from car tires (Kole et al., 2017). Recent research suggests microplastic concentrations of 88 to 605 microplastics per 30 g of dust collected in urban streets (Dehghani et al., 2017) indicating potential atmospheric fallout rates between 2 and 355 fibres per m^3 per day.

2.5 Sampling for microplastics in freshwater environments

Since microplastic research is still relatively new field, there is a general lack of standardization of methodologies for sample collection and analysis. For years, many researchers have been urging for a harmonization and/or standardization of methods (Ryan et al., 2009; Filella, 2015; Löder & Gerdts, 2015; Rocha-Santos & Duarte, 2015; Van Cauwenberghe et al., 2015; Twiss, 2016; Rochman et al., 2017; Koelmans et al., 2019), particularly since without harmonization and/or standardization comparison of data between studies is challenging. Formalizing field and laboratory techniques in both marine and freshwater environments will greatly aid the comparison of results among studies.

Many methodologies used in freshwater studies follow those used in the marine environments, and just like research conducted on microplastics in the marine environment, the ways in which samples are collected and analysed in freshwater environments vary among studies. Although there are several different sampling methodologies for collecting water and sediment samples from freshwater environments, some methods appear to be more commonly employed than others.

The most common technique used to collect water samples in rivers or lakes are net tows (either Manta or Neuston) (Eriksen et al., 2013; Free et al., 2014; Fischer et al., 2016; P. J. Anderson et al., 2017; Vermaire et al., 2017; Warrack et al., 2017), which is also commonly used to sample marine waters (Cózar et al., 2014; Eriksen, 2014; Abayomi et al., 2017; Barrows et al., 2018), and allows for the filtration of large volumes of water. Collecting grab samples either for in situ filtration or for laboratory filtration has also been used (Leslie et al., 2017; Miller et al., 2017; Forrest et al., 2019; Jiang et al., 2019; Grbić et al., 2020), however, an important limitation of this method is the low volume of water that can be filtered compared to using a net, which may increase variability between samples (Barrows et al., 2017). Although most studies use one of these two methods to collect water samples, inconsistencies are still present within both methods. Specifically, the lower size limit being examined can vary greatly (i.e. the filter or sieve size), and the volume of water that is filtered which can vary greatly between studies. The difference in mesh size and volumes sampled (with lower volumes prone to greater skews in data due to potential atmospheric contamination) can present a vast difference in the actual and potential microplastic concentrations and thus reporting these concentrations can make it difficult to draw definitive conclusions when comparing between research projects.

Similar to water samples, methods used to collect sediment samples in freshwater environments follow the methods also used in marine environments. The two main methods used to collect sediments in lakes and rivers are through collecting surface sediment samples along shorelines (Ballent et al., 2016; Fischer et al., 2016; Jiang et al., 2019), or by collecting benthic sediment samples offshore using grab samplers or corers (Castañeda et al., 2014; Ballent et al., 2016; Leslie et al., 2017; Sruthy & Ramasamy, 2017; Vaughan et al., 2017; Vermaire et al., 2017; Turner et al., 2019).

Laboratory processing of fresh water and sediment samples also tend to follow similar processing methods used in the marine environment (Masura et al., 2015), and just like sampling methods, there are many ways in which samples are processed. Water samples are often filtered and rinsed through sieves or filters before undergoing chemical digestion to rid any organic material from the sample. Next, particles are usually picked, counted and categorised from the samples using stereo microscopy, and even in this step of analysis, plastics can me missed, or incorrectly identified as organics and not extracted.

Sediment samples are typically first dried and weighed, and then undergo steps for size fractioning and/or density separation. In some cases, chemical digestion is used to remove organic material. Although most studies perform a density separation, the solutions used for this step vary greatly in the literature (Quinn et al., 2017). One of the most common solutions for density separation in the literature is sodium chloride (NaCl) (Fischer et al., 2016; Leslie et al., 2017; Sruthy and Ramasamy, 2017; Vermaire et al., 2017), while others use various different heavy liquids to obtain higher solution density than can be achieved using NaCl, such as sodium polytungstate (Ballent et al., 2016; Turner et al., 2019) or zinc chloride (Jiang et al., 2019).

Initial identification of microplastic particles typically involves visual classification of plastic particles using a stereo-microscope (Barrows et al., 2017; Vermaire et al., 2017; Warrack et al., 2017), followed by polymer identification for a subset of particles. The step of identifying and chemically characterising particles is increasingly thought to be crucial to make sure that particles are in fact microplastics, especially as more studies are now including smaller sized particles. The most common methods for particle identification and chemical characterisation are Fourier-transform infrared spectroscopy (FTIR) (Zbyszewski & Corcoran, 2011; Lusher et al., 2015; Abayomi et al., 2017; Bergmann et al., 2017; Miller et al., 2017; Mintenig et al., 2017; Primpke et al., 2017) and Raman spectroscopy (Cózar et al., 2014; Free et al., 2014; Zhao et al., 2015; Frère et al., 2016; Ghosal et al., 2018; Schymanski et al., 2018). However, other methods of identification have been published, such as the use of Scanning Electron Microscopes (SEM) in the case of Polyvinyl chloride (Anderson et al., 2017) and Nile red dye (Hengstmann & Fischer, 2019). To improve the accessibility of identifying microplastics, spectral libraries specifically geared towards plastic particles have recently been developed (Cabernard et al., 2018; Primpke et al., 2017; Munno et al., 2020).

2.6 Contamination and QA/QC

Microfibre contamination in both the field and the laboratory should also be considered while collecting and processing samples, with necessary steps taken to reduce contamination potential. Atmospheric transport and deposition of microplastics are possible throughout even the most remote locations (Free et al., 2014; Obbard, 2018; Jiang et al., 2019; Zhang et al., 2019). This highlights the importance of conducting field controls to allow for potential contamination while sampling and also collecting sufficiently large samples to distinguish an environmental signal from potential contamination, especially as one or two microfibres from contamination can great skew microplastic estimates in lower volume testing.

Indoor microfibre contamination is also important to consider, with fallout from fibers in indoor environments estimated between 1586 and 11,130 fibers/day/m^2 (Dris et al., 2017). This emphasises the importance of reducing contamination potential wile processing samples in the laboratory. Laboratory coats, made of natural microfibres if possible, and/or bright distinguishable colours should be worn at all times. If laboratory coats shed, the bright colours can be identified as contamination in samples and the natural microfibres can be removed during digestion protocols or can be discounted under Raman or FTIR analysis. All petri dishes, mesh and containers (for example beakers) should be triple rinsed with deionised water to remove any settled fibre contamination, throughout sample processing. If samples are idle at any time during processing, they should be covered and placed in fume hoods or environments where the air is extracted, thus, removing airborne microfibres away from samples. Furthermore, all sample processing where possible, should be conducted under a laminar flow hood or even better a clean room to reduce the settling potential of airborne microfibres. Microfibre contamination highlights the importance of conducting controls and blank samples throughout sampling and processing, as it establishes the detection limit and potential contamination limit of the samples.

Finally, while there should be an acknowledgement of contamination potential through controls, consideration should also be given to positive controls. During collection and processing of samples, loss of microplastics is possible. For example, various digestion protocols have been suggested to consume some polymers, thus, consideration should be given to which digestion protocol is selected, or a positive control should be applied to establish potential microplastic loss. Additionally, during visual identification stages, especially in samples with numerous microplastics, there is potential to miss microplastics in samples, therefore, utilising positive controls is a useful method to establish the potential loss of microplastics during sample processing. To date, very few studies of microplastics have employed positive controls to assess the potential loss of microplastics through processing and sorting (Koelmans et al., 2019).

2.7 Impacts of microplastics on freshwater ecosystems

Ingestion of microplastics can potentially cause chemical and physical harm to freshwater organisms (Auta et al., 2017). However, research to date on the potential impacts of microplastic pollution on organisms has largely focused on marine biota. In many cases, there is potential physical and chemical harm due to microplastic ingestion (or attachment of microplastics to biota surfaces) (Fossi et al., 2014; Barboza et al., 2018; Oliveira et al., 2018) and could be assumed to apply to freshwater organisms. Nonetheless, there is some emerging research demonstrating freshwater species can be affected by microplastics including through ingestion (Hurley et al., 2017; Windsor et al., 2019) with suggestions of potential physical harm (Rehse et al., 2016; Guilhermino et al., 2018) .

2.7.1 Bioaccumulation of Microplastics in freshwater

Microplastic interaction with biota can be somewhat dependent on microplastic shape, size, colour, aggregation and abundance and will affect their potential bioavailability (Wright et al., 2013; Van Cauwenberghe et al., 2015). The smaller sizes of microplastics increases their bioavailability to a wider array of organisms, especially indiscriminate feeders. Microplastics have the potential to sorb (and/or release) toxic chemicals, with the potential transfer through the food chain (Reisser et al., 2014; Hurley et al., 2017). Previous research has highlighted microplastics as a vector due to the sorption of waterborne pollutants, from invertebrates to higher trophic levels (Teuten et al., 2009; Zarfl & Matthies, 2010; Ivar do Sul & Costa, 2014). The accumulation of these small plastics in organisms can potentially cause adverse physical effects in addition to potential adverse chemical effects Organisms have also been found to metabolise persistent organic pollutants (POPs) from trophic surfaces of microplastics (Chua et al., 2014).

Microplastics can contain organic pollutants either added during plastic production or they are prone to contamination from water borne contaminates such as POPs and metals due to their relatively large surface area to volume ratio (Ashton et al., 2010; Cole et al., 2011). POPs prone to sorption by microplastics include dichlorodiphenyl (DDT), polycyclic aromatic hydrocarbons (PAH) and Polychlorinated biphenyls (PCB) with potential metal sorption including copper, silver, zinc, lead iron, manganese and mercury (Ashton et al., 2010; Hirai et al., 2011; Chua et al., 2014; Auta et al., 2017; P. Oliveira et al., 2018). Some of these chemicals can be found in high quantities in aquatic environments, especially on the surface, where low density microplastics can be present in large numbers (Teuten et al., 2009). Furthermore, plastics can sorb contaminants from the surrounding environment up to 100 times more than sediments, and this sorption can include organic chemicals that are persistent, bioaccumulative and toxic (Ogata et al., 2009; Ashton et al., 2010; Holmes et al., 2012; Rochman et al., 2013, 2014).

Moreover, chemical ingredients present in plastics sorbed from the environment can include pollutants as they have been identified to be bioaccumulative, persistent and/or toxic (Rochman et al., 2013), with more than 50% of the plastic polymers produced including chemical ingredients considered hazardous by the UN’s Globally Harmonized

System (Lithner et al., 2011). Nonetheless, it is important to also consider the plastic or polymer itself as the type of polymer can influence the amount of sorption of organic pollutants (Rusina et al., 2007). For example, it has been suggested polyethylene can accumulate more organic pollutants than Polyvinylchloride (Teuten et al., 2009).

The ingestion of these small plastics with toxins by organisms at the base of the food chain highlights the potential for bioaccumulation (Teuten et al., 2009). However, the research on the joint toxicology of microplastics and POPs is still limited (M. Oliveira et al., 2013; Mattsson et al., 2015) and less attention has been given to the chemical effects associated with the ingestion or bioconcentration of plastic debris, even with evidence growing of a wide range of ingestion by numerous species (Rochman et al., 2013).

Nonetheless, some researchers suggest there is no significant connection and thus importance of the cycling and bioaccumulation of organic pollutants or hydrophobic organic contaminants (HOCs) with microplastics (Lohmann, 2017). POPs are a subset of persistent HOCs and there is evidence that suggests that microplastics can and do absorb high concentrations of organic pollutants, though the significance of the sorption and transfer of organic contaminants has been suggested to be relatively low (Zarfl & Matthies, 2010; Gouin et al., 2011; Koelmans et al., 2019; Ziccardi et al., 2016). One suggestion is that organisms can uptake these contaminants already from the water, sediment and food and the added uptake from plastics does not cause any substantial increase in these chemicals for the organism (Koelmans et al., 2014). Furthermore, it is suggested that there are simply not enough microplastics in the (marine and likely freshwater) environment to outcompete the partitioning of POPs to water and natural organic matter (Lohmann, 2017).

A critical limit of microplastic pollution may not have been reached yet in most environments but could become problematic in the future or at high density locations.

Another chemical factor of microplastics to consider are the additives used in the manufacturing of polymers and plastics. As plastics have been suggested to absorb POPs and potentially released after ingestion, are plastic additives also released after ingestion? Again, this is a sparsely researched topic in freshwater ecosystems, and there needs to be more attention given to phenolic additive-derived chemicals from microplastics in the food web (Teuten et al., 2009). Nonetheless, some marine studies indicate there is no relevance of these chemicals in the uptake of organisms (Koelmans et al., 2014; Rochman et al., 2014), further suggesting that more research is needed on the interaction of microplastics, chemicals, pollutants and biota, particularly in freshwater environments where data is lacking.

2.7.2 Physical effects of microplastic ingestion by organisms in freshwater

The chemical effects of microplastics are still relatively unknown, particularly in freshwater environments. There is, however, more research into the potential physical damage of microplastics in aquatic organisms although again the focus is primarily on marine organisms and researchers are left to draw parallels to freshwater organisms.

Adverse physical reactions in organisms to microplastic ingestion, include oxidative stress (Fossi et al., 2014; Barboza et al., 2018; P. Oliveira et al., 2018), decreased feeding rates (Denuncio et al., 2011; Cole et al., 2011; Setälä et al., 2016), weight loss (Besseling et al., 2013) fitness reduction (Besseling et al., 2013), digestive tract blockages (Derraik, 2002; Galgani et al., 2010) inflammatory responses (von Moos et al., 2012) and neurotoxicity (Oliveira et al., 2018). However, additional research is required to better understand how microplastics may be impacting freshwater organisms specifically as the physical, chemical, and biological conditions of marine environments are fundamentally different from freshwater ecosystems.

With this in mind, the research will focus on a freshwater location, namely the Ottawa River watershed, to examine the temporal and spatial distribution of microplastics throughout the total watershed.

2.8 Study Region

The Ottawa River (also known as Rivière des Outaouais in French and Kitchissippi in Algonquin) runs approximately 1,272 km from its headwaters in Lac des Outaouais in Quebec (QC) to the Lake of Two Mountains, or Lac des Deux Montagnes, slightly West of Montreal City in Quebec (Figure 2.4). From its headwaters, the river flows west through very sparsely populated areas of Quebec including the La Verendrye Wildlife Reserve, with its first major population centre of note being Rouyn-Noranda, QC (a population of approximately 42,000). Rouyn-Noranda is located near the Kinojévis River, a tributary which joins the Ottawa River approximately 450 km from its headwaters. Further west, the river enters Lake Temiskaming at Notre-Dame-du-Nord, where shortly thereafter, the river becomes the Ontario and Quebec provincial border until the Carillon Dam, approximately 620 km further downstream, where the river then flows back into Quebec until its terminus.

There are 19 notable tributaries of the Ottawa River, nine in the province of Ontario, namely the South Nation, Rideau, Mississippi, Madawaska, Bonnechere, Petawawa, Mattawa, Montreal and Blanche rivers, with 10 in the province of Quebec, namely the Nord, Rouge, Petite Nation, Du Lièvre, Gatineau, Coulonge, Noire, Dumoine, Kipawa and

Kinojévis rivers (Figure 2.4). This equates to a total watershed area of approximately 140,000 km^2, where 65% of the watershed is in Quebec, and 45% of the watershed in Ontario. At the Lake of Two Mountains, the Ottawa River drains into the Saint Lawrence River, where it is the largest tributary of the Saint Lawrence watershed. Furthermore, the Saint Lawrence River drains the Great lakes, and this in combination with the Ottawa River, make it one of the most significant drainage basins of North America.

The majority of the river flows through the Canadian Shield, one of the worlds largest geologic continental shields with lower reaches of the river flowing through limestone plains and glacial deposits. The modern river and its tributaries have resulted from the retreat of the Champlain Sea, however, previously there was a much greater flow through the ancestral Ottawa River, as large Ontario glacial lakes, the upper Great Lakes, and lakes in the prairie provinces all drained into the Ottawa River. About 8000 years ago the channel shifted establishing the present-day drainage basin (Ottawa River Keeper, 2022).

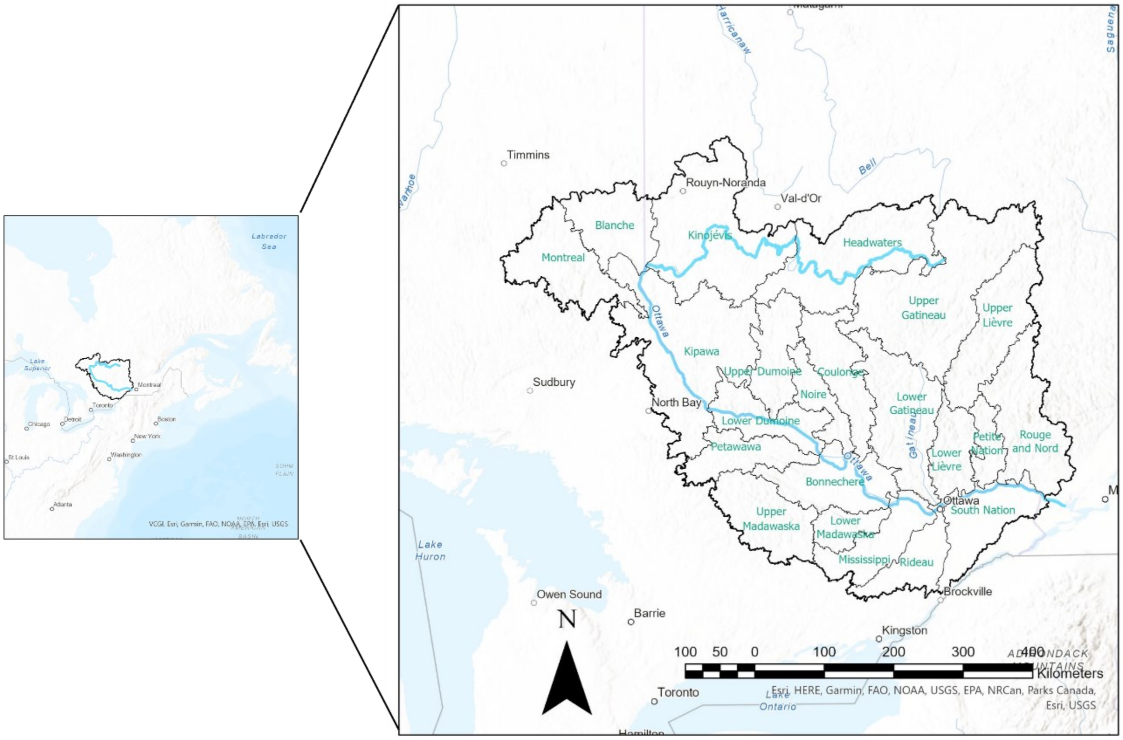

Figure 2-4 The Ottawa River watershed including the main channel and major sub basins.

3. Citizen science as a technique for measuring the spatial distribution or microplastics in water samples in a large watershed

3.1 Introduction

Global production of plastics has exceeded 300 million metric tonnes a year since 2014 (Plastics Europe, 2015) with none of the commonly used plastics being biodegradable (Geyer et al., 2017). Consequently, tiny pieces of plastics known as microplastics, small plastic pieces less than 5 mm (Masura et al., 2015) are now ubiquitous in today's ecosystems. with suggestions of potention harm River environments have recently been identified as containing microplastics (Leslie et al., 2017; Vermaire et al., 2017; Wang et al., 2017; Windsor et al., 2019). In fact, rivers are one of the major conduits (in addition to a sink and transformer) of microplastics to marine environments, with between 1.15 and 2.41 million tonnes of plastic waste entering the ocean via rivers annually (LeBreton et al. 2017). Microplastics have also recently been identified in river biota (Windsor et al., 2019), raising the level of concern for freshwater systems. Currently, eliminating plastics can only be achieved by destructive thermal treatment, such as combustion or pyrolysis (Geyer et al. 2017). With growing accumulation and no realistic method of elimination, plastic pollution is now considered as a potential geological indicator of the Anthropocene era (Zalasiewicz et al., 2016).

Microplastics can be classified as primary or secondary microplastics; primary microplastics are plastics that are manufactured, for example, microbeads in cosmetic products and industrial abrasives. Secondary microplastics are the result of breakdown or fragmentation of larger plastics, including polymer fragments from synthetic clothing. The majority of microplastic research has centred around oceans, however, there is growing evidence suggesting that microplastic pollution is also prevalent in freshwater systems (Castañeda et al., 2014; Anderson et al., 2017; Li et al., 2017; Vermaire et al., 2017; Wagner and Lambert 2018). With the establishment of microplastics in the Ottawa River (Vermaire et al. 2017), it is important to examine the spatial distribution, to understand the potential source, fate and impact of microplastics while quantifying the amount of microplastics throughout the river system. As the Ottawa River is over 1200 km in length, a single research team could find it problematic and expensive to obtain adequate spatial coverage. One option to complement microplastic research and establish a microplastic monitoring network on the Ottawa River is to engage citizen scientists, who can assist with spatial analysis. Working with citizen scientists in monitoring microplastic pollution has the added benefit of public engagement on this important environmental issue.

One example of a successful microplastic citizen science project is the International Pellet Watch (IPW), whereby volunteers report on plastic pellets (or primary microplastics) they have identified on global beaches (Yeo et al., 2015). The added advantage to the IPW project is increasing the awareness of citizen scientists to the growing concern of plastic waste (in this case primary microplastics). Additionally, previous river litter citizen science studies have acknowledged the strong correlation of volunteer and professional data (Rech et al., 2015), demonstrating the potential to utilise citizen science for spatial analysis of microplastics in river environments. Citizen science can increase the scope and spatial coverage of monitoring programs while providing access to private or restricted lands that may not normally be easily accessible. Citizen science provides a complementary monitoring approach whereby a researcher's time and resources may otherwise be limited, while offering the opportunity to establish a more extensive network of sampling locations (Hidalgo-Ruz et al., 2012).

Despite the many advantages, citizen science has been criticised, with the practice not necessarily accepted as a valid method of scientific investigation: research papers with citizen science components have traditionally faced issues to be published (Bonney et al., 2014). Concerns over the quality of data contributed by volunteers arise due to several issues including potential error, bias, reliability of data collection, lack of appropriate protocols, training or oversight, and concerns of replicability, comparability, and completeness. These concerns potentially limit citizen science data in management decisions (Conrad & Hilchey, 2011; Gillett et al., 2012). However, even with the challenges of citizen science, applying it as a tool can bring substantial benefits (Conrad and Hilchey, 2011). Future research should incorporate citizen science, where appropriate, particularly when large spatial scales are involved (Galgani et al., 2010; Bergmann et al., 2015).

Researching the spatial distribution of microplastics in the Ottawa Watershed has enormous potential to engage citizen scientists through river advocacy groups, such as the Ottawa Riverkeeper. Their vested interest in the optimal health of the Ottawa River creates a unique opportunity to engage the network of volunteers through the Ottawa Riverkeeper membership in assisting with spatial analysis of microplastics. The Ottawa Riverkeeper volunteer network in collaboration with microplastic researchers could greatly increase the spatial scope of microplastic sampling sites in the Ottawa Watershed. Therefore, the objective of this research was to quantify the spatial abundance of microplastics in the Ottawa River using the volunteer network of the Ottawa Riverkeeper. Additionally, we investigated the effectiveness of the citizen science collaboration for microplastic monitoring and provided recommendations for future freshwater microplastic pollution projects involving citizen science.

3.2 Study Region

The Ottawa River is located in the Canadian provinces of Ontario and Quebec with a large portion of the river acting as the border between the two provinces (Figure 3.1). The headwater of the river is Lac des Outaouais in Quebec which drains into the Lake of Two Mountains and the St. Lawrence Waterway West of Montreal, Quebec, approximately 1271 km downriver. The Ottawa River watershed covers an area of approximately 146,000 km^2, which is greater than the size of England (approximately 130,000 km^2). The average flow of the river measured at Carillon Dam in the southern section of the river is 1,950 m^3/s with a historical daily minimum recorded at 301 m^3/sec in 1971, and a maximum daily flow of 8862 m^3/sec recorded in 2017 (Ottawa Riverkeeper, 2021). The Ottawa River was recently designated a Canadian Heritage River by both the provinces of Quebec and Ontario, demonstrating its significant economic, cultural and ecological importance to Canada.

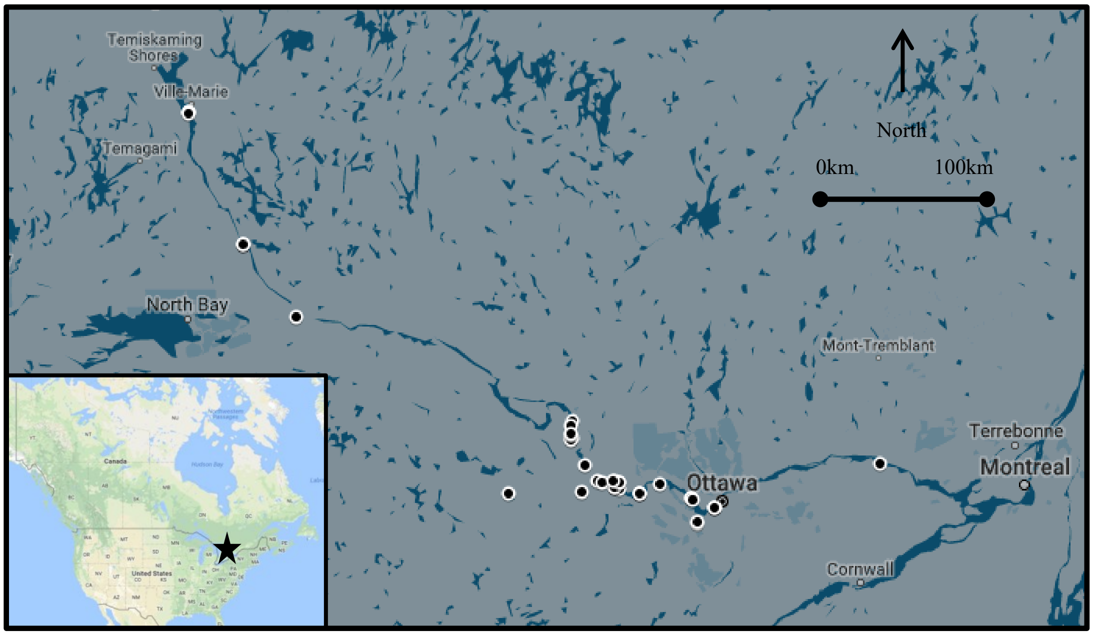

Figure 3-1 Sample locations as determined by the citizen scientists along the Ottawa River (Google Maps 2018).

3.3 Methods

The Riverwatch volunteer network representing 40 locations in the Ottawa River watershed was invited to take part in the sampling project. Citizen scientist groups and individuals representing 22 Riverwatch locations demonstrated an interest in the sampling project. These citizen scientists were given sampling kits consisting of a metal funnel, four 100-µm nitex mesh filters for the funnel and a 4-L container (Figure 3.2) to collect the river water in order to run through the funnel. The nitex mesh filters were pre-inspected under a stereomicroscope before given to the citizen scientists to ensure there was no contamination with microplastics. The sampling kits were distributed during an Ottawa Riverkeeper volunteer meeting, wherein, a 30-minute instructional presentation was given on microplastics and how to collect samples for the study. The volunteers were instructed to filter 100-L of water through their funnel, carefully remove the nitex mesh and place it in a whirlpak bag and mail the samples to the laboratory for analysis. A volume of 100-L was selected to allow for reasonable sampling time for the citizen scientists, whereby each sample could be conducted between approximately 20-30 minutes. Each volunteer was to obtain triplicate samples from the Ottawa River at or near their residence, or at their desired location and one sample of tap water at their residence to serve as a control. Filters were sealed in separate whirlpak bags and mailed to the Aquatic Ecosystems and Environmental Change Laboratory at Carleton University for analysis (Figure 3.2).

In the laboratory, the 100-µm mesh filter was washed into a clean beaker using distilled water. A 30% hydrogen peroxide (H_2O_2) solution was then added to the sample and heated to 80^oC for 7 hours to oxidize any organic material that may have been in the samples (Masura et al., 2015). After the heating cycle, the solution was cooled and then passed through a clean 100-μm mesh filter. The filter was then backwashed with distilled water into a clean petri dish for quantification of microplastics under a Leica stereomicroscope at 20 to 40X magnification, identifying the amount, type and colour of any microplastics present in the samples. In addition to the field control samples collected by the volunteers, control samples were also conducted in the laboratory with distilled water to estimate potential laboratory contamination with microplastics. Statistical analysis included examining average microplastic concentrations and if there was any relationship to the location where the samples were taken (for example, shorelines versus the channel) and any noticeable low to high concentrations moving downstream on the river.

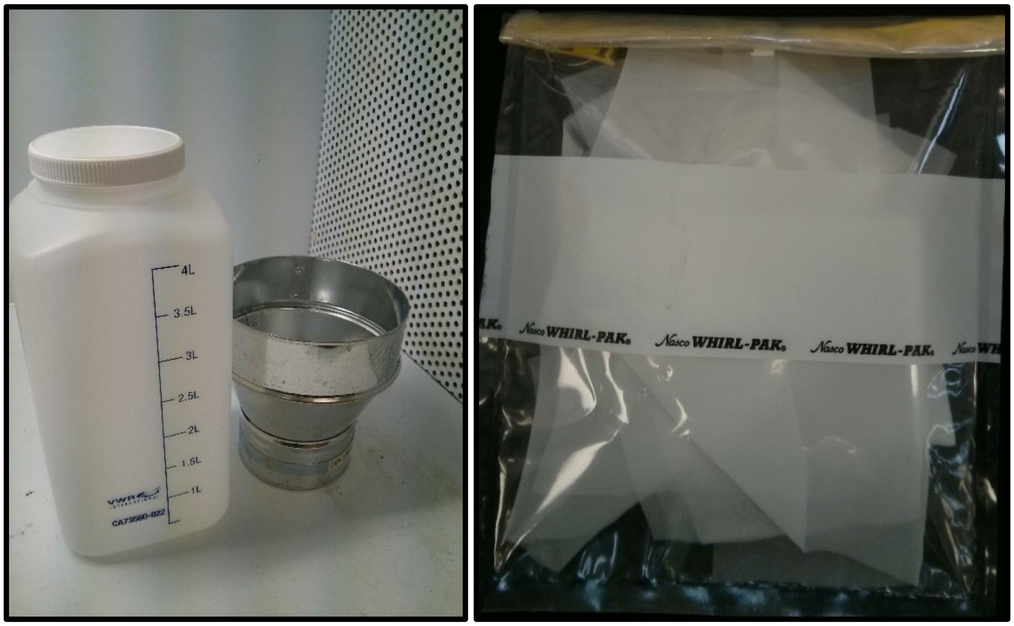

Figure 3-2 Container and filter provided to citizen scientists (a) and a whirl-pack bags (b) the citizen scientists used to post samples back to the laboratory.

3.4 Results

A total of 43 microplastic samples were received, representing 28 sample points from 17 groups of citizen scientists covering a 550 km river reach. Most samples received had the associated sample sheets filled correctly. However, two citizen scientists mailed in handwritten sample sheets with one other citizen scientist sending a different sample sheet with less than the required information. The volunteers were asked to collect three samples at their desired location and to include a tap water control. However, only six of the citizen scientists followed these instructions exactly, with three volunteers conducting four samples without a control, two volunteers conducting three samples without a control, four volunteers conducting only one sample, three of which without a control and five citizen scientists chose different locations for each of their samples (Table 3.1). Moreover, one marked a control sample that appeared to be a river sample (i.e. very high organic content), thus, some labelling errors likely occurred from the citizen scientists.

All but one field sample contained microplastics and all of the field control samples contained microplastics. One of two laboratory controls contained microplastics (Table 3.1). The mean microplastic concentration for all sample locations was 0.11 microplastics per litre (mp/L) with a standard deviation of 0.09. The Ottawa River sample average (not including tributary samples) was 0.12 mp/L. Controls presented a mean of 0.02 mp/L with a standard deviation of 0.01. The greatest concentration of sample points along the Ottawa River was from Fort-Coulonge Quebec (QC), to Ottawa, ON (approximately a 115km stretch of the river) containing 33 of the 43 samples (Figure 3.1).

The highest concentration of microplastics, 0.41 mp/L, from the citizen science sample points was measured at Pine Lodge, QC (one of the single samples) (Table 3.1).

Additional locations that demonstrated high concentrations of microplastics (over 0.2 mp/L) were Watts Creek Pond, ON, a tributary of the Ottawa River in Kanata, ON, Fort-Coulonge, QC, Woolsey Beach Camp, east of Arnprior, ON, Portage du Fort, QC, and Castleford, ON. However, all of these samples were only single samples and not a triplicate sample. Some sample locations recorded low microplastic concentrations, for example, Westboro Beach in the Ottawa urban area with a triplicate concentration of 0.02 mp/L. Another triplicate sample with a low microplastic concentration was recorded at East Arnprior with a concentration of 0.04 mp/L (Table 3.1). The highest triplicate sample concentration was 0.08 mp/L recorded at Lake Temiskaming.

There was no distinctive low to high microplastic concentration as you move downstream on the river, with concentrations varying throughout the sample points. However, two of the samples downstream of a primary wastewater treatment plant demonstrated high microplastic concentrations (0.22 and 0.26 mp/L) with one sample downstream of a secondary wastewater treatment plant also demonstrating concentration of 0.11 mp/L. However, this is still lower than the average Ottawa River concentration of 0.12 mp/L (Table 3.1). Additionally, samples that were obtained from a boat (n=7; i.e. open water) averaged 0.15 mp/L, with samples collected from the river shoreline (n=5) exhibiting lower microplastic concentrations, averaging 0.05 mp/L. A t-test to test if there was a statistical difference between shoreline and offshore samples returned alpha p-value value of 0.008, suggesting a significant difference between locations (Table 3.2). Samples obtained from a wharf or pier (n=5) had a mean of 0.10 mp/L (Table 3.2). There was no statistical difference between wharf and open water samples (p-value of 0.39) with wharf and shorelines samples demonstrating a p-value of 0.05, rather than below 0.05. Not all samples received from the citizen scientists noted their sample locations. Two volunteers noted rain during their sampling, however, very few microplastics were found in these samples ranging from 0.00 to 0.07 mp/L. Microplastics identified throughout all of the citizen scientist samples were predominantly microfibers with only five microplastics identified as non-microfiber (Figure 3.3). Therefore, microfibers accounted for approximately 98% of the microplastics identified in the samples.

Figure 3-3 Microfibres from a citizen science sample collected at Pine Lodge, Quebec.

Table 3-1 Microplastic concentration for the samples locations provided by the citizen scientists including microplastic concentration for field and laboratory samples.

Sample Location Name	**Distance upstream of Hawkesbury, Ontario (km)**	**Microplastic Concentration (mp/L)**	**Sample Location Name (Ottawa River Tributary)**	**Microplastic Concentration (mp/L)**
Hawkesbury	0	0.11	Watts' Creek Pond	0.21
Westboro Beach	100	0.02	Watts' Creek	0.08
Westboro	100	0.06	Bonnechere River (Renfrew)	0.05
Cote McKay	118	0.05	Bonnechere River Mouth	0.01
Woosley Beach	138	0.22		
Arnpior	153	0.04	**Sample Average**	**0.11**
Sand Point Wharf	165	0.17	**Ottawa River Average**	**0.12**
Norway Bay	165	0.04		
Norway Bay	166	0.01	Field Controls	
Rhoddy's Bay	168	0.07	Field Control 1	0.03
Pine Lodge	169	0.40	Field Control 2	0.05
Pine Lodge	170	0.17	Field Control 3	0.03
Castleford	178	0.27	Field Control 4	0.03
Portage du Fort	190	0.20	Field Control 5	0.02
Rocher Vendu Rapids	210	0.06	**Field Control Average**	**0.03**
Fort-Coulonge (Sullivan Island)	213	0.06		
Fort-Coulonge (La Serpent)	214	0.02	Laboratory Controls	
Fort-Coulonge (Lemione)	219	0.09	Laboratory Control 1	0.01
Fort-Coulonge (Township)	212	0.26	Laboratory Control 2	0.00
Mattawa	402	0.10	**Laboratory Control Average**	**0.01**
Temiskaming Township	453	0.04		
Lake Temiskaming	543	0.08		

Table 3-2 Microplastic concentrations in relation to sampling positions, namely shoreline, wharf or offshore with T-Test results conducted between the sample locations.

Offshore Samples	Microplastic Concentration (mp/L)	P-Value
Fort-Coulonge	0.26	
Fort-Coulonge	0.09	**Offshore vs Shoreline**
Fort-Coulonge	0.06	0.008
Fort-Coulonge	0.02	
Pine Lodge	0.41	**Wharf/Pier vs Offshore**
Pine Lodge	0.17	0.39
Rhoddy's Bay	0.07	
Offshore Sample Average	**0.15**	**Shoreline vs Wharf/Pier**
Shoreline Samples		0.05
Westboro Beach	0.02	
Westboro	0.06	
Rhoddys Bay	0.07	
Arnprior	0.04	
Temiskaming Township	0.04	
Shoreline Sample Average	**0.05**	
Wharf/Pier Samples		
Norway Bay	0.04	
Lake Temiskaming	0.08	
Sand Point Wharf	0.17	

3.5 Discussion

The citizen science collaboration provided the opportunity to greatly expand the spatial scope of the researchers, covering a 550 km stretch of the Ottawa River. A total of 9 of 17 citizen scientist groups noted specific locations of sampling sites within the river, i.e. shoreline, wharf or offshore. For the nine citizen scientists who reported a location, there was a difference in sample concentrations obtained from a boat (offshore) compared to shoreline samples suggesting, even with the small sample size, cross-sectional river flow could influence microplastic distribution. Spatial distribution of microplastics through the water column and cross-sectionally has not been examined thoroughly in previous research to date in river environments and this is an area of research that could improve our understanding of plastic flux throughout river systems. Additionally, river geography, channel shape, currents as well as river flow patterns could also affect microplastic distribution, suggested in the non-linear microplastic concentrations throughout our sample points. However, there was no consistency in timing of the samples, thus, the anomalies could suggest temporal influences due to stormwater inflows or peak river flows for example, as the citizen science samples were not all taken on the same day. Therefore, these results suggest it will be advantageous to conduct further research on the distribution of microplastics across a river profile in addition to surface and riverbed microplastic distribution, to obtain a more comprehensive understanding of the microplastic interaction and distribution within a river's water column.

Other researchers have noted that wastewater treatment plants are a conduit for microplastics to river systems (Estahbanati and Fahrenfeld 2016; Leslie et al. 2017; Talvitie et al., 2017; Ziajahromi et al., 2017;). Furthermore, the level of wastewater treatment, whether primary, secondary or tertiary, has been acknowledged to affect the amount of microplastics in the wastewater effluent. Tertiary wastewater treatment removes the most microplastics (Talvitie et al., 2017; Ziajahromi et al., 2017) and secondary and primary treatment potentially releasing a higher microplastic concentrations in their effluent. Some high concentrations of microplastics were recovered downstream of primary wastewater treatment plants (Fort-Coulonge, Mattawa and Woolsey Beach camp), but the sample downstream of the secondary wastewater treatment plant in Hawkesbury had lower concentrations of microplastic contamination. Previous research also highlights that wastewater treatment plants are a conduit for microplastics into river environments (Ziajahromi et al., 2017).

Apart from a few plastic fragments in three samples, all of the microplastics identified in the citizen science samples were microfibers. A previous study on the Ottawa River (Vermaire et al. 2017) also found that microfibers were the dominant type of microplastic found in the river, with the research noting microfibers accounting for more than 95% of microplastics found. Our samples suggest similar results with approximately 98% of the microplastics identified as microfibers. This suggests the influence of wastewater treatment plants on microfiber contamination, as secondary microplastics such as these fibres tend to be the most problematic with wastewater effluent (Estahbanati and Fahrenfeld 2016). Previous research suggests even a trace amount of microplastics in the final effluent can amount to a substantial deposition into rivers over time (Mason et al., 2016; Murphy et al. 2016; Talvitie et al., 2017; Sutton et al., 2016; Ziajahromi et al., 2017). The major source of microfibers in wastewater effluent can be attributed to fibres from synthetic clothing, where one garment can shed up to 1900 fibres during one washing cycle

(Browne et al., 2011). Furthermore, if a municipality on the Ottawa River is served by a combined sewer system, any overflow event caused by heavy rainfall would contain untreated sewage and storm water, where the microfibers and microplastics normally captured by a wastewater treatment plant, would flow directly into the Ottawa River. For example, a large proportion of the Ottawa urban area still utilises a combined sewer system, in addition to the Gatineau urban area on the Quebec side of the Ottawa River. In fact, Gatineau has an estimated 1,300 overflow events annually (Ottawa Riverkeeper, 2017). It is important to consider stormwater inputs as a conduit for microplastics into the Ottawa River, especially downstream of combined sewer overflows during an overflow event.

3.6 A Critical review of the role of citizen science in microplastic research on the Ottawa River

Engaging a group of citizen scientists to participate in microplastic research, such as the Riverwatch volunteers within the Ottawa Riverkeeper, who have a genuine interest in the ecological health of the Ottawa River, presents great opportunities and advantages. However, there are drawbacks to this method of data collection.

Citizen scientists along the Ottawa River provided a broad spatial scale of sample points from Lake Temiskaming to Hawkesbury ON, an approximate 550 kilometre stretch of the Ottawa River. This represents a large proportion of the Ottawa River, whereby a researcher or group could have increased logistical challenges and/or access challenges covering this vast spatial scale. Moreover, though the specific points were chosen by the citizen scientists, they may not necessarily represent the sampling points the researcher may have chosen, for example, a researcher may identify stormwater outflow pipes and/or sample points close to wastewater treatment plants. For this particular study, the locations were chosen by the citizen scientist, typically close to where they may reside, rather than suggested or pre-determined by the research team.

Establishing temporal consistency is potentially difficult for citizen science sampling projects. However, with growing potential and evidence for temporal variation in river systems, a researcher may have specific temporal events in mind, for example, high rainfall events, high snow melt days, sewage overflow events, or specific days of the month or year. Nonetheless, it is possible to guide citizen scientists on potential spatial and temporal objectives. This can be achieved with more specific instructions relating to the researcher's temporal and spatial sampling objectives. However, the researcher must be cautious with too many detailed instructions or demands on the citizen scientists, as this could deter the citizen from undertaking the sampling.

The sampling handouts given to the citizen scientists provided a step-by-step guide to recording information while they collected samples. However, not all of these sample sheets were used, and varying levels of detail were provided on the sheets collected, with some volunteers submitting handwritten notes. The importance of filling in the standardised form needs to be emphasised to future volunteers, as it helps ensure sample integrity. Nonetheless, the importance of filling in the sample sheets was conveyed to the citizen scientists, even with the inconsistencies on the sample sheets received.

Furthermore, not all citizen scientists conducted field-blanks and the number of samples from each sampling location varied. Control samples are particularly important for freshwater microplastic research, given the potential contamination from microfibers in the air (Ho et al., 2003; Dehghani et al., 2017). Thus, it is important to establish background concentrations with control samples. Collecting a control in addition to triplicate samples at one location was conveyed to the volunteers, however, there was still some missing controls in addition to receiving three (or even four) samples from a citizen scientist at different locations. It is important that in future microplastic monitoring programs, the importance of field-blanks is stressed to the volunteers and that samples without controls be omitted from data collection in monitoring programs.

The citizen scientists were asked to filter 100-L of water through a funnel and mesh to collect microplastic samples for analysis in the laboratory. The advantage of this sampling technique is that the process is fairly simple and easy for citizen scientists to complete. A higher volume sampling would negate some of the potential contamination from airborne microfibers, however, it was impractical to request a citizen scientist to conduct 1000-L samples (or greater volume samples) with the water bottle sample technique that was used in this study. A higher volume container, or bucket could reduce the number of iterations of passing water through the funnels and potentially increase the water volume sampled. Additionally, small pumps could be utilised, so the citizen scientists could increase their water sample volumes. Furthermore, one alternative to incorporate larger volume sampling is with a sampling net, conducted by the citizen scientist, with the volunteer still having an active role to play in the sampling. However, it is important to note that if a selected sample area demonstrates a larger percentage of fragments, the 100-L volume size could very well be an adequate sample volume for a citizen scientist. The issue for this research is that the Ottawa River is dominated by fibres, and fibres are the main concern for contamination in the field and during processing of samples. Increasing sample volume is one way to reduce contamination potential from fibres, however, this issue may not be applicable to all sample areas and thus is not necessarily a specific disadvantage to citizen science sampling.

This research on the Ottawa River highlights the potential for citizen scientists in microplastic monitoring programs. The researchers and volunteers greatly enjoyed this research, and valuable data were obtained on microplastic concentrations in the Ottawa River. Our initial approach at citizen science data collection on microplastic distribution in the Ottawa River has demonstrated some promising results, though there is always room for improvement, particularly in this study with emphasizing the importance of field-blanks to the volunteers and increasing the volume of water sampled.

3.7 Recommendations

This collaboration with citizen scientists of the Ottawa River provided a unique opportunity to demonstrate proof of project concept to gather microplastic samples over a large spatial area of the Ottawa River. The results suggested the influence of wastewater treatment plants on microplastic concentrations in addition to the first data on the spatial variability of microplastics across the Ottawa Watershed. Future microplastic monitoring programs should consider the following recommendations to strengthen the reliability and replicability of the microplastic concentration values obtained:

- A larger sample volume to reduce the influence of contamination in the laboratory or in the field. This could include boat sampling with manta nets at the volunteer's desired locations rather than increasing water bottle sampling volumes, which could become tedious and discourage participation. This study employed a 100-L sampling methodology but given the potential of atmospheric contamination by microfibers water volumes of an order of magnitude or greater are recommended.

- It is important to include both field and laboratory blanks as a control in order to determine contamination potential of the samples.
- Standardizing sample locations of interest to citizen scientists is vital. For example, samples could be collected up and down stream of wastewater treatment plants or stormwater outflows in their area of the Ottawa River. Additionally, a combination of shoreline and boat sampling could be undertaken.
- It is recommended that the citizen scientists conduct mail in samples (or hand in their samples to a processing lab), as this allows the researcher to assure quality control during sample processing, as opposed to the citizen scientists trying to analyse the samples in the field or at their own residence. Though this could cut down the processing time and reduce the potential for contamination from airborne microplastics, stringent and controlled sample processing in the laboratory conducted by the researcher or researchers is suggested. However, as the citizen scientists are not involved in the processing of the samples, it does reduce their role in the project and suggests they are more citizen samplers rather than citizen scientists.
- Coordinating sampling dates for a better comparison among sample sites.
- Encourage the use of the supplied sampling sheets for standardization of results. The sample sheet was made available for the current research, however, any subsequent citizen science research should highlight the importance of a common sampling data sheet and utilising it to achieve more consistent results.

- Ensure results are made available to the volunteers including presentations of the results, to maintain interest and a willingness to participate in the collaboration, Furthermore, after surveying the Ottawa Riverkeeper volunteers, it was noted that providing results in a timely manner was important to maintain vested interest in the citizen science program.

The collaboration with the Ottawa Riverkeeper volunteers provided a unique citizen science opportunity to spatially expand microplastic sampling on the Ottawa River. Moreover, engaging volunteers on microplastic research on the Ottawa River creates awareness of the problem, which the volunteers actively discuss with the wider community as the issue resonates with them on a personal level. The volunteers become important activists for monitoring and understanding microplastics in the Ottawa River with their continued and engaged collaboration as citizen scientists.

Though improvements are certainly needed, citizen science can offer an inexpensive method to enhance microplastic monitoring programs. Emphasizing the importance of strictly following instructions in sampling protocols is vital in obtaining useful results. Additionally, the results could be enhanced by devising a way for the citizen scientists to sample greater volumes of water without exponentially increasing the time it takes for them to sample.

4. Evaluating community science sampling for microplastics in shore sediments of large river watersheds

4.1 Introduction

Microplastics have become a global concern and have been identified from the highest point on the globe (Napper et al., 2020) to the deepest ocean trench (Peng et al., 2018). Microplastics are defined as plastic pieces less than 5mm (Masura et al., 2015) and can be classed as either primary microplastics, manufactured at this micro-scale, or secondary microplastics that have fragmented from larger plastics. Microplastic pollution continues to increase at alarming rates and now is considered the primary constituent of marine debris (Moore, 2008; Barnes et al., 2009; Cole et al., 2011). As microplastics are small, they are considered bioavailable to various organisms, thus have the potential to sorb and/or release toxic chemicals, with potential transfer through the food chain (Reisser et al., 2014; Hurley et al., 2017). To date a larger proportion of microplastic research has focused on the marine environment, however, increased attention has been given to freshwater systems, especially rivers that have been identified as the major conduit of plastics to marine areas with between 1.15 and 2.41 million tonnes of plastic waste entering oceans via rivers annually (Lebreton et al., 2017). With only limited microplastic research in freshwater systems, approximately 4% compared to marine research (Wagner and Lambert, 2018), and with even less microplastic research focused on Canadian river systems (Castañeda et al., 2014; Vermaire et al., 2017; Forrest et al., 2019; Crew et al., 2020; Bujaczek et al.,

2021) it is important to increase the scope of microplastic research in freshwater systems to enhance the understanding of microplastic inputs, outputs and sinks throughout freshwater watersheds. Microplastic concentration in river systems can be highly variable both spatially and temporally, indicating that researchers are only at the early stages of understanding these mechanisms (Forrest et al., 2020).

Citizen science is a potential technique to expand spatial monitoring for microplastics, especially across large project areas. Citizen science can be defined as scientific research and monitoring whereby members of the public collect, categorise, transcribe or analyse scientific data (Bonney et al., 2014). However, very few microplastic projects have utilised this approach to data collection. As with microplastic research in general, initial citizen science projects were focused on the marine area, including the International Pellet Watch (IPW) and citizen science projects monitoring for the plastic 'nurdle' (Tunnell et al., 2020). It can be assumed freshwater citizen science microplastic monitoring is still being evaluated as a valid complementary research tool (Forrest et al., 2019), nevertheless, it has been applied previously at the watershed scale examining microplastics in river water (Barrows et al., 2018; Forrest et al., 2019). Previous research in the Ottawa River determined that for citizen science projects larger volumes of water were required to gather representative microplastic counts in river water (Forrest et al. 2019), thus, an easier approach to utilise citizen science as a spatial research tool was to evaluate the collection of beach sediments that are more easily accessible and potentially contain a greater concentration of microplastics.

However, there has been criticism with citizen science projects, with suggestions it is not scientifically valid, with citizen science based projects struggling to be accepted in scientific journals. (Bonney et al., 2014). Some of this criticism is due to potential error, bias, reliability of data collection, which can lead to skepticism in data quality, the lack of applicable protocols and/or training of the volunteers. This can lead to concerns of replicability and subsequently, citizen scientists, and/or their data are often not considered in management decisions (Conrad & Hilchey, 2011; Gillett et al., 2012). However, citizen science can present substantial benefits (Conrad and Hilchey, 2011) and future microplastic research should strive to incorporate or utilise citizen science, particularly when larger spatial scales are involved (Bergmann et al., 2015).

The objective of this research was to work with citizen scientists to collect near-shore river sediments for the analysis of microplastics at the watershed scale. In addition, we evaluated the potential of citizen science collected samples as a viable complementary spatial analysis tool for microplastic research in freshwater systems.

4.2 Study Region

The Ottawa River is a designated heritage river in Canada due to its cultural, economic and ecological importance. The river is considered the major tributary of the St. Lawrence River and flows from its headwaters at Lac des Outaouais in Quebec Canada, to Lake of Two Mountains (Lac des Deux Montagnes), near Montreal Quebec, approximately 1272 kilometers in length (Figure 4.1). The Ottawa River watershed is large with an approximate area of 146,000 km^2 (greater than the area of England) making it difficult for single research or monitoring teams to collect representative samples from the watershed. The average flow of the Ottawa River, measured at the Carillon Dam (approximately 30 kilometers upstream of the terminus) is 1950 m^3/s, with daily minimum recorded at 306

m^3/s in 1971 and a maximum daily flow of 9217 m^3/sec in 2019 (Ottawa Riverkeeper, 2021).

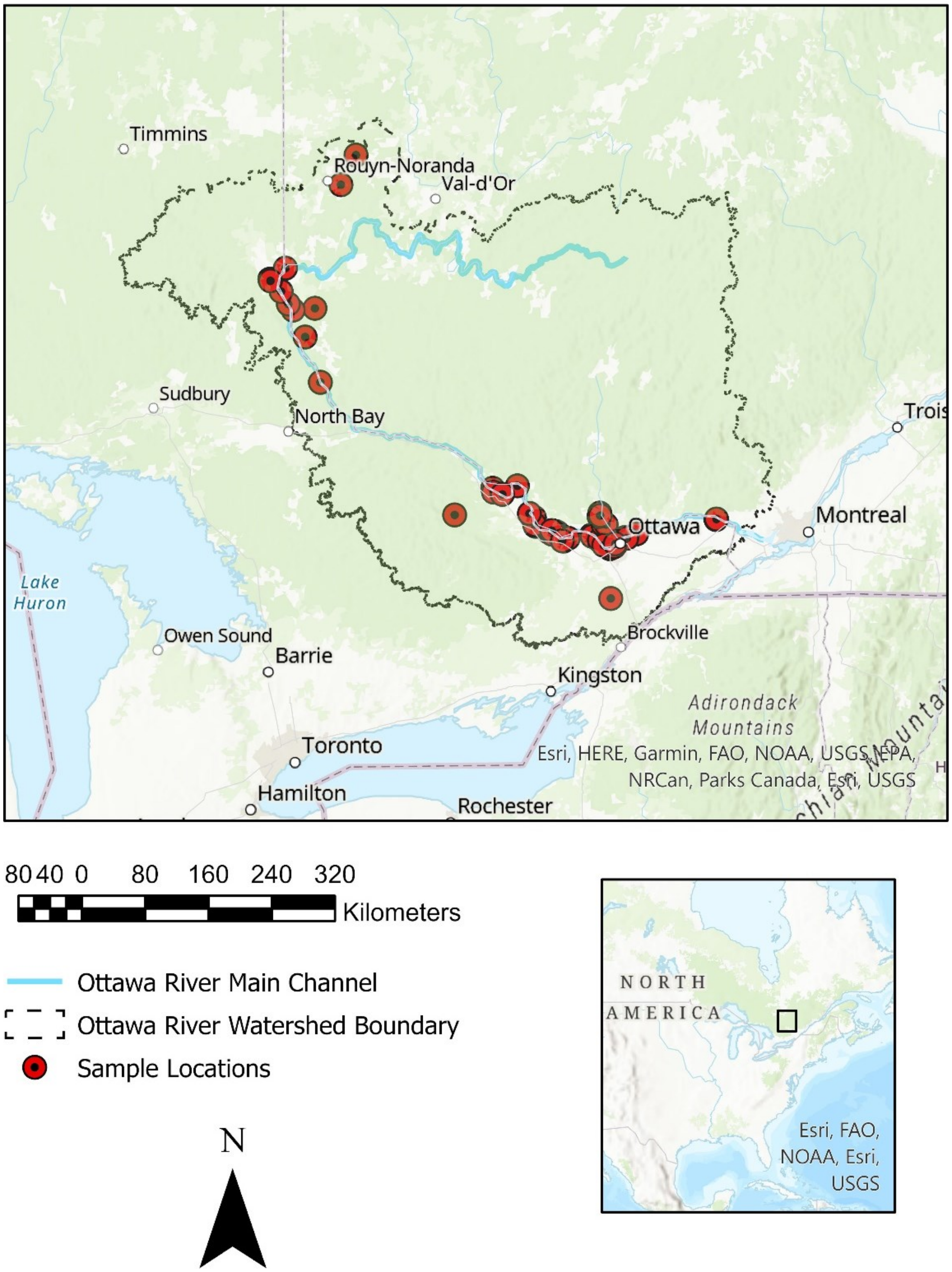

Figure 4-1 The Ottawa River watershed boundary and main channel with the citizen scientist sample locations.

4.3 The Citizen Science Network

The group of volunteers who participated in the citizen science project come from an established volunteer network through the non-governmental organization Ottawa Riverkeeper, who participate in monitoring various river water quality indices throughout the watershed including tributaries of the Ottawa River. With such an expansive watershed, this volunteer base, known as the ‘Riverwatchers’ increases the spatial scope of monitoring various pollution indicators and have previously contributed citizen science work monitoring microplastics in river water (Forrest et al., 2019). Additionally, members of the Riverwatch network have ties to other volunteer and community networks that in turn can increase the spatial reach of the research. For example, some additional groups and community members who took part in the sampling along with a Riverwatcher citizen scientist included school classes, community centre groups, a rafting club as well as interested youth participants.

4.4 Methodology

A total of 47 sediment sampling kits were distributed to Community Scientists. Sampling kits were handed to the Community Scientists in May 2019, after the Ottawa River freshet. Once the Community Scientists received the kits, they were free to sample during the late spring and summer period of 2019. Sample dates from the received sediment were between June and September (inclusive) of 2019. Each kit consisted of three containers for three bulk sediment samples, a paper 250 millilitre (ml) cup and a bright pink string. Additionally, each kit contained an instruction sheet, and all materials were contained in a post-paid box addressed to the Aquatic Ecosystems and Environmental Change Laboratory address at Carleton University in Ottawa Canada (Figure 4.2). The kits were handed out during an information session where the aims and desired protocols of the project were conveyed to the volunteers. Sampling procedures were based on the sampling protocol in Horton et al., (2017). Four 250 ml grab samples were collected at one-meter intervals parallel to the shoreline. The research team indicated the optimal place to sample the shoreline was at a distinctive debris line on the shore, where the river level would have been before receding. The Community Scientists used the container, the length of rope (coloured bright pink to distinguish any possible contamination in the processing of samples) and a paper cup that held approximately 250 millilitres of sediment, for four collections pertaining to a 1 litre bulk sample for one of the containers. The rope was pre-marked indicating one metre intervals, thus the volunteers collected one sample at the end of the rope, then at each of the two one metre marks and finally at the other end of the rope, representing the four 250 ml collections. Additionally, the rope had metal nails at each end to hold it in place during collection.

Community Scientists were asked to use their own trowel for the sediment collected and it was suggested that it was a metal trawl if possible. Each Community Scientist was provided with three containers and given the option to sample one location (utilising one to three of the containers), or at three different locations. The Community Scientists filled out the provided information sheet that included their name, GPS coordinates, location name, description of the sediments and clothing colours worn during sampling. The Community Scientists then packed the bulk sample into the aforementioned pre-paid postal box and sent the sample to the laboratory where it was stored in a dark fridge for processing.

The research team trialed the sampling protocol at various shorelines in the Ottawa River watershed to test the methodology, including laboratory processing and analysis, as an evaluation before presenting the protocol to the Community Scientists (Supplementary figures).

Additional analysis included exploring significance between urban and non-urban area concentrations in addition to comparing lake areas to higher flow sections of the river and the comparison between the main channel and tributaries. Various t-tests were performed to establish any significance between these variables.

Figure 4-2 A pre-paid postage box used by citizen scientists to return samples. B) A container, trowel, paper cup and string used for collection by the citizen scientists. C) and D) Pictures of debris layers after a spring flooding.

4.4.1 Laboratory analysis

Sediment preparation and density separation was based on the oil extraction protocol (OEP) (Crichton et al., 2017), with some modifications to adjust for processing times due to the large amount of sediment received from the citizen scientists. The OEP was chosen as it uses canola oil as reagent, thus a mode of density separation easily demonstrated, or conducted with citizen scientists, whereas other density separation techniques utilise harsher chemicals. For example, though zinc bromide ($ZnBr_2$) has demonstrated the ability to extract higher density polymers, up to 1.7g cm^{-3} (Prata et al., 2019), canola oil's lower toxicity was a factor in selection for a citizen science project as $ZnBr_2$ may pose a threat to health or the environment if mismanaged (Mani et al., 2019). Another advantage of canola oil is cost, especially for larger citizen science projects, where it can become an important factor. For example, sodium iodide (NaI) can be an effective additive for density separation, separating plastics up to 1.6g cm^{-3}, however, where canola oil may cost approximately $0.96 Canadian dollars (CDN) per sample (Crichton et al., 2017), NaI can cost approximately $90.00 CDN per sample and $ZnBr_2$ approximately $922 CDN a sample (Crichton et al., 2017). Another commonly used reagent for density separation is sodium chloride (NaCl), which can be as cost effective, approximately $10.00 CDN a sample (Crichton et al., 2017). However, NaCl typically only recovers lower density plastics, up to 1.2 g cm^{-3} and could lower recovery rates significantly for environmental samples (Prata et al., 2019). Furthermore, some of the brine solutions for density separation are based on the specific density of the solution, therefore, exclude certain polymer types with higher densities such as Polytetrafluoroethylene (PTFE) (Lechthaler et al., 2020). Recent testing on canola oil has highlighted the capability of extracting microplastics in the density range from 11–1760 kg m^{-3} and in the size range from 0.02–4.4 mm (Lechthaler et al., 2020).

An additional factor to consider for density separation for larger citizen science projects is processing time. Comparatively, the OEP has a relatively short processing time when compared to other extraction procedures. For example, the OEP does not retain high amounts of organic material, whereas other reagents such as calcium chloride ($CaCl_2$) requires overnight settling due to higher amounts of organic material that requires longer settling times (Stolte et al., 2015). Laboratory tests confirm good recovery for the OEP, with an average recovery rate of 96.2 % (± 2.2%) (Crichton et al., 2017). Additionally, other oil extraction protocols, for example castor oil (Mani et al., 2019) also exhibit good recovery rates in laboratory tests (99% ± 4%). However, when oil extraction protocols are applied to some environmental matrices, recovery rates may decline. For example, the castor oil protocol recovery rates drop to approximately 75 % (± 13%) when used on riverine suspended solids (Mani et al., 2019). Nonetheless, Crichton et al. (2017) did apply the OEP validation by spiking beach samples of various grain sizes to try and represent real word conditions for extraction, rather than examining efficiency rates purely on laboratory testing.

Each sample was prepared under a laminar flow hood, transferred to a metal tray and covered with foil before being transported to an oven for drying. Each sediment sample was dried in an oven at 70°C (Corcoran et al., 2015), considered below the melting point of all common polymers. Furthermore, 70°C would not provide conditions that could alter the inherent shape of polymers that may be in the sample (Kalpakjian & Schmid, 2008). Drying times were between 6 and 72 hours, dependent on the moisture levels of each received sample. Once a sample was dried, a dry weight of the sample was calculated and transferred to a laminar flow hood for density separation.

The samples received were a variety of grain sizes (it was stipulated in the presentation to the citizen scientists, an ideal sediment size range), thus, the first step after drying and before density separation was to put the sample through a 2mm sieve and 1mm sieve (to separate the larger materials from the sediment), with visual inspection for suspected plastic particles after each sieving. The canola oil was prepped for analysis by filtering it through a 100-micron mesh, to remove possible contamination within the canola oil vessel.

For density separation, 100g of the (dry) sediment sample was placed in a 500ml beaker. Two hundred millilitres of deionized water were then added to the beaker. A clean glass stirring rod was used to swirl the water to create a vortex, so the entire sediment fraction was submerged in the water. After a short settling period, 10 ml of filtered canola oil were added to the beaker and the sediment stirred vigorously for 30 seconds so the canola oil would come into contact with the entire sediment fraction. The mixture was then left to settle until the oil layer separated fully to the top of the water. Once separation was completed, the oil and water were decanted from the sediment into a separatory funnel. The beaker sidewalls were then rinsed to dislodge any particles and the water and oil layer decanted again into the separatory funnel. The above process was repeated until the separatory funnel was three quarters full, then shaken vigorously for 30 seconds to ensure any plastics dislodged during decanting are re-introduced back into the oil layer. The mixture was then left to settle until the oil and water layers separated. Once settling was complete, the oil layer with plastic particles was retained and the water and sediment layers were released and discarded. The oil layer was then emptied into a vacuum filtration system with an 80-micron pre-inspected mesh. The filters were then backwashed into a petri dish and the filter and petri dish contents examined for microplastics under a stereomicroscope. Identified particles were then separated into fragments and fibres, and the colours of each fragment and fibre was noted.

4.4.2 Quality control and quality assurance

During sediment sample processing, a total of 9 laboratory blanks (beakers filled with DI water) were placed in various locations under the laminar flow hood during processing, to account for the potential of airborne contamination in the laboratory. These controls averaged 0.78 fibres per blank, with the greatest concentration being 3 fibres in a single control sample. The Community Scientists were not asked to conduct field controls, as it would have complicated the sampling procedure. A previous community science project in the Ottawa River watershed noted that even with detailed and concise sampling protocol sheets and sampling methodologies that are presented thoroughly to the Community Scientists, the chance for errors in sampling, especially conducting controls is higher with volunteers (Forrest et al., 2019). Instead, the detailed sample sheet included information on clothing type and colours to determine potential contamination, and it was expressed to the volunteers during the methodology presentation to try and limit the exposure time of the containers as much as possible to reduce the potential of airborne contamination in the field. Additionally, as the postage boxes were limited in size, the research team chose to include a third sampling container rather than an empty container that could have been a control. The research team noted the colour of clothing worn by the volunteers while processing the specific samples at the laboratory and did not count fibres of the same colour if they were extracted from the sample.

4.5 Results

Of the 47 distributed kits for sediment analysis, a total of 43 were returned, representing a return rate of 91%. There were a total of 42 volunteers who submitted a total of 68 locations in the Ottawa River watershed (Figure 4.2). Of the samples received, two samples were discarded, one sample contained only sticks, leaves, and other dry material without sediment, while the other sample was damaged when received at the laboratory. Thus, 95% of the samples received were analysed. The distance between the most upstream and downstream locations was approximately 750 km. There were 56 locations sampled on the main channel of the Ottawa River with 12 locations on tributaries. These locations included five samples on the Gatineau River, one location on the Rideau River, one location on the Madawaska River, one location on the Bonnechere River, two locations on the Kipawa River and two locations on the Kinojévis River. The largest concentration of samples received were centred in or close to the Ottawa/Gatineau urban area.

Approximately 101 kg dry weight of sediment was received in the laboratory. Of the sediment received, the average dry weight of the samples was 1.5 kg. Of the suspected microplastic particles observed from the samples, 77% were fibres and 23% were fragments. Of the fibres observed, the most common were blue (approximately 69% of the fibres), green 16 %, red 5 % and black 4%. The main fragment colours were blue at 39%, white 37% and clear 20%.

All of the samples received contained suspected microplastics and/or anthropogenically modified fibres. For the concentration of identified particles, the overall average concentration was 0.06 particles per 100 grams (g) of dry weight (supplementary Tables). The highest concentration was 2.30 particles per 100g dry sediment with the lowest concentration <0.001 particles per 100g of dry sediment. The majority of the samples, 85%, were under 0.10 particles per 100g of sediment (Figure 4.3)

Although the particle counts were relatively low, several spatial characteristics were evident with the data. The average particle count per 100g of sediment in the 29 identified urban locations was 0.60, with a slight increase in the 11 Ottawa/Gatineau urban area locations. The northern end of Lake Temiskaming (the wider part of the lake) demonstrated an average of 0.74 particles per 100g of sediment from nine samples, slightly higher than the overall average. For these comparisons, a t-test was also conducted to highlight if there was any statistical difference between urban and non-urban locations (including comparing Ottawa/Gatineau sample points to the rest of the sample points), and between Lake Temiskaming and the other river locations. These three t-tests were not significant. However, a t-test was performed between tributary locations (an average of 0.25 particles per 100g of sediment) and the main channel of the river (0.64 particles per 100g of sediment), and the difference in concentration was significant with a p-value of <0.001.

The final counts of each location were added to a Google map, where the Community Scientists could access to explore the results, while comparing other locations to their own throughout the watershed. Additionally, a presentation was given to the Community Scientists where the research team talked about the process and the results to the Riverwatcher members, while giving input and evaluating the process as a whole.

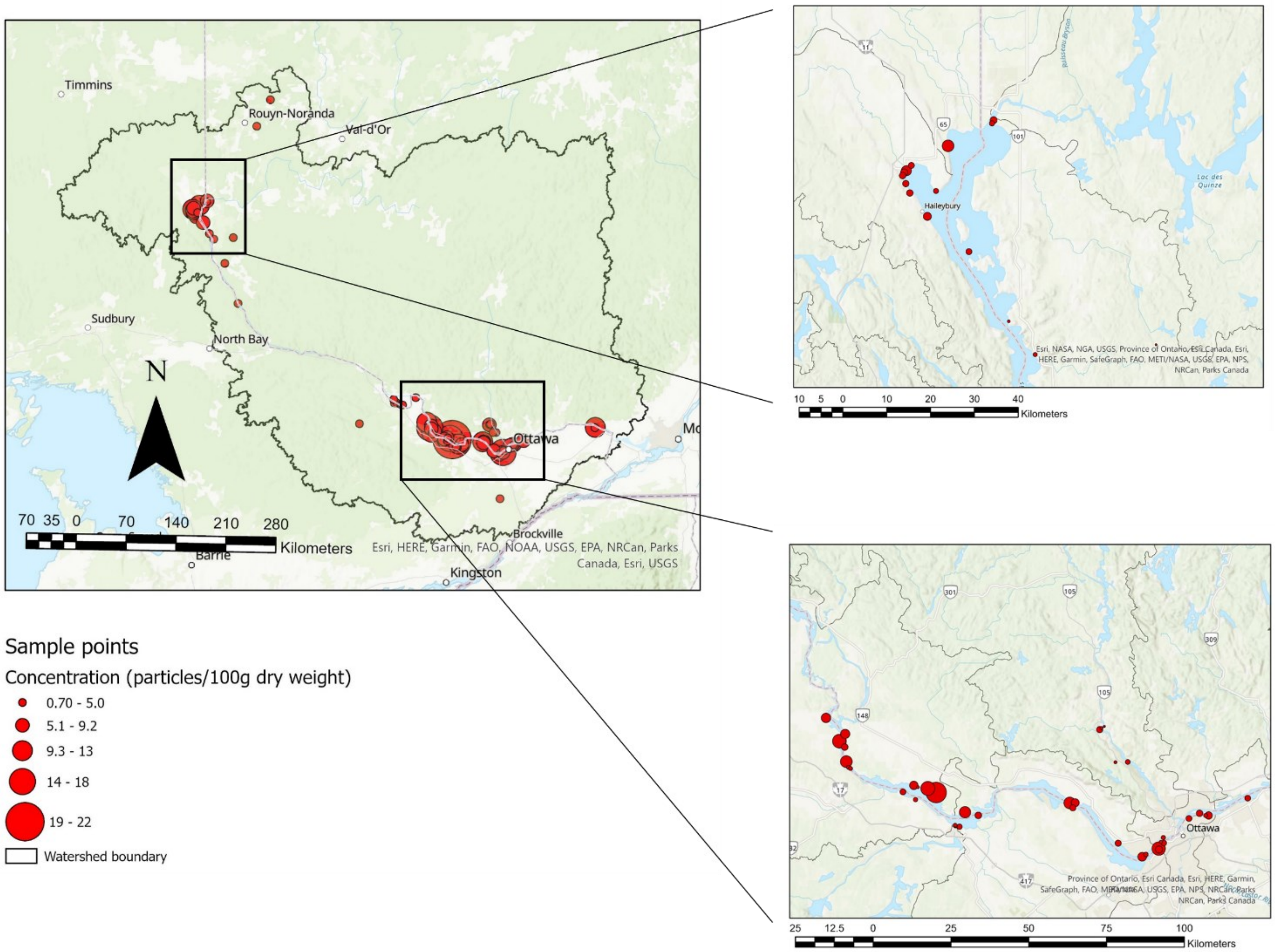

Figure 4-3 Distribution of sample concentrations using weighted circles from each community scientist location.

4.6 Discussion

Particle concentration was relatively low when compared to previous studies of concentrations in freshwater sediments. For example, the collection methodology was based off Horton et al., (2017) and dry sediment concentrations on the Thames River and tributaries in the United Kingdom presented an average concentration of 66 (±7.7) particles per 100g and a minimum value of 18.5 (±4.5) per 100g, with the equivalent maximum value in the current research of 2.30 particles per 100g and a minimum of 0.07 particles per 100g. Lake Ontario in Canada presents an even larger disparity, with Ballent et al., (2016) reporting values consistently over 50 particles per 100g with a maximum of approximately 2,800. Even previous research on the Ottawa River highlights higher concentrations, though in underwater or wet sediments, where Vermaire et al., (2017) notes a concentration of 22 particles per 100g at Petrie Island in Ottawa, Ontario. One Community Scientist sampled at this location and the dry sediment nearshore concentration was 0.50 particles per 100g. Furthermore, the Ottawa River drains into the Saint Lawrence River in Quebec and Crew et al., (2020) noted a mean wet sediment concentration of 83.2 (±15) particles per kg with values reported between 6.5 and 756.2 particles per 100g of sediment.

Several factors could contribute to the large difference in values. As highlighted, wet sediment concentrations under the water in Canadian freshwater river systems tend to present much higher concentrations than those reported in the current research. This can indicate the shoreline is not a substantial sink of microplastics in the Ottawa River watershed. This is in agreement with previous research, with Leslie et al., (2017) suggesting fibres do not tend to settle in river sediments as often as fragments.

Additionally, the constant rise and fall of river water can displace microplastics from sediments (Hurley & Nizzetto, 2018) and subsequently move them to the water column, sending microplastics further downstream to settle again, or even transported to marine environments. This is relevant in the Ottawa River watershed especially as the Ottawa River experiences two peak flows during the spring freshet season downstream and one peak flow upstream, due to the pattern of spring melts in the river basin and the unregulated inputs from the tributaries. This may move and/or remove shoreline microplastics. With such a large dynamic river system, the lessened potential for microplastic deposits settling for a significant amount of time could render shoreline microplastic deposits an insignificant microplastic sink.

While examining other river watersheds and comparing to the Ottawa River, watershed size, the population base and river discharge rates are important factors to consider. Previous research on microplastic concentration in sediment in the Thames River (Horton et al., 2017) and the Atoyac River in Mexico (Shruti et al., 2019) is compared to the Ottawa River in Table 1. Additionally, The Ottawa River watershed is in constant seasonal flux (more than rivers in more temporal zones for example) making microplastic deposition on shorelines a dynamic and constantly changing phenomenon.

The only statistically significant spatial pattern to draw from the results in the community science project was the difference in concentration along the main river channel compared to the tributary locations. Generally, the tributaries of the watershed do present lower population bases compared to the main channel, where the majority of the population of the watershed resides. However, comparing the urban areas to the average particle concentration did not present any obvious spike, which could further demonstrate dry sediments are not a significant sink in the watershed. This presents the need to examine wet sediments of the watershed at a larger scale to evaluate the potential as a microplastic sink, to gather more information on the significance of microplastic contamination in underwater sediments of a large watershed.

Table 4-1Comparing the Ottawa River to the Thames River (United Kingdom) and the Atoyac River (Mexico) in terms of population, watershed size, average discharge and particle concentrations reported in dry sediments (Thames and Ottawa rivers) and wet sediments (Atoyac River).

River Name	*Approximate population in the watershed (million)*	*Approximate watershed size (km^2)*	*Average river flow (m3/s)*	*Average concentration (per 100g)*
Thames River, England	13	12,935	65	66 (±7.7)
Atoyac River, Mexico	2	4,135	440	113 ± 7.28
Ottawa River, Canada	2	140,000	1950	0.06

4.7 Evaluating the citizen science methodology

With a well-planned methodology and sampling, citizen science can provide a spatial coverage that would otherwise be more time consuming and difficult for a research team. On the other hand, the research team has greater control over QA/QC in laboratory processing as they receive the samples from the field and can follow set protocols to minimise the potential of contamination during sample processing, which would be complicated for citizen scientists to complete without specialised equipment and training. However, sampling for microplastics in sediments did pose some difficulties and potential obstacles to address if subsequent citizen science projects attempt to use similar protocols as in the current research.

In such a large watershed, it is ideal to sample as much shoreline sediment as possible at each location, as the Ottawa River has many large beaches throughout the watershed, and it is important to get a representative sediment sample. The amount of sediment requested for each location must be balanced with the potential processing time in the laboratory, especially with time consuming sample preparation, density separation and sample inspection (including chemical analysis). These factors must be carefully considered in the project design as they can impact the practicability of a potential sediment microplastic citizen science project.

Another factor to consider is using established volunteer networks to assist with the citizen science networks. Various watersheds, especially in Canada have advocacy groups or similar that help monitor freshwater bodies of water, typically made up of people who live or have vacation properties in the watersheds. Additionally, they can provide some useful local and historical knowledge of the watershed, especially if the volunteers have had a generational presence in the watershed. This is similar to the Riverwatcher network, where the citizens are actively engaged in the monitoring of their watershed and contribute to the monitoring of the health of their freshwater ecosystem and many of the volunteers have been living in the watershed for many years including previous family generations. As microplastics are becoming a major concern for freshwater ecosystems, citizens are empowered to contribute to research in their watersheds which assists in recruiting interested participants through these networks. The Riverwatchers previously contributed to a citizen science project examining microplastics in water samples of the Ottawa River watershed (Forrest et al., 2019) and the current research was able to increase the number of participants over the previous project as the network continues to become more involved with issues that affect their river and watershed.

However, the majority of projects in citizen science in freshwater plastic pollution only request samples from volunteers without any sample processing or data analysis (Cook et al., 2021), wherein the projects are designed by scientists while the members of the public primarily contribute data. This enables the research team to have more control over QA/QC through various, often strict sample processing protocols. Nonetheless, it is suggested the path forward for citizen science is to adopt a more collaborative approach (Buytaert et al., 2014; Cundill & Fabricius, 2009; Haklay, 2013). This approach for citizen science in freshwater microplastic research empowers the citizen scientist to be involved in sampling, processing, analysis and even dissemination of the results. This would be the next logical step to increase citizen science capacity, while potentially presenting citizen science as a viable, mainstream option to research teams in microplastic research.

4.8 Conclusions

Citizen science programs provide an advantage to microplastic monitoring and sampling, especially in large watersheds where spatial scales are large. With robust project design and implementation, established citizen science or volunteer networks within watersheds can provide spatial monitoring in addition to local knowledge, advocacy while being engaged in the watershed and the issues that may affect their place of residence or vacation. However, monitoring for microplastics in dry sediments in a large watershed does present disadvantages. As beaches and shorelines in the Ottawa River watershed can be large, obtaining representative sediment samples can be difficult. Increasing the target amount of sampled sediment can be an option, however, this compromise increases sample preparation and analysis time in addition to potentially creating issues transporting large amounts of sediments to laboratories for analysis.

The results of the research presented low particle concentrations when compared to previous research in freshwater sediments. This would suggest in the Ottawa River watershed, microplastics in shoreline sediments are not a significant sink. Further research is needed to establish the potential role of wet or underwater sediments as microplastic sinks in the watershed.

Citizen science has provided a useful tool in expanding spatial coverage for microplastic monitoring in the Ottawa River watershed, however, careful research design and implementation is critical for successful implementation and to ensure research integrity. Furthermore, for sampling sediments, there needs to be an expansion to include sampling wet sediments or deposition zones in rivers, and to evaluate methodologies that can be implemented for citizen scientists. Currently citizen science in microplastic research still only focuses on a contribution approach, where citizen scientists only sample and contribute these samples to the researcher for analysis. For citizen science to expand further, while contributing useful and valid data, designing methodologies where the citizen scientist is collaborative, by also being involved in sample processing, data analysis and even data dissemination, is essential. This can increase citizen science capacity in Canada while potentially creating monitoring and watch programs where citizen scientists become an integral part of the research

5. Spatial distribution of microplastics in a large freshwater watershed: a case study of the Ottawa River watershed

5.1 Introduction

Global plastic production continues to accelerate with production exceeding 300 million tonnes a year (Plastics Europe, 2015), with the vast majority of plastics discarded after use (Rillig, 2012; Koelmans et al., 2014). Consequently, microplastics are now found throughout the majority, if not all global ecosystems. Microplastics are tiny plastic pieces smaller than 5mm (Masura et al., 2015), with research now suggesting microplastics are a threat with the potential harm of ingestion (Auta et al., 2017) as they are bioavailable for many organisms. Furthermore, microplastics have the potential to hinder mobility (Fossi et al., 2016; Avio et al., 2017; Barboza et al., 2018; Nelms et al., 2019) are said to pose threats as a vector to waterborne pollutants (Teuten et al., 2009; Zarfl & Matthies, 2010; Ivar do Sul & Costa, 2014). Microplastics also have the capacity to sorb and/or release toxic chemicals with potential trophic transfer through food chains (Hurley et al., 2017; Reisser et al., 2014). Microplastics can be classified as either primary, manufactured to be a microplastic, or secondary, fragmented from larger plastics. Microfibres are another form of secondary microplastic, with some researchers suggesting that natural fibres that have be anthropogenically modified should still be considered as an environmental contaminant (Athey & Erdle, 2021).

Freshwater microplastic research has only recently gathered momentum, with around 96% of microplastic research focused on marine areas (Wagner & Lambert, 2018). However, rivers are a major contributor of plastics to global oceans (Lebreton et al., 2017). Furthermore, there is a lack of research into the inputs, throughputs and sinks of microplastics in river systems, especially at the watershed scale. As microplastic research is relatively new, there are continued calls for more standardisation in sampling methodologies and data analysis (Löder & Gerdts, 2015; Rocha-Santos & Duarte, 2015; Van Cauwenberghe et al., 2015; Twiss, 2016; Koelmans et al., 2019; Rochman et al., 2017).

Previous research has exhibited a presence of microplastics in the Ottawa River watershed (Vermaire et al., 2017, Forrest et al., 2019), however, there is limited research examining microplastics throughout the watershed conducted under the same sampling protocols. Without standardised methodologies and data analysis, it is difficult to directly compare freshwater microplastic research. Furthermore, attempting to compare samples and research within a large freshwater watershed is also difficult, especially if there have been multiple studies in the same watershed without a standardised sampling approach. Therefore, the aim of the research is to get a synoptic view of microplastic concentrations in freshwater at the watershed scale, including the main Ottawa River channel and its major tributaries. This includes examining various landuse factors that may impact spatial distribution while employing the same sampling protocols to better understand the potential inputs, throughputs and outputs of microplastics in the watershed.

5.2 Study area

The Ottawa River watershed is approximately 140,000 square kilometres (Figure 5.1), while in itself a tributary of the Saint Lawrence drainage basin, which has the highest average water discharge in North America (Benke & Cushing, 2005). The average flow of the river measured at Carillon Dam is 1,950 m^3/s with a historical daily minimum recorded at 301 m^3/sec in 1971 and maximum daily flow recorded at 8862 m^3/sec in 2017 (Ottawa Riverkeeper, 2021). It has already been established that the Saint Lawrence contains microplastic pollution in both water and sediment (Castañeda et al., 2014; Crew et al., 2020), therefore, as the major tributary of the Saint Lawrence, understanding the spatial distribution of microplastics in relation to potential inputs, throughputs, sinks, and outputs of microplastics, provides an important perspective for Canadian watersheds. The Ottawa River watershed presents densely populated regions, namely the Ottawa/Gatineau urban area with numerous towns and small settlements along the channel and tributaries of the watershed. Furthermore, there are sparsely populated regions with minimal or no anthropogenic activity. This combined with the varying landscapes through which the Ottawa River and its tributaries flow, presents a vast array of differing environments to examine microplastic concentration.

As previously mentioned, there are 19 major tributaries of the Ottawa River, nine in the province of Ontario, and 10 in the province of Quebec (Figure 5.2 and Table 5.1). As the Ottawa River was recently designated a Canadian Heritage River by both the provinces of Quebec and Ontario, it demonstrates that it is of significant economic, cultural and ecological importance to Canada.

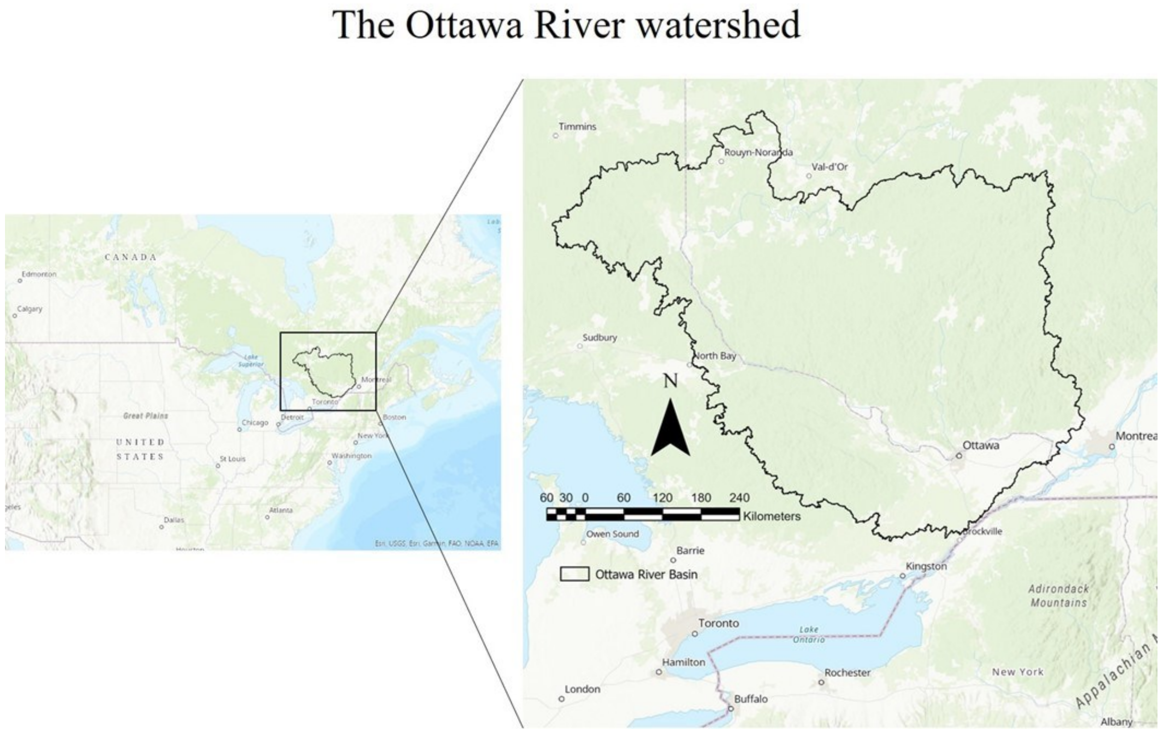

Figure 5-1 The Ottawa River watershed and its location in North America.

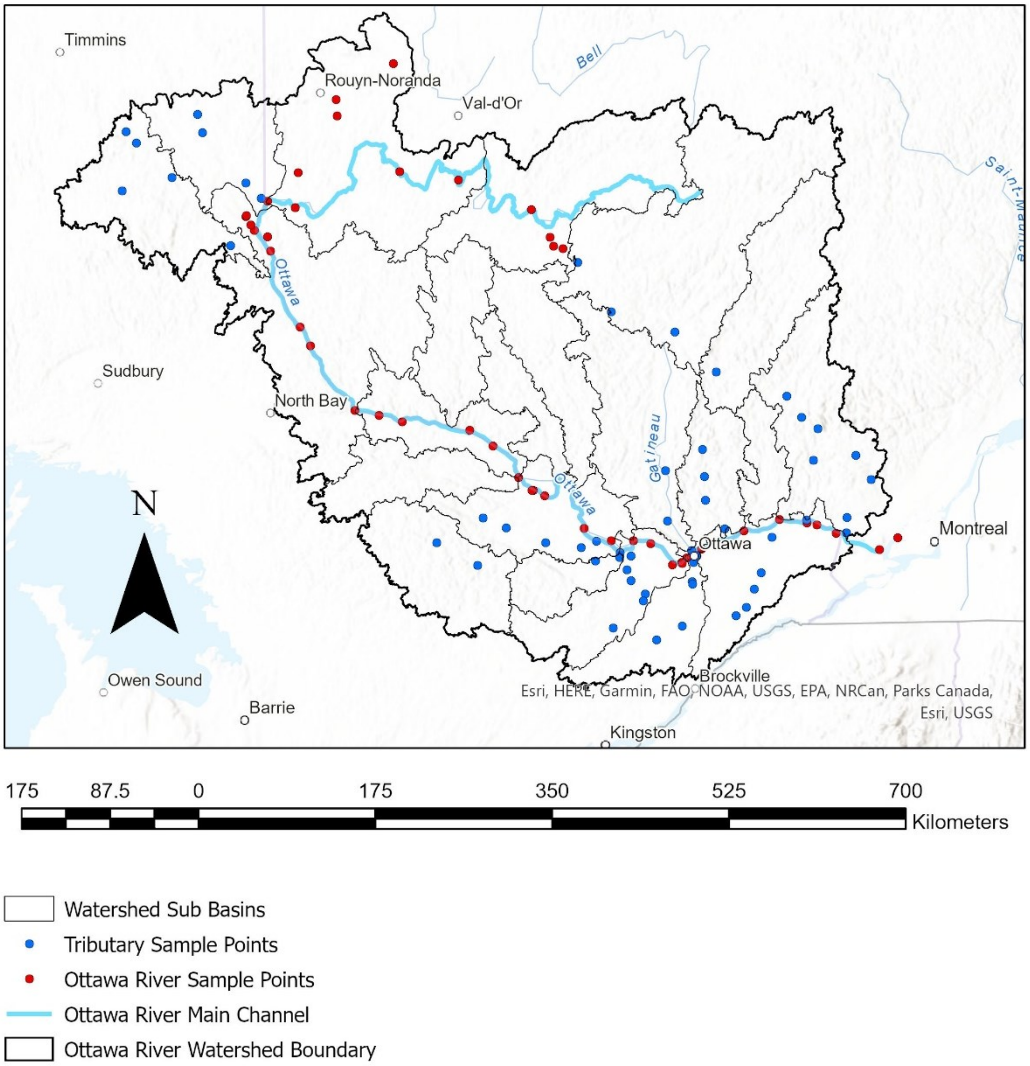

Figure 5-2 The Ottawa River watershed with sub basins and sample points on the main channel (red) and tributaries (blue).

Table 5-1 Major tributaries of the Ottawa River watershed.

Tributary name and province (Québec QC or Ontario, ON)	***Approximate river length (km)***	***Approximate distance of river mouth from the Ottawa River terminus (km)***
Rivière Nord, QC	137	21
Rivière Rouge, QC	161	55
Rivière de la Petite Nation, QC	97	90
South Nation River, ON	175	90
Rivière du Lièvre, QC	330	116
Rivière Gatineau, QC	386	138
Rideau River, ON	146	139
Mississippi River, ON	200	202
Madawaska River, ON	230	207
Bonnechere River, ON	145	226
Rivière Coulonge, QC	240	274
Rivière Noire, QC	238	284
Petawawa River, ON	187	331
Rivière Dumoine, QC	129	395
Mattawa River, ON	76	465
Rivière Kipawa, QC	180	566
Montreal River, ON	220	577
Blanche River, ON	126	628
Rivière Kinojévis, QC	135	738
Lac des Outaouais, QC (headwaters)		1272

5.3 Methodology

For consistency and to standardise results throughout the whole watershed, sampling was conducted with the same 300-micron Manta net at all the locations, with sampling conducted at, or close to the shoreline (as opposed to sampling mid-channel on a boat). A 300-micron size net was chosen as smaller mesh sizes (for example 100-micron) are more prone to clogging, especially with a wide variety of river conditions and organic content that could present itself over such a large variety of sample locations. Furthermore, shoreline sampling was favoured as it allowed access to more sampling locations, compared to sampling off a boat, where boat access can be more limited. Moreover, sampling near, or at, the shorelines was another important consistency consideration, as it has been suggested microplastics can vary between the centre of a river and the shoreline. (Corcoran et al., 2020). Additionally, sampling the whole water column where possible was the desired method, as microplastic concentration can vary throughout the water column. For example, the density and weight of a polymer can influence where in the water column it is situated (Pico et al., 2019; Waldschläger & Schüttrumpf, 2019), and various polymers may not be exclusively transported at or near the surface. This highlights the importance of sampling as much of the water-column as possible.

To reduce the impact of contamination, or atmospheric fallout skewing results while sampling in the field, large volume samples were favoured. Consequently, sampled volumes for the watershed locations ranged from 20,000 litres to over 163,000 litres depending on river flow. For stationary sampling, the Manta net was deployed one to three times between 10 to 30 minutes depending on flow and organic content in the river. Volume was calculated by using the percentage coverage of the water in the known area of the net opening. River velocity was measured with a flow meter, which indicated an average flow velocity for the duration the Manta net is submerged in the river. Therefore, to calculate the volume sampled at each location, sampling duration, area and velocity was used to calculate total volume. Once sampling was completed, the net was removed, and water was used to flush the outsides of the net to push all material to the removable cod end of the net. Once the net had been flushed, the cod end was removed, and the contents washed into a clean pre-inspected glass Mason jar for transport to the laboratory. After each sample location, the Manta net and cod end was backwashed to avoid cross-contamination between samples.

One disadvantage to stationary shoreline sampling with a Manta net, is the need for a minimum flow velocity of the river, to be able to push the water through the net. Observations from sampling along the shoreline with the Manta net, required a minimum flow of 0.1 metres a second (m/s), as velocities any lower, the river water was observed to go around the opening of the net, as the force was too low to push water through. At some sample locations, especially some of the larger beach locations on the Ottawa River, water velocity is very slow and was not significant enough to push water through the Manta net. Although the desired method was to leave the Manta net at the shoreline and let the water pass through, it would have limited the spatial extent of the sample points. Consequently, a drag method was employed where stationary shoreline samples were not possible. For this method, the Manta net was manually dragged along the accessible length of the shoreline, close to or at the shore, just off the bottom of the riverbed (not to dislodge settled sediments and potentially contaminate the sample). As with the stationary shoreline method, the flow was measured, the amount of net coverage and the time dragging was noted to calculate volume. Volumes were generally lower for this method because the net was dragged the length of the accessible shoreline and there was a pause to let the water flush or circulate through, so as not to sample the same water multiple times. Stationary sampling was approximately 30 to 45 minutes, where drag sampling was approximately 3-6 minutes. However, velocities were higher during the drag method than stationary sampling, therefore, volumes for the drag method varied between 20,000 and 97,000 litres, still representing large volume samples.

For stationary sampling, volumes ranged between 20,000 and 164,000 litres. The shoreline sampling did present a vast range in water volumes sampled (discussed in further detail in preceding sections), with the large variance dependent on the flow velocity, the duration of the sample and the percentage of net submergence in the river water. The differences in all these factors contributed to a large range of sampled volumes. However, a correlation was conducted between volume and microplastic concentration to make sure there was no dependence, thus influence, on sample volume towards microplastic concentrations.

The final consistency consideration was to try to reduce the impact of temporal influences during the sampling. This is not to discount the importance of temporal factors in microplastic distribution in watersheds. As microplastic concentrations have been demonstrated to change over a wide range of temporal scales (for example, seasonally and during rain events), to obtain a better understanding of just the spatial extent of microplastics, sampling was conducted during low flow periods during late summer and fall. Sampling during these times avoids the effects of spring freshet and enables sampling before any freezing of the river. Additionally, sampling was not planned during any significant forecasted rain event, to try to reduce the potential influence from stormwater and/or overflows at one location compared to another location.

5.3.1 Laboratory analysis

Once in the laboratory, the Mason jars were washed into a clean, pre-inspected beaker using deionised water. A 30% hydrogen peroxide solution was added to the beaker and the samples were heated to 50ºC in a water bath for digestion of organic material in the sample. Heating time varied depending on the organic level of the sample and ranged from 4 to 16 hours. Upon completion of digestion, the solution was passed through a vacuum filtration system with a clean (pre-inspected) 80-µm metal mesh filter. The filter was then washed into a clean (pre-inspected) petri dish, with visual inspection identifying potential microplastics under a Leica stereomicroscope at 20 to 40X magnification. Quality assurance and control included conducting all the sample analysis under a laminar flow hood to reduce airborne contamination while the samples were prepared and examined. Furthermore, laboratory and field blanks were used to assess the potential for contamination in the samples. For the 10 laboratory controls there was a minimum of 0, a maximum of 3 and an average of 1.1 fibres per sample. For field controls, a mesh was placed inside a metal funnel and exposed during Manta net preparation, covered up during deployments and exposed again when the net was out of the water and being prepared for the next sample. Over 15 field controls there was a minimum of 0, a maximum of 4 and an average of 1.6 fibres.

5.3.2 Statistical analysis

The concentration data from each sample point was analysed through R (R Core Team, 2020) using an analysis of co-variance (ANCOVA) to establish any significance and/or relationship between a variety of different spatial indicators in the watershed. Before the ANCOVA analysis, a log transform was applied to smooth the skews in the data. The ANCOVA examined the change in concentration from upstream to downstream in the main channel and tributaries (as a percentage of distance travelled from the headwaters), change in concentration downstream of wastewater treatment plants (WWTPs), change in concentration upstream and downstream of dam structures and microplastic concentration in relation to the amount of precipitation the day of sampling and the amount of precipitation three days before sampling. Furthermore, the ANCOVA analysis examined the categorical relationship between urban and non-urban microplastic concentrations, main channel and tributary concentrations in addition to the concentration at boat launch locations. Lastly, another ANCOVA analysis was initiated while only considering fragment microplastic concentrations for each of the variables used in the previous ANCOVA. For the analysis, urban locations are defined as a population of at least 2000 (a definition determined exclusively for the research), or if the sample location was located in a populated area close to modified surfaces and channels that may convey runoff. Furthermore, some large park or reserve areas within a town or municipality were not considered urban.

In order to ensure there was no relationship between sample volumes and microplastic concentration, a regression analysis was conducted to examine any possible bias in the concentration data to the volume of water sampled at each sampling site.

5.4 Results

Sampling was conducted during May to November, from 2019 to 2021. A total of 105 locations were sampled, with 49 locations on the main channel of the Ottawa River and 56 locations on 11 tributaries throughout the watershed (Figure 5.2). Of the 19 major tributaries noted on Table 5.1, seven were not sampled. Four of these rivers, the Coulonge, Noir, Dumoine and Kipawa in Quebec have large sections of the river where there are accessibility issues due to their remoteness. However, these rivers all begin, flow through or are close to the La Verendrye Wildlife Reserve. A total of seven other sample points were conducted in or very close to the reserve, thus, these sample points could be considered representative of the remote rivers. Only the Coulonge River has any population of note, at the headwaters where Fort-Coulonge has a population of approximately 1500 people. On the Ontario side, most of the Petawawa River flows through the Algonquin Provincial Park, another remote area with limited anthropogenic activity. As with the isolated Quebec rivers, the Petawawa River generally flows through remote areas, with Petawawa the only population base of note at the headwaters at approximately 17,000 people. The two shortest rivers in Table 5.1, the Petit Nation and Mattawa were also omitted to focus on the longer tributaries. Lastly, the Kinojévis was not directly sampled as a tributary, as some watershed maps include this river as part of the upper Ottawa catchment, or headwaters catchment area (Figure 5.2), thus it was included as a branch of the main channel and included as part of the Ottawa headwaters area with three samples conducted directly on the river. Detailed information on each sampling location can be found in the supplementary tables 1-4.

As previously mentioned, the 300-micron Manta net was deployed at shoreline locations either with a stationary sampling method or utilising a drag method. 62 locations were completed with the drag method and 43 with the stationary method. Concentrations from the drag and stationary method of sampling was compared to determine if there was any bias towards one method over the other. A t-test between the two methods presented a p-value of 0.07, thus there should have not been any bias between sampling methods. Furthermore, as the drag method, in general, sampled lower volumes compared to the stationary method, in order to assess for any bias towards larger or smaller volume samples, a regression analysis was conducted to examine if there was a relationship between particle concentration and sample volume. The regression analysis presented no significant relationship between sample volume and particle concentration observed. Therefore, there should not be any bias in the sampling towards higher or lower volume samples.

The overall distribution of particle concentration at each of the locations was broken down into each of the tributaries examined and the main channel of the Ottawa River. Box plots presents the distribution of microplastic concentrations for the main channel and tributaries (Figure 5.3) in addition to the concentration presented on a map of the watershed (Figure 5.4). The concentration as you move downstream on the main channel is represented by Figure 5.5, with the location where each tributary enters the main channel, in addition to the location of the La Verendrye wildlife reserve, Lake Timiskaming and the Ottawa/Gatineau urban area. In examining the box plots, a high proportion of the locations demonstrated particle concentrations below 1 p/m^3, with two outliers on the main channel and one outlier for the Rouge River. A similar concentration to the Rouge River outlier was noted on the Montreal River at Matachewan, however, this was not presented as an outlier and it subsequently elongated the box plot for the Montreal River, which demonstrated the largest variance. The remaining tributaries and main channel samples presented smaller variances, due to a high proportion of samples with particle concentrations below 1 p/m^3. Fibres were the dominant type of particle in the samples, representing 83% of the extracted particles. The change in particle concentration downstream (Figure 5.5) on the main channel and tributaries does not present a clear low to high gradient, as may be expected with the gradual accumulation of microplastics. The relationship presented seems complex, where there is not a simple accumulation of microplastics as you move downstream, rather there are potential interactions with several variables to consider. Therefore, an ANCOVA was preformed to examine the data for potential relationships and significance (supplementary tables). After ANCOVA analyses, the only significant relationships presented were the change in concentration in relation to the distance downstream, presenting a p-value of 0.006 and boat launch locations in relation to non-boat launch locations with a p-value of 0.002. However, the relationships were not strong with an R^2 value of 0.15 (Figure 5.6), demonstrating there are more variables to consider and/or that temporal factors could have a large bearing on the spatial distribution of microplastics in the main channel and tributaries. After analysing fragments only, the same significant relationship was presented, with the boat launch versus non-boat launch significance increasing with a p-value <0.001 and the concentration to distance downstream significance, slightly reducing to 0.02. However, the R^2 value did increase to 0.30 (Figure 5.6).

In addition to examining the significance of WWTP concentrations through the ANCOVA analysis (where there was no significance), Table 5.2 presents the change in concentration from a sample point directly upstream and downstream of a WWTP. The comparison demonstrated there is a greater mean particle concentration of downstream locations. However, this is mainly influenced by one sample point with a large comparative concentration. Only five of the nine downstream locations presented higher concentrations than upstream locations.

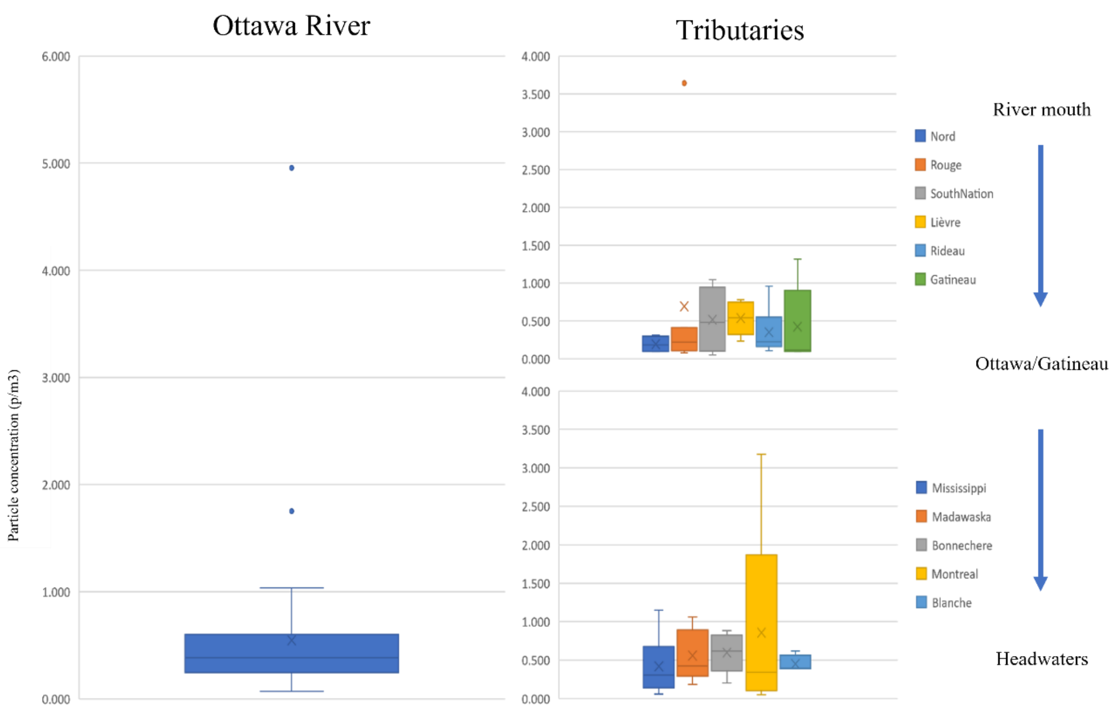

Figure 5-3 Box plots representing particle concentration (p/m^3) for the Ottawa River and sampled tributaries.

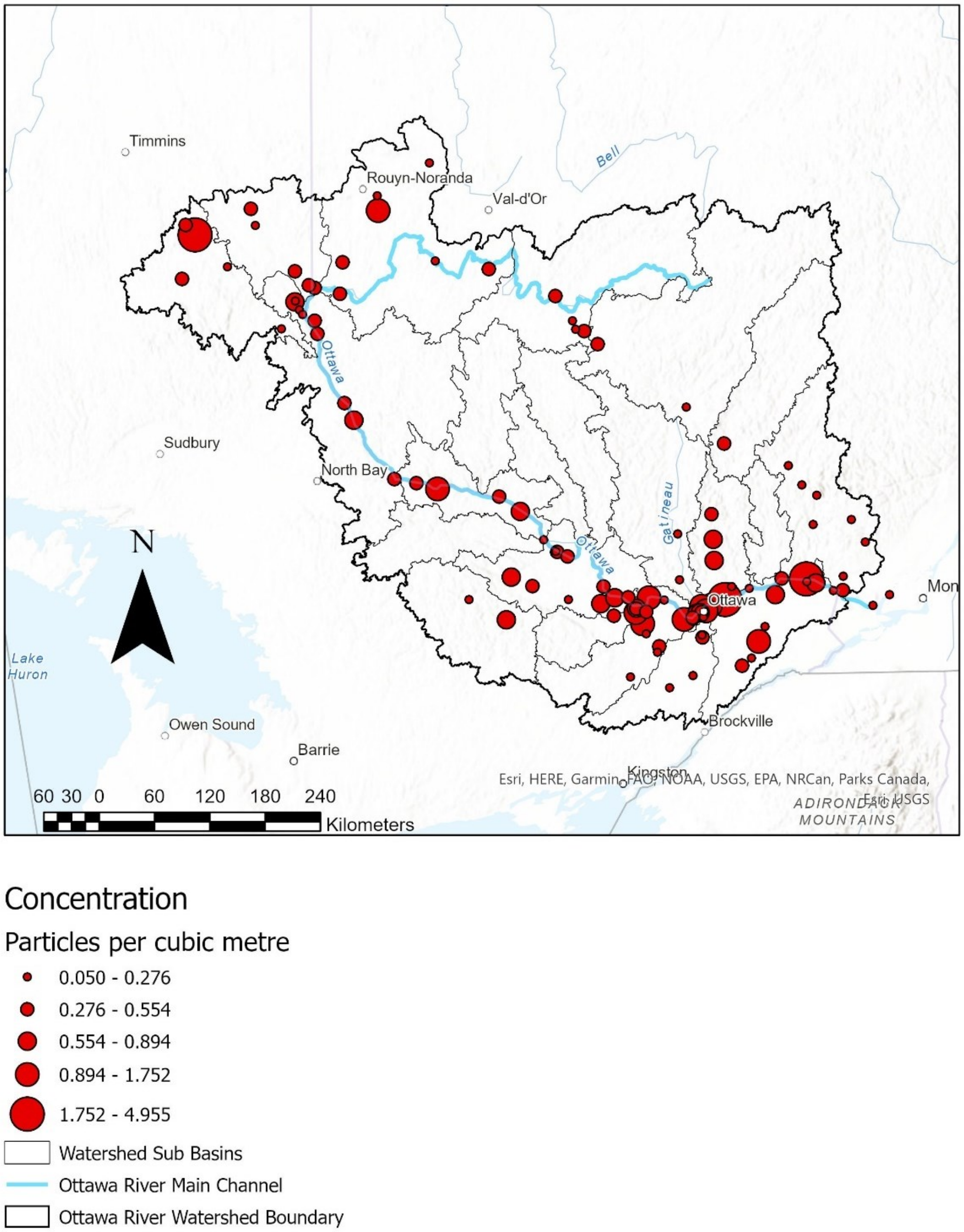

Figure 5-4 Particle concentration at each sample point throughout the Ottawa River watershed.

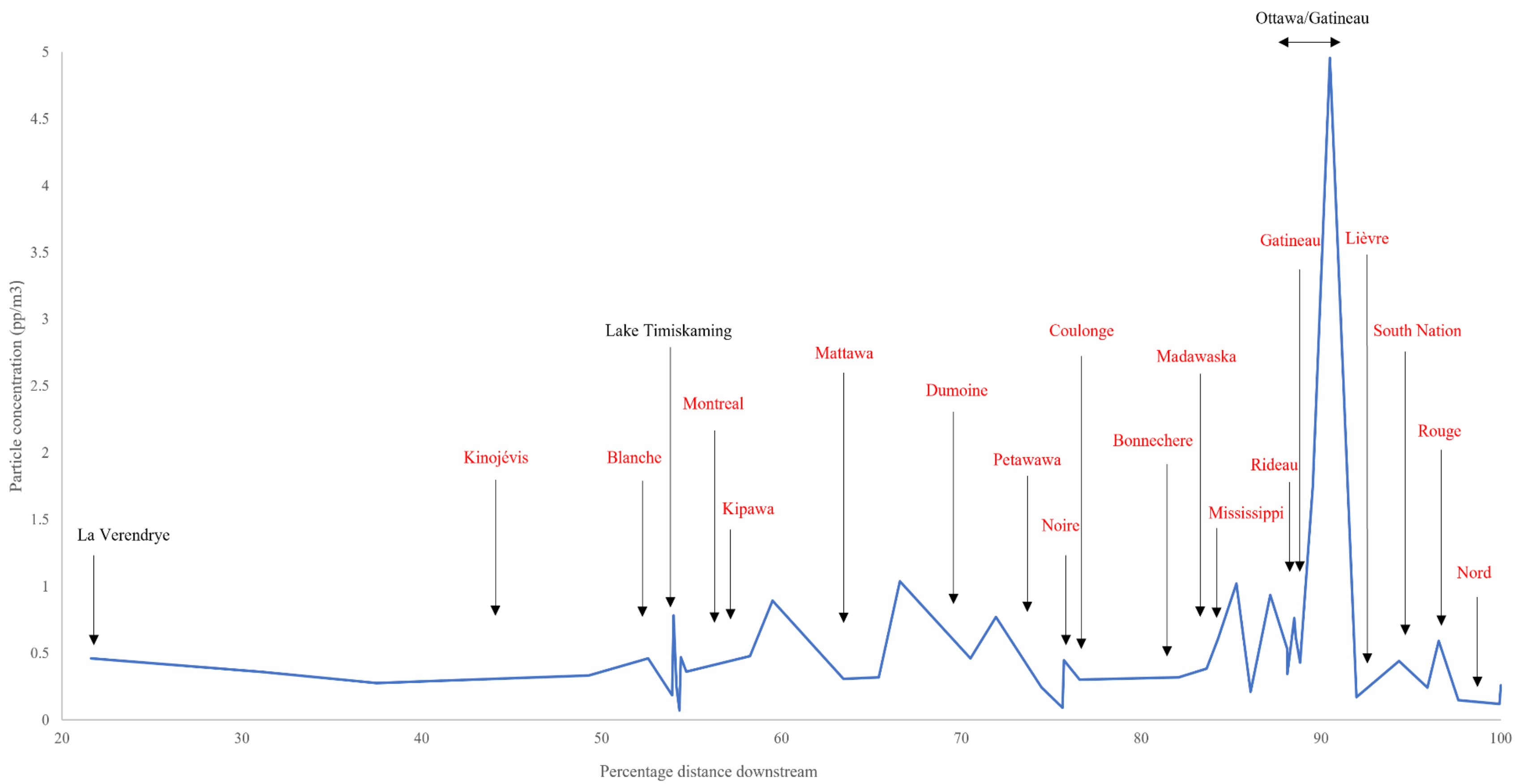

Figure 5-5 Particle concentration as you move downstream on the Ottawa River with the location of the tributaries entering the main channel and the location of the La Verendrye Wildlife Reserve, Lake Timiskaming and the Ottawa/Gatineau urban area.

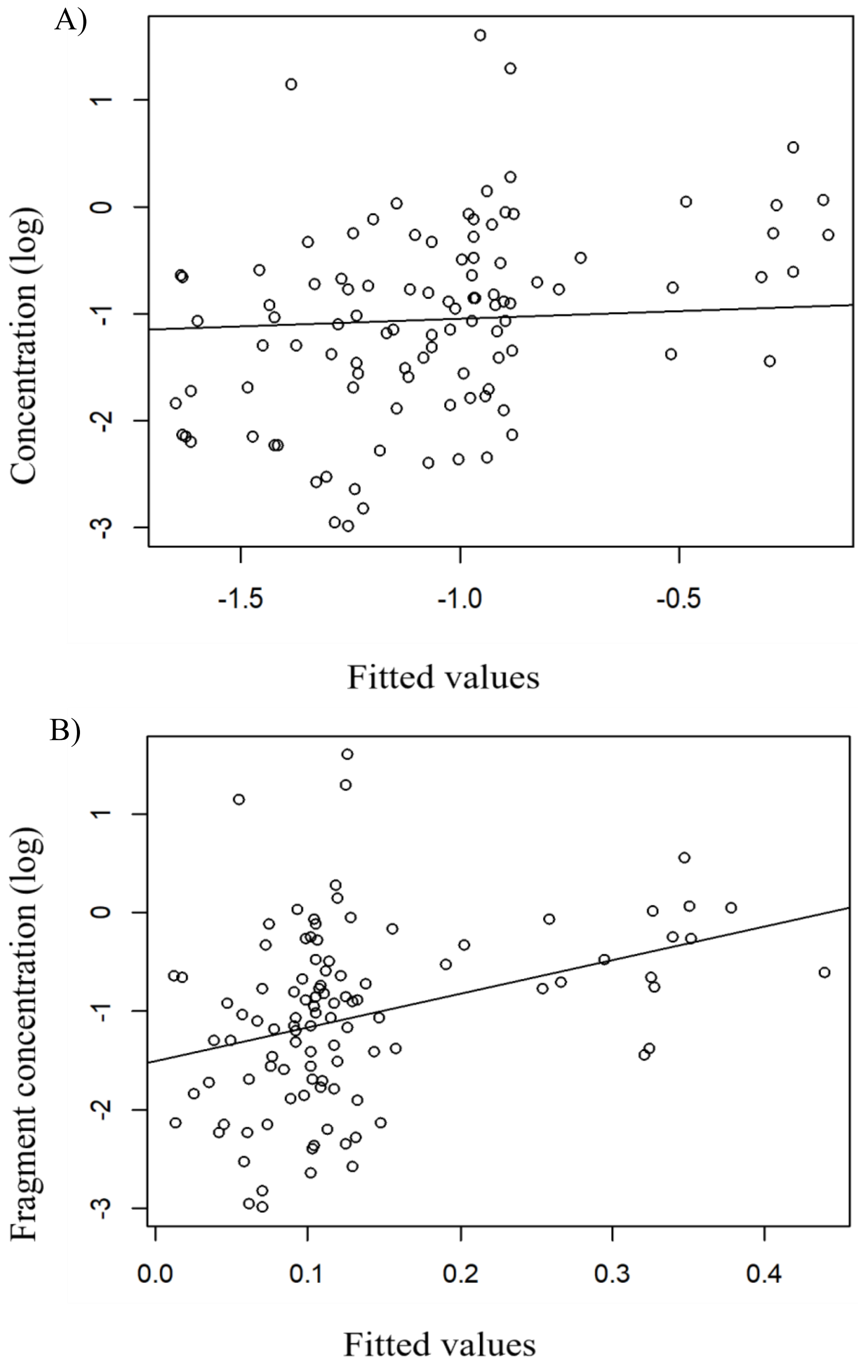

Figure 5-6 Concentrations (log) versus fitted values from the ANCOVA analysis with a linear trendline presenting the strength of the model fit Graph A) represents considered continuous and categorical variables of interest to microplastic concentrations with graph B) the same ANCOVA analysis with the same variables of interest, while only considering microplastic concentration of just fragments.

Table 5-2 Sample concentrations directly upstream and downstream of WWTP locations.

WWTP Name	Sample location name	Upstream concentration	Sample location name	Downstream concentration
Gatineau/Ottawa	Blair Road	1.752	Petrie Island	4.955
Brebeuf/Labelle	Labelle	0.080	Hubredeau	0.222
Arnprior	Highway 417	1.062	Arnprior	0.404
Almonte	Appleton	0.521	Blackney	0.167
Renfrew	Renfrew	0.885	Horton	0.765
Chesterville	Cass Bridge	0.483	Crysler	1.047
Wakefield	Wakefield	0.095	Fournier	1.316
Petawawa	Petawawa Point	0.245	Pembroke Riverside	0.092
Haileybury	Haileybury	0.251	Devils Rock	0.071
Average		0.597		1.004

5.5 Discussion

The distribution of sample concentrations was relatively consistent, with many of the noted sample locations demonstrating particle concentrations under or close to 1 p/m^3. However, there were some locations of note that were elevated compared to the majority of the samples. There were two elevated concentrations on the main channel of the Ottawa River, both were located withing the Ottawa/Gatineau urban areas., One of these sample locations presented the highest concentration of all samples, downstream of two major WWTPs. This will be further discussed later in the current section. Another outlier was noted on the Rouge River, for a location at the river mouth before it entered the Ottawa River. This location is not heavily populated and presented a concentration far greater than any other concentration upstream. Though potentially an anomaly, some precipitation was noted in the watershed in previous days, however, it could be difficult to establish if there was a temporal influence on the elevated concentration. This could warrant further research to examine the possible reason behind the high concentration at this location. One sample point on the Montreal River at Matachewan highlighted an elevated sample concentration compared to other sample points on the river and was not presented as an outlier in the analysis. This particular location is directly downstream of a large gold mining operation, which could be important due to the use of black plastic sheets, common at the bottom of tailings ponds utilised by some mining operations. Though this has not been confirmed at this particular mining location, there was a marked increase of black fibres extracted at this specific location. This result could justify further investigation around mining operations, especially those that use tailing ponds, to see if there is an influence of mining runoff as a point source of microplastics to freshwater environments.

Even remote locations, far from any major human influence, can present some level of contamination (Browne et al., 2011; Free et al., 2014; Jiang et al., 2019). This is evident when assessing sample points throughout the La Verendrye Wildlife Reserve, with four locations sampled within the reserve and one location just as the main channel exits the reserve. These samples returned an average particle concentration of 0.317 p/m^3. This is not far removed from the overall sampling average of 0.528 p/m^3. The majority of the particles in these samples were fibres, approximately 85%. Microfibres have been identified in atmospheric transport (Dehghani et al., 2017; Dris et al., 2017; Gasperi et al., 2018) and thus are capable of fallout into more remote regions of watersheds. Additionally, fragments are also potentially transported atmospherically (Cai et al., 2017; Kole et al., 2017), and some samples did contain fragments. However, though the region is remote, not all of the particles are necessary transported atmospherically, as there is still some anthropogenic activity within the reserve, for example camping and boating.

One of the standardisation objectives of the research was to reduce the possible temporal influences while sampling in the watershed. This included attempting to avoid sampling during, or after heavy precipitation. Nonetheless, rainfall amounts from the closest weather station were noted during the day of sampling and the two days prior, to examine the possibility of precipitation influencing microplastic concentrations. As some advanced sampling planning was essential, avoiding all rainfall events, especially in the summer when rainfall can be sporadic and heavy due to isolated storms, was difficult. Therefore, precipitation events did occur throughout some sampling days. Though the relationship did not present any statistical relationship in the ANCOVA analysis, it is still an important temporal factor to consider while assessing the data. WWTPs have been identified as a major conduit of microplastics to freshwater systems (Estahbanati & Fahrenfeld, 2016; Leslie et al., 2017; Talvitie et al., 2017; Ziajahromi et al., 2017), however, the current research did not present a statistical significance to particle concentration and distance downstream of WWTPs. Various reasons could explain this, including the importance of temporal influences. For example, combined sewage overflows and stormwater during rain events may have a stronger bearing on increasing concentrations downstream of WWTP or urban locations (as what will be presented in Chapter 6), rather than presenting a spatial correlation to the proximity to WWTPs. Additionally, wastewater sludge applied to agricultural areas is a known source of microplastics (Zubris & Richards, 2005; Corradini et al., 2019) with precipitation also entraining microplastics through runoff over WWTP sludge (Eriksen et al., 2013). This could be important, as agricultural areas are typically a distance from WWTP point sources, thus it may reduce potential of presenting increased particle concentration only within a close proximity to a WWTP. These factors negate a strong spatial influence to WWTP locations. Even when examining sample locations directly upstream and downstream of known WWTP in the watershed, only some of the locations noted a downstream increase in concentration. However, the largest concentration recorded in the research was noted directly downstream of two of the largest WWTPs in the watershed (Petrie Island), one servicing a large proportion of the City of Ottawa and the other servicing a large proportion of the City of Gatineau. The upstream location noted a significantly lower concentration to the location downstream. Furthermore, this location has previously been noted to present high relative microplastic concentrations in the watershed (Vermaire et al., 2017). Likewise, other WWTP locations do not service significant urban populations, with only

Petawawa's population of approximately 17,000 people, Arnprior's population of approximately 8,700 and Renfrew's of approximately 8,200 are the only other population sizes of note. This is compared to the Gatineau WWTP, which services approximately 280,000 people and the Robert O. Pickard Environmental Centre in Ottawa which processes a population of approximately 890,000. Therefore, these facilities process wastewater for a population base of over 1 million. This demonstrates that the amount of wastewater a treatment plant processes, not just the location of a WWTP, is important to consider for the potential increase of microplastics in downstream river locations.

Though urban areas have been acknowledged to influence microplastic concentration to freshwater sources, many of the examples given in previous research include highlighting temporal events in the flux of microplastics. For example, stormwater events (Grbić et al., 2020; Piñon-Colin et al., 2020; Ziajahromi et al., 2020), which tend to be short term high load events. However, the current research demonstrates no significance in spatial concentrations between urban and non-urban locations.

Another hypothesised influence was dams in the Ottawa River watershed. Previous research has noted the potential for microplastic build up behind dams (Zhang et al., 2015), however, there is a lack of sample points around dams in the current research in addition to potential unrepresentative sample points for dam analysis. For example, many of the shoreline locations in close proximity to dam structures are not accessible for sampling. Effective analysis would more likely be achieved through a mid-channel trawl behind a dam, to compare with sample points downstream of the structure. This could be a more specific analysis for future research, as the Ottawa River has over 50 dams throughout the watershed, with hundreds more water control structures throughout the main channel and tributaries, making it one of the most highly regulated rivers in Canada (Ottawa Riverkeeper, 2022). The other significant relationships noted in the ANCOVA was the increase in concentration at boat launch locations. Typically, these locations have additional human activity with vehicles and boating craft constantly entering and exiting the river from these point sources. This increase in human activity in and around these locations suggest additional research is warranted, as it is potentially an important local point source of microplastics.

The research also examined microplastic concentrations while examining fragments and discounting fibres, under the same potential spatial influences. The results suggest a similar conclusion to what was identified with total particle concentration analysis. Nonetheless, there was a slight increase in the R^2 value from the ANCOVA analysis suggesting a slightly stronger relationship to an increase of fragments at boat locations and further downstream of the tributaries and main channel. For example, as a localised point source boat launches can present more fragments due to vehicles and boat trailers shedding tire wear particles in addition to paint and fishing gear as other potential sources of fragments.

5.6 Conclusions

Sampling was conducted at 105 sample points throughout the Ottawa River watershed including tributaries and the main channel of the Ottawa River during low flow conditions. Two spatial factors examined were highlighted to be significant after an ANCOVA analysis. The first spatial factor to demonstrate a significance was microplastic concentration in relation to distance downstream on the main channel and tributaries.

However, the regression analysis only demonstrated a low R^2 value of 0.15, indicating additional factors or variables are influencing microplastic concentrations as you move downstream in the watershed. The other significant relationship was between boat launch locations and non-boat launch locations, with an increase of microplastics at boat launch locations indicating localised factors contribute to increasing concentrations at these point sources. The other variables examined, namely microplastic concentration in relation to precipitation, urban versus non-urban locations, tributary versus main channel locations, distance downstream from wastewater treatment plants and distance up and downstream from dams presented no significant relationships. These results highlight the complex interaction of microplastics in large watersheds and suggest temporal factors are also important to consider while examining spatial distribution. This emphasizes the importance of moving away from only focusing on a presence or absence methodology at a singular (sometimes random) temporal point in time. It is evident that temporal factors need to be considered in large watersheds, with a move towards focusing on more of a monitoring approach to be able to better assess the inputs, throughputs and sinks of microplastics in large watersheds.

6. Change in microplastic concentration during various temporal events downstream of a combined sewage overflow and in an urban stormwater creek

6.1 Introduction

Global plastic waste continues to accumulate at alarming rates with an estimated 60% of the 8,300 million metric tonnes of produced plastic discarded as waste (Geyer et al., 2017). Substantial amounts of plastic waste are awash in oceans with an estimated 5 trillion pieces of plastic weighing over 250,000 tonnes (Eriksen, 2014). Plastic waste is becoming a major emerging pollutant with suggestions it will be considered a geological indicator of the Anthropocene (Zalasiewicz et al., 2016). Microplastics, tiny pieces of plastics less than 5mm (Masura et al., 2015), are becoming a global concern with these tiny pollutants identified in the deepest ocean trenches (Peng et al., 2018) and at the highest point on earth (Napper et al., 2020). The majority of the research on microplastics to date has focused on marine areas and it is suggested that microplastics are a contaminant of emerging concern (Wagner et al., 2014; Eerkes-Medrano et al., 2015). However, little research has focused on freshwater ecosystems, especially as rivers are identified as the major source of plastics to oceans (Lebreton et al., 2017), with riverine environments delivering up to 80% of plastic debris to seas and oceans (Skalska et al., 2020). To date, there has been little, if any, focus on the extent of temporal variation and environmental drivers of change in microplastic concentrations from previously identified major sources, for example wastewater treatment plants (Estahbanati & Fahrenfeld, 2016; Horton et al., 2017; Vermaire et al., 2017; Windsor et al., 2019) and stormwater systems (Liu et al., 2019; Olesen et al., 2019; Grbić et al., 2020; Piñon-Colin et al., 2020; Ziajahromi et al., 2020).

One potential and major source of microplastics to freshwater systems, especially river environments, is combined sewage overflows. Sewer systems in larger urban areas are typically designed in one of two ways: a combined sewer and stormwater system; or a split-system where sewage and stormwater are conveyed separately. Traditionally, older and more established urban areas have combined sewer systems, thus, both stormwater and sewage are conveyed in the same pipe to treatment facilities. However, during larger rainfall (or snowmelt) events, treatment facilities may reach a critical volume threshold and at this point, a combined sewage overflow is activated. During these events, both sewage and stormwater are released directly into receiving bodies of water to ensure treatment facilities are not overwhelmed. These events can contain large amounts of microplastics, as typically during the wastewater treatment process, many microplastics are removed and end up in wastewater sludge (Zubris & Richards, 2005; Nizzetto et al., 2016; Corradini et al., 2019). Thus, overflow events would contain microplastics that would otherwise be removed through treatment processes.

In many newer urban areas, or in suburbs within established urban centres, sewer systems are separated. In this case, stormwater is conveyed separately from sewage. Only sewage is transported to treatment facilities whereas stormwater flows directly into receiving bodies of water via stormwater drains. In some instances, stormwater may flow into urban creeks or small tributaries of larger river systems. These creeks tend to have modified channels and/or structures controlling flow, especially during high rainfall events, but do not typically receive wastewater, only stormwater.

Some microplastic research is emerging on the role of urban creeks in conveying microplastics (Dikareva & Simon, 2019) including research on microplastics in stormwater (Grbić et al., 2020; Ziajahromi et al., 2020) and stormwater ponds (Liu et al., 2019; Olesen et al., 2019). However, research is still relatively minimal, especially within freshwater systems, with limited research to date on the change in microplastic concentrations during combined sewage overflow events. One major reason for this is that combined sewage overflow outlets are typically underwater and hard to identify with very few locations having any real-time indication of an active event. Therefore, it can be difficult to sample these events. The current research aims to temporally sample a major urban river downstream of a combined sewage overflow and an urban creek that receives stormwater, to examine the change in microplastic concentration over various weather and seasonal events.

6.2 Study Area

The research was conducted in the City of Ottawa, Ontario, Canada, at LeBreton Flats near the downtown core of Ottawa, and Graham Creek in Andrew Haydon Park in the city's West End (Figure 6.1). The LeBreton Flats location is a diversion from the main channel of the Ottawa River used by the Ottawa River Runners kayak club, where the water flows through a kayak course before joining the Ottawa River again downstream of the Chaudière Falls. At this location, the City of Ottawa has installed a light (Figure 6.1) to signal real-time sewage overflow events, so kayakers know when water quality may be impacted. This signal light was the primary reason LeBreton Flats was selected as the Ottawa River location due to a real-time indication of active sewage overflow events. The Ottawa River is a major tributary of the Saint Lawrence River and runs approximately 1272 km from its source to its terminus near Montreal, Quebec. The Ottawa/Gatineau urban area is the largest urban area on the river, at approximately 1.3 million inhabitants, and located roughly 130 km upstream from the river mouth.

Graham Creek is a small urban creek with an area of approximately 25 km^2. Graham Creek joins the Ottawa River at Andrew Haydon Park and begins at the Stony Swamp Wetland, roughly 6 km south of the Ottawa River. The creek flows through agricultural areas, however, the lower portions of the creek flow through urbanised areas with numerous channel modifications, including stormwater structures that convey stormwater runoff during rainfall and snow melt events. Stormwater outfalls are utilised in the Ottawa River watershed where there is a separate sewage system, thus, most stormwater drains are easier to identify, as they are dry during non-stormwater events, and flow during stormwater runoff. Therefore, the suburban sewer system around Graham Creek is separated and The LeBreton Flats location is in an area of the city where the sewer system is still combined.

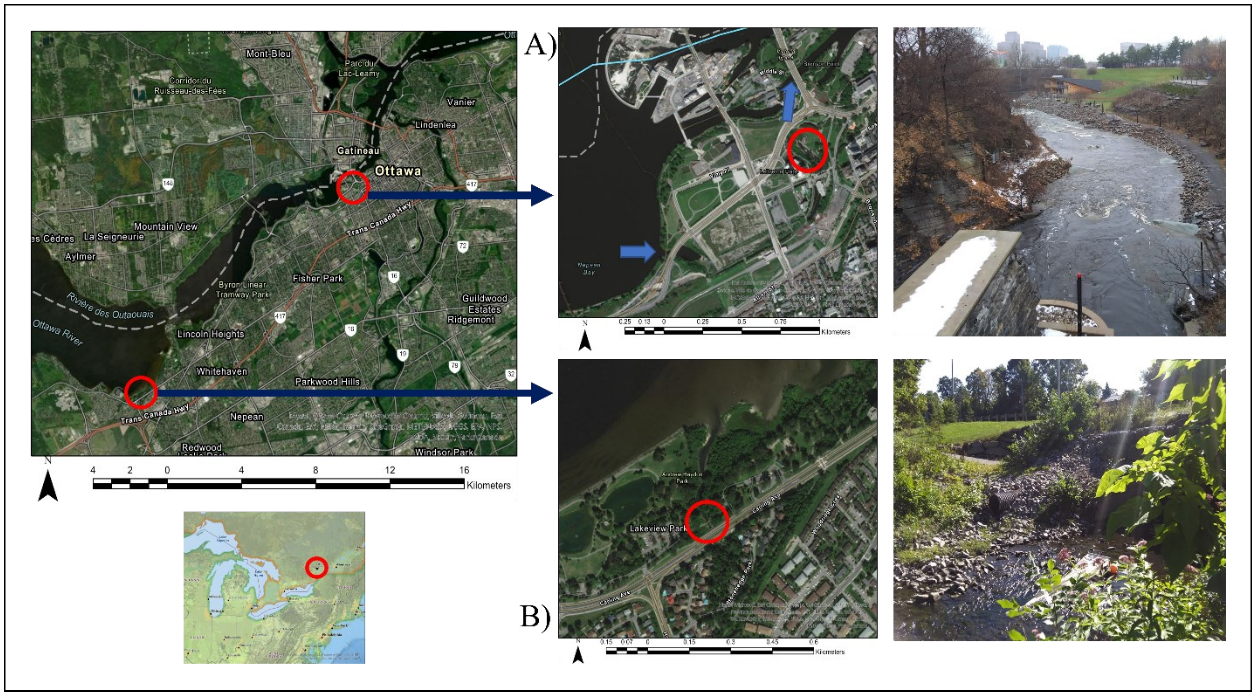

Figure 6-1 The locations of the two-research sites in the City of Ottawa A) A combined sewage overflow at LeBreton Flats, note the signal light in the foreground that is activated during real-time combined sewage events. B) Graham Creek, an urban creek, note a stormwater input near the left bank of the creek.

6.3 Methodology

Sampling at both locations was conducted from November 2019 to June 2021 during heavy rain events, low to moderate rain events, during spring snow and during a primary and secondary snowmelt event of the spring. Additionally, LeBreton Flats was sampled during an active combined sewage overflow event. Furthermore, dry, non-precipitation events were sampled at both locations to indicate potential ambient or baseline microplastic conditions, in order to compare the increase in microplastic concentrations during identified temporal events.

6.3.1 Field methods

Sampling at Graham Creek was conducted with a Manta trawl net with a 300-µm mesh and detachable cod end and a flow meter. The net was deployed into the creek for between 5-20 minutes depending on flow, organic load and debris in the creek. For example, for rain and snowmelt events, there was typically one deployment for a shorter period as organic load was high in addition to higher plastic particle counts, whereas in ambient conditions, the net could be deployed for longer periods, as the net would not clog, and it gave the opportunity to capture more particles to reduce the impact of potential airborne contamination on final particle counts. The creek was shallow enough to deploy the net on the shoreline capturing the entire water column, with the depth of the creek covering between 30 and 80% of the net opening. Between each of the sample runs, the Manta net was flushed with water along the outside of the net to move all the captured contents into the cod end of the net. The cod end was then detached, and the contents washed into a clean glass Mason jar for transport to the laboratory. After each sample was completed, the

Manta net was backwashed, with the cod end backwashed separately, to ensure there was no cross-contamination between sample dates. The volume of water sampled was calculated by taking the known area of the Manta net opening and determining the percentage coverage of the water at the net opening during sampling. The flow of the river was measured with a flow meter and averaged for the duration of the sampling time.

Graham creek was sampled on 10 different occasions between 2019 and 2021, representing a variety of temporal conditions, including the onset of heavy rain, during heavy rain, during dry and extended dry conditions, during the primary spring snowmelt in addition to a secondary snowmelt event and during snowfall. Additionally, sampling was conducted during spring, summer and fall. No winter sampling was conducted as there is no flow in the creek, as the creek is typically frozen.

The overflow location at LeBreton Flats was sampled a total of nine times between 2019 and 2021 approximately 150 metres downstream of the Fleet Street sewage overflow outlet. The representative temporal conditions included sampling during an active overflow event, during rain without an overflow event, the day after a heavy rain event, during an extended dry period, during the influence of spring freshet conditions on the Ottawa River and during the primary spring snowmelt in addition to a secondary snowmelt event. Furthermore, there were seven temporal events where both LeBreton flats and Graham Creek were sampled to compare the microplastic concentration at each location. Sampling at LeBreton Flats was conducted with a 100-µm mesh inside of a metal funnel and 1000 litres (L) of water over three samples was passed through the mesh using a bucket. Sampling was conducted on the shoreline with the bucket submerged into a shallow area of the shoreline, with good current. The bucket was submerged into approximately 50% of the water column. A control metal funnel and mesh were also present to indicate the potential for airborne contamination during sampling. Each of the samples and control mesh were then placed into a Whirl-Pak bag and transported to the laboratory and stored in a freezer until ready for processing. This method of sampling was chosen over deploying a Manta net, as the flow of the river at the LeBreton location is swift, thus, it is difficult to deploy a Manta net. Furthermore, during a combined sewage overflow event and/or heavy rainfall, fouling potential for a Manta net is high in addition to increased flow in the river. For these reasons, a grab method of sampling with a bucket was chosen.

6.3.2 Laboratory analysis

The100-µm mesh filters and the contents of the Mason jars from the Manta samples were washed into a clean beaker using filtered deionised water. A 30% hydrogen peroxide solution was added, and the sample heated to 50ºC in a water bath for digestion of organic material in the sample, keeping the temperature below 70 ºC as not to alter the inherent shape of polymers that may be in the sample (Kalpakjian & Schmid, 2008) and to avoid the potential complete loss of microplastics in wet peroxide oxidation in temperatures above 70 ºC (Munno et al., 2018). Digestion time varied depending on organic content of the samples (between 2 and 14 hours). Once digestion was completed, the solution was passed through a vacuum filtration system with a clean (pre-inspected) 80-µm metal mesh filter, that was then backwashed into a clean (pre-inspected) petri dish after the solution had completely passed through. Visual inspection identified potential microplastics under a Leica stereomicroscope at 20 to 40X magnification. Suspected microplastics were identified and classified as fibres, fragments or beads (Lusher et al., 2020). The colour of each particle was also noted (up to secondary colours including black and white), however, it has been suggested that broad colour classifications are not sufficient to indicate particle similarity, given the range of shades available, but noting microplastic colour is still recommended during visual assessment (Lusher et al., 2020) as it may give information on potential broad trends (Shaw & Day, 1994). Additionally, various fragments were put aside for further analysis to identify polymer types using micro-Fourier-transform infrared spectroscopy.

6.3.3 Quality assurance and quality control

Quality assurance and quality control (QA/QC) included strict processing protocols in the laboratory to avoid airborne contamination in addition to performing procedural blanks. All work was conducted under a laminar flow hood to reduce airborne contamination of the samples while being prepared and analysed. The research team wore natural fibre laboratory coats and minimised movement around the laminar flow hood during sample preparation and analysis. Procedural blanks in the laboratory included placing nine laboratory blanks under the laminar flow hood to account for fibre precipitation during sample preparation and analysis. Analysis of laboratory blanks returned an average of 1 fibres per blank, with a maximum count of three fibres and a minimum of zero. The LeBreton Flats field controls aimed to mimic the sampling procedure as closely as possible (Brander et al., 2020) and presented an average of 3 fibres with a maximum count of four in one control (supplementary tables). For LeBreton Flats, fibres in the controls were approximately 10-200-fold lower than those in the samples, therefore, contamination errors were considered negligeable Additionally, the concentrations were standardised with a z-score for comparison with Graham Creek concentrations. Field controls were not conducted at Graham Creek as volumes were significantly higher than LeBreton Flats, with sample volumes over 120,000 litres in ambient conditions, thus, potential atmospheric contamination influences were considered negligible.

Two different mesh sizes where utilised for sampling due to the aforementioned field sampling circumstances. It has been noted that samples collected from a 300-µm mesh compared to a 100 µm mesh, could underestimate fibres by up to four orders of magnitude (Covernton et al., 2019). However, a z-scores were calculated for both LeBreton Flats and Graham Creek (discussed in further detail in the preceding section) to allow for a comparison between the mutual temporal events. Furthermore, as the LeBreton Flats samples did not collect from the total water column like Graham Creek, the size range of microplastics is likely smaller (Barrows et al., 2017).

6.3.4 Statistical analysis

To examine if changes in microplastic concentration at the two study sites were synchronous as a result of similar environmental conditions a Pearson's correlation analysis was carried out in R (R Core Team, 2020) on the seven sampling days that overlapped between the two sites. Because of the different methods used in microplastic sampling at the two sites the data were standardized for each site by subtracting the mean and dividing by the standard deviation (z-score). Additionally, a principal component analysis was conducted in R using a square root of the variables to down weight relative abundance and to visualise the change in fibre and fragment colour types according to the temporal event.

6.4 Results

At LeBreton Flats, the nine samples averaged 88 particles per cubic metre of water (p/m^3) with a minimum of 10 p/m^3 and a maximum of 273 p/m^3. The standard deviation over the nine samples was 79 p/m^3. The active overflow event on November 1st, 2019, corresponded to a concentration of 167 p/m^3. This was after approximately 62mm of rain in the 48 hours preceding the sampling. The highest concentration of 273 p/m^3 was during the first snowmelt event of spring 2021. The weather was clear with no previous precipitation over 48 hours. However, the increased air temperature created visible overland runoff from the melting snowpack. An additional melt event six days later was also sampled and represented a concentration of 92 p/m^3. Additionally, no rain was noted during the previous 48 hours for this event. On March 26th, 2021, the location was sampled with a concentration of 49 p/m^3. Previous rainfall was noted at approximately 37mm 48 hours before the event. A spring snowfall event was sampled on April 21st, 2021, and the concentration was noted at 88 p/m^3 with the approximate amount of snow falling at 0.4 cm in the previous 24 hours. On May 14th, 2021, the concentration was noted at 29 p/m^3 with the possibility of a spring freshet influence as the river was running high with no rain or stormwater inputs. On June 4th, 2021, the location was sampled, and the concentration was 34 p/m^3. Steady rain was noted the day before with approximately 7 mm of rain falling 24 to 48 hours before, but no rainfall eight hours before sampling, in fact this rainfall broke a prolonged dry spell. After a dry spell, the river was sampled on June 11th, 2021, and the concentration was noted at 10 p/m^3, the lowest recorded concentration. The ninth sample was conducted on July 24th, 2021, with a concentration of 34 p/m^3. Some rainfall was noted eight hours before sampling, approximately 2.3 mm of rain. However, the rainfall was sporadic, and isolated, typical of summer precipitation in the watershed.

At Graham Creek, the 10 samples averaged 12.93 p/m^3 with a maximum of 52.55 p/m3 and a minimum of 0.37 p/m^3. The standard deviation over the 10 samples was 15.87 p/m3. On September 9th and 18th, 2019 sampling was conducted after dry conditions with a concentration of 0.40 and 0.37 p/m^3 respectively. On October 31st, 2019, the creek was sampled during a heavy rain event and the concentration was noted at 19.92 p/m^3. The rainfall was noted at approximately 20.6 mm over the previous 48 hours. This was the same event as the active combined sewage overflow for the LeBreton Flats location, sampled the day before. Another rain event on October 21st, 2020, was sampled with a concentration of 26.51 p/m3. As with the overflow location, samples were conducted on March 11th, March 26th, April 21st, May 14th and June 4th, 2021. The primary snowmelt event on March 11th highlighted the highest recorded concentration of 52.55 p/m^3. The secondary snowmelt event on March 26th presented a concentration of 6.96 p/m^3. The snowing conditions of April 21st had a corresponding concentration of 15.82 p/m^3, with the drier conditions of May 14th and June 4th exhibiting concentrations of 0.60 and 0.75 p/m^3 respectively (detailed sampling notes can be found in supplementary tables)

Changes in microplastic concentrations at the two sampling sites during the seven overlapping sampling days were highly correlated ($r = 0.96$, $p < 0.001$), suggesting changes in microplastic concentrations were driven by changing environmental conditions common between the two sites (Figure 6.2).

In the LeBreton combined sewage overflow samples, 96% of the extracted particles were fibres with 4% fragments. The fibres were predominately blue in colour corresponding to 65% of all identified fibres. Other colours of note for the fibres were 16% black, 10% red and 4% green. Blue fragments were also the predominant colour accounting for 53% with 16% white, 13% green and 7% black.

The Graham Creek samples were also predominantly fibres, with fibres accounting for 79% of the extracted particles (Figure 6.3). The predominant fibre colour was blue with 74%, with other common colours 17% black, 3% red and 2% green. For the extracted fragments, black was the dominant colour with 57% of the fragments, with other colours of note at 21% blue, 10% red, 5% white and 2% green.

Additionally, the relative change in fragments and fibres (according to z-scores) changed according to the temporal event (Figure 6.4), with an increase of fragments at Graham creek coinciding with the heavy rain event on October 21st, 2020, and an increase in fragments was noted at LeBreton Flats during the primary snowmelt. Additionally, an increase of fibres was also noted at LeBreton Flats during the primary snowmelt. An increase of fibres was noted at Graham creek during the heavy rain event on November 1st, 2021, and during the primary snowmelt. The PCA analysis (Figure 6.5) highlights an overall difference in fibre and fragment colours between Graham Creek and LeBreton Flats with some increase in white and black fragments during some Graham Creek dry periods, however, there is no obvious community of microplastics.

As the majority of the fibres extracted from the samples were deemed to be anthropogenically modified (as opposed to natural cotton and/or wool fibres) a selection of fragments were chosen to be analysed under micro-Fourier-transform infrared spectroscopy (Figure 6.6 and Figure 6.7). Of the 17 samples analysed with micro-Fourier-transform infrared spectroscopy, all were identified as plastics.

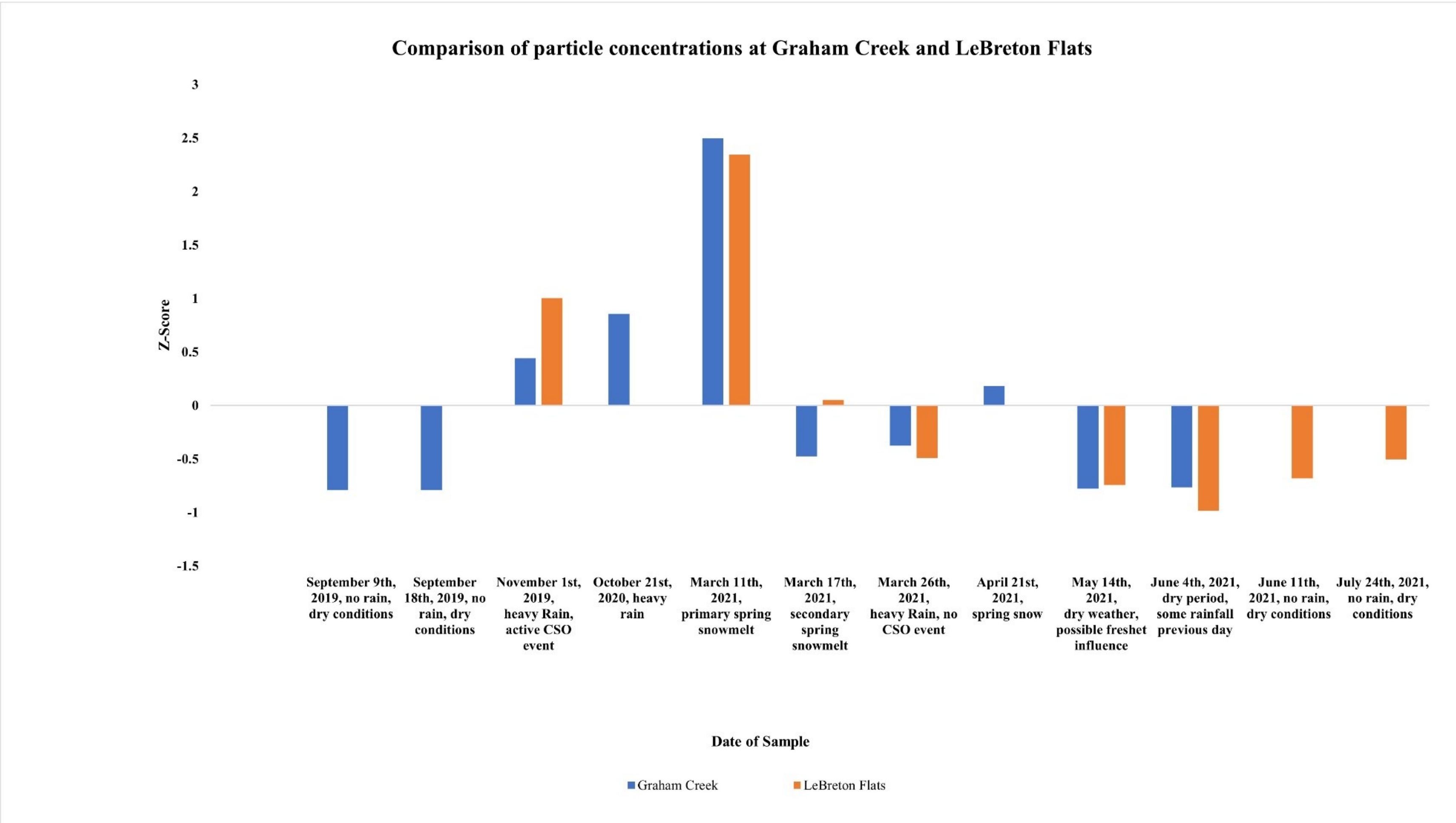

Figure 6-2 Z-score values for comparison of particle concentration between Graham Creek and LeBreton Flats during various temporal events.

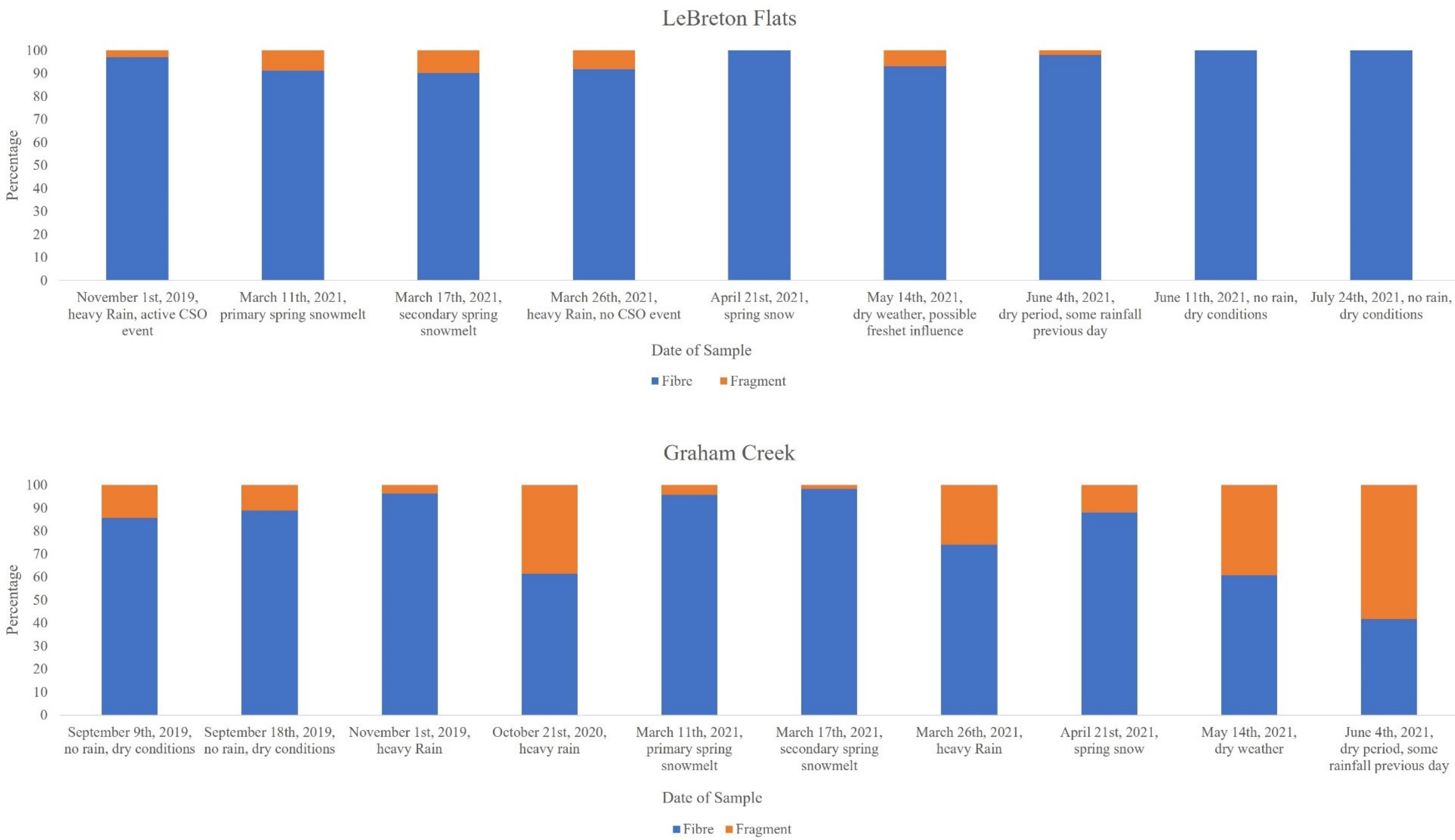

Figure 6-3 Comparison between fibres and fragments during each sampled temporal event at Le Breton Flats and Graham Creek.

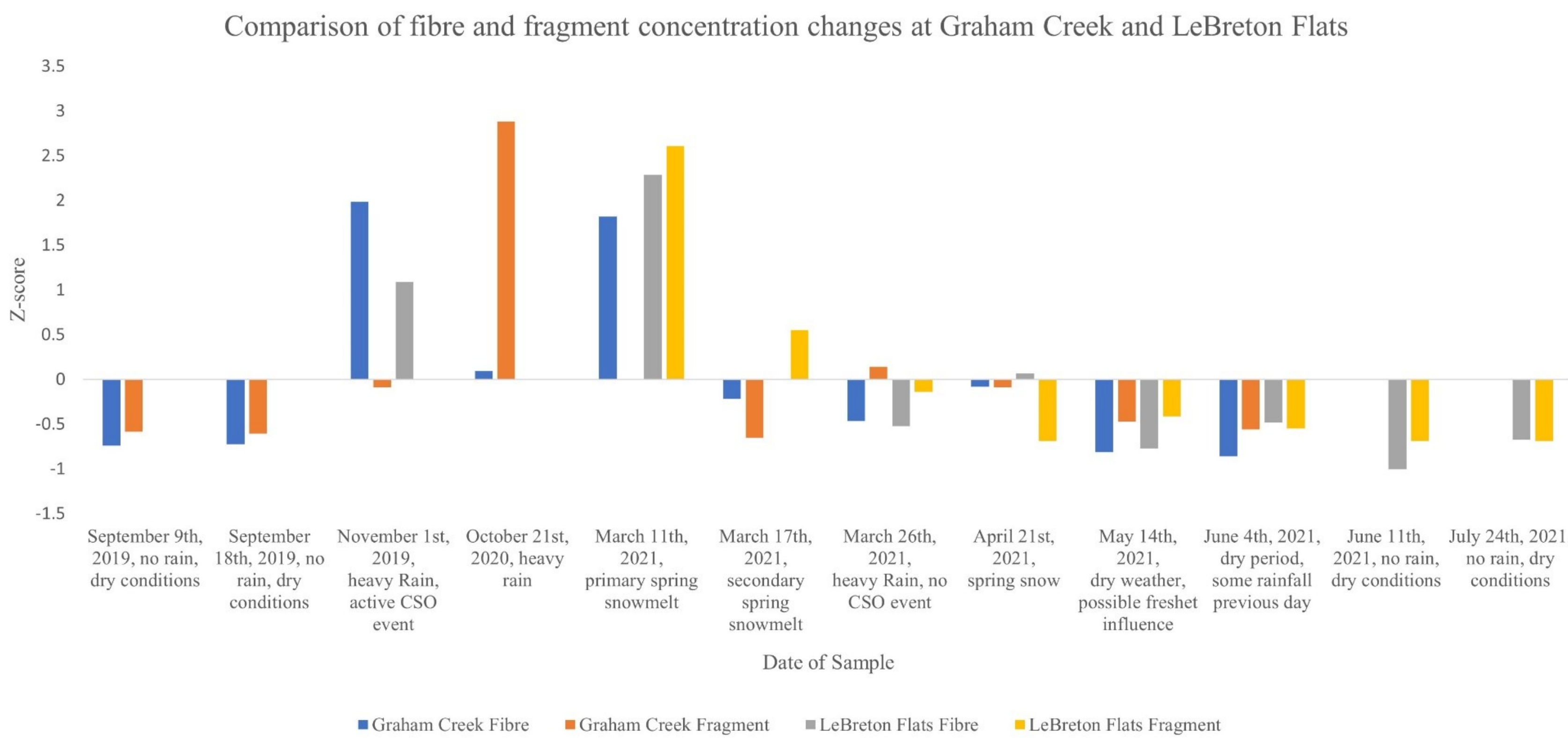

Figure 6-4 Z-score values for comparison of the change in fibres and fragments between Graham Creek and LeBreton Flats during various temporal events.

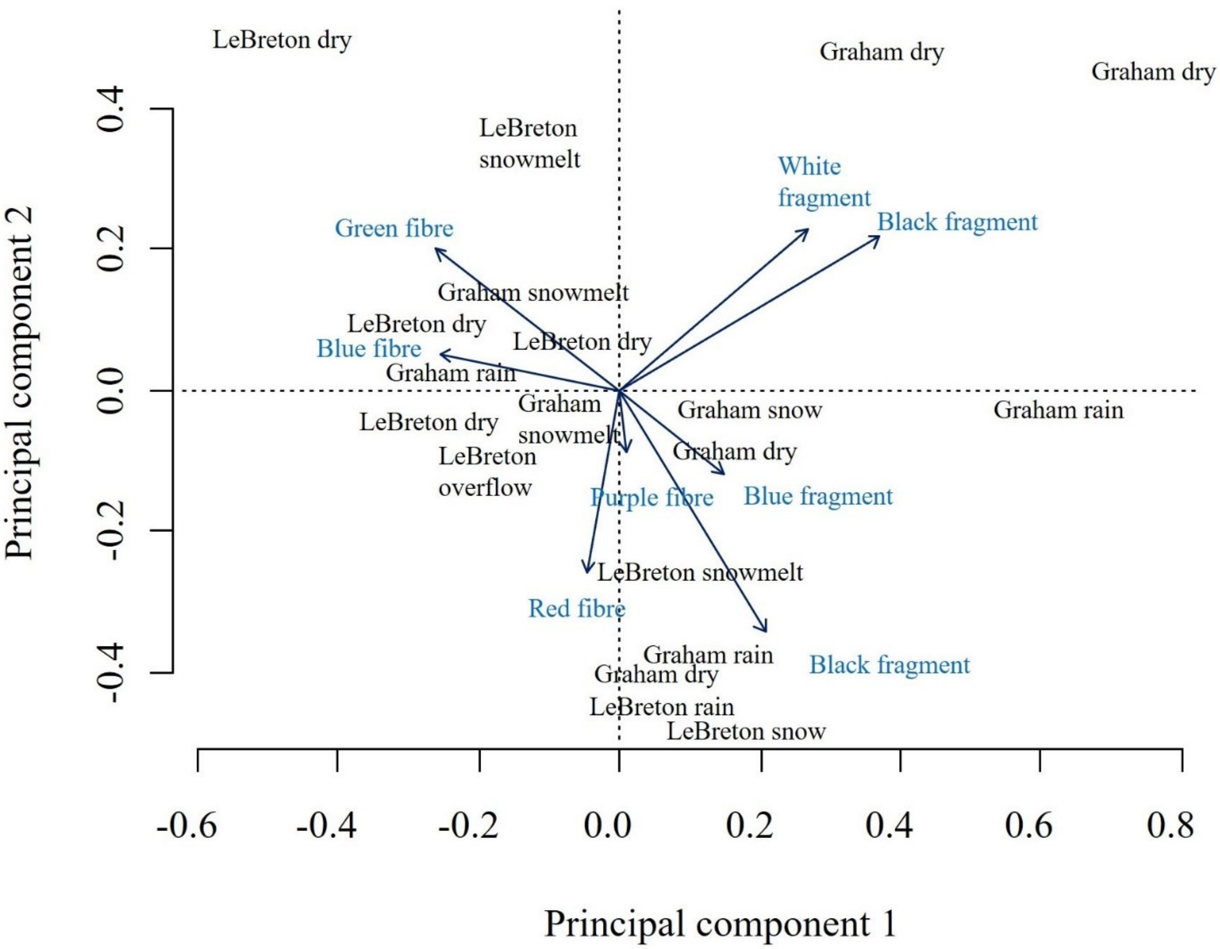

Figure 6-5 A principal component analysis highlighting the change in fragment and fibre colours according to temporal events.

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Date: Thu Apr 21 13:25:13 2022 (GMT-04:00)
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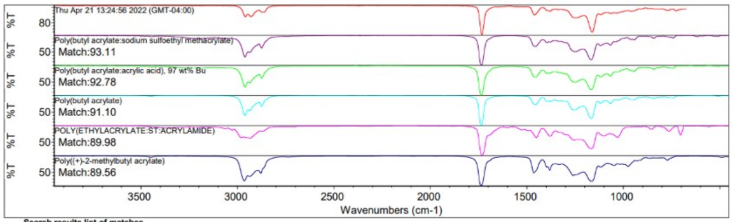
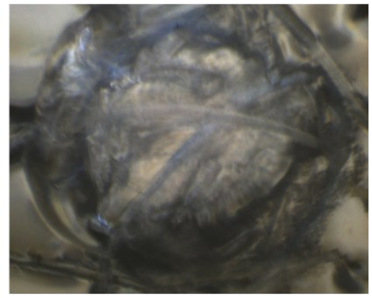
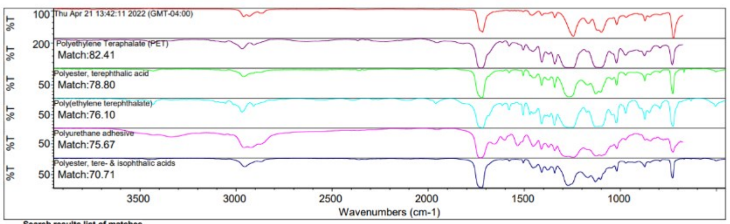


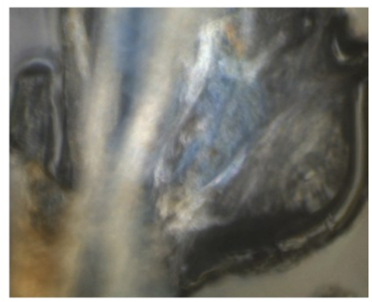


Search results list of matches

	Index	Match	Compound Name	Library Name
1	612	93.11	Poly(butyl acrylate:sodium sulfoethyl methacrylate)	HR Hummel Polymer and Additives
2	611	92.78	Poly(butyl acrylate:acrylic acid), 97 wt% Bu	HR Hummel Polymer and Additives
3	874	91.10	Poly(butyl acrylate)	HR Hummel Polymer and Additives
4	17	89.98	POLY(ETHYLACRYLATE:ST:ACRYLAMIDE)	Hummel Polymer Sample Library
5	775	89.56	Poly((+)-2-methylbutyl acrylate)	HR Hummel Polymer and Additives
6	332	88.88	Poly(ethyl acrylate)	HR Hummel Polymer and Additives
7	836	88.42	Poly(styrene/acrylate ester)	HR Nicolet Sampler Library
8	632	88.20	Poly(ethyl acrylate)	HR Nicolet Sampler Library
9	875	87.59	Poly(ethyl acrylate)	HR Hummel Polymer and Additives
10	171	87.56	Polyacrylate resin	HR Hummel Polymer and Additives

Search results for: Thu Apr 21 13:42:11 2022 (GMT-04:00)
Date: Thu Apr 21 13:42:28 2022 (GMT-04:00)
Search algorithm: Correlation
Regions searched: 3301.66-748.26





Search results list of matches

	Index	Match	Compound Name	Library Name
1	5	82.41	Polyethylene Teraphalate (PET)	Cross Sections Wizard
2	19	78.80	Polyester, terephthalic acid	HR Hummel Polymer and Additives
3	543	76.10	Poly(ethylene terephthalate)	HR Hummel Polymer and Additives
4	6	75.67	Polyurethane adhesive	Polymer Laminate Films
5	17	70.71	Polyester, tere- & isophthalic acids	HR Hummel Polymer and Additives
6	26	70.60	Polyester, tere- & isophthalic acids	HR Hummel Polymer and Additives
7	4	70.34	POLYESTER, TEREPHTHALATE	Hummel Polymer Sample Library
8	3	69.41	POLYESTER, TERE-&ISO-PHTHALATE	Hummel Polymer Sample Library
9	574	69.09	Poly(ethylene terephthalate)	HR Nicolet Sampler Library
10	29	68.24	Polyester, terephthalic acid	HR Hummel Polymer and Additives

Search results for: Thu Apr 21 13:04:40 2022 (GMT-04:00)
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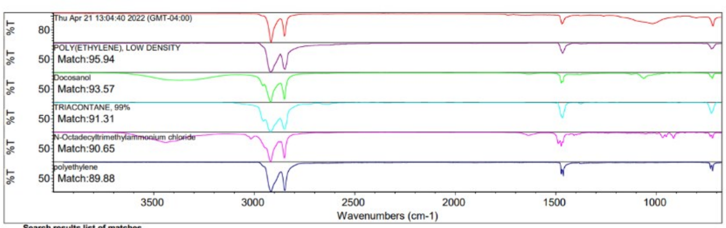

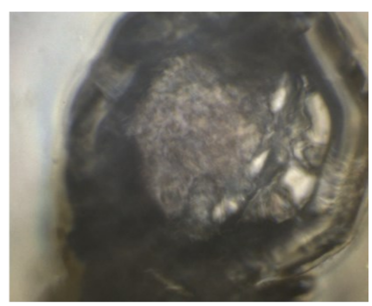

Search results list of matches

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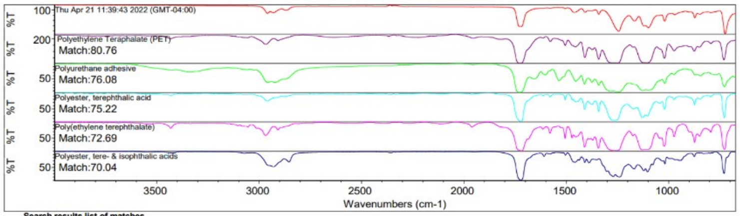
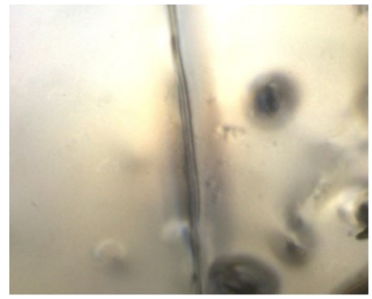
1	95	95.94	POLY(ETHYLENE), LOW DENSITY	Aldrich Condensed Phase Sample Library
2	1908	93.57	Docosanol	HR Hummel Polymer and Additives
3	1	91.31	TRIACONTANE, 99%	Aldrich Condensed Phase Sample Library
4	1911	90.65	N-Octadecyltrimethylammonium chloride	HR Hummel Polymer and Additives
5	625	89.88	polyethylene	HR Nicolet Sampler Library
6	490	89.41	Polyethylene, linear	HR Hummel Polymer and Additives
7	1912	89.10	N-Hexadecyltrimethylammonium chloride	HR Hummel Polymer and Additives
8	708	88.19	Polyethylene, LD	HR Hummel Polymer and Additives
9	821	87.69	Weston 618	HR Hummel Polymer and Additives
10	7	87.34	Polyethylene white layer (TiO2)	Polymer Laminate Films

Figure 6-6 micro-FTIR analysis of three suspected plastics from Graham Creek.

Search results for: Thu Apr 21 11:39:43 2022 (GMT-04:00)
Date: Thu Apr 21 11:40:25 2022 (GMT-04:00)
Search algorithm: Correlation
Regions searched: 3301.66-748.26

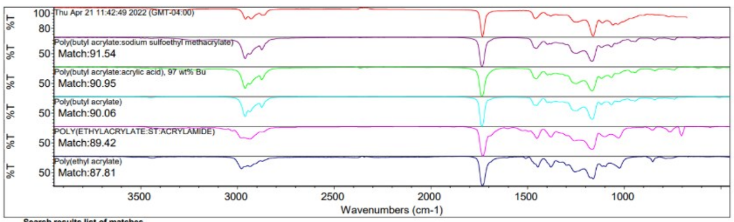


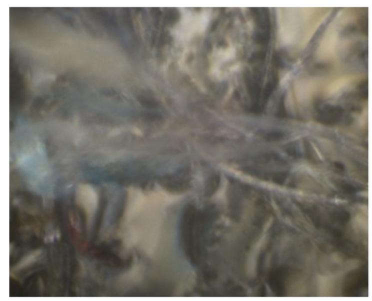


Search results list of matches

	Index	Match	Compound Name	Library Name
1	5	80.76	Polyethylene Teraphalate (PET)	Cross Sections Wizard
2	6	76.08	Polyurethane adhesive	Polymer Laminate Films
3	19	75.22	Polyester, terephthalic acid	HR Hummel Polymer and Additives
4	543	72.69	Poly(ethylene terephthalate)	HR Hummel Polymer and Additives
5	26	70.04	Polyester, tere- & isophthalic acids	HR Hummel Polymer and Additives
6	4	69.15	POLYESTER, TEREPHTHALATE	Hummel Polymer Sample Library
7	17	67.95	Polyester, tere- & isophthalic acids	HR Hummel Polymer and Additives
8	3	67.67	POLYESTER, TERE-&ISO-PHTHALATE	Hummel Polymer Sample Library
9	29	65.82	Polyester, terephthalic acid	HR Hummel Polymer and Additives
10	2	65.73	Polyurethane Adhesive	Cross Sections Wizard

Search results for: Thu Apr 21 11:42:49 2022 (GMT-04:00)
Date: Thu Apr 21 11:43:22 2022 (GMT-04:00)
Search algorithm: Correlation
Regions searched: 3301.66-748.26

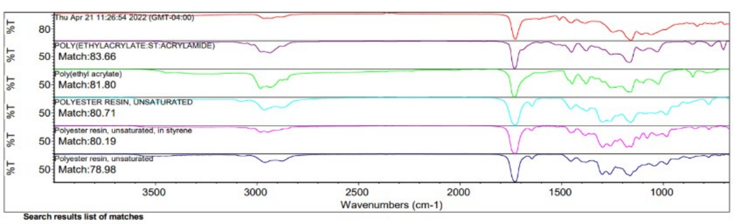


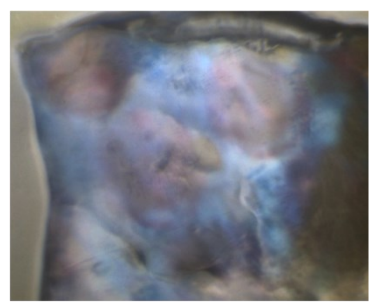


Search results list of matches

	Index	Match	Compound Name	Library Name
1	612	91.54	Poly(butyl acrylate:sodium sulfoethyl methacrylate)	HR Hummel Polymer and Additives
2	611	90.95	Poly(butyl acrylate:acrylic acid), 97 wt% Bu	HR Hummel Polymer and Additives
3	874	90.06	Poly(butyl acrylate)	HR Hummel Polymer and Additives
4	17	89.42	POLY(ETHYLACRYLATE:ST:ACRYLAMIDE)	Hummel Polymer Sample Library
5	332	87.81	Poly(ethyl acrylate)	HR Hummel Polymer and Additives
6	632	87.61	Poly(ethyl acrylate)	HR Nicolet Sampler Library
7	875	86.64	Poly(ethyl acrylate)	HR Hummel Polymer and Additives
8	775	86.09	Poly((+)-2-methylbutyl acrylate)	HR Hummel Polymer and Additives
9	836	85.45	Poly(styrene/acrylate ester)	HR Nicolet Sampler Library
10	171	85.44	Polyacrylate resin	HR Hummel Polymer and Additives

Search results for: Thu Apr 21 11:26:54 2022 (GMT-04:00)
Date: Thu Apr 21 11:29:48 2022 (GMT-04:00)
Search algorithm: Correlation
Regions searched: 3301.66-748.26





Search results list of matches

	Index	Match	Compound Name	Library Name
1	17	83.66	POLY(ETHYLACRYLATE:ST:ACRYLAMIDE)	Hummel Polymer Sample Library
2	202	81.80	Poly(ethyl acrylate)	HR Hummel Polymer and Additives
3	36	80.71	POLYESTER RESIN, UNSATURATED	Hummel Polymer Sample Library
4	459	80.19	Polyester resin, unsaturated, in styrene	HR Hummel Polymer and Additives
5	439	78.98	Polyester resin, unsaturated	HR Hummel Polymer and Additives
6	332	78.73	Poly(ethyl acrylate)	HR Hummel Polymer and Additives
7	875	78.71	Poly(ethyl acrylate)	HR Hummel Polymer and Additives
8	19	78.64	POLY(METHACRYLATE), W/OH GROUPS	Hummel Polymer Sample Library
9	169	77.58	Poly(styrene:acrylate ester)	HR Hummel Polymer and Additives
10	457	77.43	Polyester resin, unsaturated, in styrene	HR Hummel Polymer and Additives

Figure 6-7 micro-FTIR analysis of three suspected plastics from LeBreton Flats.

6.5 Discussion

The results have demonstrated how various temporal events can increase microplastic concentrations, downstream of combined sewage overflow outlets and in urban streams that receive stormwater. Snowmelt events demonstrated the highest concentrations of microplastics over all temporal events. Additionally, the combined sewage overflow event was an important point source of concentration of microplastics to the Ottawa River, especially as the LeBreton Flats location represents only one location of numerous outfalls throughout the Ottawa and Gatineau urban areas. Therefore, combined sewage overflow events are potentially a major source of microplastics to river and freshwater systems. Combined sewage overflow (CSO) inputs have previously been reported as a major conduit of microplastics to freshwater environments (Dris et al., 2018) and the current research further confirms the importance of CSO overflows as a point source. The concentrations during snowmelt events were substantially higher than non-snowmelt events, especially with a location like the Ottawa River watershed where snow melt events are throughout the watershed in spring, rather than combined sewage overflow events which are mainly confined to urban areas. However, the comparative concentration of snowmelt events to other temporal events is substantially higher, especially with a location like the Ottawa River watershed where snow melt events are throughout the watershed in spring, rather than combined sewage overflow events which are mainly confined to urban areas. Additionally, it should be noted that larger snowmelt events can also trigger combined sewage overflow events, potentially creating a much larger microplastic load event. However, as the snowmelt events were only recorded in an urban area, there are no data on the potential change in microplastic concentration from snowmelt in agricultural areas or regions of sparse population. These findings highlight more research is needed on the impact snow melt has on microplastic load to freshwater systems, especially in watersheds that receive significant winter snow.

The z-values are very close for 5 of the temporal events, demonstrating that the change in microplastic concentration during these events are linked at both locations. This demonstrates that certain temporal events have a significant influence on microplastic concentrations with these results providing useful data on how microplastic concentrations change according to a specific temporal event.

The research does highlight rain events contribute to increased microplastic concentrations, also noted in previous research in Canada (Grbić et al., 2020) in addition to being a main contributor of microplastics to water bodies (Piñon-Colin et al., 2020). Furthermore, the peak concentrations from rain events tend to occur at the onset of rainfall events with wet weather overland flow significantly increasing microplastic pollution in urban areas (Chen et al., 2020).

Both locations presented particles that were mainly fibres, consistent with previous research on Canadian rivers (Vermaire et al., 2017; Warrack et al., 2017; Forrest et al., 2019; Bujaczek et al., 2021). This could indicate the influence of wastewater discharge and/or atmospheric fallout, with these sources potentially contributing a high proportion of fibres to river environments. However, there was a change in the relative concentrations of fragments during one of the heavy rain events at Graham Creek on October 21st, 2020. During this temporal event, the dominant fragment colour was black. Many of these black fragments present in the sample particles were indicative of tire debris, however, FTIR analysis was not preformed on these particular fragments due to the potential; of interference, thus are difficult to confirm as tire or road ware particles (Grbić et al., 2020). Previous research does note tire particles as a source of microplastics to the environment (Kole et al., 2017) and as a component of stormwater particles (Ziajahromi et al., 2020). However, the PCA did indicate that black fragments were relatively high during dry periods at Graham Creek, indicating the potential for atmospheric transport of black fragments. An increase in fibres at LeBreton Flats was found to coincide with the combined sewage overflow event. Fibres have previously been noted to be the dominant type in wastewater (Gündoğdu et al., 2018; Blair et al., 2019) including samples from Canadian wastewater treatment plants (Gies et al., 2018). This can be part due to the amount of textile fibres in wastewater from laundry (Belzagui et al., 2019) entering wastewater streams. Nonetheless, Graham Creek and LeBreton flats also exhibited an increase of fibres during the primary snowmelt event, with Graham Creek exhibiting an increase during a high rainfall event. These events do not have wastewater inputs, thus, atmospheric fallout and/or atmospheric precipitation with rainfall or snow is a potential source. This can include an accumulation in the snowpack that is released in the spring during snowmelt events. Previous research has identified that atmospheric transport is an important source of microplastics, especially in urban areas (Dris et al., 2016; Cai et al., 2017; Dehghani et al., 2017) with fibres a major component of atmospheric microplastics (Dris et al., 2016, 2017; Gasperi et al., 2018). Furthermore, it represents the need to advance research on the contribution of microfibres (synthetic and anthropogenically modified) to microplastic pollution, especially in understudied compartments and geographical regions (Athey & Erdle, 2021).

The polymers identified with micro-FTIR analysis (Figure 6 and Figure 7) represent some commonly found plastics, for example polyethylene terephthalate (PET), low density polyethylene (LDPE) with other identified polymers including poly (ethyl acrylate). Some sources of the common plastics identified with micro-FTIR analysis include soda and water bottles (PET), bottle tops, garbage and plastic bags (LDPE).

Temporal events at Graham Creek sampled on dates that did not coincide with dates at LeBreton Flats involved two dry periods and another heavy rainfall event. The dry temporal events presented the two lowest particle concentrations, as there are no additional inputs from rainfall and/or overland runoff. These two events, as well as the additional dry period sampled on May 14th, 2021, is assumed to represent the normal environmental, or background particle concentrations of Graham Creek. The average concentration of the three sampled dry events is 0.46 p/m^3. To compare this background concentration to major temporal events, it is noted a heavy rainfall event in summer or fall, with strong stormwater drain outflows (October 31st, 2019, and October 21st, 2020) increases particle concentrations by approximately 50 times (from 0.46 p/m^3 to 23.21 p/m^3). Additionally, the first snowmelt event of the season increases particle concentrations by approximately 114 times (from 0.46 p/m^3 to 52.55 p/m^3) with secondary melt events increasing concentrations by approximately 12 times. Additionally, snowfall, when the creek is not frozen and flowing in early spring, presents a significant increase in particle concentration, approximately 34 times from background concentrations (from 0.46 p/m^3 to 15.82 p/m^3). This highlights that even though urbanised creeks may not have any wastewater treatment inputs, they can still be a significant point source of microplastics to the environment.

Additional events sampled at LeBreton Flats independent from Graham Creek included two supplementary samples during dry periods. As with Graham Creek, sampling during dry periods with no assumed significant temporal inputs presents a potential background concentration to compare major temporal events. The background concentration at LeBreton Flats was noted at 24 p/m^3, averaged from May 14th, June 11th and July 24th, 2021. A combined sewage overflow event potentially increased particle concentrations by seven times (from 24 p/m^3 to 167 p/m^3) with the highest increase potentially 11 times during the primary spring snowmelt (from 24 p/m^3 to 273 p/m^3). The secondary snowmelt and spring snow presents a potential increase of concentration up to four times (from 24 p/m^3 to 92 and 88 p/m^3 respectively). Additionally, rainfall events during a non combined sewage overflow event still increase concentrations by approximately double. Furthermore, it should be noted that the city of Ottawa constructed overflow tunnels in the downtown core during the sampling period. These tunnels direct active combined sewage overflow events into temporary tunnel storage and not directly into a receiving body of water, like the LeBreton Flats location. This is significant, as the rainfall event on March 26th, 2021, in the eight hours prior to sampling, demonstrated similar rainfall amounts to the sampled active combined sewage overflow event. Though the eventual rainfall amounts were higher up to two days before the active combined sewage overflow event, what would have potentially been an additional combined sewage overflow event at LeBreton Flats on March 26th, 2021, was possibly avoided by the storage tunnels.

As climate trends continue to warm and urban populations continue to grow, the impact on urban river systems will intensify as summer baseline flows decrease and wastewater flows increase (Woodward et al., 2021). Additionally, the impact of snowmelt in urban areas necessitates additional research into microplastic release during these temporal events.

6.6 Conclusions

A major river downstream of a combined sewage overflow outlet and an urban creek were sampled under various temporal indicators that were suspected to contribute to a change in microplastic concentrations. An urban creek receiving stormwater inputs demonstrated an increase of suspected microplastics up to 50 times during heavy rain events and up to 34 times during spring snow. The first snowmelt event of the spring contributed to a particle increase of up to 114 times, with a secondary melt event demonstrating an increase of 12 times. For the Ottawa River, a location directly downstream of a combined sewage overflow outlet demonstrated a particle increase of up to seven times during an active combined sewage overflow event after heavy rainfall. During a rainfall event without an active combined sewage overflow, the concentration doubled, possibly suggesting recent storage tunnels built in Ottawa to manage combined sewage overflow events are reducing particle concentrations by up to two-thirds. During the primary snowmelt of spring, concentrations were recorded at approximately 11 times the normal concentration.

These results demonstrate that snowmelt events are the most significant event in terms of increased concentration of microplastics in urban rivers and streams. However, combined sewage overflow events are contributing the greatest load of microplastics to urban rivers, as demonstrated in the Ottawa/Gatineau urban area, due to the numerous combined sewage overflow locations and frequent discharge events. This research establishes further temporal analysis would be beneficial to freshwater microplastic research as these results indicate temporal events produce significant microplastic loads to freshwater environment

7. Summary, implications and future work

7.1 Introduction

This thesis has examined microplastic pollution in the Ottawa River watershed, improving the understanding of the spatial and temporal extent of microplastics within this freshwater environment. Furthermore, the thesis has evaluated citizen science as a tool to assist and potentially improve spatial monitoring for microplastics in freshwater systems.

Two citizen science projects examined how citizen scientists can assist in monitoring for microplastics in river water and dry shoreline river sediments. Furthermore, microplastic concentration was examined over a watershed scale to determine if there are any significant spatial factors to consider with microplastic concentrations in river water. Finally, microplastic concentrations were measured during various temporal events to determine flux, and how this may contribute to microplastic loads to river environments. These temporal events included heavy rainfall that trigger stormwater flow and combined sewage overflow events, snowfall and snowmelt events.

7.2 Summary of findings, significance, and directions for future research

7.2.1 Citizen science as a tool in microplastic sampling

One of the main objectives of the thesis was to evaluate the role of citizen science in spatial monitoring for microplastics in freshwater environments. For monitoring microplastics in watersheds, citizen scientists provided the added advantage of increasing the spatial scale of monitoring, whereby a research team would have to spend added time and resources to cover the same sampling area. Furthermore, citizen scientists who sample the river where they work and/or play are typically more engaged in the process as they are concerned about pollution at their river location and are eager to assist and learn from the citizen science process. Engaging an established volunteer network through programs like the Riverwatchers in the Ottawa River watershed enables a more streamlined recruitment process, while providing a platform to disseminate findings. Subsequent sampling or monitoring projects can also benefit from utilising established volunteer networks as it can encourage additional participants. This was evident in the second citizen science project in this thesis, where participation increased.

Nonetheless, it is essential that project design and implementation is carefully planned, as the scientific integrity of citizen science projects can be questioned. This is where citizen science microplastic projects can have the added benefit of ensuring sample processing is conducted by the research team in the laboratory. This can ensure that the research team can follow strict quality assurance and quality control guidelines. However, this does pose the question whether this deems the volunteers as citizen samplers, rather than citizen scientists.

Project design should present a methodology that allows for ease of sampling for the citizen scientist, so not to overburden or discourage the volunteer while collecting samples. This can be difficult where higher volume samples may be required. For example, grab samples as utilised in the project described in this thesis may not be conducive to high volume samples. Additionally, large volumes of sediment collected by volunteers for analysis could also be impractical. Furthermore, microplastic counts in dry sediments were found to be low along the Ottawa River, thus, sampling higher volumes of sediment would be advantageous. However, this could pose fiscal difficulties when trying to sample high water and sediment volumes. To sample more water and/or sediment, more expensive sampling and processing equipment may be needed to achieve the desired volumes to uphold the scientific integrity and results of citizen science projects in large watersheds. Therefore, cost is potentially a major hurdle for some microplastic citizen science projects.

This thesis has demonstrated citizen science is an effective tool for microplastic sampling in freshwater environments as it extents the spatial scope of monitoring. Future citizen science projects should carefully consider project design and implementation to uphold scientific integrity. This includes the research team who should follow strict quality assurance and quality control protocols in the laboratory while processing samples. Establishing a successful citizen science network also has the added advantage of incorporating a temporal aspect to analysis, potentially creating the opportunity to continually monitor microplastic concentrations in watersheds through continued citizen science collaboration. Citizen science is a useful tool and should be considered more often for microplastic research.

However, the citizen science projects presented in this thesis only focus on the citizens or community members doing the sampling. Citizen science within the field of freshwater plastic pollution still remains niche, with the majority of projects only following a contributory model of participation. Citizens can and should play an important role in data collection, processing and developing toolkits and contribute to catchment scale monitoring, that is important for the health and resilience for freshwater ecosystems (Cook et al., 2021). Nonetheless, this is currently difficult in microplastic citizen science projects, as strict QA/QC protocols generally need to be controlled by the research team. However, water resource monitoring has seen the emergence of new low-cost sensing equipment creating opportunities for citizen science monitoring in freshwater environments (Buytaert et al., 2014; Baalbaki et al., 2019), and this could be an important step for citizen science in microplastic monitoring, the development of tools where volunteers can engage in more of the research process, including sample processing, data interpretation and dissemination.

7.2.2 Spatial distribution of microplastics in river water throughout the Ottawa River watershed

This thesis has demonstrated that in the Ottawa River watershed, the only two significant spatial variables when compared to microplastic concentration were the increase of microplastic concentration as you move downstream of the main channel and tributaries and an increase of microplastics at boat launch locations. However, these were only weak relationships, and it highlights the potential for additional influences on the spatial distribution of microplastics in the watershed. Factors that could contribute to the weaker relationship include (but not limited too) temporal influences, numerous sinks, potential outputs and various source points (not examined in the ANCOVA analysis) throughout the length of the main channel and tributaries.

The spatial research presented in this thesis has demonstrated it may only be one important element to consider for microplastic pollution in large watersheds. The research has underlined other temporal factors (further discussed in the preceding section) need to be considered in combination with spatial factors in future research. The other important factor that could be concluded from the spatial analysis is the role sinks play in freshwater environments. If sinks did not play an important role, one might expect a consistent accumulation of microplastics as rivers flow through watersheds. As the research has demonstrated, spatially, there is not a distinctive accumulation of microplastics from the headwaters to the terminus of the Ottawa River. Previous research has alluded to this, with suggestions a large proportion of plastic waste entering the environment does not make it to the world's oceans, potentially more than 98% of plastics leaked into the environment are retained (Meijer et al., 2021) and rivers are in fact plastic reservoirs (van Emmerik et al., 2022). Though these suggestions have focused on macroplastics, microplastics could indeed follow the same pattern, thus, more research in microplastic sinks in freshwater systems is imperative.

7.2.3 Temporal flux of microplastics during precipitation and snowmelt events

The thesis also examined how major temporal events can contribute to a change in microplastic concentrations to river environments. Active CSO events were identified as a major contributor to microplastic load to river environments. In the Ottawa/Gatineau urban area there are numerous overflow locations that are triggered multiple times a year, and the extra microplastic concentration downstream of these locations during an active event contribute a very high load to the Ottawa River. Additionally, snowmelt events exhibit very high concentrations of microplastics. The first snowmelt event of the spring typically exhibits the highest concentration of microplastics compared to other temporal events, however, it is only a one-off annual event. Nonetheless, secondary snow melt events also contributed to very high microplastic concentrations compared to baseline or ambient river conditions. This demonstrates that snow on the ground throughout the winter in Canadian cities is a major temporary sink of microplastics for freshwater environments. Previous research has acknowledged the potential of snowmelt and that it ‘could’ greatly increase microplastic loads (Kapp & Yeatman, 2018) in streams and rivers (Baldwin et al., 2016). However, this research presented a larger more focused temporal analysis, by comparing snowmelt directly to other precipitation events and drier ambient conditions. Therefore, the research concludes snow and snowmelt are potentially one of the more significant sources and sinks of microplastics in urban areas.

Stormwater is another major temporal contributor of microplastics to river environments, especially during heavy rainfall events in urban areas where sewage systems may be separated. In these situations, stormwater is untreated and conveyed directly to river environments. This thesis has demonstrated that these shorter-term temporal events greatly increase microplastic concentration and thus microplastic load in urban streams and rivers. Previous research in Canadian urban centres has acknowledged stormwater is a major contributor of plastics to freshwater (Grbić et al., 2020), however, the current research has compared multiple precipitation events against baseline or ambient conditions to give insight on how each of these compare to each other in terms of microplastic concentrations.

This thesis has demonstrated that snow is potentially a major temporary sink of microplastics in freshwater environments, especially in urban locations. More research is essential to further establish the importance of snowmelt and snowfall in the accumulation and release of microplastics to freshwater environments. Accumulated snow in urban areas is potentially an important factor that has not been considered in any detail in previous microplastic research. This is especially important in Canada where major cities experience snow throughout winter months. Furthermore, the research has further confirmed that stormwater and CSO events are important point sources of microplastics to river environments.

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9. Supplementary figures and tables

Supplementary table 1

Sample Location Name	Concentration	Particle Breakdown	% Fragments	%Fibres	Urban?	Boat Launch?
Ottawa						
Laval	0.260	Fragments: 4 blue, 1 green, 1 black. Fibres: 3 blue, 1 black.	0.60	0.40	Y	
Oka	0.119	Fragments: 0. Fibres: 4 blue.	0.00	1.00	Y	
Voyager	0.149	Fragments: 0. Fibre: 10 blue.	0.00	1.00		
Hawkesbury	0.591	Fragments: 1 white, 2 black, 4 grey, 6 yellow, 6 blue, 3 green, 1 red, 1 silver. Fibres: 3 blue, 1 white, 1 black.	0.77	0.16	Y	
L'Oringal	0.243	Fragments: 2 pink, 1 red, 1 Green, 1 black. Fibres: 2 blue, 7 black, 1 green.	0.33	0.67		
Montebello	0.441	Fragments: 3 white, 1 green, 1 blue, 1 black. Fibres: 2 green, 1 red bundle.	0.67	0.33		
Clarence	0.172	Fragments: 1 blue, 1 grey. Fibres: 4 blue.	0.33	0.67		
Petrie Island	4.955	Fragments: 5 blue, 2 white. Fibres: 148 blue, 22 black, 32 red, 22 green, 1 yellow, 1 purple.	0.03	0.97	Y	
Blair Road	1.752	Fragments: 2 white, 2 Blue. Fibres: 34 blue, 10 black, 1 green, 3 red.	0.08	0.92	Y	Y
Le Breton	0.429	Fragments: 2 green, 6 blue, 6 white, 1 red, 1 yellow. Fibres: 12 black, 21 blue, 6 white, 2 green, 4 red.	0.25	0.75	Y	
Limieux Island	0.620	Fragments: 2 black. Fibres: 9 blue, 1 black, 1 white.	0.15	0.85	Y	
Bate Is.	0.763	Fragments: 1 blue. Fibres: 21 blue, 9 black, 4 red.	0.03	0.97	Y	
Deschene	0.343	Fragments: 0. Fibres: 5 blue, 3 black.	0.00	1.00	Y	
Brittania	0.530	Fragments: 2 blue. Fibres: 5 blue, 3 black, 2 red	0.17	0.83	Y	
Shirleys Bay	0.937	Fragments: 2 green, 1 blue, 2 red, 1 orange .Fibres: 28 blue, 18 black, 1 red.	0.11	0.89		

Constance bay	0.210	Fragments: 1 blue. Fibres: 7 blue, 2 black.	0.10	0.90		
Quyon	1.020	Fragments: 23 black, 3 blue, 1 green, 1 red. Fibres: 4 black, 4 blue, 1 green.	0.76	0.24		Y
Robert Simpson	0.613	Fragments: 1 black. Fibres: 16 blue, 2 green, 2 red, 4 black.	0.04	0.96	Y	
Norway Bay	0.385	Fragments: 5 blue, 2 black. Fibres: 1 red, 3 blue 4 black.	0.47	0.53		
Portage du Fort	0.319	Fragments: 1 black, 1 blue, 2 red. Fibres: 4 blue, 1 green, 1 black, 1 red.	0.36	0.64		
George Matherson	0.301	Fragments: 2 black, 2 blue. Fibres: 2 blue, 3 black.	0.44	0.56		
Pembroke Waterfront	0.447	Fragments: 2 black. Fibres: 12 blue, 2 black.	0.13	0.88	Y	
Pembroke Riverside	0.092	Fragments: 0. Fibres: 2 blue, 1 green	0.00	1.00	Y	
Petawawa Point	0.245	Fragment: 0. Fibres: 7 blue, 1 black.	0.00	1.00	Y	
Deep River	0.772	Fragments: 3 blue, 1 pink, 1 red. Fibres: 8 blue, 5 black, 1 red, 1 orange, 1 pink.	0.24	0.76		
Dumoine	0.461	Fragments: 0. Fibres: 24 Blue, 4 red, 1 yellow, 1 black	0.00	1.00		
Deux-Rivieres	1.037	Fragments: 8 black. Fibres: 24 blue, 8 black.	0.20	0.80		
Klock	0.320	Fragments: 2 red, 2 white. Fibres: 5 blue, 1 black.	0.40	0.60		
Mattawa	0.309	Fragments: 1 black. Fibres: 3 blue, 3 red.	0.14	0.86	Y	
Temiscaming	0.894	Fragments: 3 white, 1 blue. Fibres: 2 green, 19 blue, 1 red, 2 black	0.14	0.86		
Opemican	0.478	Fragments: 0. Fibres: 40 blue, 1 red, 2 purple, 3 black	0.00	1.00		
Vieux Fort	0.361	Fragments: 1 blue, 1 green. Fibres: 13 blue, 4 black.	0.11	0.89		
De ille	0.469	Fragments: 1 red. Fibres: 18 blue, 4 black.	0.04	0.96		Y
Devils Rock	0.071	Fragments: 0. Fibres: 3 blue, 2 black	0.00	1.00		

Haileybury	0.251	Fragments: 1 blue. Fibres: 5 blue, 6 black.	0.08	0.92	Y	Y
New Liskeard	0.783	Fragments: 3 black, 4 blue, 1 green. Fibres: 11 black, 8 blue, 1 white, 2 red.	0.26	0.71	Y	
Waterfront Inn	0.184	Fragments: 2 green, 2 red, 2 blue, 1 white. Fibres: 3 blue, 2 black.	0.58	0.42	Y	
Notre Dame	0.460	Fragments: 2 black. Fibres: 2 blue, 5 black, 1 red.	0.20	0.80	Y	
Angliers	0.334	Fragments: 2 white. Fibres: 24 blue, 2 green, 3 red	0.06	0.94		
Remingy	0.522	Fragments: 0. Fibres: 27 blue, 7 black	0.00	1.00		
Forester	0.180	Fragments: 1 red, 1 black, 1 blue. Fibres: 1 blue, 2 black.	0.50	0.50		
St. Agnes	0.939	Fragments: 10 blue, 1 green. Fibres: 3 blue, 1 red, 7 black.	0.50	0.50		
Rapide Sept	0.276	Fragments: 1 blue. Fibres: 3 blue, 1 black, 1 red.	0.17	0.83		
Twin Rapids	0.358	Fragments: 0. Fibres: 4 red, 3 green, 21 blue	0.00	1.00		
Kinojevis	0.159	Fragments: 0. Fibres: 20 blue, 1 green, 1 red, 1 black	0.00	1.00		
Picnic	0.460	Fragments: 3 blue, 1 red, 1 white, 3 black. Fibre: 3 blue, 6 black, 1 green.	0.44	0.56		Y
Whiskey Creek	0.117	Fragments: 1 blue. Fibres: 1 black, 1 blue.	0.33	0.67		
Verendrye 30	0.119	Fragments: 0. Fibres: 2 blue, 1 green	0.00	1.00		
Camatose	0.531	Fragments: 1 blue. Fibres: 10 blue, 1 black, 1 red.	0.08	0.92		
Nord						
St. Andrews	0.311	Fragments: 2 green, 3 blue, 8 white. Fibres: 6 green, 11 blue, 1 black, 1 blue	0.41	0.59	Y	
Lachute	0.095	Fragments: 1 blue. Fibres: 1 black.	0.50	0.50		

Piedmont	0.254	Fragments : 1 blue, 1 green, blue bundle. Fibres: 1 blue, 1 green, 2 black, 1 red.	0.25	0.75		
Val David	0.111	Fragments 1 blue, 2 white. Fibres: 2 blue, 2 black.	0.43	0.57		
Rouge						
Highway 50	3.640	Fragments: 5 white, 1 red, 5 blue. Fibres: 156 blue, 137 black, 21 red, 10 white 2 green	0.03	0.97		
Hubredeau	0.222	Fragments: 0. Fibres: 4 blue, 6 green, 3 black, 1 purple.	0.00	1.00		
Tremblant	0.276	Fragments: 3 white, 2 black, 2 green. Fibres: 2 blue, 3 black.	0.58	0.42	Y	
Labelle	0.080	Fragments: 0. Fibres: 2 blue	0.00	1.00		
Riviere-Rouge	0.107	Fragments: 2 black, 1 white, 2 red. Fibres: 2 black, 1 blue, 1 white.	0.56	0.44		
South Nation						
Plantagenet	0.843	Fragments: 4 orange, 3 white. Fibres: 37 blue, 7 black, 4 green, 2 red	0.02	0.98		
Casselman	0.153	Fragments: 1 blue. Fibres: 6 blue, 1 black.	0.13	0.88	Y	
Crysler	1.047	Fragments: 7 black, 1 white, 4 blue. Fibres: 17 blue, 4 red, 1 pink, 1 green, 6 black.	0.29	0.71	Y	Y
Chesterville	0.052	Fragments: 1 black, 1 white. Fibres: 2 green.	0.50	0.50	Y	
Cass Bridge	0.483	Fragments: 8 black, 2 blue, 2 red, 1 green. Fibres: 4 blue, 1 green.	0.72	0.28		Y
Lièvre						
Buckingham	0.233	Fragments: 3 white, 2 gold, 1 blue. Fibres: 31 blue, 1 green, 4 black	0.12	0.88	Y	

Notre Dame du Salette	0.715	Fragments: 4 black, 1 red, 1 orange, 1 green. Fibres: 5 blue. 3 black	0.47	0.53		
Val des Bois	0.781	Fragments: 6 black, 1 red, 2 white, 2 blue. Fibres: 5 black, 1 blue.	0.65	0.35		Y
Notre Dame du Laus	0.543	Fragments: 6 black, 2 blue, 1 green. Fibres: 8 blue, 1 black, 1 blue bundle.	0.47	0.53		Y
Mont Laurier	0.414	Fragments: 6 black, 2 white. Fibres: 1 blue.	0.89	0.11		
Gatineau						
Fournier	1.316	Fragments: 3 blue, 1 white, 1 orange. Fibres: 33 blue, 7 black, 2 green, 2 red.	0.10	0.90	Y	
Wakefield	0.095	Fragments: 7 red, 1 blue, 1 silver. Fibres: 3 blue.	0.75	0.25	Y	
Lac St. Marie	0.116	Fragments: 1 blue. Fibres: 2 blue.	0.33	0.67		
Baskatong	0.102	Fragments: 0. Fibres: 3 blue, 1 black	0.00	1.00		
Lac Rapide	0.495	Fragments: 2 blue, 3 black. Fibres: 13 blue, 1 black.	0.26	0.74		Y
Rideau						
Vanier	0.958	Fragments: 16 white, 1 green, 1 red. Fibres: 35 blue, 18 green	0.24	0.76	Y	
Carleton University	0.181	Fragments: 2 white, 3 blue, 1 black. Fibres: 7 blue, 1 green, 1 red, 3 black, 1 white.	0.32	0.68	Y	
Jocks Landing	0.236	Fragments: 1 white. Fibres: 7 blue, 1 black.	0.11	0.89	Y	Y
Manotick	0.414	Fragments: 1 blue 1 black. Fibres: 4 red, 22 blue, 2 green, 10 black.	0.05	0.95	Y	
Burritts Rapids	0.210	Fragments: 1 black, 1 blue, 1 red. Fibres: 4 blue, 1 red.	0.38	0.63	Y	
Smiths Falls	0.107	Fragments: 1 blue. Fibres: 7 blue, 1 black.	0.11	0.89	Y	

Mississippi						
Galetta	0.343	Fragments: 12 blue, 3 red 2 white. Fibres: 24 blue, 9 black	0.24	0.76		
Packenham	1.150	Fragments: 3 blue, 1 white, 1 orange, 2 black. Fibres: 43 blue, 10 black, 6 red, 1 green.	0.10	0.90		
Blackney	0.167	Fragments: 1 black. Fibres: 6 blue.	0.14	0.86		
Appleton	0.521	Fragments: 1 red, 5 blue, 2 black, 1 white. Fibres: 5 blue, 3 green.	0.53	0.47		Y
Carleton Place	0.268	Fragments: 3 white, 2 blue, 1 black. Fibres: 8 blue, 5 black, 1 green.	0.30	0.70	Y	
Lanark	0.060	Fragments: 1 black.Fibres: 3 blue, 1 black.	0.20	0.80		
Madawaska						
Arnprior	0.404	Fragments: 2 black. Fibres: 5 blue, 1 black, 2 green, 1 red.	0.18	0.82	Y	
Highway 417	1.062	Fragments: 15 black. Fibres: 6 blue, 3 black, 1 red, 1 green.	0.58	0.42		Y
Burnstown	0.424	Fragments: 1 silver, 1 red. Fibres: 4 blue, 1 red, 2 black, 1 white.	0.20	0.80		
Combermere	0.722	Fragments: 4 blue, 1 pink, 2 red, 3 white, 3 grey, 1 black. Fibres: 4 black.	0.78	0.22		
Madawaska Town	0.184	Fragments: 0. Fibres: 4 blue	0.00	1.00		
Bonnechere						
Horton	0.765	Fragments: 2 white, 4 blue, 1 green, 12 black. Fibres: 4 black, 3 blue.	0.73	0.27		Y
Renfrew	0.885	Fragments: 7 white, 5 red, 1 yellow, 1 blue. Fibres: 2 red, 14 blue, 2 black, 4 green	0.39	0.61	Y	

Bonnechere Caves	0.204	Fragments: 1 white. Fibres: 5 blue, 2 black.	0.13	0.88	
Golden Lake	0.515	Fragments: 0. Fibres: 9 blue, 2 green, 2 black, 1 red.	0.00	1.00	
Round Lake	0.618	Fragments: 16 blue, 1 green, 1 red. Fibres: 1 blue, 1 red, 1 green.	0.86	0.14	
Montreal					
Latchford	0.157	Fragments: 1 green, 1 red, 2 blue. Fibres: 6 blue, 2 white, 1 green, 1 red.	0.29	0.71	
Elk Lake	0.050	Fragments: 1 white, 1 red. Fibres: 0	1.00	0.00	
Matachewan	3.178	Fragments: 2 white. Fibres: 33 green, 86 black, 78 blue, 4 red	0.01	0.99	
Mistinikon Lake	0.554	Fragments: 0. Fibres: 15 blue, 5 black, 1 purple, 1 red	0.29	0.71	
Gowganda	0.343	Fragments: 1 pink. Fibres: 11 blue, 1 red, 4 black.	0.06	0.94	
Blanche					
Highway 65	0.397	Fragments: 0. Fibre: 1 red. Frag: 1 white, 7 red	0.89	0.11	
Hillard	0.386	Fragments: 1 black. Fibre: 6 blue, 1 black.	0.13	0.88	
Round Lake	0.618	Fragments: 16 blue, 1 green, 1 red. Fibres: 1 blue, 1 red, 1 green.	0.86	0.14	
Swastika	0.400	Fragments: 4 white. Fibres: 24 blue, 5 green, 3 red, 9 black	0.09	0.91	Y

Supplementary table 2

River	Sample Location	Rain day of (mm)	Rain 2 days previous (mm)	Total (mm)
Ottawa	Laval	0	0	0
Ottawa	Oka	8	2	10
Ottawa	Voyager	6	0	6
Ottawa	Hawkesbury	3	22.7	25.7
Ottawa	L'Orignal	8	2	10
Ottawa	Montebello	0	0	0
Ottawa	Clarence	0	0	0
Ottawa	Petrie Island	0.8	5.4	6.2
Ottawa	Blair Road	0.8	5.4	6.2
Ottawa	Bate Is.	0	0.4	0.4
Ottawa	Limieux Island	0	0	0
Ottawa	Le Breton	0	0	0
Ottawa	Dechenes	0	3.6	3.6
Ottawa	Britannia	0.2	5.4	5.6
Ottawa	Shirleys Bay	0	0.4	0.4
Ottawa	Constance bay	0	0	0
Ottawa	Robert Simpson	2.1	2.1	4.2
Ottawa	Quyon	1.2	0.2	1.4
Ottawa	Norway Bay	1.2	0.2	1.4
Ottawa	Portage du Fort	1.2	0.2	1.4
Ottawa	George Matherson	0.2	0	0.2
Ottawa	Pembroke Waterfront	0.2	0	0.2
Ottawa	Pembroke Riverside	0	4	4
Ottawa	Petawawa Point	0	4	4
Ottawa	Deep River	0	4	4
Ottawa	Dumonie	0	7.5	7.5
Ottawa	Deux-Rivieres	3.6	0.4	4
Ottawa	Klock	3.6	0.4	4
Ottawa	Mattawa	0	0	0
Ottawa	Temiscaming	0	0.6	0.6
Ottawa	Opemican	0.8	11.5	12.3
Ottawa	Vieux Fort	0.8	11.5	12.3
Ottawa	du Ille	0.8	11.5	12.3
Ottawa	Devils Rock	0.1	11.4	11.5
Ottawa	Haileybury	0.1	11.4	11.5
Ottawa	New Liskeard	0.1	11.4	11.5
Ottawa	Waterfront Inn	11.4	0.7	12.1

Ottawa	Notre Dame	0	1.7	1.7
Ottawa	Angliers	0	1.8	1.8
Ottawa	Remingy	0	1.8	1.8
Ottawa	St-Agnes	0	6.4	6.4
Ottawa	Forester	0	6.4	6.4
Ottawa	Rapides Sept	0	0	0
Ottawa	Twin Rapids	0	4.9	4.9
Ottawa	Kinojevis	0	4.9	4.9
Ottawa	Picnic	0	0	0
Ottawa	Whiskey Creek	4.4	6.2	10.6
Ottawa	Verendrye 30	0	0	0
Ottawa	Camatose	0	0	0
Mississippi	Galetta	0	10.4	10.4
Mississippi	Packenham	0	3.4	3.4
Mississippi	Blackney	2.1	2.1	4.2
Mississippi	Appleton	0	2.3	2.3
Mississippi	Carleton Place	0	0	0
Mississippi	Lanark	0	0	0
Rideau	Vanier	1.3	3.1	4.4
Rideau	Carleton University	0	0	0
Rideau	Jock Landing	0	0	0
Rideau	Manotick	0.2	0.2	0.4
Rideau	Burritts Rapids	0	2.3	2.3
Rideau	Smiths Falls	0	0	0
Nord	St. Andrews	0	4.4	4.4
Nord	Lachute	0	1.3	1.3
Nord	Piedmont	0.2	32.4	32.6
Nord	Val David	0.2	32.4	32.6
Rouge	Highway 50	0	2.8	2.8
Rouge	Hubredeau	0	12	12
Rouge	Tremblant	0	0	0
Rouge	Labelle	0	0	0
Rouge	Riviere-Rouge	5.9	0	5.9
Lièvre	Mont Laurier	5.9	0	5.9
Lièvre	Notre Dame du Laus	36.6	0	36.6
Lièvre	Val des Bois	0	5.9	5.9
Lièvre	Notre Dame du Salette	36.6	0	36.6
Lièvre	Buckingham	0	2.8	2.8
South Nation	Plantagenet	0	14.8	14.8
South Nation	Casselman	2.7	0	2.7
South Nation	Crysler	2	25.8	27.8
South Nation	Chesterville	0	0	0

South Nation	Cass Bridge	2	25.8	27.8
Gatineau	Fournier	0	0.6	0.6
Gatineau	Wakefield	2.3	2.9	5.2
Gatineau	Lac St. Marie	1.4	11.2	12.6
Gatineau	Baskatong	18.6	0	18.6
Gatineau	Lac Rapide	2.2	4.3	6.5
Gatineau	Lac Rolland	2.2	4.3	6.5
Madawaska	Arnprior	2.1	2.1	4.2
Madawaska	Highway 417	2.1	2.1	4.2
Madawaska	Burnstown	0	6.6	6.6
Madawaska	Combermere	0	6.6	6.6
Madawaska	Madawaska Town	2.5	6.6	9.1
Bonnechere	Horton	2.1	2.1	4.2
Bonnechere	Renfrew	0	0.1	0.1
Bonnechere	Bonnechere Caves	0	0	0
Bonnechere	Golden Lake	9.1	2.1	11.2
Bonnechere	Round Lake	9.1	2.1	11.2
Montreal	Latchford	0	0	0
Montreal	Elk Lake	0	1.7	1.7
Montreal	Matachewan	1.8	0.6	2.4
Montreal	Mistinikon Lake	10.4	14.3	24.7
Montreal	Gowganda	10.4	14.3	24.7
Blanche	Highway 65	0	1.7	1.7
Blanche	Hillard	0	1.7	1.7
Blanche	Round Lake	10.4	14.3	24.7
Blanche	Swastika	1.8	0.6	2.4

Supplementary table 3

River	Location Name	Latitude	Longitude	Date of Sample	Drag	Stationary
Ottawa	**Laval**	45.535539	-73.878571	27th August 2020		ST
Ottawa	**Oka**	45.462367	-74.043337	June 30th 2021	DR	
Ottawa	**Voyager**	45.562930	-74.427169	June 30th 2021	DR	
Ottawa	**Hawkesbury**	45.616246	-74.599431	26th August 2020		ST
Ottawa	**L'Orignal**	45.627056	-74.685791	June 30th 2021	DR	
Ottawa	**Montebello**	45.647692	-74.933706	July 5th 2021	DR	
Ottawa	**Clarence**	45.579781	-75.247135	23rd July 2020		ST
Ottawa	**Petrie Island**	45.501931	-75.486241	1st November 2019		ST
Ottawa	**Blair Road**	45.463479	-75.625584	1st November 2019		ST
Ottawa	**Bate Is.**	45.410070	-75.756495	9th September 2019		ST
Ottawa	**Limieux Island**	45.413578	-75.728153	11th June 2021		ST
Ottawa	**Le Breton**	45.415747	-75.71115	5th July 2019		ST
Ottawa	**Dechenes**	45.380485	-75.800281	24th July 2020		ST
Ottawa	**Britannia**	45.374682	-75.796943	9th August 2019		ST
Ottawa	**Shirleys Bay**	45.366316	-75.883103	24th April 2021	DR	
Ottawa	**Constance bay**	45.499067	-76.075456	17th may 2021	DR	
Ottawa	**Robert Simpson**	45.443968	-76.350125	10th May 2021	DR	
Ottawa	**Quyon**	45.516170	-76.227960	24th June 2021	DR	
Ottawa	**Norway Bay**	45.517660	-76.427590	24th June 2021	DR	
Ottawa	**Portage du Fort**	45.592710	-76.669020	24th June 2021	DR	
Ottawa	**George Matherson**	45.797460	-77.019830	24th June 2021	DR	
Ottawa	**Pembroke Waterfront**	45.828580	-77.118230	24th June 2021	DR	
Ottawa	**Pembroke Riverside**	45.830559	-77.131353	6th September 2020	DR	
Ottawa	**Petawawa Point**	45.910203	-77.251240	6th September 2020	DR	
Ottawa	**Deep River**	46.103299	-77.479663	6th September 2020	DR	

Ottawa	**Dumonie**	46.200720	-77.684856	22nd August 2019		ST
Ottawa	**Deux-Rivieres**	46.253129	-78.287160	1st August 2021	DR	
Ottawa	**Klock**	46.291888	-78.492496	1st August 2021	DR	
Ottawa	**Mattawa**	46.320824	-78.708845	8th August 2020	DR	
Ottawa	**Temiscaming**	46.716554	-79.103260	2nd October 2019		ST
Ottawa	**Opemican**	46.831084	-79.192127	31st July 2021	DR	
Ottawa	**Vieux Fort**	47.290116	-79.455288	31st July 2021	DR	
Ottawa	**du Ille**	47.376659	-79.481628	31st July 2021	DR	
Ottawa	**Devils Rock**	47.416871	-79.602835	30th July 2021	DR	
Ottawa	**Haileybury**	47.447475	-79.630012	30th July 2021	DR	
Ottawa	**New Liskeard**	47.499735	-79.677191	30th July 2021	DR	
Ottawa	**Waterfront Inn**	47.506272	-79.669676	29th July 2021	DR	
Ottawa	**Notre Dame**	47.591865	-79.483678	6th August 2020	DR	
Ottawa	**Angliers**	47.553029	-79.238848	4th October 2019		ST
Ottawa	**Remingy**	47.760921	-79.210047	4th October 2019		ST
Ottawa	**St-Agnes**	48.098476	-78.865923	July 28th 2021	DR	
Ottawa	**Forester**	48.195226	-78.873976	July 28th 2021	DR	
Ottawa	**Rapides Sept**	47.768981	-78.307175	25th September 2020	DR	
Ottawa	**Twin Rapids**	47.717097	-77.787497	5th October 2019		ST
Ottawa	**Kinojevis**	48.408685	-78.366249	4th October 2019		ST
Ottawa	**Picnic**	47.539629	-77.139233	24th September 2020	DR	
Ottawa	**Whiskey Creek**	47.375665	-76.972991	27th July 2021	DR	
Ottawa	**Verendrye 30**	47.320592	-76.938238	26th September 2020		ST
Ottawa	**Camatose**	47.306244	-76.859017	24th September 2020	DR	
Mississippi	**Galetta**	45.420939	-76.250285	4th July 2019		ST
Mississippi	**Packenham**	45.335875	-76.287205	7th June 2020		ST
Mississippi	**Blackney**	45.267436	-76.250182	10thMay 2021		ST
Mississippi	**Appleton**	45.183250	-76.124980	12th May 2021	DR	

Mississippi	**Carleton Place**	45.142514	-76.144164	14th June 2020		ST
Mississippi	**Lanark**	44.971065	-76.407559	14th June 2020		ST
Rideau	**Vanier**	45.426163	-75.668898	11th September 2019		ST
Rideau	**Carleton University**	45.381593	-75.696540	24th July 2020		ST
Rideau	**Jock Landing**	45.260473	-75.707814	18th may 2021	DR	
Rideau	**Manotick**	45.245584	-75.702946	26th June 2020		ST
Rideau	**Burritts Rapids**	44.982070	-75.794900	12th May 2021	DR	
Rideau	**Smiths Falls**	44.896684	-76.025728	14th June 2020		ST
Nord	**St. Andrews**	45.566649	-74.335436	21st July 2019		ST
Nord	**Lachute**	45.662821	-74.333784	July 5th 2021	DR	
Nord	**Piedmont**	45.895515	-74.115754	25th August 2020	DR	
Nord	**Val David**	46.046141	-74.252484	25th August 2020		ST
Rouge	**Highway 50**	45.645046	-74.689759	20th July 2019		ST
Rouge	**Hubredeau**	46.015020	-74.627717	24th August 2020		ST
Rouge	**Tremblant**	46.211428	-74.589326	July 4th 2021	DR	
Rouge	**Labelle**	46.281377	-74.734044	July 3rd 2021	DR	
Rouge	**Riviere-Rouge**	46.410186	-74.868691	23rd August 2020		ST
Lièvre	**Mont Laurier**	46.558874	-75.494424	23rd August 2020	DR	
Lièvre	**Notre Dame du Laus**	46.083880	-75.616710	25th June 2021	DR	
Lièvre	**Val des Bois**	45.913873	-75.597997	22nd August 2020	DR	
Lièvre	**Notre Dame du Salette**	45.769280	-75.591050	25th June 2021	DR	
Lièvre	**Buckingham**	45.592262	-75.420031	20th July 2019		ST
South Nation	**Plantagenet**	45.537238	-74.996097	21st July 2019		ST
South Nation	**Casselman**	45.318638	-75.092981	22nd July 2020		ST
South Nation	**Crysler**	45.215342	-75.156257	June 15th 2021	DR	
South Nation	**Chesterville**	45.101107	-75.224889	20th September 2020		ST
South Nation	**Cass Bridge**	45.049295	-75.319978	June 15th 2021	DR	
Gatineau	**Fournier**	45.456638	-75.711916	9th September 2019		ST
Gatineau	**Wakefield**	45.638871	-75.929063	28th September 2020		ST

Gatineau	**Lac St. Marie**	45.950208	-75.944429	27th July 2021	DR	
Gatineau	**Baskatong**	46.801527	-75.862846	27th September 2020	DR	
Gatineau	**Lac Rapide**	47.221885	-76.726467	27th July 2021	DR	
Gatineau	**Lac Rolland**	46.923405	-76.425567	27th July 2021	DR	
Madawaska	**Arnprior**	45.439947	-76.348051	10th May 2021	DR	
Madawaska	**Highway 417**	45.412615	-76.352938	10th May 2021	DR	
Madawaska	**Burnstown**	45.391354	-76.568797	5th September 2020	DR	
Madawaska	**Combermere**	45.363439	-77.617053	5th September 2020	DR	
Madawaska	**Madawaska Town**	45.503442	-77.980283	5th September 2020	DR	
Bonnechere	**Horton**	45.514547	-76.557021	10th May 2021	DR	
Bonnechere	**Renfrew**	45.475011	-76.696661	10th July 2019	DR	
Bonnechere	**Bonnechere Caves**	45.504351	-77.010315	1st August 2020		ST
Bonnechere	**Golden Lake**	45.595720	-77.359825	7th September 2020	DR	
Bonnechere	**Round Lake**	45.657210	-77.565090	7th September 2020	DR	
Montreal	**Latchford**	47.322850	-79.809351	19th August 2019		ST
Montreal	**Elk Lake**	47.732003	-80.332611	5th August 2020	DR	
Montreal	**Matachewan**	47.939579	-80.650748	3rd October 2019		ST
Montreal	**Mistinikon Lake**	48.005026	-80.741850	29th July 2021		ST
Montreal	**Gowganda**	47.651865	-80.776399	29th July 2021	DR	
Blanche	**Highway 65**	47.608886	-79.540142	6th August 2020	DR	
Blanche	**Hillard**	47.699959	-79.673722	6th August 2020	DR	
Blanche	**Round Lake**	48.000139	-80.061084	29th July 2021	DR	
Blanche	**Swastika**	48.109498	-80.104715	3rd October 2019		ST

Supplementary table 4

Location Name	Concentration	Total Length	Distance Upstream	Distance Travelled	Percentage Travelled of total length
Laval	0.260	1272	0	1272	100
Oka	0.119	1272	1	1271	100
Voyager	0.149	1272	30	1242	98
Hawkesbury	0.591	1272	44	1228	97
L'Oringal	0.243	1272	52	1220	96
Montebello	0.441	1272	72	1200	94
Clarence	0.172	1272	102	1170	92
Petrie Island	4.955	1272	121	1151	90
Blair Road	1.752	1272	133	1139	90
Le Breton	0.429	1272	142	1130	89
Limieux Island	0.620	1272	145	1127	89
Bate Is.	0.763	1272	146	1126	89
Deschene	0.343	1272	151	1121	88
Brittania	0.530	1272	151	1121	88
Shirleys Bay	0.937	1272	163	1109	87
Constance bay	0.210	1272	177	1095	86
Quyon	1.020	1272	187	1085	85
Robert Simpson	0.613	1272	200	1072	84
Norway Bay	0.385	1272	208	1064	84
Portage du Fort	0.319	1272	228	1044	82
George Matherson	0.301	1272	298	974	77
Pembroke Waterfront	0.447	1272	309	963	76
Pembroke Riverside	0.092	1272	310	962	76
Petawawa Point	0.245	1272	325	947	74
Deep River	0.772	1272	357	915	72

Dumoine	0.461	1272	375	897	71
Deux-Rivieres	1.037	1272	425	847	67
Klock	0.320	1272	440	832	65
Mattawa	0.309	1272	465	807	63
Temiscaming	0.894	1272	515	757	60
Opemican	0.478	1272	531	741	58
Vieux Fort	0.361	1272	576	696	55
De ille	0.469	1272	580	692	54
Devils Rock	0.071	1272	581	691	54
Haileybury	0.251	1272	583	689	54
New Liskeard	0.783	1272	585	687	54
Waterfront Inn	0.184	1272	586	686	54
Notre Dame	0.460	1272	603	669	53
Angliers	0.334	1272	645	627	49
Remingy	0.522	701	669	32	5
Forester	0.180	820	750	70	9
St. Agnes	0.939	820	762	58	7
Rapide Sept	0.276	1272	795	477	38
Twin Rapids	0.358	1272	875	397	31
Kinojevis	0.159	820	800	20	2
Picnic	0.460	1272	997	275	22
Whiskey Creek	0.117	1091	1034	57	5
Verendrye 30	0.119	1091	1041	50	5
Camatose	0.531	1091	1049	42	4
Galetta	0.343	200	4	196	98
Packenham	1.150	200	15	185	93
Blackney	0.167	200	24	176	88
Appleton	0.521	200	40	160	80
Carleton Place	0.268	200	47	153	77

Lanark	0.060	200	86	114	57
Vanier	0.958	145	3	142	98
Carleton University	0.181	145	10	135	93
Jocks Landing	0.236	145	25	120	83
Manotick	0.414	145	27	118	81
Burritts Rapids	0.210	145	65	80	55
Smiths Falls	0.107	145	100	45	31
St. Andrews	0.311	135	6	129	96
Lachute	0.095	135	21	114	84
Piedmont	0.254	135	71	64	47
Val David	0.111	135	126	9	7
Highway 50	3.640	160	1	159	99
Hubredeau	0.222	160	50	110	69
Tremblant	0.276	111	80	31	28
Labelle	0.080	160	86	74	46
Riviere-Rouge	0.107	160	109	51	32
Buckingham	0.233	330	9	321	97
Notre Dame du Salette	0.715	330	35	295	89
Val des Bois	0.781	330	55	275	83
Notre Dame du Laus	0.543	330	77	253	77
Mont Laurier	0.414	330	149	181	55
Plantagenet	0.843	175	10	165	94
Casselman	0.153	175	59	116	66
Crysler	1.047	175	73	102	58

Chesterville	0.052	175	90	85	49
Cass Bridge	0.483	175	100	75	43
Fournier	1.316	386	2	384	99
Wakefield	0.095	386	29	357	92
Lac St. Marie	0.116	88	66	22	25
Baskatong	0.102	386	148	238	62
Lac Rapide	0.495	332	282	50	15
Arnprior	0.404	230	1	229	100
Highway 417	1.062	230	4	226	98
Burnstown	0.424	230	25	205	89
Combermere	0.722	230	136	94	41
Madawaska Town	0.184	230	176	54	23
Horton	0.765	145	1	144	99
Renfrew	0.885	145	16	129	89
Bonnechere Caves	0.204	145	44	101	70
Golden Lake	0.515	145	72	73	50
Round Lake	0.618	145	105	40	28
Latchford	0.157	220	40	180	82
Elk Lake	0.050	220	105	115	52
Matachewan	3.178	220	141	79	36
Mistinikon Lake	0.554	220	161	59	27
Gowganda	0.343	220	201	19	9
Highway 65	0.397	125	6	119	95
Hillard	0.386	125	21	104	83
Round Lake	0.618	125	71	54	43

Swastika	0.400	125	88	37	30

Supplementary table 5

Date	Fibre	Fragment	total	% fragment	% fibre	Total per L	Total per m3
November 1st 2019	162	5	167	0.03	0.97	0.167	167
March 11th 2021	249	24	273	0.09	0.91	0.273	273
March 17 2021	83	9	92	0.10	0.90	0.092	92
March 26 2021	45	4	49	0.08	0.92	0.049	49
April 21 2021	88	0	88	0.00	1.00	0.088	88
May 14th 2021	27	2	29	0.07	0.93	0.029	29
July 24 2021	48	1	49	0.02	0.98	0.049	49
June 11 2021	10	0	10	0.00	1.00	0.01	10
June 4 2021	34	0	34	0.00	1.00	0.034	34

Supplementary table 6

Date	Total per m3	Rainfall 1 hour before (mm)	Rainfall 2 hours before (mm)	Rainfall 4 hours before (mm)	Rainfall 8 hours before (mm)	Rainfall 24 hours before (mm)	Rainfall 48 hours before (mm)	Comments
November 1st 2019	167	0.3	0.3	0.5	18.0	55.2	61.6	Heavy rain the previous day and day before. Sampling during drizzle in early morning, CSO signal light activated.
March 11th 2021	273	0.0	0.0	0.0	0.0	0.0	0.0	Fine and sunny. Snowmelt, visible overland runoff from melting snowpack.
March 17 2021	92	0.0	0.0	0.0	0.0	0.0	0.0	Fine and sunny, second snowmelt day, with less snowpack. Some overland runoff.
March 26 2021	49	1.8	4.8	18.2	27.8	28.0	36.8	Rain. Sampled after some heavy downpours. Almost all of snowpack gone. No active CSO.
April 21 2021	88	0.0	0.0	2.0* Snow	2.0* Snow	4.0* Snow	10.7* Snow	Snowing (cm). Sampled during snow. Spring, so a wet snow.
May 14th 2021	29	0.0	0.0	0.0	0.0	0.0	0.0	Dry weather, no significant rain previous days. River running high with no storm drain input. Possible freshet influence.
June 4th 2021	48	0	0	0	0	7.1	7.1	Steady rain the day before. Previous long dry spell. River still running high, potential second freshet from upstream melt.
June 11 2021	10	0	0	0	0	7.1	7.1	Long dry spell, however, river level still up a bit. Possible tail end of second freshet.
July 24th 2021	34	0.0	0.0	0.0	2.3	2.3	2.6	Dry spell. Two days previous some heavy showers that broke a long dry spell.

Supplementary table 7

Date	Fibre	Fragment	total	% fragment	% fibre	Total per L	Total per m3
November 1st 2019	162	5	167	0.03	0.97	0.167	167
March 11th 2021	249	24	273	0.09	0.91	0.273	273
March 17 2021	83	9	92	0.10	0.90	0.092	92
March 26 2021	45	4	49	0.08	0.92	0.049	49
April 21 2021	88	0	88	0.00	1.00	0.088	88
May 14th 2021	27	2	29	0.07	0.93	0.029	29
July 24 2021	48	1	49	0.02	0.98	0.049	49
June 11 2021	10	0	10	0.00	1.00	0.01	10
June 4 2021	34	0	34	0.00	1.00	0.034	34

Supplementary table 8

Date	Total per m3	Rainfall 1 hour before (mm)	Rainfall 2 hours before (mm)	Rainfall 4 hours before (mm)	Rainfall 8 hours before (mm)	Rainfall 24 hours before (mm)	Rainfall 48 hours before (mm)	Comments
September 9th 2019	0.40	0.0	0.0	0.0	0.0	0.0	0.0	No rain, dry conditions, good flow in the creek.
September 18th 2019	0.37	0.0	0.0	0.0	0.0	0.0	0.0	No rain, dry conditions, good flow in the creek.
October 31st 2019	19.92	2.7	4.8	10.3	12.3	19.9	20.6	Heavy rain. Strong flow out of strom drains.
October 21st 2020	26.51	3.1	4.1	5.5	11.4	11.4	17.8	Heavy rain. Strong flow out of strom drains.
March 11th 2021	52.55	0.0	0.0	0.0	0.0	0.0	0.0	Fine and sunny. Snowmelt, visible overland runoff from melting snowpack.
March 17th 2021	5.36	0.0	0.0	0.0	0.0	0.0	0.0	Fine and sunny, second snowmelt day, with less snowpack. Some overland runoff.
March 26th 2021	6.96	1.8	4.8	18.2	27.8	28.0	36.8	Rain. Sampled after some heavy downpours. Almost all of snowpack gone.
April 21st 2021	15.82	0.0	0.0	2.0* Snow	2.0* Snow	4.0* Snow	10.7* Snow	Snowing (cm). Sampled during snow. Spring, so a wet snow.
May 14th 2021	0.60	0.0	0.0	0.0	0.0	0.0	0.0	Dry weather, no significant rain previous days. Creek flowing well.
June 4th 2021	0.76	0	0	0	0	7.1	7.1	Steady rain the previous day that broke a long dry spell.

Supplementary table 9

Date	Fragments	Fibre	Total	% Fragment	% Fibre	Total per L	Total per m3
September 9th 2019	6	36	42	0.14	0.86	0.000400	0.40
September 18th 2019	5	40	45	0.11	0.89	0.0003677	0.37
October 31st 2019	28	746	774	0.04	0.96	0.019923	19.92
October 21st 2020	159	253	412	0.39	0.61	0.0265122	26.51
November 3rd 2021	32	703	735	0.04	0.96	0.0525526	52.55
March 17th 2021	3	172	175	0.02	0.98	0.0053625	5.36
March 26th 2021	38	108	146	0.26	0.74	0.00695934	6.96
April 21st 2021	28	208	236	0.12	0.88	0.015819391	15.82
May 14th 2021	11	17	28	0.39	0.61	0.000600601	0.60
June 24th 2021	7	5	12	0.58	0.42	0.00075706	0.76

Supplementary table 10

ANCOVA analysis – returned variables – executed with R		
(Tested variables included microplastic concentration in relation to percentage distance downstream, distance from a wastewater treatment plant, distance upstream and downstream of dam structures, precipitation amount day of and three days previous to sampling and concentrations from urban and non-urban locations, boat launch and non-boat launch locations and main channel and tributary locations)		
Tested variable	t-value	p-value
Distance downstream	2.795	0.00621 **
Boat launch locations	-3.124	0.00233 **
Significance codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1		
Multiple R-squared: 0.1457, Adjusted R-squared: 0.1288		

ANCOVA analysis – returned variables – executed with R (fragment only)		
(Tested variables included microplastic concentration in relation to percentage distance downstream, distance from a wastewater treatment plant, distance upstream and downstream of dam structures, precipitation amount day of and three days previous to sampling and concentrations from urban and non-urban locations, boat launch and non-boat launch locations and main channel and tributary locations)		
Tested variable	t-value	p-value
Distance downstream	2.291	0.0241 *
Boat launch locations	-3.124	1.19e-07 ***
Total precipitation	1.861	0.0657
Significance codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1		
Multiple R-squared: 0.2977, Adjusted R-squared: 0.2766		

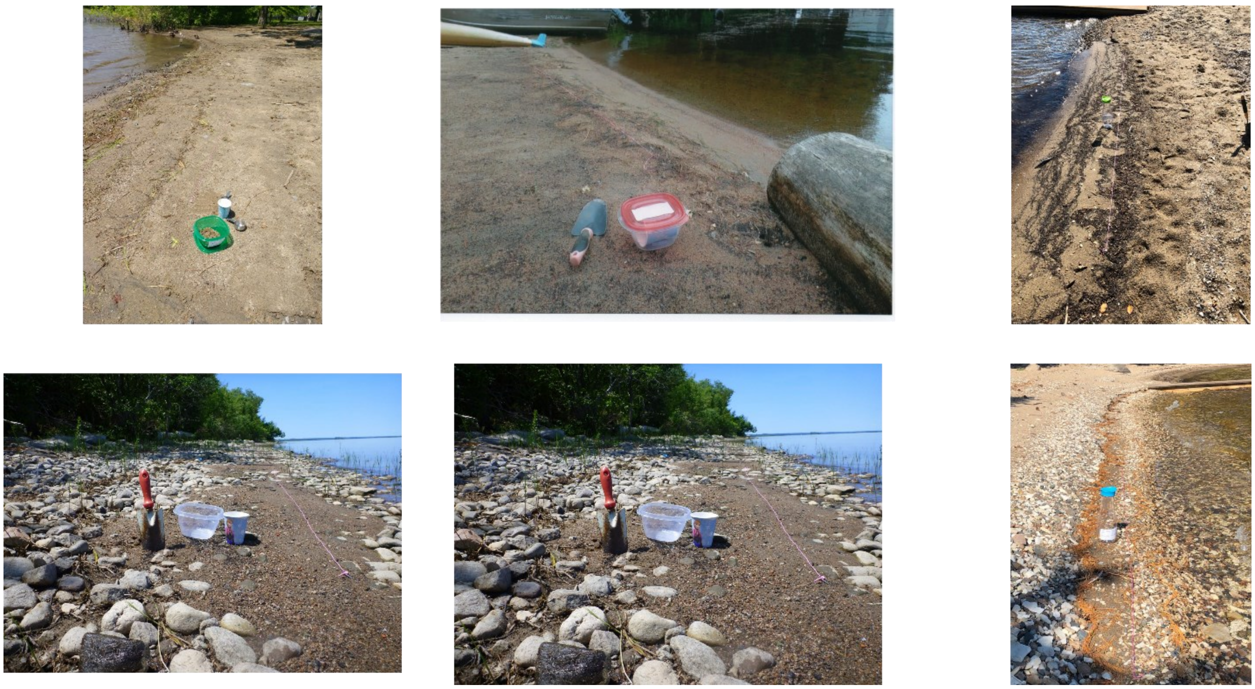
Supplementary table 11

Location name (downstream to upstream)	Approximate distance from river mouth (km)	Particle concentration (per kg of dry sediment)	Latitude	Longitude
Ottawa River				
Chenal Island	48	11.78	45.61598	-74.59928
Hawkesbury	48	4.29	45.61181	-74.60356
Petrie Island	123	5.00	45.49850	-75.50522
Rebecca Overflow	133	6.83	45.46333	-75.61794
Blair Road	134	2.99	45.46326	-75.62665
Kettle Island	135	5.78	45.46771	-75.64425
New Edinburgh Club	138	5.19	45.45730	-75.67482
Parc Mousette	146	3.34	45.41866	-75.74849
Remic Rapids	147	4.74	45.40756	-75.74706
Bate Island	148	2.35	45.40961	-75.75565
Westboro Downstream	150	13.71	45.39606	-75.76212
Westboro Upstream	151	6.59	45.39443	-75.76143
Rue Huole	154	3.55	45.38427	-75.79906
Guillot Park	155	8.41	45.37967	-75.80977
Queens park	163	5.45	45.40710	-75.87907
Luskville	176	7.60	45.48913	-76.00327
Baskins Beach	176	5.43	45.47880	-76.00991
Allen Island	177	11.79	45.48861	-76.01861
Morris Island	203	5.93	45.46336	-76.28290
Pontiac Station	206	11.31	45.46999	-76.32127
Mclean Park	207	4.62	45.44046	-76.33712
Robert Simpson Park	207	2.90	45.44357	-76.34985
Norway Bay East	215	21.95	45.50983	-76.40434
Norway Bay West	217	14.55	45.51794	-76.42848
Braeside	219	2.29	45.49536	-76.46410
Bristol	220	1.77	45.52060	-76.45717
Haughton’s Bay	221	8.16	45.52436	-76.46902
Chats lake	223	5.15	45.51112	-76.50055
Baie Indian Island	236	3.00	45.55848	-76.65183
Baie Indian	237	3.13	45.56061	-76.65703
Webster’s Beach	239	11.95	45.57221	-76.66378
Portage-du-Fort	242	6.04	45.60180	-76.66855
McLarens	243	14.22	45.61373	-76.68370
Portage-du-Fort Nord	244	8.96	45.62841	-76.66697
Harmony Bay	250	9.23	45.66053	-76.72246
Hennessey Bay	283	1.72	45.87888	-76.85974
Morrison Island East	308	0.88	45.81160	-77.02999
Morrison Island West	309	3.81	45.81639	-77.04698
Pembroke Waterfront	314	4.48	45.82987	-77.09668
Pembroke Riverside	316	4.94	45.82975	-77.13057
Murphy’s Point	318	2.63	45.86000	-77.13900
Thorne	508	0.83	46.69719	-79.09838
Ville-Marie	584	1.76	47.29700	-79.45990
Oster Island	597	5.58	47.39391	-79.54176
Haileybury	604	8.04	47.44277	-79.62703
Dawson Point	606	4.39	47.47803	-79.60933
Dixon Creek	607	6.07	47.47530	-79.66300
Wabi bay	608	6.02	47.48824	-79.67104
New Liskeard West	609	6.15	47.49932	-79.67760
New Liskeard Centre	610	7.01	47.50369	-79.67444
New Liskeard East	611	10.44	47.50618	-79.67003
Bakers Bush	612	5.61	47.51341	-79.65965
Sutton bay	616	12.31	47.54050	-79.58460
Notre-Dame-du-Nord Sud	621	4.51	47.57171	-79.49453
Notre-Dame-du-Nord	622	6.72	47.57613	-79.49099

Supplementary table 12

Location name (downstream to upstream)	Approximate distance from river mouth (km)	Particle concentration (per kg of dry sediment)	Latitude	Longitude
Tributaries				
Burnett Road (Gatineau)	18	3.83	45.57168	-75.85125
Meech Creek (Gatineau)	21	1.81	45.57096	-75.88701
Lepeche (Gatineau)	40	5.58	45.63689	-75.93221
Turntable Park (Gatineau)	41	1.94	45.64021	-75.92727
Macintyre Beach (Gatineau)	42	0.74	45.64356	-75.91900
Burritts Rapids (Rideau)	60	4.55	44.98179	-75.79893
Robert Simpson Park (Madawaska)	1	2.95	45.44230	-76.34889
Round Lake (Bonnechere)	90	3.28	45.64846	-77.57176
Laniel (Kipawa)	15	1.81	47.04288	-79.26851
Lake Moran (Kipawa)	63	0.70		
Rouyn-Noranda (Kinojévis)	15	2.06	47.26438	-79.15818
Aiguebelle	70	2.01	48.43144	-78.69156

Supplementary figure 1



Supplementary figure 2

10. Appendices

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