Microplastic contamination in Te Tai Tokerau-Northland (Aotearoa-New Zealand) beach sediments

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Executive summary

The problem

This study aims to identify and quantify microplastics from sand sediments across the Northland region of New Zealand. This region is framed by the Pacific Ocean and the Tasman Sea and known for its famous beaches among surfers and beachgoers. This report characterises the microplastic distribution within this region.

This project

This study falls under the umbrella of *Aotearoa Impacts and Mitigation of Microplastics* (AIM[2]) with the purpose of investigating microplastics in the New Zealand environment and the implications for ecosystems. The national research programme is composed of three research objectives: (1) understanding the distribution of microplastics in NZ environments, (2) identifying associated risks and impacts to animals, people, and ecosystems, and (3) identifying solutions ranging microbial mitigation of existing pollution, to prevention of new pollution through outreach and education. This endeavour falls within the first research objective of the program.

Key results

This project aims to characterise and quantify microplastic distribution in sand sediments from the Northland region across 9 open coastal sites, one dune lake and one estuary site. Microplastic contamination was further investigated under varied beach location (north/south), coast side (east/west), and season (summer/winter). The mean large microplastic particle (LMPP) abundance in Northland sand sediments was 3 MP/kg DW (dry weight) (equivalent to 229 particles/m^2 and 4587 particles/m^3). Results showed that there was a varying degree of particle abundance in different Northland sites. Microplastic abundance varies among locations within the beach and freshwater sites. Higher microplastic concentrations were seen in beach sites like Mangawhai and Sandy Bay, while lower concentrations were obtained from a lake and an estuary site like Taharoa and Onerahi, respectively. Statistical analysis confirmed significant differences in mean particle abundance based on site using one-way ANOVA. Moreover, in comparison to the microplastic concentration of the Auckland beaches (6 MP/kg DW or 459 particles/m^2) investigated by Bridson et al., (2020), the overall mean particle concentration of Northland sites (3 MP/kg DW or 229 particles/m^2) was found to be significantly lower compared to that of the Auckland beaches. Three (3) large particle morphologies were distinguished from the samples, with fibres forming the majority of particles (51%), followed by fragments (36%), and then by films (13%). Characterisation identified the presence of common polymers, polyethylene (PE) polypropylene (PP), and polyethylene terephthalate (PET), in these sand sediments. A significant portion of cellulose and regenerative cellulose (C & RC) was evident as well.

Further analysis showed that the overall abundance of small microplastic particles (SMPP) observed during winter was lower with an average of 405 MP/kg DW, ranging from 0-6044 MP/kg DW compared to the particles seen during summer with an average of 1417 MP/kg DW, ranging from 0-9062 MP/kg DW. However, statistics shows that the difference between the two seasons is not significant (p=0.065) based on a 95% confidence level. Analysis of SMMPs also revealed that 82% of particles were ≤25 μm and were predominantly polyethylene and polypropylene.

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Introduction

Humans have been heavily reliant on plastics since their mass production at the beginning of the 1950s. Hundreds of millions of tonnes of plastics are produced every year and only a small proportion of it is recycled. Most of the plastics are disposed of to landfill, however some end up in the ocean. It has been reported that in 2016, up to 23 million metric tons of plastics are floating in the world's oceans [1,2].

According to the U.S. National Oceanic and Atmospheric Administration (NOAA) and the European Chemicals Agency (ECHA), microplastics are miniscule plastics measuring less than 5 mm in length. They are found in various forms such as nurdles, fibres, fragments, and films. These microparticles are globally recognised as contaminants in marine and terrestrial environments. Microplastic can be classified as either primary, where the particles are industrially designed for commercial use (e.g. plastic nurdles used for plastic manufacturing and glitter), or secondary, where the small pieces of particles were formed through various weathering processes resulting in the fragmentation of larger plastic items or pieces. Microplastics have been identified as derived from plastics of different origins and compositions such as polyethylene (PE), polypropylene (PP), polystyrene (PS), polyester (PET), polyamide (PA), cellulose acetate (CA), and poly(vinyl chloride) (PVC) [3,4]. Microplastics are insoluble in water, nondegradable, and possess various physicochemical properties (size, shape, buoyancy, chemical composition) that determine their bioavailability and ecotoxicity to organisms [5-7]. The following sources have been identified as dominant in releasing microplastics into the environment: abrasion of car tyres, synthetic textiles, litter, marine coatings, road markings, personal care products and plastic pellets [1-6].

Plastic marine debris continuously degrades in the environment, subsequently creating smaller fragments due to weathering factors such as UV radiation (sun), physical and biological degradation. An increasing number of studies have reported the ingestion of these microplastics by a variety of marine organisms across all trophic levels such as birds, marine mammals, fish, and invertebrates [3, 7]. When ingested, the microplastics can remain in the digestive tract and form clumps that can hinder the passage of food and block the digestive system. It can also give a false sense of satiation thus leading to starvation. The release of additives (e.g. plasticisers) and ambient pollutants from the plastics can cause harmful effects by affecting the hormonal and reproductive system, damaging the gastrointestinal tract, causing delayed growth, genotoxicity and in some cases death [8-11]. Microplastics can also act as a carrier for dispersal of chemicals and biota such as invasive species and pathogens thereby potentially endangering marine biodiversity [12]. While there have been many studies reported on the effects of microplastics on marine life the impacts and mechanisms involved are still not fully understood [19-23]. Even less is known about the potential human health effects, with this area of research even more in its infancy, although preliminary findings suggest that life wildlife, there are deleterious effects [24].

Microplastics either sink or float in the marine environment depending on their density. Positively buoyant polymers, such as polypropylene and polyethylene, generally float in seawater as their densities are less than that of seawater. However, factors such as biofouling can cause a usually buoyant polymer to become heavy and accumulate in the sediment or sink to the bottom of the ocean [12]. This manifestation is evident in a recent study reporting microplastic presence in sediments from beaches [9, 13] and freshwater sources [27] of New Zealand. Other studies that investigated the microplastic distribution in New Zealand include the discovery of plastics in a third of turtles and birds found washed up on the shore dead [14], and the sighting of a plastic patch larger than Greenland, which was believed to have originated from New Zealand by a group of researchers looking at the microplastic abundance in the South Pacific [15].

The primary sources of microplastics have been linked to marine and domestic land-based activities (e.g. laundry discharges) [32], and industries including production and waste management. It is widely known that the ocean is considered as a sink for microplastics. However, the marine environment is not always the final fate of microplastics since some studies proved that microplastics can become resuspended into the atmosphere from the sea surface. Therefore, resulting in the continuous cycling between terrestrial and aquatic systems [39,40].

This study aims to contribute to the understanding of the microplastic distribution in Aotearoa New Zealand's beach ecosystems. Specifically, this intends to create baseline data for the Te Tai Tokerau - Northland region coastline and freshwater sites.

Materials and methods

Sampling sites

Sand sediment samples were supplied to Scion by Northland Regional Council (NRC) via courier. Upon arrival at Scion the samples were immediately stored at -20°C until required for analysis. Details of the sampling coordinates are provided in Appendix A. The 11 locations sampled for microplastics encompassed the east and west coast of the Te Tai Tokerau - Northland region (Figure 1). Information provided by NRC for each location is provided in Appendix B.
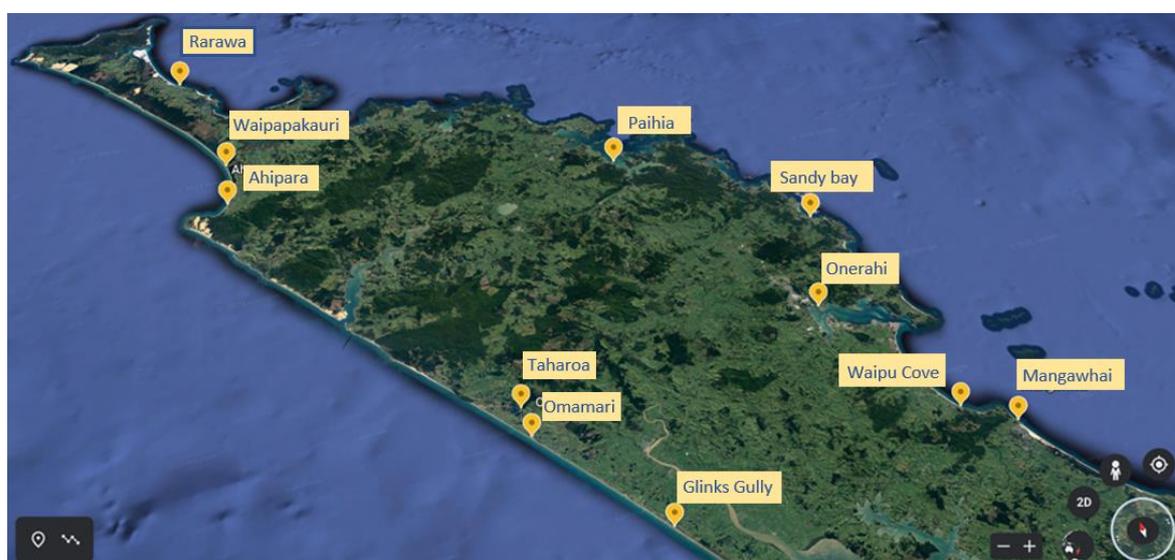
Figure 1. *Map of the Northland locations sampled for microplastic analysis.*

Sample collection

The samples were collected during the summer period: November 2019- February 2020 and winter period: August - September 2020. All locations were sampled on an out-going tide to ensure that there was plenty of beach area to sample from. Depending on the size of the beach at least three to four transects (spacing between each transect 50 m) were sampled. A 10 m transect was laid along the last high tide mark (Figure 2a). One metre either side of the transect was marked out and the 2 x 10 m area was divided into a grid consisting of eighty (80) half-metre quadrants (0.5 x 0.5 m) (Figure 2b). The number randomisation function in Excel was used to choose five quadrats from each transect for sampling (see Figures 2a and 2b). These quadrants represented replicates for each transect.

Figure 2a. *Example of a transect along the beach*
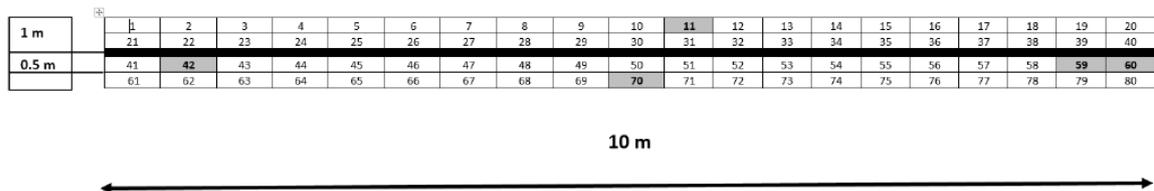
Figure 2b. *Transect (centre thick line) and the 80 x 0.5m² quadrants.*

Within each of the 5 quadrants, 5 sand cores were taken (from each corner and the centre of the quadrant) with steel corers (5 x 5 cm) to a depth of 5 cm. The 5 cores of sand were combined in a pre-rinsed aluminium tray and sealed with the foil-lined paper lid and wrapped in aluminium foil.

Quality Control

To minimise the risk of any possible contamination during field sampling, the staff wore natural fibres and minimised the use of plastics where possible. Moreover, the staff positioned downwind during sample collection to avoid cross contamination of fibres from clothes shedding. During separation process in the laboratory, all glassware and equipment were rinsed first with tap water then twice with ultra-pure water (distilled water which had passed through a 0.45 µm filter). All washed vessels were covered with aluminium foil when not in use. A cotton laboratory coat was worn during the handling of the sediment and analysis of samples. The use of plastic apparatus was minimised as much as possible. All samples were processed in a controlled room with minimal foot traffic.

Procedural blanks were used throughout the isolation process to assess any potential cross-contamination. An analysis using a procedural blank showed an average of 5 MPs per sample. Analysis showed the presence of microplastics identified as polytetrafluoroethylene (PTFE) and blue polypropylene (PP) fragments. During the investigation, it was found out that the sodium iodide salt from LOBA-ACS reagent, which was used at the end of the peroxidation step was, in fact, contaminated with microplastics identified as PTFE and blue PP. The LOBA chemical was used

only in the summer samples and its use was discontinued thereafter. Due to a substantial amount of procedural blank contamination, the PTFE films and distinct blue PP fragments were excluded from the quantitative analysis of beach sediments. The results expressed in Figure 3 were calculated with the omission of PTFE contaminants and the distinct blue PP in affected samples.

Environmental blanks were collected to monitor for the presence of airborne plastic contamination inside the laboratory. Three (3) blank filter membranes in glass petri dishes were placed on the lab work bench and inside the fume hood. Inspection showed no microplastic in the blanks which indicates a low risk of environment contamination.

Separation of microplastics

The large microplastic particles (LMPP 300 µm - 5 mm) were separated from the sediment following the NOAA protocol [16], using sodium iodide solution as the flotation media. The separation process involved a series of steps which are detailed below.

Extraction
Wet sediments in the trays was dried in an oven set at 70°C. The dried sediment (400 g) was weighed into an 800 mL beaker and 300 mL of sodium iodide solution (5.3 mol/L (797 g NaI per 1 L water); ρ = 1.6 g/mL) was added to it. The mixture was stirred for 10 minutes using a glass rod and allowed to stand for 1 hour after which the floating matter was decanted onto a cascade of 300 µm and 32 µm sieves. The collected matter on the 300 µm sieve was washed into a pre-weighed 600 mL beaker and dried at 70°C overnight. Any large non-plastic material was thoroughly washed with ultra-pure water before discarding to ensure no microplastic was accidentally lost. The material collected on the 32 µm sieve was washed into a pre-weighed jar and dried at 70°C overnight. Both fractions were later processed separately and in a slightly different manner. From here on the microplastic collected on the 300 µm sieve are referred to as large microplastic particles (LMPP) and the microplastic collected on the 32 µm sieve are referred to as small microplastic particles (SMPP).

Wet Peroxidation

LMPP
The wet peroxidation step was included to digest any remaining organic matter. Iron sulphate (20 mL, 0.05 mol/L, Sigma-Aldrich) and 20 mL of hydrogen peroxide (30%, Merck) solutions were added to the 600 mL beaker containing the dried matter. The mixture was left to stand for 5 minutes during which it bubbled and frothed. The beaker was placed in a water bath at 75 °C and shaken for 30 mins and an additional aliquot of 20 mL hydrogen peroxide added if any organic matter was still visible. This step was repeated until no organic matter was evident. Sodium iodide (Merck, LOBA and ECP, ACS reagent) was added to the mixture (15 g NaI per 20 mL of the solution) to increase the density of the solution. The mixtures were heated and shaken in a 75 °C water bath until most of the salt dissolved. The samples were then cooled to room temperature in preparation for the density separation.

SMPP
Ten (10) mL each of iron sulphate solution (0.05 mol/L, Sigma-Aldrich) and hydrogen peroxide solution (30%, Merck) were added to the 600 mL beaker containing the dried matter. The mixture was left to stand for 5 minutes during which time it bubbled and frothed. The beaker was placed in a water bath at 75°C and shaken for 30 mins, and an additional aliquot of 10 mL hydrogen peroxide was added if any organic matter was still visible. The addition of hydrogen peroxide aliquot was repeated until no organic matter was evident. Sodium iodide (ECP, ACS reagent) was added to the mixture (15 g NaI per 20 mL of solution) to increase the density of the solution. The mixture was heated and shaken in the water bath until all the salt had dissolved. The samples were then cooled to room temperature in preparation for density separation.

Density separation

LMPP
The room temperature peroxidation was allowed to cool before being transferred to a density separator. To ensure that all microplastics were transferred to the density separator the beaker was

thoroughly rinsed with NaI solution (5 mol/L). The funnels were covered with foil and left to settle overnight at room temperature. The settled matter was discarded, and the remaining solution was filtered through a PCTE filter membrane (Whatman Track-Etched Polycarbonate Membrane, 4.7 cm, 10 μm). The inner wall of the beaker was thoroughly rinsed with Milli-Q-water to ensure complete transfer of microplastics and the surface of the filter membrane was washed thoroughly to ensure complete dissolution of the NaI crystals. The filter membrane were transferred to a labelled glass petri dish and stored in a cool dark place until required for the quantification and analysis step.

SMPP
The same procedure used for LMPP for the transfer of the peroxidised solution and the density separator to rinsing using NaI solution (5 mol/L) was followed for the SMPP mixture. After overnight sedimentation, the settled matter was discarded, and the remaining solution was collected in a pre-washed and pre-weighed capped glass vial (25 mL). Using a dropping pipette, a few drops of the well mixed solution were transferred onto an aluminium oxide filter membrane (Whatman Anodisc, 25 mm, 0.2 μm). The inner wall of the beaker was thoroughly rinsed with ultra-pure water to ensure complete transfer of microplastics and the surface of the filter was washed thoroughly to ensure complete dissolution of the NaI crystals. The weight of the solution transferred onto the membrane was recorded. The filter membrane was transferred to a labelled petri dish and stored in a cool dark place until required for the quantification and analysis steps.

Identification, quantification and analysis of microplastics

LMPP
The collected matter on the filter membranes was examined under a Zeiss Stemi Dv4 stereo microscope with 8-32x magnification to identify potential LMPPs. Plastic particle selection was performed according to previously published guidelines [41], specifically: size <5 mm, absence of cellular or organic structures, fibres of equal thickness throughout entire length, particles of clear and homogenous colour throughout. Fourier Transform Infrared (FTIR) Spectroscopy (Bruker Tensor 27) with attenuated total reflectance (Bruker, single bounce diamond cell) was used for the identification and quantification of the LMPP. All particles were selected for analysis by FTIR spectroscopy. Spectra were processed using the Bruker OPUS software, using the rubberband baseline correction algorithm. The spectra were searched against BioRad KnowItAll library databases for polymers, coatings and fibres using the Euclidean distance algorithm. Using an approach adapted from Kroon et al. (2018), database hits of >70% were positively identified as microplastics, while database hits of <70% were reviewed by an experienced spectroscopist to confirm the identity of the particle [42].

SMPP
Analysis of SMPPs was performed using a Bruker Hyperion 3000 FTIR-microscope equipped with a 64 x 64 focal plane array (FPA) detector. SMPPs were quantified using the automated approach of Primpke *et al.*, (2017). The Anodisc membrane was placed between two 25 x 2 mm CaF_2 windows and mounted on the microscope stage. A visual image of the samples was collected using the 4x vis objective, followed by FTIR imaging using the 15x cassegrain objective in transmission. Data collection was performed using OPUS version 8.2.28 (Bruker Optics GmbH) software with 4 x 4 binning at a resolution of 8 cm^{-1} with 6 co-added scans. Spectra were manipulated using the OPUS cut, and baseline correction functions prior to data processing.

Data processing was performed using an offline computer running OPUS 7.2. Spectra were compared against a database of common plastic spectra using OPUS search function as previously described (Primpke *et al.*, 2018). Image analysis was performed using Python and Simple ITK image processing modules as previously described [15, 17, 18].

Validation

<u>LMPP</u>
The validity of the method was evaluated using polymer-spiked sediment samples. Coloured polymers were used in the experiment for easy visual sorting during identification. The characteristics and properties of the three (3) polymers used in the spiking experiment are shown in Table 1. In the visual differentiation of microplastics and confirmation of particle count, a low magnification microscope was used. Positive controls were prepared by spiking clean sediments with thirty (30) particles of each polymer type. The negative controls (blank), on the other hand, were devoid of polymers. All experiments were prepared by two analysts and performed in triplicate. Samples were processed following the LMPP quantification described in the methods above; after which the percent recovery of the polymers was determined.

Table 1. *Properties of microplastics used in the LMPP validation.*

Analysis	Polymer type	Size range (µm)	Colour	Morphology	Density (g/cm^3)
LMPP	Polystyrene	500-1000	Purple	Fragment	1.05
	Polyamide	500-1000	Green	Fragment	1.15
	Polypropylene	500-1000	Pink	Fragment	0.92
SMPP	Polyethylene	38-45	Orange	Sphere	0.96
	Polyamide	30-100	Green	Fragment	1.15
	Polystyrene	100-300	Purple	Fragment	1.05

<u>SMPP</u>
The validation of instrumental parameters was conducted to determine the rate of detection by the analysis method described above for SMPP. Ten (10) particles of each polymer (refer to Table 1) were applied to a small area of an Anodisc filter using a low magnification microscope. This procedure was repeated to make triplicates. The negative controls (blank), on the other hand, were prepared similarly but with no added polymer.

Results and discussion

Validation results

LMPP

During validation, a chemical change was observed on the polymers, polyamide and polypropylene, after the peroxidation step. FTIR analysis confirmed that the discoloration is a result of oxidation attributed to the presence of an -OH stretch signal evident at the 3400 cm^{-1} band. The number of microplastic particles was recorded through visual inspection, taking note of the altered colours and the identified chemical nature using FTIR-ATR. A high overall recovery (95%) of the spiked polymers was achieved (Table 2).

Table 2. *Results of Validation Experiment for LMPP.*

Polymer type	Analyst	Recovered amount per replicate			Average (n=3) recovery (%)
		1	2	3	
Polystyrene	1	28	27	29	95
	2	28	28	30	
Polyamide	1	27	29	27	92
	2	27	28	28	
Polypropylene	1	30	27	29	98
	2	30	30	30	
Overall recovery					**95%**

SMPP

Validation shows a high rate of success, at 96%, in the detection of artificially spiked microplastic fragments through FTIR-FPA (Table 3). This translates to high reliability of particle counting and identification of the instrument.

Table 3. *Results of instrumental parameter validation for SMPP.*

Polymer type	replicate no.	No. of fragments spiked	No. of spiked successfully identified	Average (n=3) recovery (%)
Polyethylene	1	10		
	2	10	30	100
	3	10		
Polyamide	1	10		
	2	6	26	87
	3	10		
Polystyrene	1	10		
	2	10	30	100
	3	10		
Success rate				**96%**

LARGE MICROPLASTIC PARTICLE ANALYSIS

Microplastic abundance

The abundance of microparticles at different sites of the Northland region is expressed in three (3) different units (MP /kg DW, MP/m2, and MP/m3) to be consistent with the literature (see Appendix C). Table 4 summarizes the abundance in terms of MP/kg DW. Concentrations were computed from the average of 3, 5 or 6 replicates of summer samples and 3 replicates of all winter samples. Percent (%) abundance for each type of polymer at each location is also represented graphically in Figure 3.

Table 4. *LMPP abundance expressed as MP / kg DW for Northland summer samples.*

Site Name	Zone	Abundance (MP/kg DW)	
		Summer	*Winter*
Ahipara North	West	1 ± 1	6 ± 6
Ahipara South	West	*nd*	4 ± 5
Waipapakauri North	West	3 ± 3	3 ± 4
Waipapakauri South	West	5 ± 4	2 ± 0
Glinks Gully North	West	2 ± 1	2 ± 4
Glinks Gully South	West	5 ± 6	*nd*
Omamari North	West	3 ± 1	3 ± 4
Omamari South	West	7 ± 11	2 ± 0
Paihia North	East	1 ± 2[a]	6 ± 1
Paihia South	East	--	3 ± 1
Rarawa North	East	2 ± 2	2 ± 2
Rarawa South	East	2 ± 3	2 ± 2
Sandy North	East	8 ± 10	9 ± 6
Sandy South	East	7 ± 4	2 ± 1
Waipu North	East	1 ± 1	12 ± 7
Waipu South	East	*nd*	3 ± 1
Mangawhai North	East	7 ± 4	7 ± 4
Mangawhai South	East	3 ± 1	10 ± 2
Onerahi (North)	Estuary site	*nd*	1 ± 1
Onerahi (South)	Estuary site	*nd*	1 ± 1
Taharoa North	Dune lake	1 ± 1	*nd*
Taharoa South	Dune lake	1 ± 1	1 ± 1

[a]*Average value for North and South*
nd - none detected

Results showed that there was a varying degree of LMPP particle abundance in different Northland sites (Table 4). Microplastic abundances vary between locations within the beach, estuary and freshwater sites. High microplastic concentrations were seen in beach sites like Mangawhai and Sandy Bay, while lower concentrations were obtained from a lake and an estuary site like Taharoa and Onerahi, respectively. Statistical analysis confirmed significant differences in mean particle abundance based on site using one-way ANOVA (see Appendix D-1).

On the basis of season, particle abundance during summer and winter ranged from 0-8 MP/kg DW and 0-12 MP/kg DW, respectively. These quantified to a mean abundance of LMPP of 3 MP/kg DW (188 MP/m^2) during summer and 4 MP/kg DW (270 MP/m^2) for winter (see Appendix C). No significant difference in particle abundance was seen, however, between summer and winter samples (Appendix D-2A) and on the east and west side of the coast (Appendix D-2B) at a 95% confidence level.

In comparison to the microplastic concentration of Auckland beaches (6 MP/kg DW or 459 particles/m^2) investigated by Bridson et al., (2020), the overall mean particle concentration of Northland sites (3 MP/kg DW or 229 MP/m^2) was found to be significantly lower compared to that of Auckland beaches (Appendix D-2D). It should be noted, that the flotation media used for the Auckland study was of lower density (ρ = 1.2 g/L) compared to that used in this study (ρ = 1.6 g/L), as such abundance values from the Northland study should if anything be bias towards higher values than reported by Bridson *et al.*, 2020. The lower abundances observed in the Northland region correlate with the lower population density of the region compared to Auckland. In addition, the relative abundance of microplastics between the east and west coasts was different for the Auckland (west coast > east coast) and Northland (east coast > west coast) regions. (see Appendix D - 2E & 2F). This observation suggests that much of the microplastic debris on the Auckland west coast has originated from local sources, rather than from the more populous regions of East Australia and southeast Asia carried by the East Australian current, as hypothesised by Bridson et al., 2020 [7].

Polymer composition

In the characterisation of the particles, polyethylene (PE) was seen in the majority of the analysed sediment samples in both seasons. Polypropylene particles (PP) followed next as the most dominant polymer type while PET and PS were identified in relatively lower quantities. This order of relative mass compositions (PE>PP>PS) conforms to the characteristics of the plastics generated globally and those that are captured from the water and benthic environments seen previously in similar studies [35]. Negligible levels of PVC were observed, although this study used a high-density floatation solution suitable for the recovery of PVC. While PVC is produced and used widely (4th highest production polymer) [37], its negative buoyancy likely limits transport and distribution in aquatic environments, settling and depositing into sediment rapidly, rather than being transported to beach environments.

Although the exact sources of the contamination remain unknown, identified polymers are commonly associated with outdoor, recreational, personal and industrial use. PP and PS, for example, are commonly used in outdoor gear and disposable food containers and utensils. PE is used in a variety of household and industrial applications, while PET is used in common fabrics.
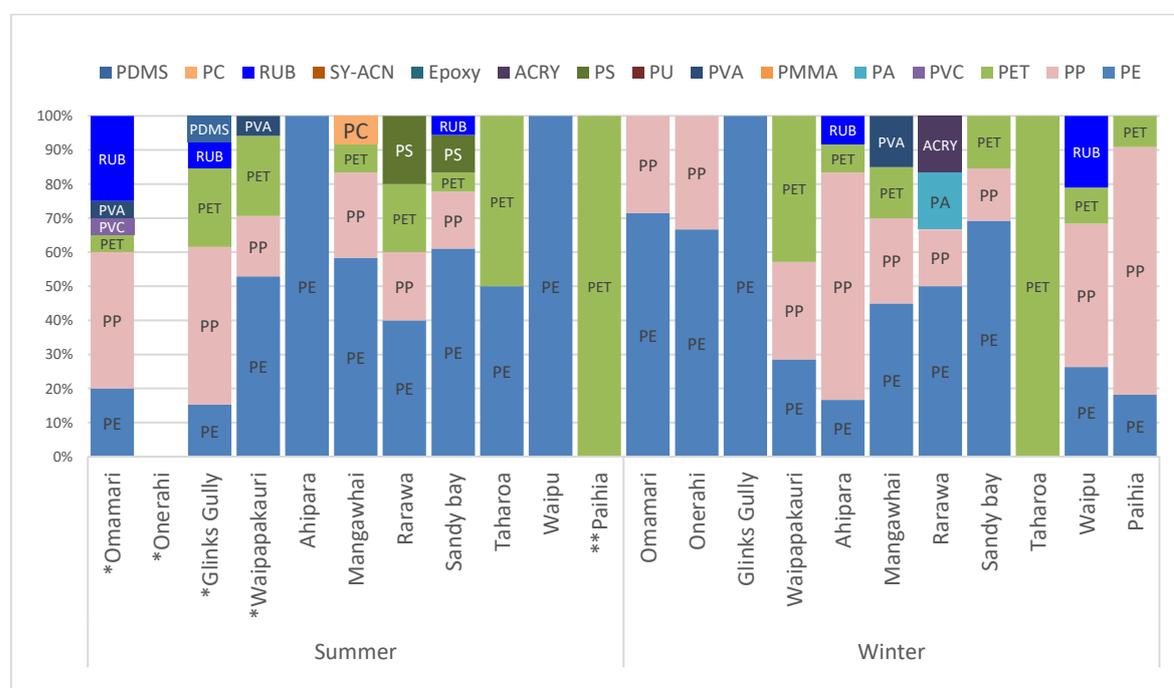
Figure 3. Relative abundance (%) of polymer type of *LMPPs identified at the sampling sites in summer and winter. An average of 3 samples were conducted for each site, unless indicated where 5* or 6** were done. Polymer type: Polymers:* RUB = Rubber; PVA = Polyvinyl acetate; PP = Polypropylene; PE = Polyethylene; PET = Polyethylene terephthalate; PVC = Polyvinyl chloride; PA = Polyamide; PC = Polycarbonate; PS = Polystyrene; PDMS = Polydimethylsiloxane; ACRY = Acrylic.

Accounting for other particle presence, significant portions of cellulose and regenerative cellulose (C & RC) in the form of fibre, were evident during analysis (see Figure 4). Cellulose fibres are unlikely to be harmful to the environment, but the associated dyes may pose a risk [33]. Based on the result of this study, the fibres identified as cellulose and regenerated cellulose were 93% colourless and the remaining 7% contained black, red, blue and green pigments. Hence, in this study, particles composed of C & RC were not considered as microplastics and were omitted from the overall calculation.
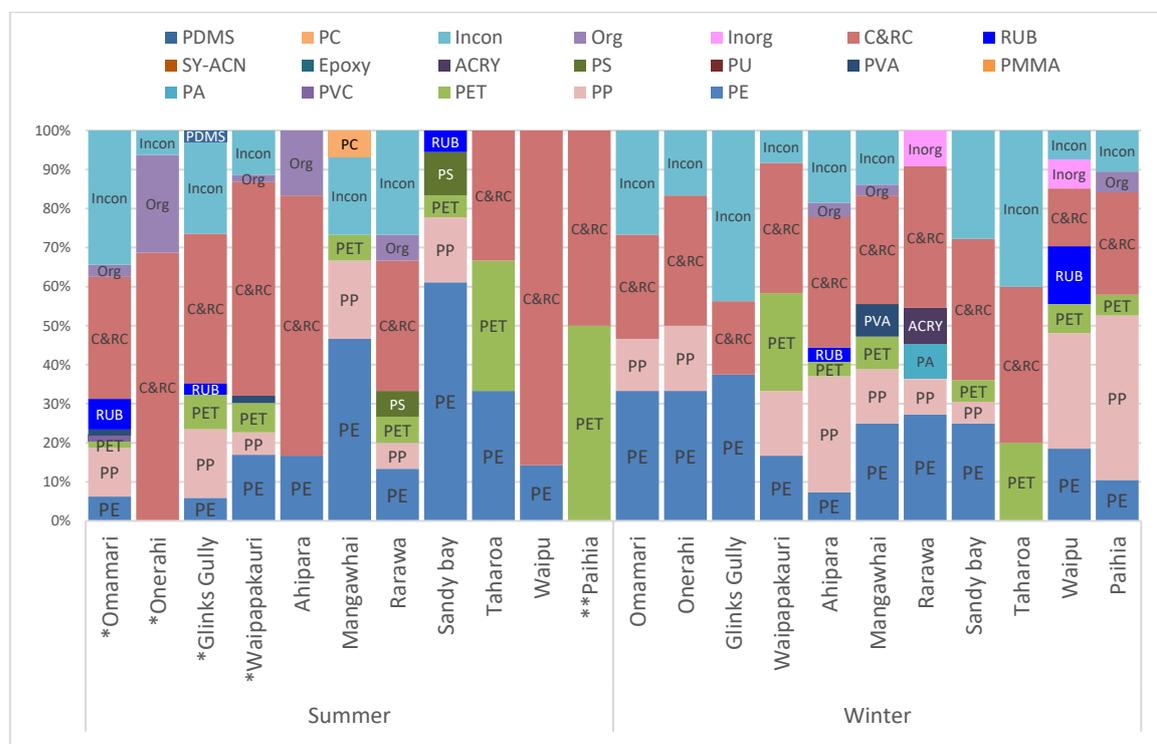
Figure 4. *LMPP (%) for various polymer types including non-microplastic particles (C & RC, Organic, Inorganic and Inconclusive) at the various sampling locations.*

Morphological features

In terms of morphology, three (3) microplastic morphologies were distinguished: film, fibre, and fragment. Fibre was the most prevalent morphotype and a large proportion was attributed to the presence of C & RC particles in both summer and winter. Fragments were identified as PE was predominantly present in summer, while both PP and PE were prevalent in winter. Moreover, PE was also the predominant polymer in the form of film observed in summer and winter. These findings were consistent with previous reports [7].

Table 5. *Characteristics of large microplastics across the variables of shape and polymer type for all sites.*
[1]*including rubber*

Variable	Category	Percentage	
		Summer	*Winter*
Morphology	Film	5	23
	Fibre	61	39
	Fragment	34	38
Polymer type	PE	16	20
	PP	10	18
	PET	6	6
	PS	1	-
	Other[1]	5	5
	C & RC	39	29

SMALL MICROPLASTIC PARTICLE ANALYSIS

A subset of the locations was analysed for SMPP, based on the highest and lowest number of total particles in the eastern and western beach sites in the region: Omamari, Taharoa, Onehari, and Mangawhai sites.

The mean abundance of SMPP across the four sites (911 MP/kg DW) was significantly higher *(p=0)* than for LMPP (3 MP/kg DW). This highlights the limitations of commonly used visual identification and quantification methods and the importance of identifying smaller (<300 µm) particles for understanding the extent of microplastic pollution in the environment.

Across these four sites, the mean abundance of SMPP was lower in winter (average of 405 MP/kg DW, ranging from 0-6044 MP/kg DW) compared to summer (average of 1417 MP/kg DW, ranging from 0-9062 MP/kg DW). However, statistical analysis demonstrated that the difference between the two seasons is not significant (*p=0.065*) based on a 95% confidence level, as similarly observed for LMPP.

Table 6. *SMPP abundance result for selected Northland summer and winter samples expressed as MP/kg DW.*

Site	Season	Polymer excl rubber[1]	Total rubber[2]	Total all polymers
Omamari	Summer	339 ± 483	*nd*	339 ± 483
	Winter	65 ± 117	19 ± 46	84 ± 114
Taharoa	Summer	871 ± 1576	27 ± 66	898 ± 1569
	Winter	279 ± 430	110 ± 147	389 ± 524
Onerahi	Summer	1162 ± 1293	45 ± 75	1207 ± 1366
	Winter	231 ± 305	60 ± 105	291 ± 311
Mangawhai	Summer	3297 ± 3695	126 ± 308	3423 ± 3936
	Winter	1043 ± 2450	*nd*	1043 ± 2450

[1]*All identified plastics (excluding rubber)*
[2]*Mostly ethylene–propylene–diene rubber*
 nd - none detected

Polyethylene was predominant at all sites at both seasons except for Lake Taharoa during winter. The types of polymers found from the analysis of SMPP are consistent with that of the LMPP. Smaller particles were likely derived from the physical and chemical degradation of large microplastic particles in the environment. However, it should be noted that fragmentation of LMPP can occur during sample processing. Meanwhile, some polymer types were exclusively seen in significant proportions based on the result of SMPP analysis. For example, Lake Taharoa (summer), a popular coastal highway for vehicle users was found to have a relatively high fraction of polychloroprene (neoprene), while Mangawhai (summer) and Omamari (summer) were both seen with high proportions of polyamide, none of which were detected in the LMPP analysis.
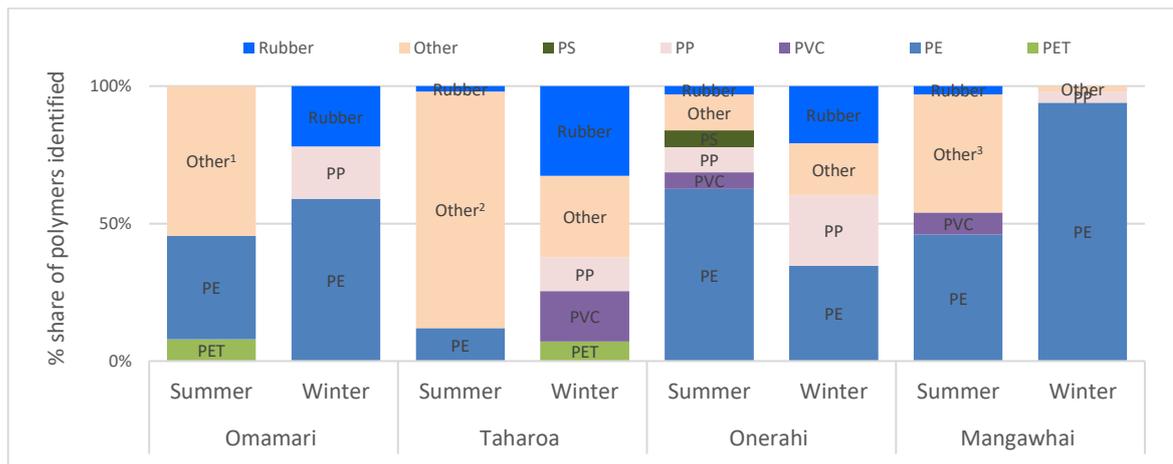
Figure 5. *Proportion (%) of polymer types identified at selected sampling sites from SMPP analysis.*
Polymer type: PP = Polypropylene; PE = Polyethylene; PET = Polyethylene terephthalate; PVC = Polyvinyl chloride; PA = Other[1] mostly polyamide, Other[2]- mostly polychloroprene, Other[3]- polyamide and Acrylic.

Furthermore, SMPP analysis revealed that particle size of ≤25 μm accounted for 82% of the small microplastics. Figure 6 below shows that all particles from Omamari Winter and Taharoa Summer fell within the size range with 225-300 μm particles only evident in the Taharoa winter samples.
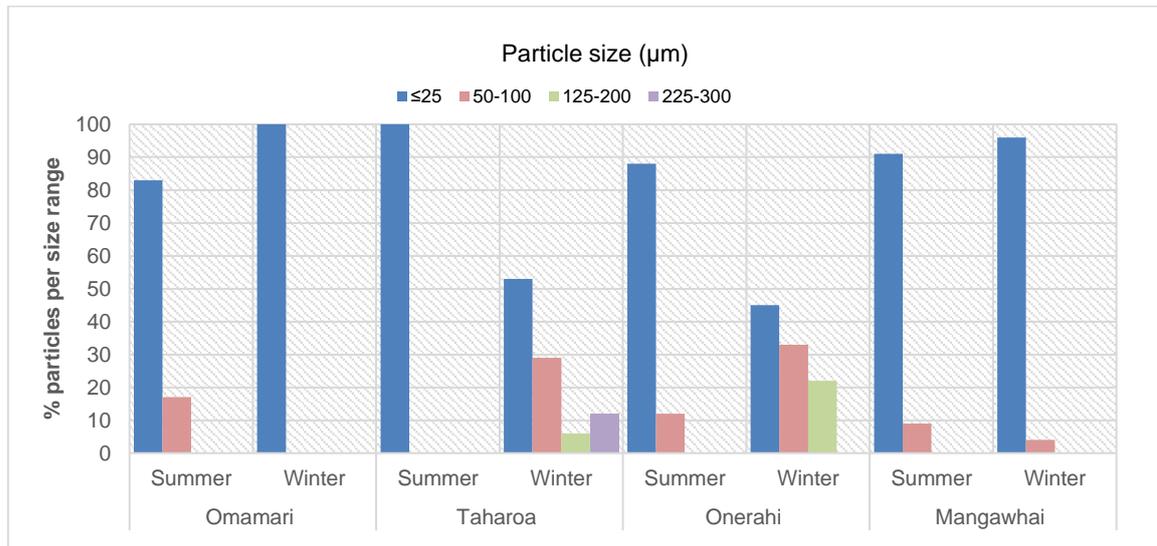
Figure 6. *Size distribution of SMPPs identified in sand samples from four sites in summer and winter.*

MICROPLASTICS TRANSPORT MECHANISM

Data suggests that contamination of large microplastics (LMPP) in all Northland sites were evident except for the samples from Onerahi, an estuary site known as downstream of the stormwater and wastewater treatment plant (WWTP) in Whangarei. This was surprising given the abundance of microplastic debris observed at this site during sampling. However, sand samples from this site were high in organic debris which made it difficult to digest via peroxidation. It was acknowledged that the abundance of microplastics in Onerahi was likely underestimated due to the obstructing organic matter during scanning. This inference is supported by the small particle analysis confirming the presence of microplastics with an overall mean abundance of 1293 MP/kg DW during summer and 231 MP/kg DW during winter. Comparing the SMPP result of Onerahi to the rest of the selected sites during summer, its abundance was relatively high with a variety of polymers identified. Potential sources of the contamination were possibly from runoff and/or from the WWTP effluent.

Also noteworthy is the higher number of microplastics in the samples from Ahipara beach from summer (1 MP/kg DW) to winter (6 MP/kg DW) which can be explained by the heavy rain event before the sampling during winter. It is likely that microplastics were deposited from the nearby creek during the heavy rain.

Lake Taharoa has a relatively low number of large microplastics in both summer and winter. The microplastics found on the lake's shore are composed of 90% fibres attributed to cellulose/regenerated cellulose, polyester, and polyethylene. The remaining 10% were disregarded due to uncertain characteristics. Lake Taharoa has no known natural inlets or outlets and it is believed that 70% of its water is sourced from rainfall [34]. The sources of microplastics in the freshwater lake remain unknown but it is likely that the recreational activities on the lake contributed to the deposition rate.

Moreover, overall data revealed the presence of large particle fibres in 91% of the samples (in both seasons), with an overwhelming fraction (69%) found during summer. Fibres are of concern due to their ability to cycle between the marine environment and atmosphere. Significant portions of cellulose and regenerated cellulose and polyester were commonly seen. The composition is consistent with the quantities used in textiles and common fabrics for clothing. Clothes are known to shed significant amounts of microfibers during normal wear and are directly released into the atmosphere [32], hence this explains that potential emissions from tourists and beachgoers especially during summer possibly contributed to the contamination.

Conclusion

This study revealed that large and small microplastic particles were ubiquitous in the coastal and freshwater sites of the Te Tai Tokerau-Northland region of Aotearoa-New Zealand. Microplastics were found to have consistent polymer characteristics, and were predominantly PP, PE, and PS, which are commonly associated with outdoor, recreational, personal and industrial use. Fibres were the predominant particle morphology observed in sample taken during the summer, however the proportion of fibres was similar to fragments in winter samples. In addition, SMPP were significantly *(p=0)* higher than LMPP, which are likely generated from the physical and chemical fragmentation of larger microplastics in the coastal environment.

Microplastic abundance did not significantly differ between seasons. Seasonal variations also had no significant effect on the type of polymers identified. Meanwhile, microplastic levels were seen to significantly vary across all sites. The east coast also generally had a higher LMPP count compared to the west. Potential contributors of the plastic contamination include marine and domestic land-based activities, water runoffs, airborne transport and atmospheric fallouts.

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Te Aupouri – Niki Conrad

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Appendix A

Sampling location coordinates

	LATITUDE	LONGITUDE
OMAMARI NORTH	-35.8673477	173.662825
OMAMARI SOUTH	-35.8723164	173.66753
ONERAHI NORTH	-35.7666025	174.357334
ONERAHI SOUTH	-35.7670962	174.35752
PAIHIA NORTH	-35.2854109	174.094349
PAIHIA SOUTH	-35.286821	174.096942
RARAWA NORTH	-34.7175429	173.075975
RARAWA SOUTH	-34.7200117	173.079188
SANDY NORTH	-35.5572355	174.473246
SANDY SOUTH	-35.5595612	174.47769
TAHAROA NORTH	-35.8097499	173.661959
TAHAROA SOUTH	-35.8113558	173.661773
WAIPU NORTH	-36.0288554	174.505372
WAIPU SOUTH	-36.0317037	174.508267
WAIPAPAKAURI N	-35.0399015	173.16749
WAIPAPAKAURI S	-35.0432365	173.168516
GLINKS GULLY NORTH	-36.0820297	173.854919
GLINKS GULLY SOUTH	-36.0905891	173.861943
AHIPARA NORTH	-35.1570216	173.15765
AHIPARA SOUTH	-35.1611912	173.15477
MANGAWHAI NORTH	-36.0801013	174.596865
MANGAWHAI SOUTH	-36.0828291	174.598486

Appendix B

Summary of locations provided by Northland regional Council

Site name	Popular activities
Ahipara WC	High Seasonal recreational use (beach access for driving and surfing all year) (WAUC/DC) prevailing wind SW, W
Waipapakauri WC	High Seasonal recreational use (beach access for driving and shellfish collection all year) West Auckland Current/D'Urville Current (WAUC/DC) prevailing wind SW, W
Glinks Gully WC	High Seasonal recreational use (beach access for driving, fishing and shellfish collection) (WAUC/DC) prevailing wind SW, W
Omamari WC	High Seasonal recreational use (beach access for driving and fishing) (WAUC/DC) prevailing wind SW, W
Lake Taharoa	Dune Lake-very popular during summer, High recreational use-Lake, site on west facing shore adjacent to camping and lake access, prevailing wind SW, W
Paihia EC	Semi-suburban, High Rec Use year-round, busy tourist location (EAUC) prevailing wind E, NE
Rarawa EC	High Seasonal Recreational Use, East Auckland Current (EAUC), prevailing wind E, NE
Sandy Bay EC	High Recreational Use (year-round) popular and significant surf break (EAUC) prevailing wind E, NE
Onerahi Upper Whangarei Harbour	Suburban, adjacent to large vessel maintenance facilities and multiple Stormwater outlets, prevailing wind SW, W, E, NE
Waipu Cove Bay EC	High Rec Use (year-round) popular surf break (EAUC) busy Campground adjacent to sites, prevailing wind E, NE, SW
Mangawhai EC	High Rec Seasonal Use, popular surf break (EAUC) busy swimming/surfing beach, prevailing wind E, NE, SW

EC - East coast
WC - West coast

Appendix C

Large microplastic particles: Mean Concentrations per Site (MP/kg DW, MP/m^2, MP/m^3)

Site Name	Zone	Summer			Winter		
		MP / kg DW	MP / m^2	MP / m^3	MP / kg DW	MP / m^2	MP / m^3
Ahipara North	West	1 ± 1	56 ± 98	1127 ± 1951	6 ± 6	369 ± 385	7386 ± 7708
Ahipara South	West	nd	nd	nd	4 ± 5	295 ± 363	5904 ± 7254
Waipapakauri North	West	3 ± 3	256 ± 205	5119 ± 4095	3 ± 4	225 ± 255	4493 ± 5103
Waipapakauri South	West	5 ± 4	356 ± 251	7118 ± 5029	2 ± 0	171 ± 2	3418 ± 43
Glinks Gully North	West	2 ± 1	107 ± 98	2142 ± 1956	2 ± 4	182 ± 315	3641 ± 6307
Glinks Gully South	West	5 ± 6	203 ± 237	4057 ± 4730	nd	nd	nd
Omamari North	West	3 ± 1	217 ± 82	4344 ± 1642	3 ± 4	242 ± 276	4834 ± 5522
Omamari South	West	7 ± 11	501 ± 787	10029 ± 15744	2 ± 0	183 ± 6	3659 ± 126
Paihia North/ South	East	1 ± 2	88 ± 150	1770 ± 3003	6 ± 1	371 ± 107	7416 ± 2138
Paihia North/ South	East	nd	nd	nd	3 ± 1	217 ± 95	4332 ± 1896
Rarawa North	East	2 ± 2	182 ± 178	3644 ± 3557	2 ± 2	159 ± 152	3179 ± 3034
Rarawa South	East	2 ± 3	122 ± 211	2434 ± 4216	2 ± 2	174 ± 177	3485 ± 3534
Sandy North	East	8 ± 10	629 ± 762	12578 ± 15249	9 ± 6	697 ± 469	13939 ± 9379
Sandy South	East	7 ± 4	516 ± 312	10315 ± 6230	2 ± 1	235 ± 203	4692 ± 4063
Waipu North	East	1 ± 1	56 ± 98	1129 ± 1956	12 ± 7	814 ± 432	16285 ± 8646
Waipu South	East	nd	nd	nd	3 ± 1	213 ± 90	4251 ± 1798
Mangawhai North	East	7 ± 4	486 ± 275	9712 ± 5496	7 ± 4	530 ± 298	10605 ± 5952
Mangawhai South	East	3 ± 1	237 ± 102	4746 ± 2048	10 ± 2	686 ± 156	13715 ± 3112
Onerahi (North)	East	nd	nd	nd	1 ± 1	57 ± 100	1149 ± 1991
Onerahi (South)	East	nd	nd	nd	1 ± 1	60 ± 104	1197 ± 2073
Taharoa North	West	1 ± 1	68 ± 118	1365 ± 2364	nd	nd	nd
Taharoa South	West	1 ± 1	65 ± 112	1297 ± 2246	1 ± 1	66 ± 114	1319 ± 2285

ND- none detected

Appendix D

A. Statistical Results (LMPP)

1. ONE WAY ANOVA – NORTHLAND SITES

NORTH VS SOUTH
One-way ANOVA: MP/kg DW versus Location

Method

Null hypothesis	All means are equal
Alternative hypothesis	Not all means are equal
Significance level	α = 0.05

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
Location	11	Ahipapara, Glinks, mangawhai, Omamari, Onerahi, Paihia, Rarawa, Sandy, Taharoa, Waipapakauri, Waipu

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Location	10	538.8	53.88	3.29	0.001
Error	137	2240.7	16.36		
Total	147	2779.4			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
4.04417	19.38%	13.50%	5.84%

Means

Location	N	Mean	StDev	95% CI
Ahipapara	12	2.71	4.32	(0.40, 5.02)
Glinks	16	2.499	3.975	(0.500, 4.499)
mangawhai	12	6.66	3.59	(4.36, 8.97)
Omamari	16	4.21	6.17	(2.21, 6.21)
Onerahi	16	0.312	0.854	(-1.687, 2.312)
Paihia	12	2.915	2.574	(0.607, 5.224)
Rarawa	12	2.253	2.204	(-0.056, 4.561)
Sandy	12	6.46	6.16	(4.15, 8.77)
Taharoa	12	0.625	1.130	(-1.684, 2.933)
Waipapakauri	16	3.749	2.886	(1.750, 5.748)
Waipu	12	4.17	5.96	(1.86, 6.47)

Pooled StDev = 4.04417

Boxplot of MP/kg DW
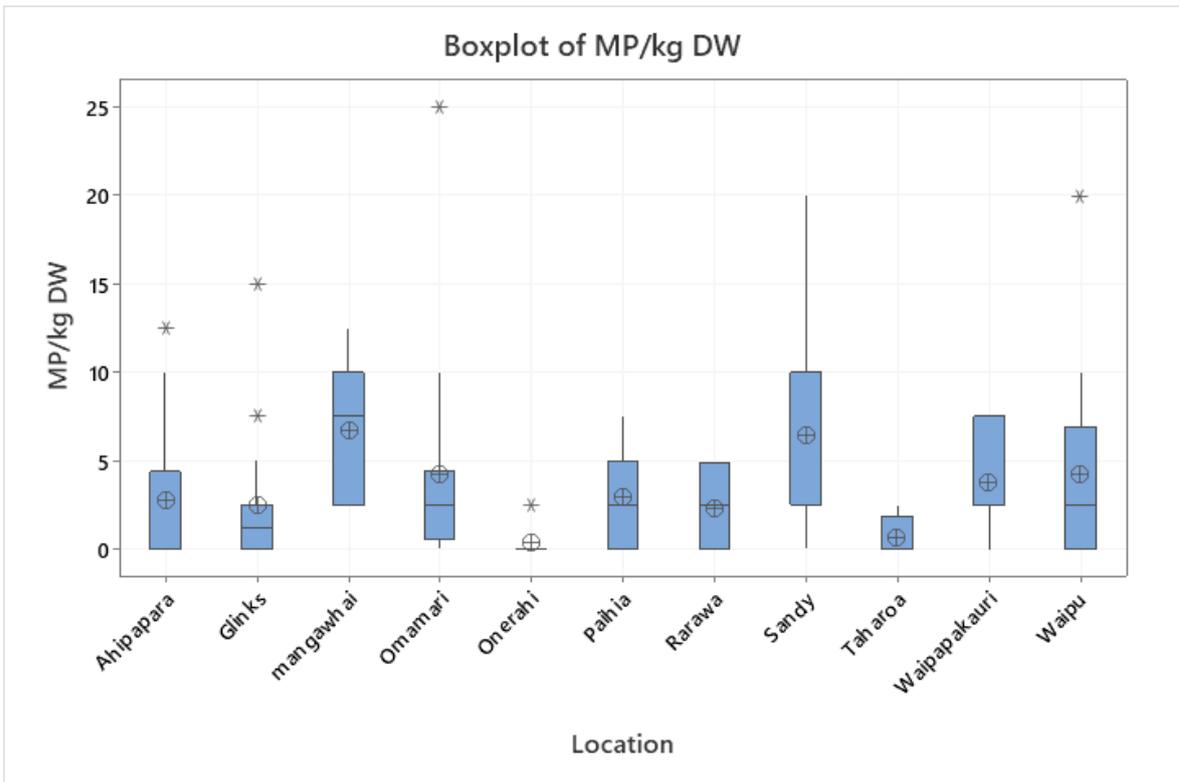
T-TEST COMPARISONS

2A. Summer vs Winter

Method

Null hypothesis	All means are equal
Alternative hypothesis	Not all means are equal
Significance level	α = 0.05

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
Season	2	Summer, Winter

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Season	1	37.61	37.61	2.00	0.159
Error	146	2741.83	18.78		
Total	147	2779.43			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
4.33355	1.35%	0.68%	0.00%

Means

Season	N	Mean	StDev	95% CI
Summer	82	2.803	4.397	(1.858, 3.749)
Winter	66	3.817	4.253	(2.763, 4.872)

Pooled StDev = 4.33355

Interval Plot of MP / kg DW vs Season
95% CI for the Mean

The pooled standard deviation is used to calculate the intervals.
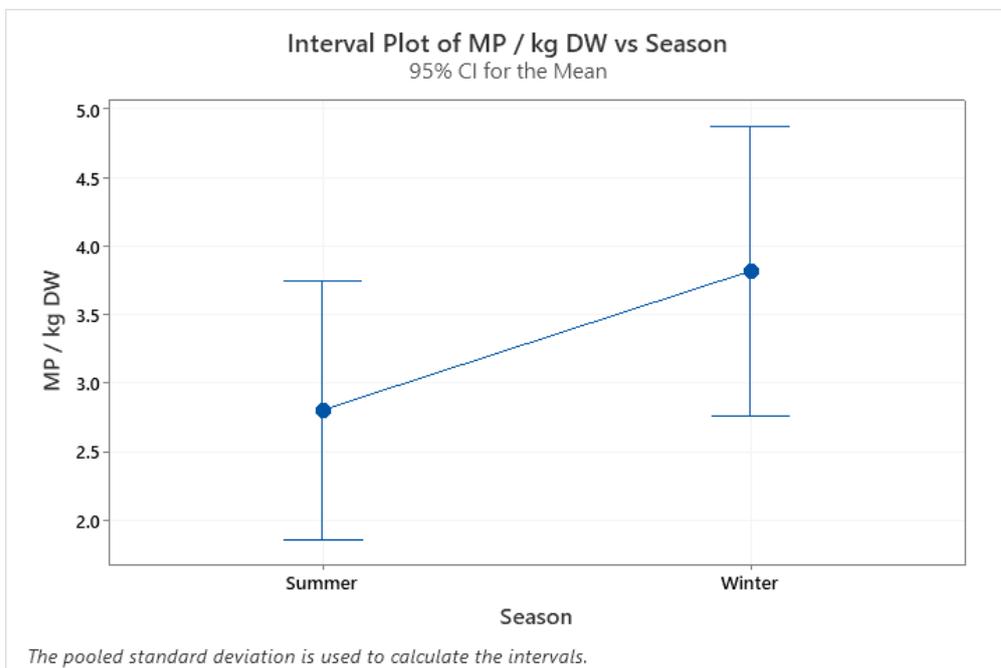
2B. East vs West (excluding Taharoa and Onerahi)

Method

Null hypothesis   All means are equal
Alternative hypothesis   Not all means are equal
Significance level   α = 0.05

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
Coast	2	East, West

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Coast	1	40.34	40.34	1.95	0.165
Error	118	2440.90	20.69		
Total	119	2481.24			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
4.54814	1.63%	0.79%	0.00%

Means

Coast	N	Mean	StDev	95% CI
East	60	4.491	4.642	(3.328, 5.654)
West	60	3.331	4.452	(2.169, 4.494)

Pooled StDev = 4.54814
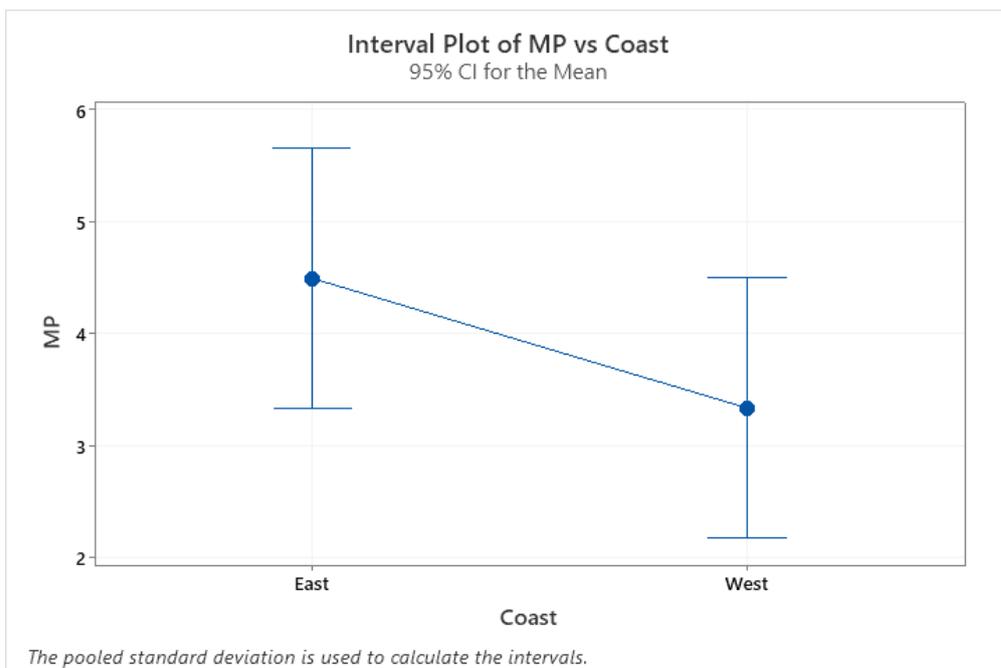
2C. North vs South

One-way ANOVA: MP/kg DW versus Site

Method

Null hypothesis	All means are equal
Alternative hypothesis	Not all means are equal
Significance level	α = 0.05
Rows unused	6

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
Site	2	North, South

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Site	1	10.99	10.99	0.57	0.453
Error	140	2721.42	19.44		
Total	141	2732.41			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
4.40893	0.40%	0.00%	0.00%

Means

Site	N	Mean	StDev	95% CI
North	71	3.619	4.485	(2.584, 4.653)
South	71	3.062	4.332	(2.028, 4.097)

Pooled StDev = 4.40893

2C. Auckland vs. Northland

Two-Sample T-Test and CI: MP/kg DW, Region
Method

μ_1: population mean of MP/kg DW when Region = Auckland
μ_2: population mean of MP/kg DW when Region = Northland
Difference: $\mu_1 - \mu_2$

Equal variances are not assumed for this analysis.

Descriptive Statistics: MP/kg DW

Region	N	Mean	StDev	SE Mean
Auckland	55	6.03	7.20	0.97
Northland	148	3.26	4.35	0.36

Estimation for Difference

Difference	95% CI for Difference
2.77	(0.71, 4.83)

Test

Null hypothesis	H_0: $\mu_1 - \mu_2 = 0$
Alternative hypothesis	H_1: $\mu_1 - \mu_2 \neq 0$

T-Value	DF	P-Value
2.68	69	0.009

Boxplot of MP/kg DW
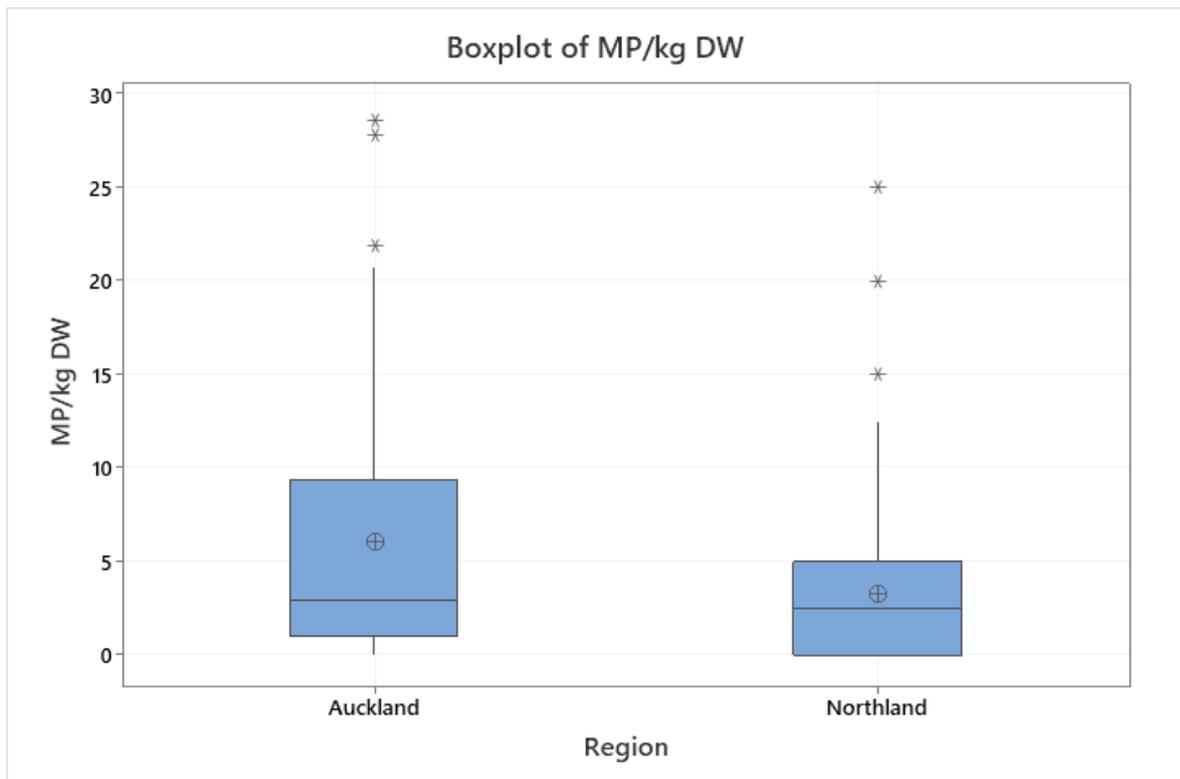
B. Statistical Results (SMPP)

One-way ANOVA: Concentration versus Site

Method

Null hypothesis	All means are equal
Alternative hypothesis	Not all means are equal
Significance level	α = 0.05

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
Site	4	Mangawhai, Omamari, Onerahi, Taharoa

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Site	3	26961067	8987022	2.81	0.050
Error	44	140806981	3200159		
Total	47	167768048			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1788.90	16.07%	10.35%	0.12%

Means

Site	N	Mean	StDev	95% CI
Mangawhai	12	2170	3213	(1129, 3211)
Omamari	12	202	364	(-839, 1243)
Onerahi	12	697	1019	(-344, 1737)
Taharoa	12	575	1144	(-466, 1616)

Pooled StDev = 1788.90

WORKSHEET 1
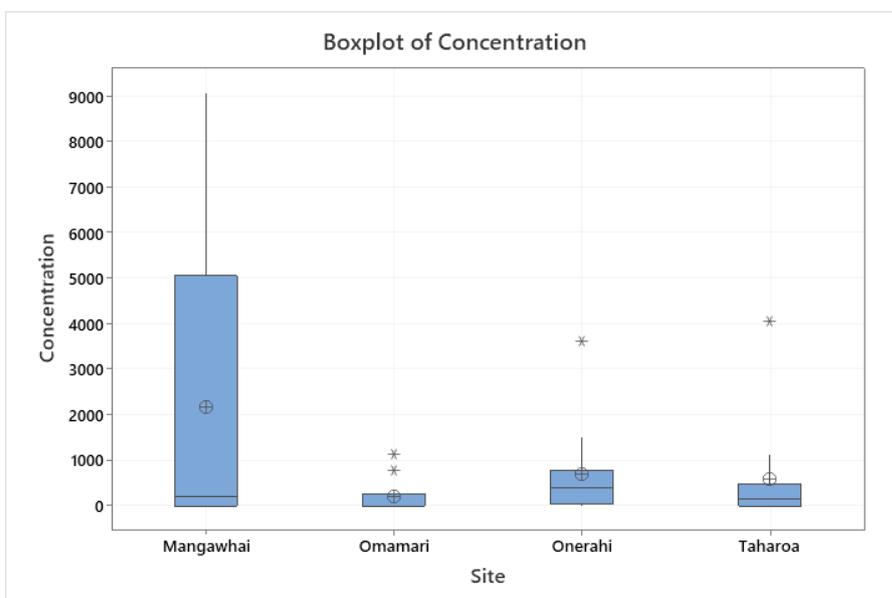
T- test comparison: Concentration vs. Season

Method

μ_1: population mean of Concentration when Season = Summer
μ_2: population mean of Concentration when Season = Winter
Difference: $\mu_1 - \mu_2$

Equal variances are not assumed for this analysis.

Descriptive Statistics: Concentration

Season	N	Mean	StDev	SE Mean
Summer	24	1417	2290	467
Winter	24	405	1232	251

Estimation for Difference

Difference	95% CI for Difference
1013	(-65, 2090)

Test

Null hypothesis	H_0: $\mu_1 - \mu_2 = 0$
Alternative hypothesis	H_1: $\mu_1 - \mu_2 \neq 0$

T-Value	DF	P-Value
1.91	35	0.065

Boxplot of Concentration
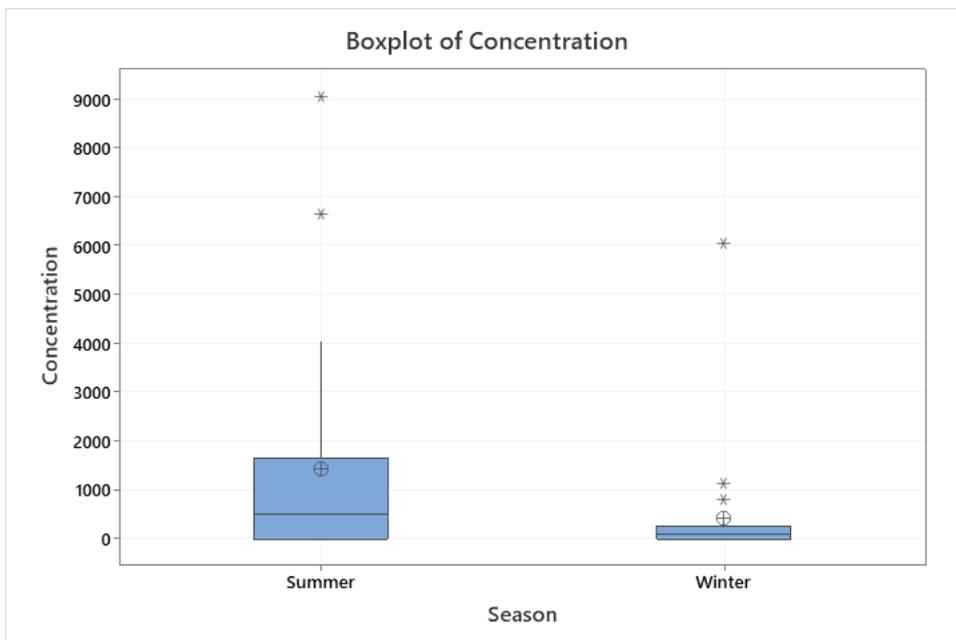
Two-Sample T-Test and CI: Concentration vs. sampling location

One-way ANOVA: MP/kg DW versus Site

Method

Null hypothesis	All means are equal
Alternative hypothesis	Not all means are equal
Significance level	α = 0.05

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
Site	2	North, South

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Site	1	10.99	10.99	0.57	0.453
Error	140	2721.42	19.44		
Total	141	2732.41			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
4.40893	0.40%	0.00%	0.00%

Means

Site	N	Mean	StDev	95% CI
North	71	3.619	4.485	(2.584, 4.653)
South	71	3.062	4.332	(2.028, 4.097)

Pooled StDev = 4.40893

Boxplot of MP/kg DW