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OPTIMISATION OF A METHOD TO QUANTIFY  
MICROPLASTICS IN INTER-TIDAL SEDIMENTS  
AROUND JERSEY, CHANNEL ISLANDS

by

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A dissertation submitted in partial fulfilment of the requirements for the  
degree of M.Sc. Oceanography by instructional course.

## DECLARATION

As the nominated University supervisor of this M.Sc. project by Hannah Brittain, I confirm that I have had the opportunity to comment on earlier drafts of the report prior to submission of the dissertation for consideration of the award of M.Sc. Oceanography.

Signed.....

Supervisor's name: XXXXXXXXXX

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## ABSTRACT

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Microplastics are microscopic pieces of plastic between 1  $\mu\text{m}$  – 5 mm. They are an emerging threat to marine environments worldwide, occurring primarily through degradation of larger items of plastic. A number of adverse effects have been documented in marine species following exposure to microplastics, so it is important to monitor microplastic concentrations in the marine environment to assess potential impacts to marine ecosystems and commercial fisheries. In an attempt to address the current lack of consensus on standardised and robust methods for microplastics quantification, this study aimed to optimise a method to extract microplastics from sediment samples. A method that had been proposed in the literature with promising preliminary results was selected, then several adaptations were experimentally applied to optimise the method. An amended method was finalised, involving three steps; 1. Volume reduction via elutriation; 2. Extraction of microplastics via floatation and 3. Visual sorting using a dissection microscope. The method was applied to intertidal samples from beaches around Jersey, Channel Islands, which had not, to date, been quantified for microplastic contamination. A microplastic profile was catalogued using visual sorting under a dissection microscope, based on size, shape and colour of individual particles observed. Microplastic profiles for West and East Jersey beaches were similar. Fragments were the most common shape, and brown and black were the most common two colours observed across both sites. However, the method had a low extraction efficiency of 31 %, which varied across size, shape and polymer type, so the profiles observed are not likely to be fully representative of microplastics in the environment. A number of additional method limitations were identified, including an especially poor extraction efficiency for microplastics > 1 mm (22 %), background contamination in the laboratory, several potential loss steps, and the inability to confirm the synthetic polymer origin of particles resembling microplastics. Suggested improvements were provided to avoid similar limitations in future work. Overall these findings highlight the implicit variance in microplastics data and substantiate the importance of clean laboratory spaces and standardised methods for the quantification of microplastics.

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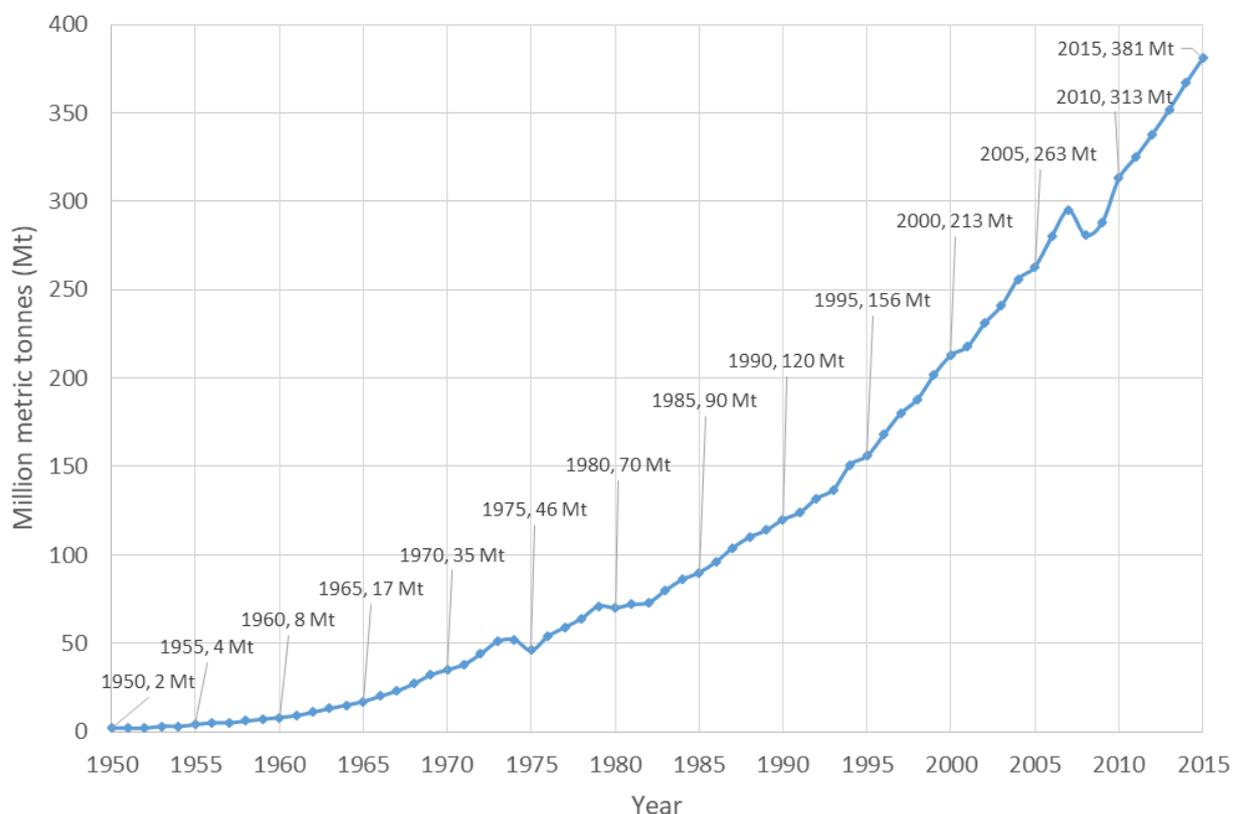
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# 1 INTRODUCTION

'Plastics' are synthetic materials composed of many recurring smaller molecules, also known as synthetic polymers (Crawford and Quinn, 2016). Plastics are manufactured from organic and inorganic raw materials (i.e. carbon, silicon, hydrogen, oxygen and chloride) which are typically extracted from oil, coal and natural gas (Shah et al., 2008). The first modern plastic material, Bakelite (chemical name: polyoxybenzyl methylene glycol anhydride), was developed in 1907 (Cole et al., 2011). Soon after this, manufacturing techniques were developed through the 1940s to allow for the mass-production of plastics. Plastic production has increased exponentially since 1950 (Figure 1.1), with an estimated 381 million metric tonnes (Mt) produced in 2015, compared to 2 Mt in 1950; an almost 200-fold increase within 65 years (Geyer et al., 2017). There are many different types of plastic in existence today, with a range of useful properties. Some common properties of plastics include durability, malleability, low thermal conductivity, high strength to weight ratio and biological inertness (Andrady, 2011). Ultimately, plastic materials have changed the way we live, and have become universal to industries and everyday domestic settings alike.

With an exponential increase in production, waste plastic has also accrued at a similar rate, with around 10 % of this waste entering the ocean every year (Barnes et al., 2009).



**Figure 1.1** Annual global plastic production from 1950 – 2015 in million metric tonnes (Mt) (Geyer et al., 2017).

The majority of marine plastic debris originates from land-based sources (80 %), with plastic waste being generated primarily from densely populated and industrialised areas (Li et al., 2016). The other 20 % of plastic debris in the marine environment is ocean-based, originating primarily from commercial fishing activities. The first reports of plastics within marine debris date back to the 1970s (Buchanan, 1971; Carpenter and Smith Jr., 1972; Colton et al., 1974; Gregory, 1978). These studies did not garner much attention from the scientific community at the time. However, evidence mounted in the following years of a variety of ecological consequences posed by plastic marine debris, such as entanglement of large marine animals, such as turtles, in larger pieces of plastic debris (Barnes et al., 2009; Gall and Thompson, 2015). Using worldwide data on waste and population statistics, Jambeck et al. (2015) estimated that 4.8–12.7 million metric tonnes of plastic waste from the land entered the marine environment in 2010 alone, with further increases expected as plastic demand increases. The use of 'single-use', disposable plastic products, such as straws and cups, has exacerbated the problem of plastic waste by increasing the rate at which plastic becomes waste material (Ivar Do Sul and Costa, 2014).

More recently, the focus of academics has shifted towards the arguably more insidious issue of microplastics in the marine environment (GESAMP, 2015). Microplastics are microscopic pieces of plastic, between 1  $\mu\text{m}$  – 5 mm across their widest diameter (Germanov et al., 2018). Primary microplastics are deliberately manufactured at a microscopic size (Boucher and Friot, 2017). This includes industrial pellets, which are used to manufacture plastic products (Gregory, 1983), and microbeads, which have been used widely in cosmetics products such as toothpaste and facial scrubs (Andrady, 2011). Analysis of outfall water has indicated that microbeads from cosmetics are able to enter the environment via wastewater treatment plants (Murphy et al., 2016). Microbeads are currently being phased out in cosmetics in the UK following the introduction of new legislation proposing a microbead ban in 2017 (Draft Statutory Instruments, 2017). Secondary microplastics are more common than primary microplastics in the marine environment, and occur as a result of degradation of larger plastic items (mesoplastics and macroplastics) via chemical and physical processes (Sundt et al., 2014). In the marine environment, the predominant processes, resulting in macroplastic degradation into microplastics, include physical weathering through wave action and solar UV photodegradation (Li et al., 2016). Secondary microplastic fibres have also been found to leach from clothing during wash cycles, with a single garment being able to produce >1900 fibres per wash (Browne et al., 2011).

## 1.1 GLOBAL SIGNIFICANCE AND IMPACTS OF MARINE MICROPLASTIC POLLUTION

Microplastics have been labelled as an environmental contaminant of concern, with a number of recorded impacts on marine species (Teuten et al., 2009; Wright et al., 2013). These impacts can be caused by microplastics as a pollutant in its own right, including changes in behaviour, gene expression or physiological function following the ingestion of microplastics by various marine species. For example, an exposure experiment by Sussarellu et al. (2016) indicated that exposing the Pacific Oyster (*Crassostrea gigas*) to microplastics for 2 months, at environmentally realistic concentrations, resulted in a reduction in feeding, gamete quality and fecundity via ingestion.

There are also indirect impacts caused by microplastics in the marine environment. This includes the ability of microplastics to absorb a range of persistent organic pollutants (POPs) onto their surface, such as polychlorinated biphenyls (PCBs), which are toxic to most marine organisms at high doses and associated with reduced fecundity at lower doses (Teuten et al., 2009). For example, a study by Besseling et al. (2013) found that weight loss and bioaccumulation of PCBs occurred in polychaetes (*Arenicola marina*) following the ingestion of microplastic particles laced with PCBs. In addition, it has been hypothesized that POPs accumulate in megafauna (i.e. mobulid rays, whale sharks and baleen whales), through the indiscriminate filter feeding of water containing microplastics that have absorbed POPs (Germanov et al., 2018). Environmental observations supporting this theory include the presence of plastic additives and POPs in samples of basking shark muscle, fin whale blubber and whale shark skin (Fossi et al., 2017, 2014, 2012). Potential impacts to megafauna include altered reproductive fitness, endocrine disruption and general disruption to biological processes (Germanov et al., 2018). Another impact that has been hypothesised is that biofilms which form on microplastics could play host to harmful bacteria such as *Vibrio* spp. which are capable of harbouring putative oyster pathogens (Frère et al., 2018; Harrison et al., 2014; Kirstein et al., 2016; Zettler et al., 2013). There is also growing concern for microplastics becoming a threat to human health, through trophic transfer of microplastics and absorbed POPs to commercial species (Farrell and Nelson, 2013; Van Cauwenberghe and Janssen, 2014).

Considered in the light of their persistence in the marine environment, the impacts of microplastics are a pervasive threat to all marine environments. Microplastics are ubiquitous to marine environments globally (Eriksen et al., 2014; Germanov et al., 2018). They have been detected throughout the water column and sediments worldwide, and also within many marine organisms and seabirds (Andrady, 2011; Wright et al., 2013).

Lower density microplastics (specific gravity  $< 1 \text{ g cm}^{-3}$ ), such as expanded polystyrene/Styrofoam (EPS), tend to be positively buoyant in seawater. These microplastics can therefore be transported thousands of miles via surface waters from their source location due to oceanic and wind-driven currents (Baztan et al., 2014). Eriksen et al. (2014) estimate that there are 5.25 trillion plastic particles currently floating in the oceans, equivalent to 268,940 tonnes, with microplastics contributing 92.4 % by number of particles and 13.2 % by weight. Subtropical gyres in particular are known to be regions where microplastics accumulate due to oceanic currents (Cozar et al., 2014; Eriksen et al., 2013b; Moore et al., 2001). In addition, deep sea sediments have been hypothesised as a major sink for higher density microplastics (Van Cauwenberghe et al., 2013b; Woodall et al., 2014). Microplastics are also prone to sinking due to biological interactions with fouling fauna and slow sinking aggregates (Kaiser et al., 2017; Long et al., 2015). It is hypothesized that coastal transport of microplastics, which regulates their spatial and temporal distribution, is a major controlling process in the environmental fate and risks posed to marine species by microplastics (Zhang, 2017).

## **1.2 RECORDED CONCENTRATIONS IN COASTAL SEDIMENTS**

Microplastics are present in marine sediments worldwide and have been found to accumulate in coastal regions (Zhang, 2017). A summary of recorded concentrations of microplastics in coastal sediments is provided in Table 1.1. This covers a range of locations around the world, but is by no means an exhaustive list. Research quantifying microplastics in sediment has been primarily focused on intertidal and littoral zones of beaches. Table 1.1 includes over 30 examples of beach-focused studies, spanning the continents of Africa, America, Asia and Europe (Baztan et al., 2014; Ivar do Sul et al., 2009; Kaberi et al., 2013; Ng and Obbard, 2006). Other coastal environments that have been quantified for sediment microplastic concentrations include mangroves, estuaries, harbours and subtidal bays (Claessens et al., 2011; Fok and Cheung, 2015; Mohamed Nor and Obbard, 2014; Vianello et al., 2013).

The field of microplastics research is relatively new, with the majority of key papers published within the last decade. As such, there has been a lack of consensus in the literature, as the field has developed, with regards to standardised measurement units for microplastic concentrations and the size range for microplastics (Hidalgo-Ruz et al., 2012). This has led to a range of literature results that are difficult to compare directly with one another, on account of the various units of measurement and size ranges documented (Van Cauwenberghe et al., 2015b).

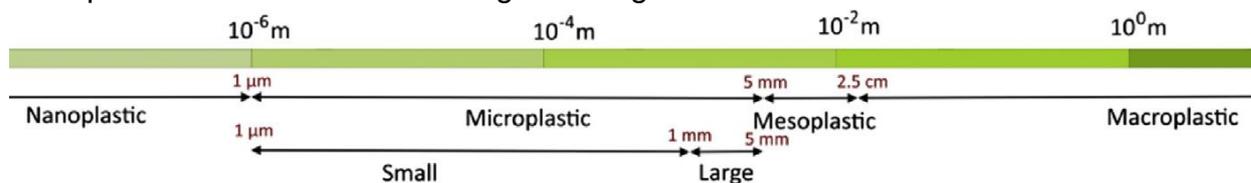
The most commonly used units of measurement for microplastic concentration in sediments were microplastics per square metre (MP m<sup>-2</sup>), typically reported in studies which used quadrants to sample an area for abundance per unit of surface (Table 1.1). Other commonly used units include microplastics per kilogram of dry sediment (MP kg<sup>-1</sup> DW), and microplastics per litre of sediment (MP L<sup>-1</sup>). Sediment samples are likely to

Continent	Location	Specific location	Size range	Predominant Type	Concentration	Reference	
Africa	Canary Islands	Beach	1 mm – 5 mm	Fragment, pellets	< 1 – 109 g L <sup>-1</sup>	Baztan et al., 2014	
	South Africa	Beach	65 µm – 5 mm	Fibres 90%	688.9 – 3308 MP m <sup>-2</sup>	Nel and Froneman, 2015	
America	Canada	Beach	< 1 mm – > 5 mm	PE predominant	< 10 MP m <sup>-2</sup>	Gregory, 1983	
	Bermuda	Beach	< 1 mm – > 5 mm	PE predominant	> 5000 MP m <sup>-2</sup>		
	Hawaii	Beach	1 mm – 4.75 mm	Fragment	43.4 MP L <sup>-1</sup>	McDermid and McMullen, 2004	
	US	Florida, subtidal		250 µm – 4 mm	Fragment	116 – 215 MP L <sup>-1</sup>	Graham and Thompson, 2009
		Maine, subtidal		250 µm – 4 mm	Fragment	105 MP L <sup>-1</sup>	
	Brazil	Noronha, Beach	2 mm – 5 mm	Fragment 65%	15 MP kg <sup>-1</sup>	Ivar do Sul et al., 2009	
	Hawaii	Ka Milo, Beach	250 µm – 4 mm	PE 85%	211.8 MP m <sup>-3</sup>	Carson et al., 2011	
	Chile	Beach	1 mm – 4.75 mm	Fragment 89%	27 MP m <sup>-2</sup>	Hidalgo-Ruz and Thiel, 2013	
	Canada	Nova Scotia, Beach	0.8 µm – 5 mm	Plastic fibres	2000 – 8000 MP kg <sup>-1</sup>	Mathalon and Hill, 2014	
	Brazil	Beach	47 µm – 5 mm	Fibres, fragments	12 – 1300 MP m <sup>-2</sup>	de Carvalho and Neto, 2016	
	Gulf of Mexico	Marine-dominated		200 µm – 5 mm	Fibres, fragments	50.6 MP m <sup>-2</sup>	Wessel et al., 2016
		Freshwater-dominated		200 µm – 5 mm	Fibres, fragments	13.2 MP m <sup>-2</sup>	
	Asia	Oman Gulf	Beach	2 mm – 5 mm	PE, pellets	> 100 MP m <sup>-2</sup>	Khordagui and Abu- Hilal, 1994
Arabian Gulf		Beach	2 mm – 5 mm	PE, pellets	< 80,000 MP m <sup>-2</sup>		
Japan		Beach	2 mm – 5 mm	Fragment 41%	8 – 17 MP m <sup>-2</sup>	Kusui and Noda, 2003	
Singapore		Beach	1.6 µm – 5 mm	PE and PS	< 3 MP kg <sup>-1</sup> DW	Ng and Obbard, 2006	
India		Ship-breaking yard	1.6 µm – 5 mm	Fragment 100%	81.4 mg kg <sup>-1</sup>	Reddy et al., 2006	
India		Beach		1 mm – 5 mm	Fragment	68.8 MP m <sup>-2</sup>	Jayasiri et al., 2013
		Beach dry season		1 mm – 5 mm	PS expanded >96%	8205 MP m <sup>-2</sup>	Lee et al., 2013
		Beach rainy season		1 mm – 5 mm	PS expanded >96%	27,606 MP m <sup>-2</sup>	
Singapore		Mangrove	1.6 µm – 5 mm	PE, PP, nylon & PVC	36.8 MP kg <sup>-1</sup> DW	Mohamed Nor and Obbard, 2014	
South Korea		Beach	50 µm – 5 mm	PS expanded	56 – 285,673 MP m <sup>-2</sup>	Kim et al., 2015	
Hong Kong		Pearl River estuary	315 µm – 5 mm	PS expanded 92%	5595 MP m <sup>-2</sup>	Fok and Cheung, 2015	
New Zealand		Beach	1 mm – 5 mm	PE and PP	> 1000 MP m <sup>-2</sup>	Gregory, 1978	
Europe		Russia	Beach	2 mm – 5 mm	Fragment 55.6%	5 – 10 MP m <sup>-2</sup>	Kusui and Noda, 2003
	UK	Beach		1.6 µm – 5 mm	Fibres	8 MP L <sup>-1</sup>	Thompson et al., 2004
		Estuary		1.6 µm – 5 mm	Fibres	48 MP L <sup>-1</sup>	
		Subtidal		1.6 µm – 5 mm	Fibres	112 MP L <sup>-1</sup>	
	Sweden	Subtidal	80 µm – 5 mm	Fibres	20 – 3320 MP L <sup>-1</sup>	Norén, 2007	
	UK	Tamar estuary	1.6 µm – 1 mm	PVC 26%; PE 35%	< 8 – 413 MP L <sup>-1</sup>	Browne et al., 2010	
	UK	North Sea beach		38 µm – 1 mm	Fibres	4 – 16 MP L <sup>-1</sup>	Browne et al., 2011
		English Chl. beach		38 µm – 1 mm	Fibres	8 – 20 MP L <sup>-1</sup>	
		Subtidal		38 µm – 1 mm	Fibres	112 MP L <sup>-1</sup>	
	Belgium	Harbour		38 µm – 1 mm	Fibres 59%	166.7 MP kg <sup>-1</sup> DW	Claessens et al., 2011
		Beach		38 µm – 1 mm	Fibres 59%	92.8 MP kg <sup>-1</sup> DW	
	Portugal	Beach	1.2 µm – 5 mm	PE, Polyester, PS	133.3 MP m <sup>-2</sup>	Martins and Sobral, 2011	
	Malta	Beach	1.9 mm – 5.6 mm	PE, pellets	> 1000 MP m <sup>-2</sup>	Turner and Holmes, 2011	
Italy	Venice, subtidal	0.7 µm – 1 mm	PE + PP 82%	672–2175 MP kg <sup>-1</sup> DW	Vianello et al., 2013		
Germany	Tidal flat		1.2 µm – 5 mm	Granules	210 MP kg <sup>-1</sup>	Liebezeit and Dubaish, 2012	
	Tidal flat		1.2 µm – 5 mm	Fibres	461 MP kg <sup>-1</sup>		
Greece	Beach		1 mm – 2 mm	Fragment 68%	57 – 602 MP m <sup>-2</sup>	Kaberi et al., 2013	
	Beach		2 mm – 4 mm	Pellets	10 – 575 MP m <sup>-2</sup>		
Belgium	Low tide line		38 µm – 1 mm	Granules, fibres	9.2 MP kg <sup>-1</sup> DW	Van Cauwenberghe et al., 2013a	
	High tide line		38 µm – 1 mm	Fibres, granules	17.6 MP kg <sup>-1</sup> DW		
Germany	Beach	< 1 mm	PP, PE, PET	1.3 – 2.3 MP kg <sup>-1</sup> DW	Dekiff et al., 2014		
Slovenia	Beach		0.25 mm – 5 mm	Fibres, fragments	177.8 MP kg <sup>-1</sup> DW	Laglbauer et al., 2014	
	Infralittoral		0.25 mm – 5 mm	Fibres, fragments	170.4 MP kg <sup>-1</sup> DW		
North Sea	Beach	35 µm – 1 mm	LDPE, HDPE and PS	0.3 – 11.7 MP kg <sup>-1</sup>	Van Cauwenberghe et al., 2015		
France	Subtidal	207 µm – 2 mm	PE 53.3%	0.97-MP kg <sup>-1</sup> DW	Frère et al., 2017		
Scotland	Beach		0.7 µm – 5 mm	Fibres	2300 MP kg <sup>-1</sup> DW	Blumenröder et al., 2017	
	Beach		0.7 µm – 5 mm	Particles	730 MP kg <sup>-1</sup> DW		

**Table 1.1** Worldwide environmental concentrations of microplastics detected in coastal sediments. Sampling continent, location, specific location, and size range, morphology and/or polymer and concentration of microplastics are listed with their corresponding studies. MP = microplastics (i.e. number of fragments, microbeads, pellets, fibres, foams or films); DW = dry weight (of sediment). Plastic polymer types: PS = Polystyrene; (HD/LD)PE = (High Density/Low Density) Polyethylene; (U)PVC = (Un-plasticised) Polyvinyl chloride; PP = Polypropylene; PET = Polyethylene terephthalate.

contain different water content depending on temporal and spatial variables (i.e. location on the beach, whether it was collected immediately before or after a high tide) and sediment porosity (Van Cauwenberghe et al., 2015b). For this reason, a number of authors have chosen to dry sediment samples before analysis, to remove water content as a variable and allow for a more consistent comparison of data, using units of MP kg<sup>-1</sup> DW (Claessens et al., 2011; Dekiff et al., 2014; Frère et al., 2017; Laglbauer et al., 2014; Mohamed Nor and Obbard, 2014; Ng and Obbard, 2006; Van Cauwenberghe et al., 2015a, 2013a; Vianello et al., 2013). This study also elected to use MP kg<sup>-1</sup> DW for microplastics concentration measurements.

Figure 1.2 shows the standard nomenclature for plastic debris in the environment, including the now largely accepted size range for microplastics, 1 – 5 mm (MSFD GES Technical Subgroup on Marine Litter, 2013). The most common two size ranges used to quantify microplastics in the environment are < 1 mm and 1 – 5 mm, therefore it has been suggested that microplastics are split into two categories to reflect this (Figure 1.2). The concentrations of large microplastics (2 – 5 mm) reported for Japanese beaches (8 – 17 MP m<sup>-2</sup>), Russian beaches (5 – 10 MP m<sup>-2</sup>) and Noronha, Brazil (15 MP m<sup>-2</sup>) were low, compared to other studies reporting in the same units (Ivar do Sul et al., 2009; Kusui and Noda, 2003) (Table 1.2). Generally, studies that considered a size range encompassing smaller microplastics, reported much higher concentrations present in the environment. For example, Lee et al. (2013) reported concentrations of large microplastics (1 – 5 mm) at 8,205 and 27,606 MP m<sup>-2</sup> in South Korea beach sediments during the dry and rainy seasons, respectively. A later study from Kim et al. (2015) reported particularly high microplastic concentrations of up to 285,673 MP m<sup>-2</sup> on South Korea beaches. However, the size range of microplastics considered in this later paper was 50 µm – 5 mm, thereby including an additional size range between 50 µm – 1 mm not covered by Lee et al. (2013). Other studies considered only small microplastics (< 1 mm), including a number of European studies, which typically consider a size range of 38 µm – 1 mm for microplastics in sediment (Browne et al., 2011; Van Cauwenberghe et al., 2015a, 2013a). With these differences in reporting in mind, this study sought to quantify a size range of microplastics in sediments covering both large and small size fractions.



**Figure 1.2** Plastic debris nomenclature based on size, including microplastics, as proposed by the European MSFD Technical Subgroup on Marine Litter (2013). Microplastics are further split into two size categories; small microplastics (1 µm – 1 mm) and large microplastics (1 – 5 mm), to differentiate between two commonly used size ranges of microplastics in literature. Adapted from Van Cauwenberghe et al. (2015b).

### 1.3 EXISTING METHODS TO EXTRACT MICROPLASTICS FROM MARINE SEDIMENTS

Several techniques are employed by the scientific community to extract microplastics from sediment samples. For studies focused on intertidal areas of beaches, sediment samples are generally collected using metal implements (i.e. iron spoon or spade) (Van Cauwenberghe et al., 2015b). Following sample collection, a range of methods to extract microplastics from the natural sediment matrix (typically sand) can be used. The majority of these methods use a density separation approach, which utilises the differences in density between plastic and natural sediment particles to isolate microplastics from sediment. One of the simplest and most widely used methods was pioneered by Thompson et al. (2004). This method involves agitating a sediment sample in saturated sodium chloride (NaCl) salt solution to release microplastic particles from the sediment matrix, which float to the surface. However, only microplastics consisting of low density polymers ( $< 1.2 \text{ g cm}^{-3}$ ) are able to be extracted using this method, as common salt solution will not surpass a density of  $1.2 \text{ g cm}^{-3}$ . Therefore higher density polymers will not float to the surface and will remain in the sediment. Subsequent studies have used different types of salt to attain a higher density salt solution and increase the extraction efficiency for higher density polymers, such as polyvinyl chloride (PVC) ( $1.14 - 1.56 \text{ g cm}^{-3}$ ), which comprises 17 % of European plastic demand (PlasticsEurope, 2015). Zinc chloride ( $\text{ZnCl}_2$ ) solution ( $1.5 - 1.8 \text{ g cm}^{-3}$ ) has been used in some studies (Coppock et al., 2017; Liebezeit and Dubaish, 2012) and sodium iodide (NaI) solution ( $1.3 - 1.8 \text{ g cm}^{-3}$ ) has been used in others (Claessens et al., 2013; Coppock et al., 2017; Dekiff et al., 2014; Van Cauwenberghe et al., 2013a). High density microplastics are the first to sink and intersperse with sediments (seawater density is  $1.02 \text{ g cm}^{-3}$ ), therefore it is important that the methods used to analyse sediments are capable of extracting them (Van Cauwenberghe et al., 2015a). One limitation in using different salt solutions is the cost of materials. Coppock et al. (2017) provided estimate costs for NaCl,  $\text{ZnCl}_2$  and NaI solutions of different densities. NaI and  $\text{ZnCl}_2$  solutions ( $1.5 \text{ g cm}^{-3}$ ) were 41.5 and 15.6 costs units, respectively, compared to the standard cost unit for NaCl solution ( $1.2 \text{ g cm}^{-3}$ ). A new method was recently proposed by Claessens et al. (2013), which included a prior step to reduce the overall sample size before performing a floatation with high-density salt solution, similar to the process described above. This involved elutriation, an upward stream of water that separates out lighter particles from denser ones. This volume reduction step allowed for a fraction of high-density salt solution to be used per sample, compared to the standard density separation method, which reduces the cost of required materials significantly. In addition, the extraction efficiency of this new two-step

method was reported by (Claessens et al., 2013) to be more efficient than using the flotation method alone. Claessens et al. (2013) tested the extraction efficiency of their method by using sediments spiked with a known amount of microplastics. Retrieval rates for microplastics were 100 % for microplastic granules, 98 % for fibres, and 100 % for PVC fragments, compared to 75 %, 61 % and 0 %, respectively, for the standard floatation method of Thompson et al. (2004).

#### **1.4 RESEARCH AIMS AND OBJECTIVES**

Considering the residing lack of consensus on standardised methods and reporting units for sediment analysis, the overarching aim of this research project was to develop a method to quantify the microplastic content of sediments. Based on the promising results in their 2013 paper, the method proposed by Claessens et al. (2013) was used as a starting point for method optimisation.

A need for quantification of microplastics in sediments around Jersey was highlighted in a project proposal from the States of Jersey's Department of the Environment (DoE). Contact was made with the DoE, who collaborated on this research project in order that the optimised method could be applied to Jersey intertidal sediment samples to assess microplastic contamination around the island.

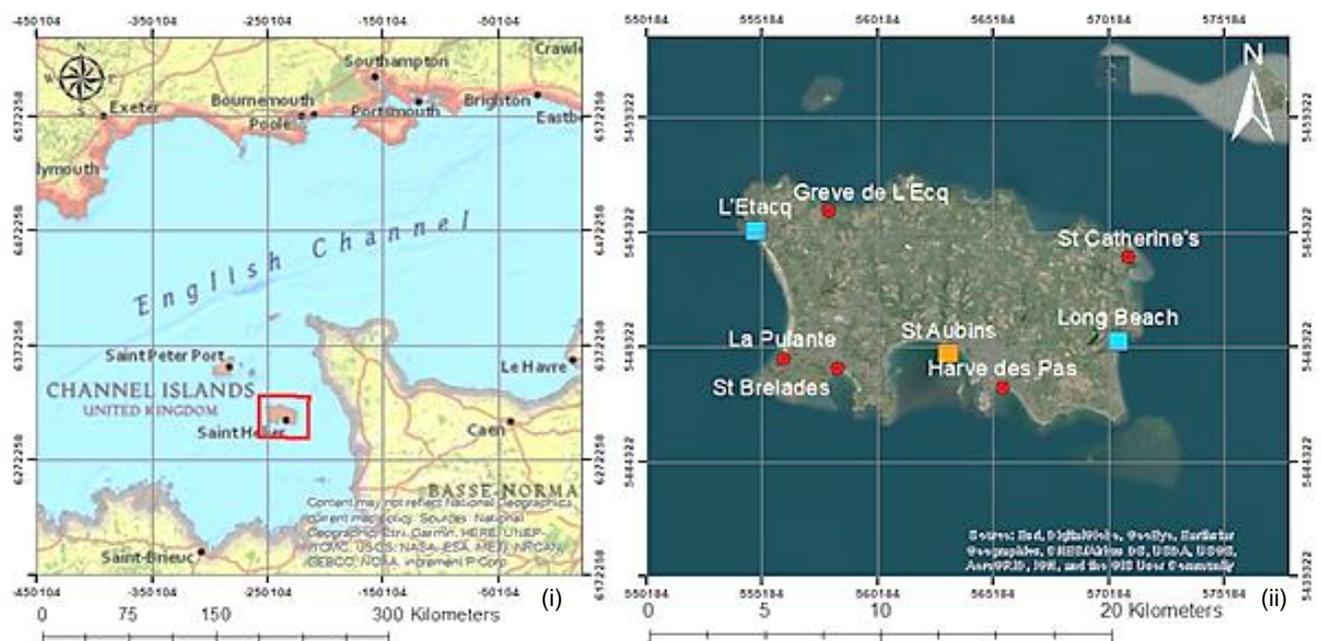
Objectives:

1. Optimise a method to analyse sediment samples for microplastic content, based on the method put forward by Claessens et al. (2013).
2. Achieve a consistent method efficiency (microplastic recovery rate) of > 90 %
3. Apply the optimum method to intertidal sediment samples from Jersey and quantify microplastic contamination.
4. Create a microplastic profile for Jersey beaches (i.e. size, morphology and colour of microplastics).

## 2 METHODS

### 2.1 AREA OF STUDY

Jersey is a self-governed island, situated in the English Channel 23 km from mainland France (Figure 2.1 (i)). The Bailiwick of Jersey is a Crown Dependency; a territory that is under the sovereignty of the British Crown but does not form part of the UK (Ministry of Justice, 2014). Following the introduction of draft legislation by the Department of the Environment and Rural Affairs (DEFRA) in 2017, banning the manufacture of plastic microbead scrubbers in personal care products in the UK, Jersey's Environment Minister publicly announced that Jersey would follow the UK's example (JEP, 2017). Quantifying and monitoring microplastics in the marine environment is an important part of understanding the extent of the problem of microplastics pollution. However, no research to date has set out to quantify microplastics in sediments, surface waters or outfall discharges around Jersey. For this reason, one of the main objectives of this research project was to apply an optimised method of quantifying microplastics in sediments to samples from intertidal sites on a selection of Jersey's beaches. Jersey experiences a hypertidal range of up to 12 m during spring tides, which is surpassed during storm surges. This makes for an interesting and dynamic environment in which to monitor microplastics contamination in intertidal sediments.



**Figure 2.1** Map showing the study area and sample sites. (i) Jersey, Channel Islands, indicated by the red box. (ii) Intertidal sediment sampling sites for the project. The Map Key (right) indicates the method stage to which samples were analysed (1. drying -> 2. separation -> 3. microscopy).

## 2.2 SAMPLE COLLECTION, PREPARATION AND STORAGE

The States of Jersey Department of the Environment (DoE) collaborated on this research project and, as part of this collaborative effort, very kindly collected and shipped a number of sediment samples to the National Oceanography Centre upon request. Figure 2.1 (ii) indicates the sample collection sites on a map of Jersey, and Table 2.1 provides the exact coordinates and a description of each of the intertidal sites selected by the author.

Beach name	Abbrev.	Latitude	Longitude	Date collected	Description of intertidal site
Long Beach	LB	49.195	-2.030	29/05/2018	East, RAMSAR site
L'Etacq	LE	49.240	-2.245	30/05/2018	West, storm washed
St Aubins	SA	49.191	-2.131	30/05/2018	South, near outfall source
Harve des Pas	HP	49.177	-2.100	29/05/2018	South beach
St Catherine's	SC	49.228	-2.024	29/05/2018	North East sheltered bay
Greve de L'Ecq	GE	49.247	-2.202	30/05/2018	North bay
La Pulante	LP	49.190	-2.230	30/05/2018	West, near outfall
St Brelades	SB	49.185	-2.198	30/05/2018	South West bay

**Table 2.1** Jersey intertidal sites sampled. Beach names are provided along with an abbreviations for the samples from each intertidal site. Exact coordinates of where the sample was collected are provided in latitude and longitude along with the date each site was sampled.

These sites were chosen to provide a spatial range across the island with varied levels of anthropogenic impact in different sites i.e. some sites are close to outfall sources, which are well-documented as sources of microplastics to the environment in the literature (Browne et al., 2011; Lourenço et al., 2017; Stolte et al., 2015). Samples were collected by the States of Jersey DoE on 29 – 30 May 2018. A total of 4 x 500 g samples were collected for each site, along a 4 m transect parallel to the tide line. Each sample was collected approximately 1 m apart to 100 mm depth.

Samples were then shipped to the National Oceanography Centre Southampton in separate sealed polyethylene bags. Upon arrival in the laboratory, sediment samples were transferred to glass beakers which had been cleaned previously in an acid wash (Hydrochloric acid; HCl) and covered in aluminium foil to minimise airborne contamination. All samples were prepared for analysis by drying in an oven or autoclave to remove excess water content. Full drying regime details in the Appendix (Table i).

Due to time constraints imposed by an extended period of method optimisation, three sites of the eight sampled were prioritised for further analysis; Long Beach (LB), L'Etacq (LE) and St Aubins (SA). These sites were prioritised because they offered a broad spread of locations around the island. This included one western, storm-washed site, one southern site in close proximity to an outfall source, and one eastern beach within a RAMSAR site. Following the extraction of microplastics from sediment, each remaining sediment sample was recovered and transferred to a glass beaker. During grain size analysis, sediments were stored in disposable aluminium trays with paper lids.

## 2.3 CLAESSENS ET AL.'S METHOD

The method in this study was optimised from a method presented by Claessens et al. (2013), described below.

Claessens et al. (2013) developed a device to carry out elutriation on sediment samples, using an upward flow of water to separate lighter particles in the sediment matrix, including microplastics, from denser ones. The aim of elutriation was to achieve a sample volume reduction before undergoing floatation in high density salt solution. The device used was a PVC column, with tap water entering from the base and an aeration stone arrangement at the bottom of the column to ensure efficient separation of sediment particles. Sediment samples were washed through a 1 mm sieve into the column, then tap water was forced in through the base. It was experimentally determined that the flow rate for tap water should be set at 300 L hr<sup>-1</sup> and run for 15 minutes. This rate was found to be adequate to keep sand particles in the tube whilst other material, including microplastics, flowed over the edge. Lighter particulates were transported to the top of the column with the rising water, and eventually flowed out with the supernatant water. Solids were retained on a 35 µm sieve.

The second step following volume reduction through elutriation, was floatation. Solids retained on the 35 µm sieve were transferred to a 50 mL centrifuge tube and 40 mL of high density NaI solution (1.6 g cm<sup>-3</sup>) was added. This was followed by vigorous manual shaking and centrifugation for 5 minutes at 3,500 g. The top layer of salt solution containing microplastics was then vacuum filtered over 5 µm sieve. This floatation step was repeated 2 – 3 times to ensure all microplastics were extracted from the sample. Visual inspection of the filter was carried out using a dissection microscope.

Claessens et al. (2013) also carried out a method validation phase to determine the extraction efficiency of their newly developed method and compare with the method pioneered. This phase involved evaluating both techniques using sediments spiked with a known concentration of fibres or granules before subjecting these sediments to either one of the techniques. Clean sediment was obtained by subjecting sediment to several elutriations to remove all microplastics present in the sediment matrix. The microplastics used to spike the clean sediment samples were polyvinyl chloride (PVC) granules, polyethylene (PE) granules and fibres (polymer(s) unknown) that had been previously extracted from environmental sediment samples. 50 particles or fibres were used to spike each sediment sample. As mentioned previously, the results of this method validation indicated that retrieval rates for microplastics were 100 % for microplastic granules, 98 %

for fibres, and 100 % for PVC fragments, compared to 75 %, 61 % and 0 %, respectively, for the standard floatation method of Thompson et al. (2004).

## **2.4 THE STANDARDISED SIZE COLOUR SORTING (SCS) SYSTEM**

The Standardised Size Colour Sorting (SCS) System (Crawford et al., 2017) was used to categorise all microplastics based on their size and appearance (Figure 2.2). The SCS System is able to categorise any plastic, but for the purposes of this study, only the microplastics size range (1  $\mu\text{m}$  – 5 mm) was utilised.

### **Step 1: Category (size)**

The first step in using the SCS System was to sort plastics into categories, based on their size. Size was measured as the entire length for fibres, and the widest diameter for other microplastics. The microplastics (MP) category covers all plastics between < 5 mm – 1 mm, and the mini-microplastic (MMP) category covers all plastics between < 1 mm – 1  $\mu\text{m}$ , along their longest dimension. All MP category microplastics were measured using ImageJ.

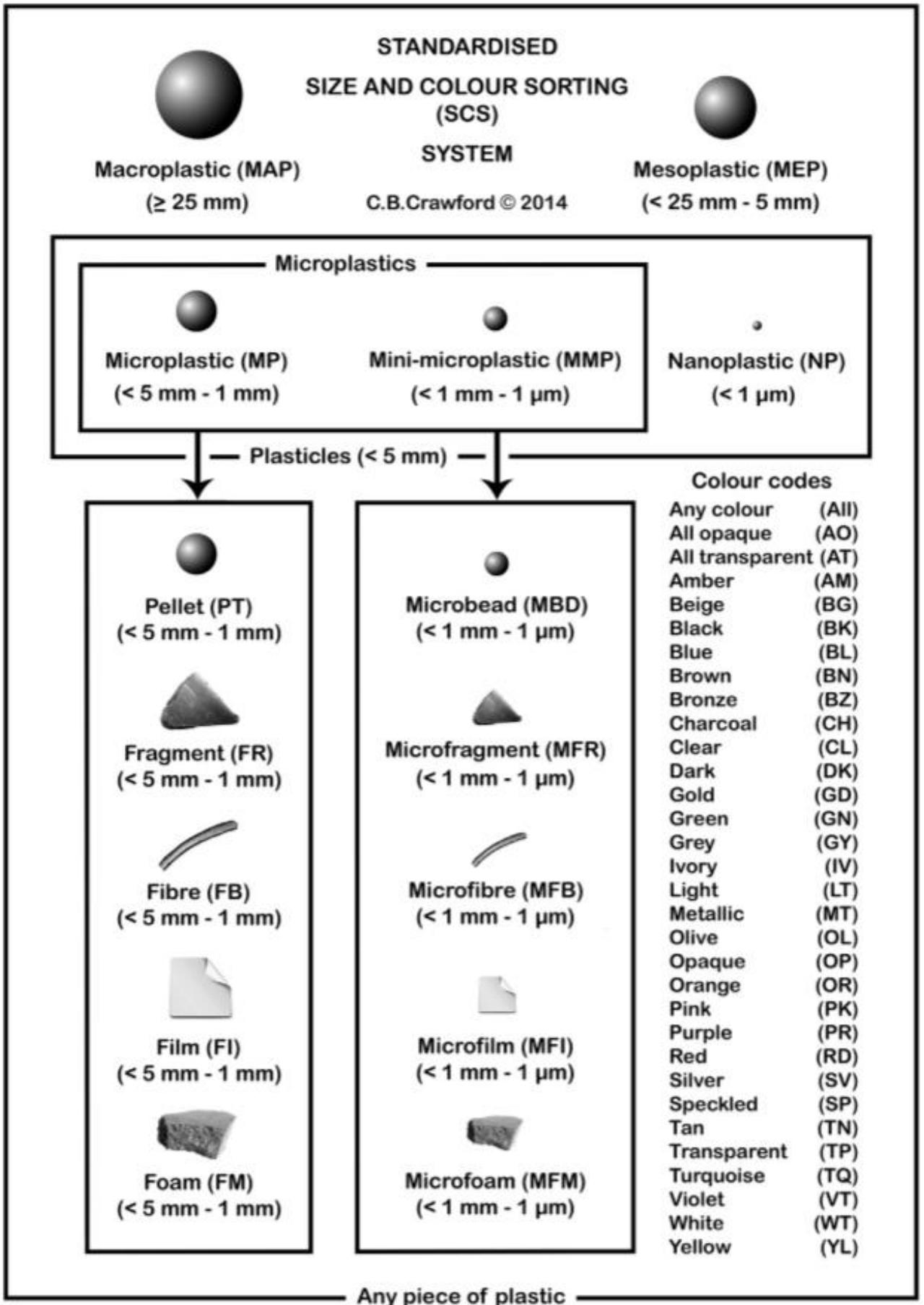
### **Step 2: Type**

Microplastics were then categorised based on their morphology, with five subcategories under each size category (MP and MMP). Under the MP category, spherical pieces of plastic were labelled 'Pellet' (PT), irregular shaped pieces of plastic were labelled 'Fragment' (FR), strands or filaments of plastic were labelled 'Fibre' (FB), thin sheets or membrane-like pieces of plastic were labelled 'Film' (FI), and pieces of sponge, foam, or foam-like plastic material were labelled 'Foam' (FM). Under the MMP category, spherical pieces of plastic were labelled 'Microbead' (PT), irregular shaped pieces of plastic were labelled 'Microfragment' (FR), strands or filaments of plastic were labelled 'Microfibre' (FB), thin sheets or membrane-like pieces of plastic were labelled 'Microfilm' (FI), and pieces of sponge, foam, or foam-like plastic material were labelled 'Microfoam' (FM).

### **Step 3: Colour**

Next, microplastics were all given an individual colour code from the listed codes in the right-hand panel on Figure 2.2.

**Example:** An irregularly shaped piece of plastic, 0.8 mm in length across the widest diameter, which is green in colour would be given the label 'MMP/MFR/GN' according to the SCS System.



**Figure 2.2** The Standardised Size Colour Sorting (SCS) System to categorise plastic found in the environment (Crawford et al. 2017). Microplastics are first categorised by size, then type, and finally by colour to give a SIZE/TYPE/COLOUR code.

## **2.5 METHOD OPTIMISATION**

A major portion of this research project was devoted to method optimisation. This was conducted by testing a range of adaptations to try and improve different aspects of the method put forward by Claessens et al. (2013).

### **2.5.1 Nested vs Single Sieves**

In order to cover the full range of microplastics, it was decided that the method should be amended to extract microplastics up to 5 mm. Claessens et al. (2013) sieved their sediment samples down to 1 mm before elutriation, therefore only microplastics < 1 mm were considered. The use of nested sieve filters, at 1 mm and 38 µm apertures, was tested for the elutriation step to keep these larger and smaller size fractions of microplastics separate from the outset. The outcome of these tests indicated that nothing was gained from adding an additional mesh to the sieve (1 mm), as very little material was retained > 1 mm, and the size of larger particulates could be confirmed using visual microscopy with the use of a single sieve to retain material following elutriation. Therefore a single sieve at 38 µm was used, as in Claessens et al. (2013).

### **2.5.2 Considerations for microplastics < 38 µm**

A protocol to recover microplastics < 38 µm was researched, and tested, where possible. It was hoped that an additional size range of 1.2 – 38 µm could be quantified using an amended method. This size range of microplastics is the most likely to impact on benthic species important to Jersey's commercial fisheries, such as the Pacific Oyster (*Crassostrea gigas*) and the King scallop (*Pecten maximus*). This is due to their similar size to filter-fed particulate matter, making these smaller microplastics more likely to be ingested by these species via filtration (Brillant and MacDonald, 2000; Sussarellu et al., 2016; Van Cauwenberghe and Janssen, 2014).

#### **I. Smaller Mesh for Elutriation**

Due to the flow rate of the elutriation (300 L hr<sup>-1</sup>), it was not possible to simply add or replace the existing 38 µm mesh with a smaller mesh. This is because the flow rate would be likely to exceed the filtration rate at such a small aperture (1.2 µm), and more markedly so with the accumulation of material on the filter throughout the process of elutriation. This would therefore greatly increase a risk of overflow, resulting in sample loss. Other protocols to tackle this size range were therefore considered.

## **II. Vacuum Filtration of Collected Water**

One protocol was tested, which involved vacuum filtration of the water that had been through an elutriation step. A 200 L glass tank was cleaned (rinsed thoroughly with tap water) and used to collect the 75 L of water which had been through elutriation. Foil was used to cover the tank to reduce airborne contamination. This water was then vacuum filtered onto several 1.2  $\mu\text{m}$  glass fibre filters to retain particulates (including microplastics) between 1.2 – 38  $\mu\text{m}$ . This additional step added approximately 15 hours to a 1 hour protocol, per sample. Furthermore, this method was subject to additional contamination on account of the length of time taken to complete the filtration, which allowed for dust to settle out and contaminate the water in the tank overnight. This protocol was therefore discarded, on account of its time-consuming nature and unreliability of the data collected due to contamination.

## **III. Tangential Flow Filtration**

Another protocol was considered, but was not possible to test within the scope of this project. This proposed the use of a Tangential Flow Filtration (TFF) system, which has been used in previous studies to separate microbes and viruses from marine water samples (Cai et al., 2015). It was suggested that this principle could be used to separate microplastics from water samples, specifically from the water which had undergone elutriation. Unfortunately it was not possible to source a TFF System within the scope of this project.

The results of this research indicated that the options for processing microplastics < 38  $\mu\text{m}$  were limited, and difficult to apply to Claessens et al.'s method (2013). Therefore it was decided that only microplastics > 38  $\mu\text{m}$  would be considered.

### **2.5.3 Low Cost, High Density Salt Solution**

The approximate costs to make salt solution with 1.5  $\text{g cm}^{-3}$  density are £35.10  $\text{L}^{-1}$  for  $\text{ZnCl}_2$  and £172.95  $\text{L}^{-1}$  for  $\text{NaI}$  (Coppock et al., 2017). Due to the considerable difference in material costs, yet relatively similar density that could be achieved,  $\text{ZnCl}_2$  solution (1.5  $\text{g cm}^{-3}$ ) was chosen as the floatation medium, in substitution of  $\text{NaI}$  solution (1.6  $\text{g cm}^{-3}$ ), which was used by Claessens et al. (2013).

### **2.5.4 Transferring Retained Solids to Zinc Chloride Salt Solution**

Claessens et al. (2013) state that the step following each elutriation is to transfer the solids to a 50 mL centrifuge tube for the floatation step. However, it is not explicitly detailed in the paper how to do so. Therefore several different protocols were considered.

### **I. Scrape Material off Filter**

Firstly, the use of a metal implement to scrape material from the filter to the centrifuge tube was considered. This protocol, or similar, was assumed to be the method used by Claessens et al. (2013), despite the ambiguity of the transfer method detailed in the paper, hence was the first to be considered. As this method would rely on visual inspection of the filter to ensure all material was transferred, it was deemed to add an unnecessary potential loss step for smaller microplastics, which are difficult to see with the naked eye and thus ensure their transfer to the tube. Therefore other protocols were considered which involved transferring the filter to the tube along with any retained solids.

### **II. Add Whole Filter to Tube**

A second consideration was to transfer the filter as a whole to the tube. However, as the circular filter had 15 cm diameter, it needed to be folded before adding it to the tube. This meant that it was difficult to achieve a transfer without trapping retained material (including microplastics) within the folds of the filter, thus reducing the extraction efficiency of the floatation step. This protocol was therefore deemed impractical.

### **III. Cut Up and Add Filter to Tube**

In this protocol, filters were cut up before floatation was performed. Firstly, any visible material retained on the filter was scraped into the tube using a clean metal spatula. Then the filters were cut into approx. 0.5 – 1 mm pieces in a clean glass container being added to the centrifuge tube. This aimed to reduce the potential for microplastics being trapped during floatation whilst ensuring that the majority of retained material was transferred to the centrifuge tube.

Protocol III. was used for all subsequent  $\text{ZnCl}_2$  floatation steps for sample analysis.

#### **2.5.5 Blanks Using Water of Different Origin and Purity**

Blanks were carried out using different water mediums, to determine which would be the most suitable for the method by minimising contamination. The water mediums tested were of different origins and purity, and included sea water (filtered through sand to remove large particulates), tap water and reverse osmosis (RO) water. Three blanks were carried out, for each water medium, through the full method protocol (without a sediment sample). Microplastic contamination on the filters following the blanks being carried out was categorised using the SCS System. Based on the results of these tests

(section 3.1), tap water was chosen as the water medium to take forward for spiked sediment testing and sample analysis. Incidentally, this is the same water medium used by Claessens et al. (2013).

## **2.6 MINIMISATION OF CONTAMINATION**

Microplastics tend to be present in laboratory settings in the form of airborne fibres and other small particulates, which settle on equipment and surfaces and can contaminate samples (Wesch et al., 2017). Several measures were therefore put in place to minimise microplastic contamination throughout the laboratory experiments. Sediment samples were stored in clean glass beakers and covered with aluminium foil to block airborne contaminants. The elutriation column was cleaned before the first use and in between each elutriation. This involved removing the bolts at the base of the column so the upper tube could be removed. The residual sediment on the base sieve was then removed and the sieve rinsed thoroughly with tap water, as were the air stones. The column was also rinsed thoroughly with tap water before being reassembled and filled with tap water supplied from the base. This water entered the column base at a flow rate of 300 L hr<sup>-1</sup> as in the elutriations, but without a sediment sample or the retainer sieve. The tap water was left to flow out from the column brim for 5 minutes to wash out any residual material from the inner tube. During each elutriation, an aluminium foil lid covered the top and outflow opening of the column to reduce airborne contamination. A new 38 µm mesh was replaced on the retainer sieve for every elutriation. Mesh for the retainer sieve was prepared in bulk ahead of time and wrapped in aluminium foil. When used mesh sieves were removed from the retaining filter assemblage, they were folded in half on a sheet of blue roll to remove excess moisture then wrapped in aluminium foil. Fresh 1.2 µm glass fibre filters were used for each individual sample and centrifugation during the floatation step. All filters used were made of stainless steel (elutriation) or glass fibre (floatation) to avoid additional sources of plastic contamination. Used glass fibre filters were stored in individual petri dishes and sealed with tape around the lid to prevent airborne contamination.

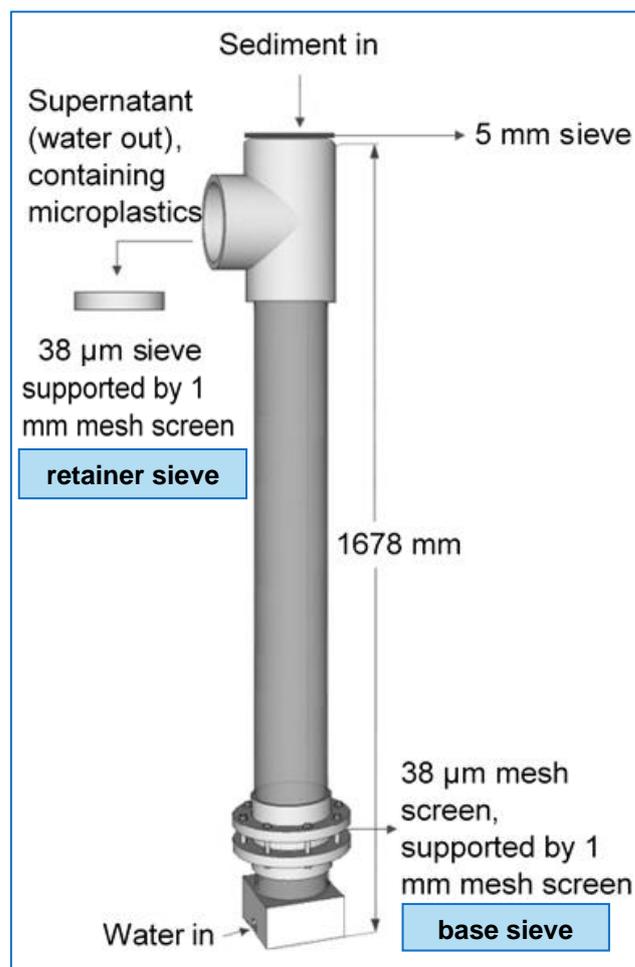
## 2.7 AMENDED METHOD PROTOCOL

Following the method optimisation phase, an amended method protocol was established for the extraction of microplastics from sediment samples.

### 2.7.1 Volume Reduction via Elutriation

A custom-made PVC column was made to the specification of Claessens et al. (2013) to carry out elutriation on sediment samples (Figure 2.3). The column and airstones were cleaned with tap water prior to use. A 500 g dry sediment sample was washed through a 5 mm mesh into a 2 L beaker to remove larger particles from the sediment, then carefully washed into the elutriation column from the top. Airstones were then turned on and placed into the column from the top, and the column openings were covered with aluminium foil (without blocking the supernatant outflow) to reduce airbourne contamination. Tap water flow rate was measured to 300 L hr<sup>-1</sup> using a measuring flask and timer (12 second to fill up to the 1 L mark). The tap water was then supplied to the column via a pipe attached to the base. Elutriation was carried out for 15 minutes from the time the supernatant water started to exit the overflow, with lighter solids (including microplastics) being retained on the 38 µm (retainer sieve). During elutriation, the filter was monitored to ensure retained

material did not block the flow of water and cause an overflow. At the end of the 15 minute elutriation, the tap water supply was removed from the base of the column and water allowed to flow out. Remaining sediment was retained on the base sieve, and was retrieved by removing the column from the base. Lighter solids that were retained on the retainer sieve were removed with the mesh from the retainer sieve holder. The mesh was carefully folded in half to keep solids from being inadvertently lost, then placed on a piece of blue roll to remove excess moisture and wrapped in aluminium foil.



**Figure 2.3** Elutriation column schematic, amended from Claessens et al. (2013).

### **2.7.2 Floatation using 7M Zinc Chloride Salt Solution**

Following volume reduction of a sample through elutriation, microplastics were extracted from the material retained on the 38  $\mu\text{m}$  sieve using 7M zinc chloride solution ( $\text{ZnCl}_2$ ) ( $1.5 \text{ g cm}^{-3}$ ).

Preparation of  $\text{ZnCl}_2$  solution was carried out in a fume cupboard and was made to the specifications of (Coppock et al., 2017). 1 L of Milli-Q ultrapure water was added to a 5 L conical flask. Following this,  $\text{ZnCl}_2$  powder (Arcos Organics Zinc Chloride 98+% extra pure) was weighed out to 972 g in a fume cupboard, then added to the Milli-Q water. This was then manually stirred for approximately 5 minutes (or until all solids had visibly dissolved). The process of dissolving the salt powder in water resulted in an exothermic reaction, thus the solution was left in the fume cupboard for 60 minutes to cool. The  $\text{ZnCl}_2$  solution was then vacuum filtered using 1.2  $\mu\text{m}$  glass fibre filters to remove any undissolved salt crystals. Prepared  $\text{ZnCl}_2$  was stored in 50 mL centrifuge tubes in batches of 40 mL, ready for floatation.

The solids and 38  $\mu\text{m}$  sieve filter were then transferred to a 50 mL centrifuge tube filled with 40 mL 7M  $\text{ZnCl}_2$  solution using the method described in section 2.5.4 (III. Cut Up and Add Filter to Tube). This was followed by vigorous manual shaking and centrifugation for 5 minutes at 3,500 g (Hettich Zentrifugen Rotana 460R. Settings: 18°C; 3,500 g; 05:00). The top layer of salt solution (containing microplastics) was then vacuum filtered over 1.2  $\mu\text{m}$  sieve using glass pipettes that been altered so that the wider aperture end could be used to collect larger material floating in the salt solution. This floatation step was repeated 2 times to ensure all microplastics were extracted from the sample.

### **2.7.3 Visual Sorting using Light Microscopy**

Visual inspection of the filter was carried out using a dissection light microscope (Olympus BH-2) and a photographic catalogue was kept of each section of the filter where microplastics were present using a Nikon D5000 camera. Microplastics were sorted according to the SCS System (section 2.4) (Crawford et al., 2017). Details of the microplastics observed were catalogued in an Excel spreadsheet for each sample, which included the date, photo number, sample (site and #repeat), #centrifugation, size (MP/MMP), type (morphology), colour, count (# microplastics of the same SCS code), and exact size for microplastics > 1 mm (MP only).

## 2.8 SPIKED SEDIMENT EXTRACTION EFFICIENCY TESTS

Bulk sediment for the spiked sediment tests was collected at Hayling Island, (50°47'37.5"N, 1°01'29.9"W). Prior to being spiked, sediment was put through several elutriations to remove any microplastics present, before being dried at 60 °C for 24 hrs. Clean dry sediment was then weighed out to 500 g samples and stored in glass beakers covered in aluminium foil, ready to be spiked with a known amount of microplastics. Three polymer types were used for the spiked sediment tests; nylon/ polyamide (PA), polystyrene (PS) and polyvinyl chloride (PVC) (Table 2.2). These polymer types were used as they are commonly found in marine sediments (Table 1.1). PS and PVC microplastics were created using a band saw to cut fragments and microfragments from larger plastic items (PS coffee cup lid/ tray and PVC column offcut). PA microplastics were created by distressing tulle fabric to create fibres and microfibrils. Microplastics were sorted into MP and MMP size fractions by sieving through a 1 mm mesh, then collecting the different size fraction in glass vials. Three sediment samples were used for the spiked sediment tests, with a different polymer in each sample. Each sediment sample was spiked with 50 x MP and 50 x MMP of a polymer type, to a total of 100 microplastics, which were counted out with the aid of a dissection microscope and fine tweezers. Spiked sediments were then put through the full amended method to determine the extraction efficiency by the amount of microplastics extracted.

Polymer name	Symbol	Density (g cm <sup>-3</sup> )	Common sources for microplastics in the marine environment	Plastic item(s) used	Microplastics created
Polystyrene (PS)		1.05	Packaging foam, food containers, plastic tableware, disposable cups, plates, cutlery, building insulation	White coffee cup lid & white tray	FR and MFR
Polyvinyl Chloride (PVC)		1.38	Plumbing pipes and guttering, shower curtains, window frames, flooring, films	Grey elutriation column offcut	FR and MFR
Polyamide/ Nylon (PA)		1.15	Discarded fishing gear, toothbrush bristles, car engine mouldings, films for food packaging	Fluorescent yellow tulle fabric	FB and MFB

Table 2.2 Polymers used in spiked sediment tests. Density and common sources from Li et al. (2016).

## 2.9 GRAIN SIZE ANALYSIS

Grain size analysis was carried out on all sediment samples that had been through the elutriation protocol. Samples were dried for 24 hours at 60°C, then separated using nine stacked sieves on a shaking plate for 10 minutes. Sieves decreased in pore size from 1 mm to 63 µm (0 – 4 φ in fractions of 0.5 φ). Sediment retained on each of the sieves was weighed and recorded in a spreadsheet. This data was then analysed using GRADISTAT Version 8.0 (Blott and Pye, 2001) to provide mean grain size, % loss of sediment sample weight and an overall sediment description.

### 3 RESULTS

#### 3.1 SEAWATER, TAP WATER AND REVERSE OSMOSIS WATER BLANKS

Contamination on the blanks varied with the use of three water sources of different origin and purity (Table 3.1). The initial count of particles retained on glass fibre filters included all particles visible at 4 x magnification under the light microscope ('All'). However, the technique of visual microscopy to identify microplastics becomes increasingly subjective with decreasing particle size. Therefore small dark fragments smaller than 100 µm (listed as MMP/MFR/DK under the SCS System) were disregarded from the data to remove some speculation of whether particles are of plastic origin ('> 100 µm'). In addition, a count for microplastics larger than 1 mm observed was conducted ('> 1 mm') to indicate the split of large (1 – 5 mm) and small (< 1 mm) microplastics found in the blanks ('Ratio of Large vs Small Microplastics').

Water medium	Repeat	Total microplastic particle count (2 x filters)			Ratio of Large vs Small Microplastics
		All	> 100 µm	> 1 mm	> 1 mm : 100 µm – 1 mm
Sea water	1	453	83	11	11 : 72
	2	1143	293	9	9 : 284
	3	792	282	2	2 : 280
	<b>Mean</b>	<b>796</b>	<b>219.3</b>	<b>7.3</b>	<b>7.3 : 212</b>
Tap water	1	221	131	9	9 : 122
	2	330	160	2	2 : 158
	3	244	54	6	6 : 48
	<b>Mean</b>	<b>265</b>	<b>115</b>	<b>5.7</b>	<b>5.7 : 109.3</b>
Reverse osmosis (RO) water	1	624	385	17	17 : 368
	2	195	145	2	2 : 143
	3	296	166	1	1 : 165
	<b>Mean</b>	<b>371.7</b>	<b>232</b>	<b>6.7</b>	<b>6.7 : 225.3</b>

**Table 3.1** Results from the blanks, run using three water sources of different origin and purity. Three repeats were carried out with each water source; sea water, tap water and reverse osmosis (RO) water. The initial total of observed particles is listed (All), along with those larger than 100 µm (> 100 µm) and those larger than 1 mm (> 1 mm). A ratio of large:small microplastics is also provided (> 1 mm : 100 µm – 1 mm).

The mean average counts of particles observed, in all three size categories, indicated that the use of tap water for elutriation resulted in the least contamination, compared to sea water and reserve osmosis (RO) water. In addition, the contamination of blanks carried out with tap water was more consistent across the three repeats, with a lower standard deviation ( $\pm 54.8$ ), compared to that of sea water ( $\pm 118.2$ ) and RO water ( $\pm 132.9$ ). Therefore, tap water was used for all subsequent elutriations.

### 3.2 SPIKED SEDIMENT EXTRACTION EFFICIENCY TESTS

The results of the method validation tests using spiked sediment gave a mean average extraction efficiency of 31% with one elutriation and two subsequent extractions via floatation (Table 3.2). This is in contrast to the results of the method validation phase of the study by Claessens et al. (2013), which indicated an extraction efficiency of 98 – 100% following one elutriation of spiked sediment containing 50 microplastics (fibres, granules or PVC particles), and three subsequent extractions.

Of the three polymer types, PA fragments had the highest extraction efficiency (41%), followed by PVC fragments (30%) and PS fibres (23%). Conversely, Claessens et al. (2013) used PVC granules, PE granules and fibres previously extracted from the environmental sediment samples (polymer(s) unknown) to spike sediment, and noted little difference in extraction efficiency observed between polymer types.

The extraction efficiencies for MFR and FR (50 of each in each spiked sample) differed greatly, with 36% for PVC MFR and 44% for PS MFR, and 16% for PVC FR and 10% for PS FR. This was a 2.8-fold, and 3.6-fold, decrease between the extraction efficiency of MFR and FR of PVC, and PS, respectively. For PA fibres, however, the extraction efficiency for MFB and FB was very similar (42% and 40%, respectively). The method developed by Claessens et al. (2013) sieved sediment to < 1 mm, removing all large debris, thus MP were removed from sediment samples before analysis was carried out.

Polymer	Type	Small microplastics 40 µm – 1 mm (%)	Large Microplastics 1 – 5 mm (%)	Mean extraction efficiency (%)
PVC	Fragments (FR/MFR)	44	16	30
PS	Fragments (FR/MFR)	36	10	23
PA	Fibres (FB/MFB)	42	40	41
<b>Mean extraction efficiency (%)</b>		<b>41</b>	<b>22</b>	<b>31</b>

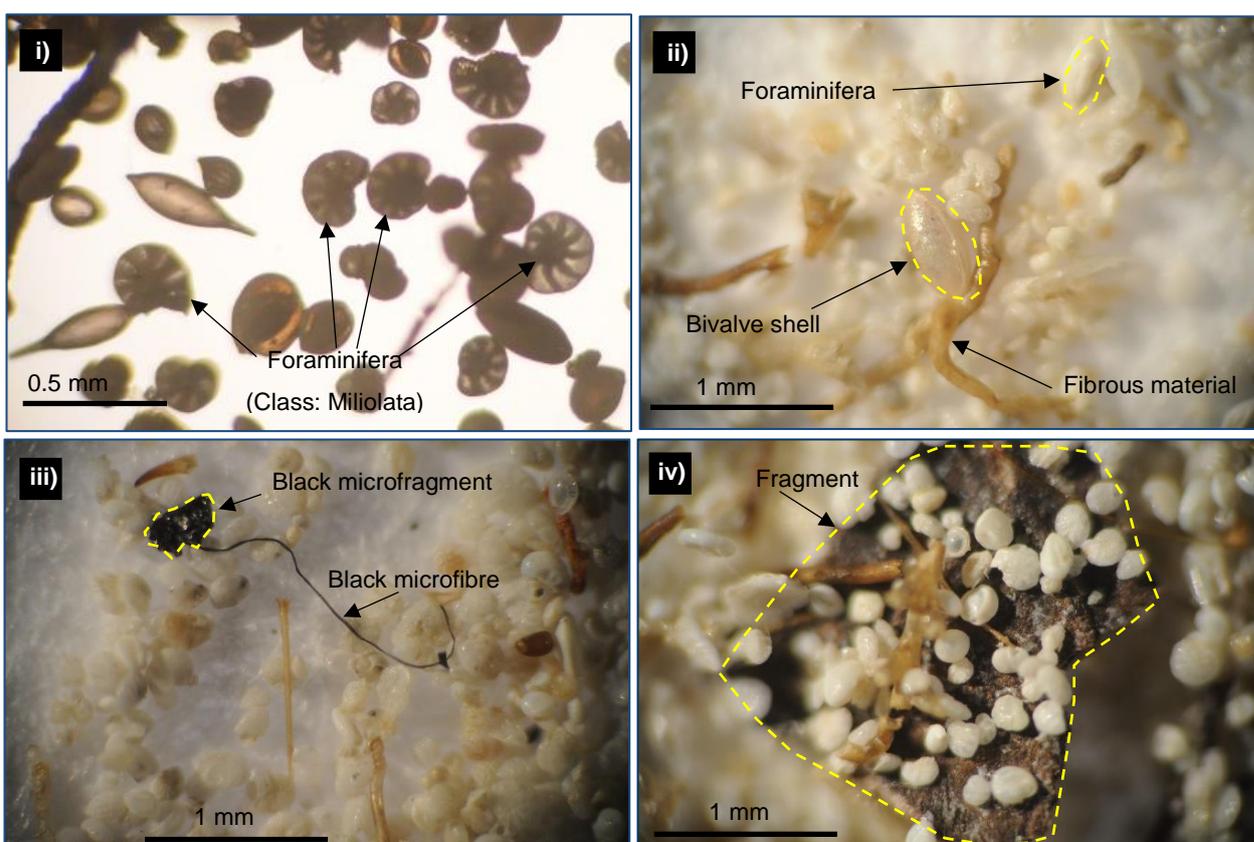
**Table 3.2** Extraction efficiencies of the method using microplastics of different polymer type and size. Efficiencies were determined by running sediment spiked with 100 pieces of microplastic (one polymer type; 50 MMP, 50 MP) through one elutriation and two subsequent extractions using saturated zinc chloride salt solution.

### 3.3 JERSEY INTERTIDAL SEDIMENT SAMPLE ANALYSIS: INITIAL OBSERVATIONS

Jersey sediment samples from St Aubins (SA), L'Etacq (LE) and Long Beach (LB) were subjected to the full method protocol to determine their microplastic content. Visual microscopy revealed some initial observations of the material extracted from these three intertidal sites.

#### 3.3.1 Abundance of Material on St Aubins (SA) Filters

A high volume of material was retained following each elutriation of sediment samples from St Aubins (SA). The volume of material retained was sufficiently high to justify separating the bulk of material retained and cut up filter into two different centrifuge tubes, as the two parts would not fit into a 50 mL tube together (as the method dictates). These floatations resulted in a retention of substantially higher volumes of material on the glass fibre filters when compared to the other beaches analysed. Upon inspection under the light microscope, the majority of particulates retained appeared to be biological in nature (Figure 3.1). Foraminifera (forams) were the most commonly observed items, with forams in the Class Miliolata (with multi-chambered shells) appearing most frequently (Figure 3.1, i). Fibrous material (likely of plant origin) and bivalves (likely juvenile *Mytilus edulis*) were also commonly observed throughout the filters (Figure 3.1, ii). Microplastics did



**Figure 3.1** Particulate material retained on filters from St Aubins sediment samples. Photo i) Material includes foraminifera (forams), along with some bivalve shell(s) and unidentified fibrous material. Photo ii) Light from below highlights morphological features of retained material, including forams (likely class: Miliolata; identified by their multi-chambered shells). Photo iii) Large fragment (possibly of synthetic polymer origin) covered in multiple layers of particulate biological material. Photo iv) Black plastic microfragment and microfibre intersperse biological material.

appear to be present (Figure 3.1, iii), however, the sheer volume of material created an issue with overlap of particulates (Figure 3.1, iv). This was deemed likely to result in a number of microplastics being missed from the particulate count. In addition, it was difficult to differentiate forams and other biological material from microplastics without using further separation techniques or more advanced microscopy techniques. Therefore SA samples were unable to be analysed further to determine their microplastic content.

### **3.3.2 Total Counts of Particles on Filters**

Particles extracted from Long Beach (LB) and L'Etacq (LE) sediment samples were visually sorted following the extraction of microplastics from sediment samples using the amended method. Microplastics were observed under a light dissection microscope and assigned a code using the SCS System (detailed in section 2.4). A photographic catalogue was recorded for each filter observed under the microscope. Microplastics that looked to be close to or above 1 mm were measured to confirm their size and allow microplastics to be split into two size fractions; large microplastics (MP, 1 – 5 mm) and mini-microplastics (MMP, < 1 mm).

An initial count of microplastics included small fragments of indiscernible colour (< 100 µm), which were listed as MMP/MFR/DK. Following this initial count, the total number of microplastics observed across the two beaches was 18,574 for 2 kg of sediment. The total for 1 kg of LB sediment was 11,571 microplastics, and for 1 kg of LE sediment was 7,002 microplastics. The two repeats for LB yielded very different counts, with 8,929 particles in LB1 and 2,642 particles in LB2 (500 g sediment each), whereas the two repeats for LE yielded similar results (3,775 and 3,227 particles in LE1 and LE2, respectively, from 500 g sediment). However, the characteristics of the particulates that had been labelled MMP/MFR/DK were difficult to distinguish, on account of their small size. This made it very difficult to rule out biological or mineral origin and verify synthetic polymer origin for these fragments. MMP/MFR/DK were therefore removed from further analyses, and the above totals were disregarded due to ambiguity of data for microplastics < 100 µm. The total counts were recalculated for microplastics > 100 µm to reduce ambiguity and improve the reliability of the data. The results of this secondary count revealed a total count of 2,827 microplastics extracted from 2 kg sediment across both beaches. The total for 1 kg of LB sediment was 1,473 microplastics, and for 1 kg of LE sediment was 1,354 microplastics. The two repeats for LB yielded different counts, with 849 microplastics in LB1 and 624 microplastics in LB2 (500 g sediment each). Similarly, the two repeats for LE yielded counts of 574 microplastics in LE1 and 780 microplastics in LE2 (500 g sediment each).

### 3.4 CHARACTERISTICS OF MICROPLASTICS ON JERSEY BEACHES

Microplastics > 100 µm from LE and LB were individually labelled with codes using the SCS System. The results of microplastics SCS System classification are summarised below, covering each classification, size, morphology and colour.

#### 3.4.1 Size

Microplastics were categorised into two size classes; large microplastics (MP), between 1 – 5 mm, and mini-microplastics (MMP), between 100 µm – 1 mm. MP constituted 1.2% (33 microplastics of 2,827) of the material observed under the microscope from LE and LB samples, with MMP constituting the other 98.8% (2,794 microplastics of 2,827). This indicates that just over 1 MP was observed in every 100 microplastics between 100 µm – 5 mm. The trends observed were similar when comparing the two sites together and separately.

#### 3.4.2 Morphology

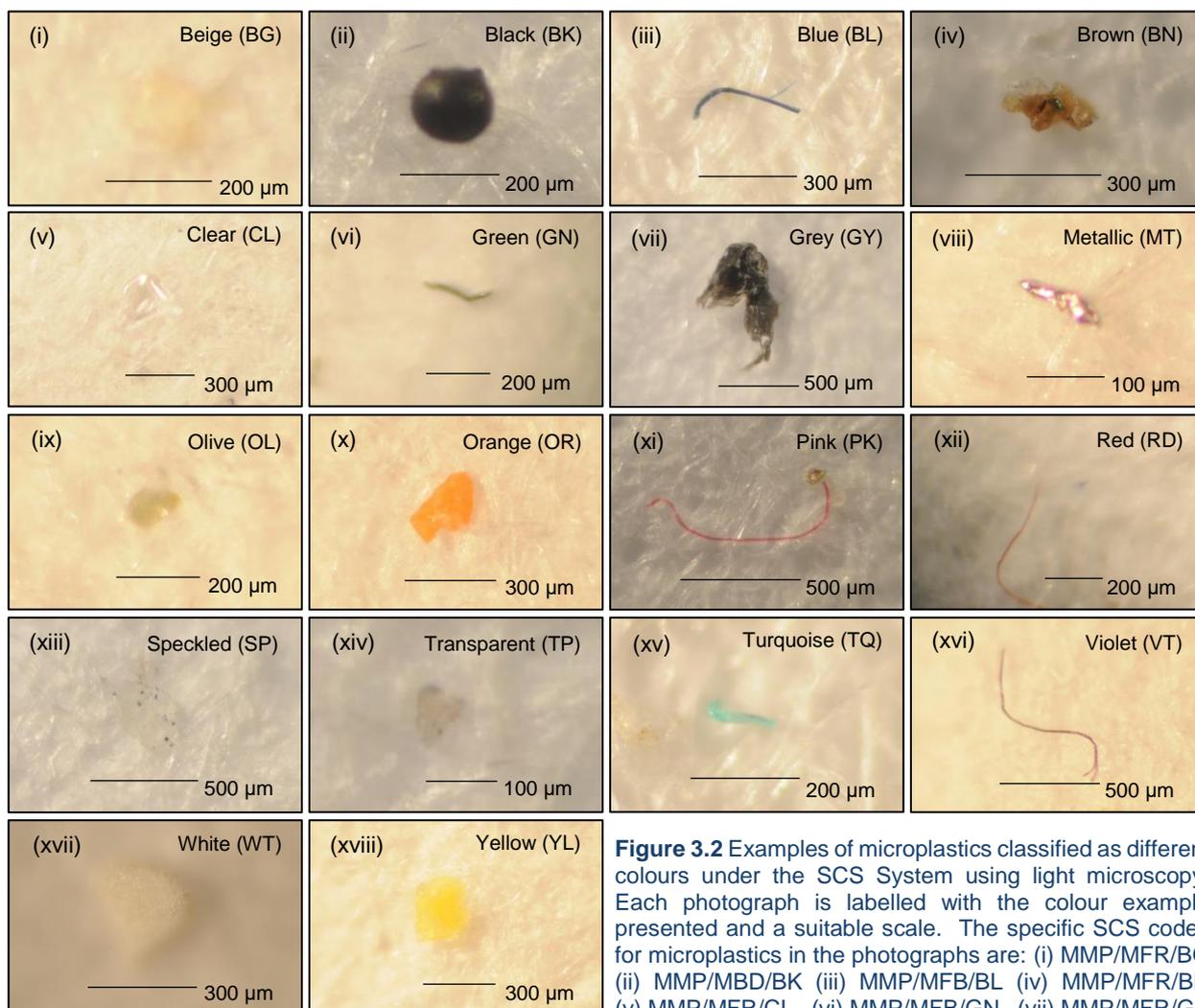
One of ten individual morphology codes under the SCS System were applied to each microplastic observed (Table 3.3). Of the ten codes, three were not observed at all across all samples analysed, which included PT (pellets, 1 – 5 mm), FI (film, 1 – 5 mm) and FM (foam, 1 – 5 mm). In addition, FR were not observed in LB samples. The majority of MP were FB across both intertidal sites. The most common MMP observed were MFR, which constituted 85.0% of LB microplastics and 84.1% of LE microplastics. In both LB and LE samples, the next most common MMP were MFB (7.8 & 5.8 %, respectively). MFM constituted 0.1% of LB microplastics and 1.8% of LE microplastics. MBD were observed less in LE than LB (1.5 & 3.1 % respectively) whereas MFI were observed less in LB than LE (3.0 & 5.6 % respectively).

Intertidal Site	Microplastic Morphology Code							
	1 – 5 mm		100 µm – 1 mm					
<b>Long Beach (LB)</b>	FB	FR	MBD	MFB	MFI	MFM	MFR	Total
Count (> 100 µm)	17	0	45	115	44	1	1250	1472
Proportion (%)	1.2	0.0	3.1	7.8	3.0	0.1	85.0	100
	1 – 5 mm		100 µm – 1 mm					
<b>L'Etacq (LB)</b>	FB	FR	MBD	MFB	MFI	MFM	MFR	Total
Count (> 100 µm)	14	2	21	80	77	24	1151	1369
Proportion (%)	1.0	0.1	1.5	5.8	5.6	1.8	84.1	100

**Table 3.3** Profile of morphology types for microplastics > 100 µm from Jersey intertidal sediments (2 x 500 g samples). Microplastic morphology codes are from the SCS System. A count for each morphology code is listed, along with the proportion (%) that each morphology code contributes to the total microplastics count.

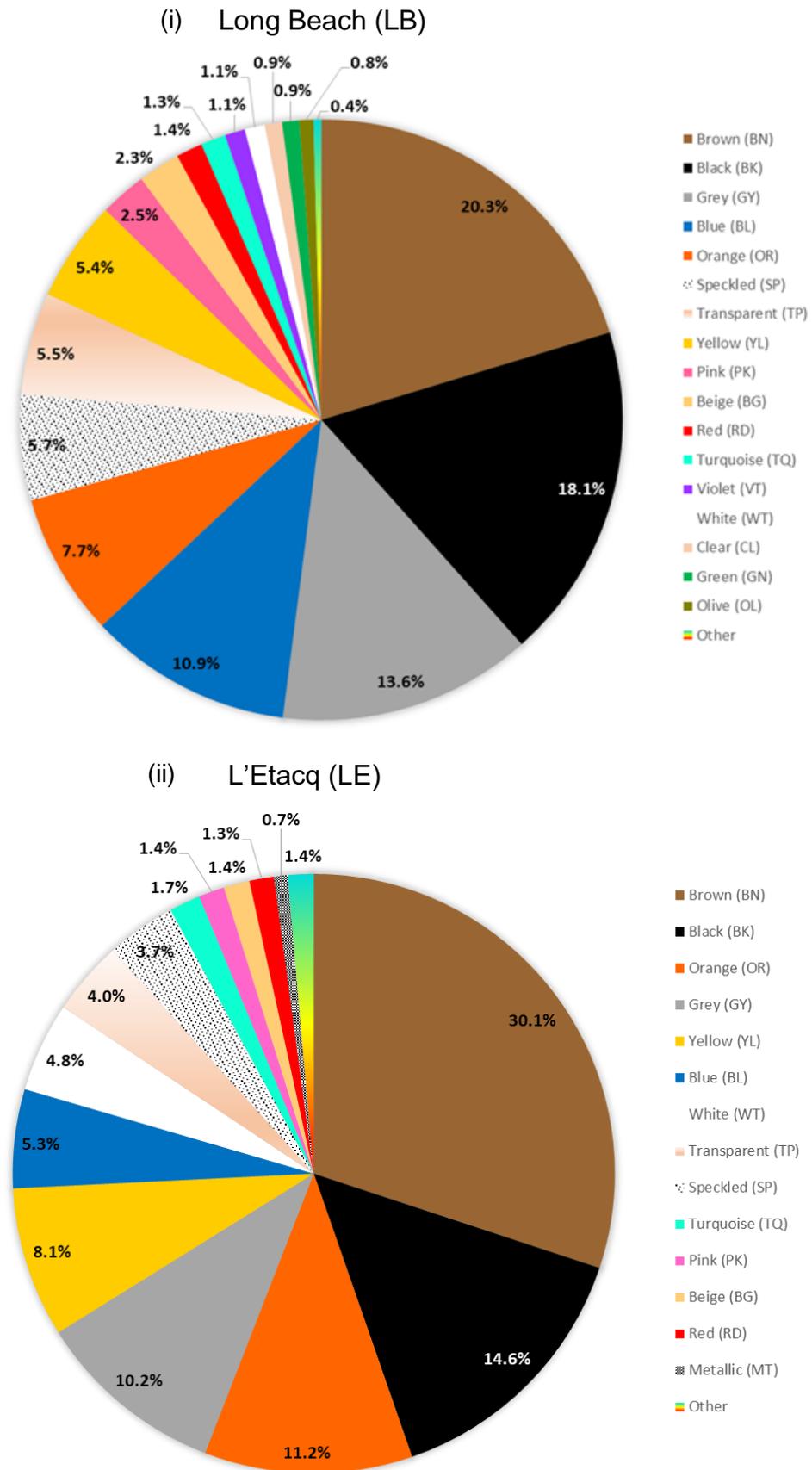
### 3.4.3 Colour

18 different colour classifications under the SCS System were observed at levels higher than 0.5 % of microplastics per site sample. A photographic example of each of these colour classifications is provided in Figure 3.2, (i) – (xviii), in alphabetic order. In addition to exhibiting the full range of colours observed during sample analysis, a range of different microplastic sizes and morphologies are shown. All photographs are of microplastics extracted from LB or LE samples. A full account of the photo number and date corresponding to each photograph, along with the specific filter the microplastic was observed on, is detailed in the Appendix.



**Figure 3.2** Examples of microplastics classified as different colours under the SCS System using light microscopy. Each photograph is labelled with the colour example presented and a suitable scale. The specific SCS codes for microplastics in the photographs are: (i) MMP/MFR/BG (ii) MMP/MBD/BK (iii) MMP/MFB/BL (iv) MMP/MFR/BN (v) MMP/MFR/CL (vi) MMP/MFB/GN (vii) MMP/MFR/GY (viii) MMP/MFR/MT (ix) MMP/MFR/OL (x) MMP/MFR/OR (xi) MMP/MFB/PK (xii) MMP/MFB/RD (xiii) MMP/MFR/SP (xiv) MMP/MFR/TP (xv) MMP/MFB/TQ (xvi) MP/FB/VT (xvii) MMP/MFM/WT (xviii) MMP/MFR/YL.

The percentage of different colours observed in each intertidal site is shown in Figure 3.3. A range of colours were observed across LB (i) and LE (ii) samples. The colour composition of microplastics extracted from each site were somewhat similar. In both LB and LE samples, over half of the microplastics observed fell under three colour categories. For LB samples, 52.0 % of microplastics were either brown (BN), black (BK) or grey (GY). For LE samples, 55.9 % of microplastics were BN, BK, or orange (OR). In



**Figure 3.3** Percentage of different colours observed in microplastics > 100 µm extracted from Jersey intertidal sediments LB and LE. (i) Colours of 1,473 microplastics extracted from LB sediment samples (2 x 500 g). Other category includes: opaque (OP), charcoal (CH), metallic (MT) and purple (PR). (ii) Colours of 1,354 microplastics from LE sediment samples (2 x 500 g). Other category includes: olive (OL), green (GN), violet (VT), purple (PR), opaque (OP), and charcoal (CH).

both LB and LE samples, BN was the most commonly observed colour (20.3 & 30.1 %), followed by BK (18.1 & 14.6 %). Turquoise (TQ) and red (RD) microplastics constituted similar proportions in LB and LE samples, with 1.3 & 1.7 % TQ and 1.4 & 1.3 % RD microplastics observed across the two sites. Common rare colours observed in both sites ('Other': < 0.5 % of site samples) were opaque (OP), charcoal (CH), and purple (PR).

There were also some differences observed between LB and LE samples. More than double the proportion microplastics were blue in LB compared to LE (10.9 and 5.3 %). The difference in proportion of white microplastics was even more pronounced, constituting 4.8 % of LE microplastics and 1.1 % of LB microplastics. In addition, whilst abundance of each colour did not differ substantially and was generally similar between sites, the order of colours, from most common to least common, was different in each site (colours are listed in order according to % composition in Figure 3.3 (i) and (ii)).

### 3.4.4 Size and Characteristics of Large Microplastics (MP)

MP constituted 1.2 % of the material observed under the microscope from LB and LE samples (Table 3.3). Of these MP, most were fibres of various colours (93.9 %) and the others were brown fragments (6.1 %).

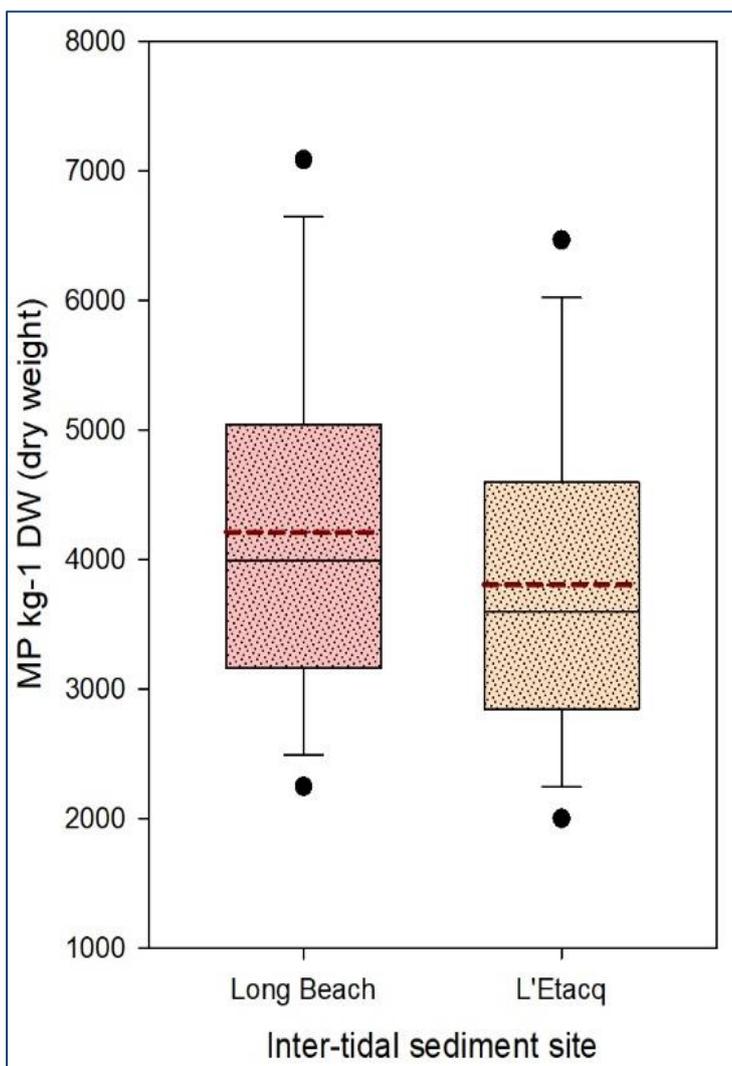
Sample	Description	SCS Code	Size (mm)	Sample	Description	SCS Code	Size (mm)
<b>Long Beach (LB)</b>				<b>L'Etacq (LE)</b>			
LB2 C2	Black fibre	MP/FB/BK	1.053	LE1 C2	Black fibre	MP/FB/BK	1.236
LB1 C2	Blue fibre	MP/FB/BL	1.001	LE1 C1	Blue fibre	MP/FB/BL	1.177
LB2 C1	Blue fibre	MP/FB/BL	1.130	LE1 C2	Blue fibre	MP/FB/BL	1.276
LB2 C1	Blue fibre	MP/FB/BL	1.188	LE1 C2	Blue fibre	MP/FB/BL	1.291
LB2 C1	Blue fibre	MP/FB/BL	1.280	LE1 C2	Blue fibre	MP/FB/BL	1.339
LB2 C2	Blue fibre	MP/FB/BL	1.287	LE1 C1	Blue fibre	MP/FB/BL	1.886
LB1 C1	Blue fibre	MP/FB/BL	1.325	LE1 C1	Brown fragment	MP/FR/BN	2.846
LB2 C2	Blue fibre	MP/FB/BL	1.728	LE1 C1	Brown fragment	MP/FR/BN	1.025
LB2 C2	Blue fibre	MP/FB/BL	1.812	LE1 C2	Grey fibre	MP/FB/GY	2.635
LB2 C2	Blue fibre	MP/FB/BL	2.020	LE2 C2	Pink fibre	MP/FB/PK	1.006
LB2 C2	Green fibre	MP/FB/GN	2.751	LE1 C1	Purple fibre	MP/FB/PR	2.914
LB2 C2	Green fibre	MP/FB/GN	3.108	LE2 C2	Turquoise fibre	MP/FB/TQ	1.160
LB2 C1	Grey fibre	MP/FB/GY	1.422	LE2 C2	Turquoise fibre	MP/FB/TQ	1.286
LB2 C2	Pink fibre	MP/FB/PK	1.223	LE2 C1	Turquoise fibre	MP/FB/TQ	2.329
LB1 C1	Pink fibre	MP/FB/PK	1.234	LE1 C1	Violet fibre	MP/FB/VT	1.002
LB2 C2	Pink fibre	MP/FB/PK	1.702	LE1 C1	Violet fibre	MP/FB/VT	2.503
LB1 C1	Turquoise fibre	MP/FB/TQ	1.335				

**Table 3.4** Detailed catalogue of microplastics > 1 mm. Microplastics are listed under the intertidal sediment they were extracted from, with the specific sample (LB1, LB2, LE1 or LE2) and #centrifugation (C1 or C2) given in the left-hand column. The description gives the same information as the SCS code in a more digestible format in the centre two columns. Each microplastic was measured along the longest diameter (fragment) or length (fibre) three times using ImageJ, with the mean average listed in the right-hand column.

MP from LE samples ranged between 1.002 – 2.914 mm in length. Of these microplastics, 14 were fibres (FB) and 2 were fragments (FR). Blue (BL) was the most common colour (5 FB), followed by turquoise (TQ) (3 FB). MP from LB samples ranged from 1.001 – 3.108 mm in size. All 17 of these microplastics were fibres. BL was the most common colour (9 FB), followed by pink (PK) (3 FB).

### 3.5 ESTIMATES FOR MICROPLASTIC CONTAMINATION IN THE ENVIRONMENT

In order to estimate the true concentrations of microplastics in sediment from each site, it was necessary to apply known constraints to account for external contamination and method extraction efficiency. The two repeats for each site (i.e. LB1 and LB2) were analysed separately. First, the mean average contamination was subtracted from the total count for microplastics > 100 µm on a sample. The mean average contamination was 115 microplastics per sample ( $\pm 54.78$  SD), determined from the tap water blanks. Following this, the mean average extraction efficiency was applied to the residual microplastics observed after an assumed level of contamination had been removed from the count. The mean average extraction efficiency for the method was 31% ( $\pm 9$  % SD), determined from the spiked sediment tests. Finally, the resulting value was doubled to give the value applicable to 1 kg sediment to fit the units (MP kg<sup>-1</sup> DW). Applying the effects of contamination ( $\pm$  SD) & extraction efficiency ( $\pm$  SD) and fitting to the units resulted in nine estimate values for microplastic concentrations for each replicate, and a total of 18 estimates per site.



**Figure 3.4** Box plots showing the estimate microplastic concentration in Jersey intertidal sediments. Units are presented as microplastics per kilogram of dry sediment (MP kg<sup>-1</sup> DW). Calculated estimates are based on an application of standard contamination ( $\pm$ SD) the extraction efficiency ( $\pm$ SD). The red dashed line indicates the mean average, the black line in the centre of each box is the median.

These data are presented for each site as box plots (Figure 3.4). The range of values obtained for LB was 2,248.2 – 7,081.1 MP kg<sup>-1</sup> DW, whilst the range for LE was 2,000.7 – 6,467.2 MP kg<sup>-1</sup> DW. The mean average concentration of microplastics (100 µm – 5 mm) in LB sediments was estimated to be higher than that of LE sediments. The mean average for LB was 4,209 (± 1,377) MP kg<sup>-1</sup> DW and the mean average for LE was 3,806 (± 1,258) MP kg<sup>-1</sup> DW.

### 3.6 GRAIN SIZE ANALYSIS AND SAMPLE LOSS

Grain size analysis was carried out for all sediments put through elutriation (Table 3.5). This included three sediment samples (collected at Hayling Island) used for spiked sediment tests with three different polymers (PA, PS and PVC), and 2 repeat samples from each of the three Jersey intertidal sites analysed (LB, LE and SA). Sediments were sieved through a 5 mm mesh prior to elutriation and some material > 5 mm was retained for most samples. Therefore grain size analysis results would have been slightly different if conducted before elutriation. However, the primary purpose of grain size analysis was to quantify the grain size of sediments as they would have been during elutriation, not as it was when collected from the environment. Additional contamination from stacked sieves and other equipment is a further reason this analysis was not carried out before elutriation. Spiked sediment samples (SPA/SPS/SPVC) were given the description ‘moderately well-sorted, fine sand’, and were very similar in composition, with close mean grain sizes and % sand and mud content. This was expected, as these three samples were sourced from the same location. Similarly, the repeat samples for each Jersey intertidal site (LB1/LB2, LE1/LE2, SA1/SA2) had similar mean grain sizes and sand/mud

Sediment Sample	Mean grain size		Sand (%)	Mud (%)	Loss (%)	Overall sediment description
	µm	φ				
SPA	191.3	2.341	100.0	0.0	1.8	Moderately well sorted, fine sand
SPS	192.0	2.355	99.9	0.1	1.5	Moderately well sorted, fine sand
SPVC	188.2	2.365	99.9	0.1	3.1	Moderately well sorted, fine sand
LB1	329.0	1.751	100.0	0.0	1.3	Moderately sorted, medium sand
LB2	319.2	1.768	100.0	0.0	2.4	Moderately sorted, medium sand
LE1	201.9	2.355	100.0	0.0	51.6	Very well sorted, fine sand
LE2	205.5	2.328	100.0	0.0	2.8	Very well sorted, fine sand
SA1	104.1	3.309	98.2	1.8	11.2	Very well sorted, very fine sand
SA2	104.5	3.301	98.6	1.4	18.9	Very well sorted, very fine sand

**Table 3.5** Grain size analysis results. Samples from the spiked sediment tests are prefixed with ‘S’, followed by the abbreviation for the polymer used to spike the sample. Samples from Jersey intertidal sites are prefixed with the site abbreviation, followed by the number repeat. The arithmetic (µm) and logarithmic (φ) mean grain size is provided for each sample, along with sand and mud content (%), overall sample loss from elutriation and sieving (%), and an overall sediment description.

content between repeat 1 and 2. However, the sediment description differed between the three sites. LB1 and LB2 were given the description 'moderately sorted, medium sand'. LE1 and LE2 were given the description 'very well sorted, fine sand'. SA1 and SA2 were given the description 'very well sorted, very fine sand'. Each sample was 500 g DW prior to elutriation. Therefore grain size analysis allowed for the change in dry weight of sediment samples to be documented. The percentage loss in dry weight of sediment is presented in the 'Loss (%)' column of Table 3.5. The loss for LE1 appears to be an outlier, at 51.6 %, compared to an average loss of 5.4 % for all other samples. The loss of SA samples appear to be substantially higher than others in the data set (not including the 51.6 % outlier of LE1).

Full details of grain size analysis and the resulting data set is provided in the Appendix.

## 4 DISCUSSION

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### 4.1 MICROPLASTICS IN JERSEY INTERTIDAL SEDIMENTS

The ratio of the two size fractions of microplastics (MP:MMP) was the remarkably similar for LB and LE sediments (3:247 approx. for both). This could be indicative of the presence of a greater number of microplastics in the smaller size category (MMP) in the environment, which is consistent with the literature (see Table 1.1). However, there was a considerable difference in extraction efficiency between MP and MMP during the spiked sediment tests (22 and 41 %, respectively). This is likely to have reduced the amount of MP extracted from sediment samples and therefore contributed to the pronounced difference in counts for each size category.

Primary microplastics (PT and MBD) were rare in both sites, with the majority of microplastics appearing to be of secondary origin, with most of these in the form of MFR or MFB. Secondary microplastics occur as a result of degradation from larger plastic items. This suggests that the majority of microplastics found in Jersey sediments are the result of weathering of post-consumer plastic items present in the marine environment. The microplastics colour profiles for each site were also somewhat similar, suggesting that a similar assemblage of microplastics exist in sediments to the East and West of the island. The majority of microplastics observed in both sites were brown, followed by black. These colours are common to natural materials (biological and mineral) in the marine environment. For example, granite is particularly common in Jersey sediments and can be black in colour. This could suggest some level of misidentification during visual sorting. However, even if a marginal portion these particles are of synthetic polymer origin, this may pose a considerable threat to marine species, as black microplastics have been shown to be preferentially ingested by marine species compared to other colours (Ory et al., 2018).

Overall, the microplastics extracted from Jersey intertidal sediments appeared to be similar for western (LE) and eastern (LB) beaches. The estimate microplastic concentrations for LB and LE were marginally different (mean average  $4,209 \pm 1,377$  MP  $\text{kg}^{-1}$  DW and  $3,806 \pm 1,258$  MP  $\text{kg}^{-1}$  DW, respectively). However, as these estimates were subject to two major assumptions (external contamination levels and method extraction efficiency) the range of estimated values varied greatly. Thus it was difficult to quantify the significance of the marginal differences observed. In addition, when the estimated microplastic concentrations for intertidal sites were compared to the literature,

it was found that they were one order of magnitude (or even two) in excess of the concentrations from comparable studies. For example, two studies that quantified the microplastic concentrations of marine sediments in Belgium, and considered a similar size range of microplastics to this study (38  $\mu\text{m}$  – 1 mm), reported concentrations at 166.7 MP  $\text{kg}^{-1}$  DW (harbour), 92.8 MP  $\text{kg}^{-1}$  DW (beach), 17.6 MP  $\text{kg}^{-1}$  DW (high tide line) and 9.2 MP  $\text{kg}^{-1}$  DW (low tide line) (Claessens et al., 2011; Van Cauwenberghe et al., 2013a). Similarly, the microplastic concentrations of Slovenian marine sediments have been recorded at 177.8 MP  $\text{kg}^{-1}$  DW (beach) and 170.4 MP  $\text{kg}^{-1}$  DW (Infralittoral) for microplastics between 250  $\mu\text{m}$  – 5 mm. All of these reported concentrations are at least one order of magnitude lower than the estimates for Jersey intertidal sites. A study by Frère et al. (2017) considered microplastics between 0.7  $\mu\text{m}$  – 1 mm and found concentrations of 0.97 MP  $\text{kg}^{-1}$  DW in subtidal sediments from the Bay of Brest. This is very low compared to the results found for Jersey samples, particularly considering that Frère et al. (2017) included a lower limit for microplastic size that was 2 orders of magnitude smaller than the lower limit for LB and LE microplastics (0.7  $\mu\text{m}$  vs 100  $\mu\text{m}$ ). Conversely, the differences observed between the Bay of Brest and Jersey sediments could be due to the different site sources; subtidal and intertidal, respectively. Microplastics, in particular low density polymers, are known to accumulate on beaches and thus higher concentrations of microplastics are generally found in beach sediments compared to subtidal sediments (Zhang, 2017). One study from Canada with comparable results reported microplastic concentrations on a Nova Scotia beach between 2,000 – 8,000 MP  $\text{kg}^{-1}$  (Mathalon and Hill, 2014). However, similarly to Frère et al. (2017), Mathalon and Hill (2014) consider a lower size limit for microplastics that is 2 orders of magnitude smaller than the lower limit for LB and LE microplastics (0.8  $\mu\text{m}$  vs 100  $\mu\text{m}$ ), thus a greater concentration of microplastics, and not a comparable value, would be expected. These comparisons with data sourced from recent literature call into question the validity and robustness of the data obtained for Jersey intertidal sediments in this study.

With this in mind, it is of note that the amended method had a number of limitations, identified throughout the method optimisation and sample analysis phases of the research. These impacted on the validity of the data, which made it difficult to draw robust conclusions from the results, due to the level of implicit and observed variance. Therefore the remainder of this section is dedicated to a discussion around the various method limitations and suggestions for improvements where relevant.

## 4.2 SOURCES OF CONTAMINATION IN THE LABORATORY

Although steps were taken to minimise contamination (see section 2.6), samples were nonetheless exposed to varying levels of contamination throughout the process of laboratory analysis. This was evident from the results of blanks carried out with tap water, seawater and RO water, with filters from every blank containing microplastics in varying concentrations. It was assumed, prior to the blanks being carried out, that RO water would result in the cleanest blanks. This is because RO water is the purest water source of the three tested mediums and microplastics have been found at trace levels in tap water (Mintenig et al., 2019; Pivokonsky et al., 2018). However, the results indicated otherwise, with tap water resulting in marginally purer blanks than RO water, overall. These results suggested that any microplastics within tap water did not impact on the overall contamination levels observed between tap water and RO water. The base levels of contamination observed across tap water and RO water blanks are therefore likely to originate primarily from laboratory surfaces, equipment and airborne microplastics. Potential sources of microplastic contamination included blue fibres leached from the nylon rope that held the elutriation column in place, grey PVC fragments from the custom-made PVC elutriation column, airborne synthetic fibres, and microplastics in dust that had settled on equipment, hoses and laboratory worktop surfaces.

Microplastics > 1 mm from blank and sample filters were compared (Table 4.1). The morphology of each MP observed was almost exclusively fibres (FB) for both the blanks and samples. In addition, total counts for each of the blanks and analyses were not markedly different.

Blank or sample analysis name	MP Count (> 1 mm)	Morphologies observed	Size Range (mm)
B1	9	FB	1.12 – 5.94
B2	2	FB	1.14 – 1.20
B3	6	FB	1.00 – 1.87
LB1	4	FB	1.00 – 1.34
LB2	13	FB	1.13 – 3.11
LE1	12	FB, FR	1.00 – 2.91
LE2	4	FB	1.00 – 1.29

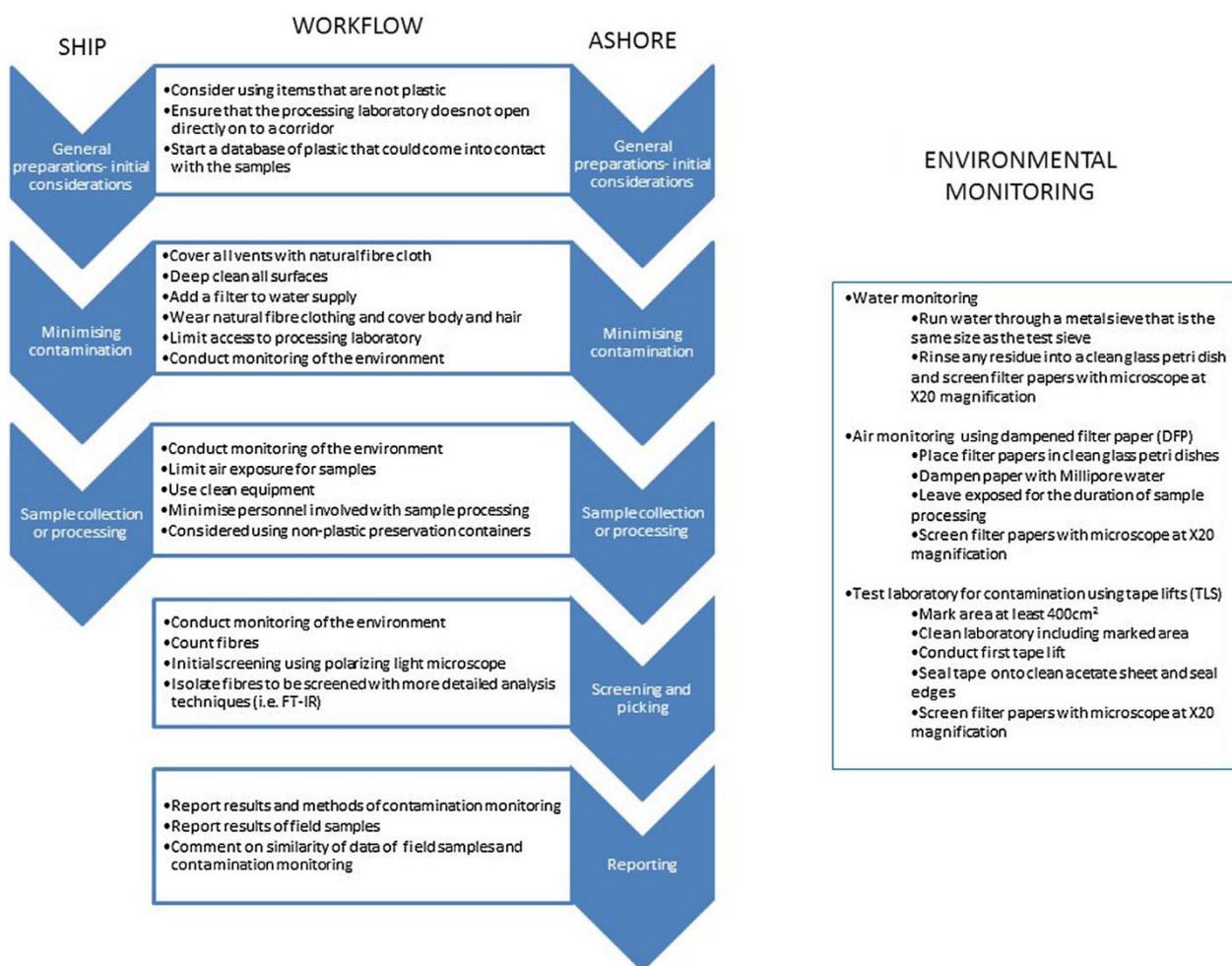
**Table 4.1** Comparison of microplastics > 1 mm observed on filters from tap water blanks and sample analyses. The total count of microplastics > 1 mm is provided along with the morphology codes for microplastics observed and the size range (measured using ImageJ).

The similarity to the blanks suggests that the majority of fibres observed on sample analysis filters originated from external contamination rather than from the environmental sample.

In addition, microscopic materials other than plastics may have been mistaken for microplastics under the microscope. For example, microscopic pieces of the steel 38 µm pore size filters could have been released following them being cut and placed into ZnCl<sub>2</sub>

solution, then transferred to the glass fibre filters following the floatation step. Indeed, the results from visual microscopy indicated a small number of ‘metallic’ microplastics that fit the description of a microscopic steel fragment (see Figure 3.2, viii). Steel is a dense material ( $8.05 \text{ g cm}^{-3}$ ), which should have settled at the base of the 50 mL tube following centrifugation. Therefore it is likely that any steel fragments observed occurred as a result of becoming stuck to the inside of the 50 mL tube, then accidentally removed with the supernatant  $\text{ZnCl}_2$  solution following floatation. It remains uncertain as to whether any of the ‘metallic microplastics’ observed are of synthetic polymer or metal origin, as it is difficult to confirm the composition of microscopic particulates using light microscopy (discussed further in section 4.5).

In order to minimise the impacts of contamination of metal fragments, greater scrutiny of metallic coloured particles should be applied to determine their material. The larger issue of microplastics contamination from laboratory equipment, surfaces and airborne fibres could be tackled using a forensic approach, as described by Woodall et al. (2015) (Figure 4.1). The measures to minimise and monitor contamination in this approach include, and are not limited to, monitoring the contamination in the laboratory by leaving a filter out



**Figure 4.1** Forensic approach workflows for research quantifying microplastics in environmental samples (Woodall et al. 2015).

during sample analysis, wearing only cotton laboratory coats and clothing, and covering all vents with natural fibre cloth. In addition, clean air filters have been shown to reduce airborne microfibre contamination in the laboratory by up to 96.5 % (Wesch et al., 2017). Efforts to wear only 100 % cotton laboratory coats and clothing during analysis of samples is commonplace in the more recent studies quantifying microplastics in the environment (Frère et al., 2017; Steer et al., 2017). In addition, the method could be amended to reduce the amount of plastic equipment used and thereby minimise contamination further. For example, the PVC elutriation column could be replaced with a custom-made metal or glass elutriation column.

### **4.3 SAMPLE LOSS**

The amount of sediment collected from intertidal sites was specified at 500 g per sample, with 500 g DW required for each sediment analysis. As part of sample preparation, sediments were dried in separate containers to remove excess water content. This resulted in 63 % of the intertidal sediment samples prepared weighing less than 500 g after drying (see Appendix, Table i). For the two replicates analysed for each site, SA and LE samples were > 500 g, but one LB sample was < 500 g once dried (490.4 g DW). This sample was topped up with excess dry sediment from the other sample collected from LB (519.4 g DW). The mean average weight loss per sample after drying for all samples prepared was 14.3 %, with a maximum loss of 20.9 %. With this in mind, it would be advised for future research that additional sediment (perhaps 25 % extra, so 625 g minimum if collecting 500 g) is collected in the field in order to ensure the sample size is sufficient to carry out analysis in the laboratory.

As part of the grain size analysis, the total weight of sediment samples that had been analysed was compared to the original 500 g weight to calculate the % loss of sediment following elutriation. All samples reduced in weight to some degree. A small portion of this loss could be attributed to the loss of lighter material (including microplastics) via elutriation. This appears to have impacted SA samples (SA1 and SA2), which were observed to retain higher volumes of material on the retaining sieve. SA1 and SA2 experienced weight losses of 11.2 and 18.9 %, respectively compared to an average loss of 2.2 % in all other samples (excluding an outlier of 51 % loss). However, there are also other steps where loss could have occurred throughout the method. For example, the mesh supporting the sediment at the base of the column had 38 µm apertures, which would have allowed sediment grains < 38 µm to escape at the base of the column. In addition, as the supporting mesh was removed, cleaned and then replaced between

elutriations, it is possible that the sieve could have been replaced ineffectively, leaving a gap for sediment to escape. It was found that one of the samples had reduced in weight by 51 % (LE1), which was likely due to a misplacement of the base sieve mesh.

In addition to sediment loss, there were other steps where microplastics in the sediment could have been lost. All samples were dried prior to elutriation, with some dried in an oven at 60 °C and some dried in an autoclave. After drying was complete, it was noted that the standard temperature of the autoclave was approximately 120 °C. This could have resulted in the melting together of microplastics consisting of polymers with a low melting temperature (i.e. some grades of PE have a melting temperature as low as 80 °C). The presence of microplastics with lower melting temperatures would likely reduce the overall count of microplastics extracted from a sample. This would only be applicable to LE and SA samples, which were dried in the autoclave, and not to LB samples, which were dried in the oven.

During elutriation, microplastics could have become stuck to the inside of the PVC column due to static interactions. In addition, microplastics which exited the column via supernatant water may have stuck to the retainer filter edges or column outflow lip. Between elutriation and floatation, filters were stored in aluminium foil. Despite the removal of excess water from the filters by dabbing the clean side on blue roll prior to being wrapped, the foil degraded quickly on account of the residual moisture present on the filters. This may have resulted in a slight loss of solids retained on the elutriation filters due to gaps in the foil wrap caused by degradation. In addition, the preparation of filters for floatation by cutting into 0.5 – 1 mm squares also could have resulted in the loss of microplastics. During floatation, microplastics may have become trapped inside the glass pipette used to transfer the surface layer to the filter. They could have also become stuck to the sides of the Büchner funnel during vacuum filtration, despite rinsing with RO water to minimise this. In addition, microplastics may have remained within ZnCl<sub>2</sub> solution or stuck to the edges of the centrifuge tube near the surface of the solution.

Further measures could be taken to overcome some of these loss steps throughout the method. This includes securing the base sieve filter in place with bolts before adding sediment to the PVC column, which would minimise the loss of sediment during elutriation. Fewer transfer steps would also reduce the potential for sample loss. For example, instead of wrapping filters in foil then carrying out floatation at a later stage, floatation could be carried out immediately after elutriation to remove the need for wrapping filters between these two method steps. Alternatively, other methods involving

a single step for microplastics extraction from sediments could be considered, such as the use of a Sediment-Microplastic Isolation (SMI) unit, proposed by Coppock et al. (2017). This technique involves a single step floatation using  $ZnCl_2$  solution, thus reducing the potential loss steps through transfer of solids to different containers.

#### **4.4 LOW METHOD EXTRACTION EFFICIENCY**

The spiked sediment tests resulted in varied extraction efficiencies across size range, morphology and polymer type (22 – 41 %). Compared to the results of spiked sediment tests presented in Claessens et al. (2013) (98 – 100 %), these results indicated that the amended method had a very low method extraction efficiency (mean average 31 %). This was substantially lower than the target extraction efficiency of > 90 % that was set as an objective of this study. The issues around extraction efficiency arguably presented the most considerable method limitations, as the efficiency was not only low, but had substantial variation between size and polymer type. This meant that the microplastics profile observed on the filters following extraction from intertidal sediments was not likely to be fully representative of the microplastics in the environment.

PA fibres were recovered from spiked sediment samples at a higher % than fragments of PVC and PS (41 % vs 30 % and 23 %, respectively). This suggests that microplastics of different morphology types and polymers were recovered from sediment samples at different extraction efficiencies. Also, as mentioned previously, microplastics between 1 – 5 mm were recovered at around half the % of microplastics between 100  $\mu m$  – 1 mm (22 and 41 %, respectively). This likely impacted the results of sample analysis by underestimating the contribution of large microplastics to microplastic contamination in sediments. This particular method involving elutriation was therefore deemed to be incompatible with the extraction of microplastics > 1 mm.

Ideally, the extraction efficiency for microplastics should be consistently > 90% for microplastics of varying characteristics (i.e. morphology, polymer, size) found in the environment. Further method optimisation is required to try and achieve a higher extraction efficiency of microplastics from sediments. This could include different aeration stone setups, and/or different water flow rates for elutriation. Another suggested amendment to the method would be to change the initial sieve, used for sorting sediments prior to elutriation, back to 1 mm, as in Claessens et al. (2013). Following this, the sediment sample < 1 mm would be run through the method as described (elutriation, floatation, microscopy). The sediment > 1 mm retained on the sieve would then be sieved through a 5 mm mesh to remove larger debris and put through floatation in high-density

salt solution (no elutriation necessary). In addition, more robust and varied spiked sediment tests should be carried out to assess a wider range of polymer types (i.e. PE, Polyethylene terephthalate; PET) and morphologies (i.e. MBD, MFI, MFM).

#### **4.5 POTENTIAL IMPACTS OF SEDIMENT GRAIN SIZE ON METHOD SUITABILITY**

Grain size analysis revealed that sediments varied in characteristics between intertidal sites (Table 3.5). It is likely that grain size impacted on the results (or lack thereof) obtained from SA sample analysis. SA sediments were the finest of the three Jersey intertidal sediments analysed, assigned as 'very well sorted, very fine sand' following grain size analysis (mean grain size 104.3  $\mu\text{m}$ ). Following elutriation and floatation, the material extracted from SA samples was of substantially more volume than that of the other samples, and was found to be primarily biological material (forams, bivalves, etc.). The volume and abundance of material that was not of synthetic polymer origin rendered analysis of these filters difficult, and thus visual sorting of microplastics was unsuccessful. It is likely that the high-density  $\text{ZnCl}_2$  solution used during the floatation step was sufficiently dense to float the small biological structures within the sediment such as bivalve and foraminifera shells (Figure 3.1). In addition, grain size could have impacted on the overall efficiency of the method. This could be confirmed through further tests involving spiked sediment with different grain size distributions. It is important to have a method which is relatively consistent across different grain sizes, as sediment grain size may also impact the distribution of microplastics found in marine sediments (Martins and Sobral, 2011).

To overcome the prevalence of non-plastic material extracted from finer sediments, further laboratory techniques to isolate microplastics could be employed. A number of studies have used hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) to treat samples prior to analysis in order to remove biological material (Cole et al., 2014; Foekema et al., 2013; Mathalon and Hill, 2014; Wesch et al., 2016). However, this was demonstrated to result in incomplete dissolution of biological tissue and a significant loss of microplastics from samples (Nuelle et al., 2014). The use of technical grade enzymes, including proteinase, chitinase, cellulose and lipase, could offer a more effective approach to remove biological content of sediments. For example, the Basic Enzymatic Purification Protocol (BEPP) proposed by Löder et al. (2017), which involves several stages of enzymatic purification to remove various types of biological material at each stage (shells, exoskeletons, cell walls, etc.). This technique is preferable because enzymes are biological agents and thus do not

destroy or impact on the microplastics present with the sample, as they are not easily impacted by biological processes.

#### **4.6 IDENTIFICATION OF MICROPLASTICS USING LIGHT MICROSCOPY**

Visual sorting using a microscope to define the type, morphology, and colour of microplastics is one of the most common methods of microplastics identification, and is suggested as a first step for microplastics analysis (Hidalgo-Ruz et al., 2012). This method is cost-effective and provides some idea of the range and number of microplastics extracted from sediment samples. Due to a lack of availability of other analytical techniques to identify microplastics for the specific scope of this study, visual sorting using a light microscope was used as the sole method to identify and quantify microplastics in Jersey intertidal sediments. This presented a number of method limitations and issues.

Firstly, visual sorting was extremely time-consuming (approx. 3 – 4 hours per filter). It is a subjective method, which is highly dependent on light levels, the person examining the filters, and the quality and magnification of the dissection microscope. This is particularly true with regard to identifying colour (Song et al., 2015). In addition, visual identification does not allow for the verification of material type. This meant that polymer type could not be confirmed for particles extracted from Jersey samples, and, more importantly, it was not possible to ascertain that the particles visually resembling microplastics were of synthetic polymer origin. The importance of using analytical laboratory techniques to determine the chemical composition the particles resembling microplastics (i.e. FT-IR or Raman spectroscopy, detailed below) was highlighted in a recent case study by Löder and Gerdts (2015). In the case study, only 1.4% of particles that resembled microplastics under the microscope were of synthetic polymer origin (particles between 100 – 500 µm, approx.). The majority of other particles extracted were confirmed as quartz sand granules, using FT-IR analysis and comparison of spectra to the IR spectrum of laboratory quartz. Taking the visual observations into account on their own would therefore result in error rate of 98.6 %. Even considering larger particle sizes (> 500 µm), which are somewhat easier to categorise under the microscope, the error rate of visual sorting reported in the literature ranges from 20 % (Eriksen et al., 2013a) to 70 % (Hidalgo-Ruz et al., 2012). With a lack of laboratory analysis techniques available as part of the scope of this project, it was not possible to confirm the chemical composition of particles observed on the filters following extraction from Jersey intertidal sediments. Therefore it is highly likely that the estimate totals for microplastics concentrations (Figure 3.4) are a gross overestimation of microplastics in the environment. Assuming a high error rate of

98.6%, as presented by Löder and Gerdts (2015), the mean average estimates for LB and LE (without standard deviation) would be altered from 4,209 MP kg<sup>-1</sup> DW and 3,806 MP kg<sup>-1</sup> DW, respectively, to 58.9 MP kg<sup>-1</sup> DW and 53.3 MP kg<sup>-1</sup> DW, respectively. Interestingly, this is more in line with recorded concentrations for sediments published in the literature (Table 1.1). However, it was not possible to determine the actual error rate for microplastic identification in Jersey intertidal samples. Thus these postulations only serve to highlight the implicit variance in the microplastic concentration estimates.

Another fundamental drawback of visual sorting is the size limitation. In this study, initial counts included smaller particles (< 100 µm), which were listed as dark microfragments (MMP/MFR/DK). However, later in the study, it was decided that these fragments should not be included in the final counts, because their minuteness meant that they were unable to be visually discriminated as microplastics with any degree of certainty. The total count without MMP/MFR/DK was 84.8 % lower than the initial count across the two sites (individually, the second count for LB was 87.3 % lower, and for LE was 80.7 % lower than the initial count). Conversely to the issue of error rates described above, this limitation could result in an underestimation of microplastics observed in Jersey sediments, by excluding the size fraction between 38 – 100 µm.

Visual sorting of microplastics is therefore recommended to be used as a first step for microplastics analysis only. New techniques have been developed to assist with visual sorting, including the use of Nile red dye to quantify PE, PP, PS and nylon particles by fluorescence (Erni-Cassola et al., 2017). However, visual sorting is only suitable for identifying larger microplastics. Some studies suggest a particle size limit > 500 µm (Löder and Gerdts, 2015), whilst others suggest a more conservative limit of > 1 mm (Hidalgo-Ruz et al., 2012). Material extracted from intertidal sediment samples should be subject to further analyses to verify synthetic polymer origin of particles observed. As mentioned above, Fourier-Transform Infra-Red spectroscopy (FT-IR) and Raman spectroscopy are common methods that have been used in previous studies to do this (Elert et al., 2017). Both techniques involve obtaining a spectrum for a particle and comparing this to a library of known synthetic polymer spectra. FT-IR is the arguably the most reliable and straightforward method of differentiating between plastic and non-plastic items, and is also capable of identifying particles down to a few µm (Hidalgo-Ruz et al., 2012). Thus FT-IR is the most widely used technique across the literature (Cincinelli et al., 2017; Kunz et al., 2016; Mohamed Nor and Obbard, 2014; Syranidou et al., 2017; Yu et al., 2018). Raman spectroscopy recommended for particles < 20 mm in size, as it provides higher resolution spectra (Shim et al., 2017).

## 5 SUMMARY OF FINDINGS

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A method for extracting microplastics from environmental sediment samples was developed and optimised to cover a wide size range of microplastics (100 – 5 mm). However, the method was subject to a number of limitations. The mean average extraction efficiency achieved using sediments spiked with microplastics was 31 %. This was substantially lower than the target extraction efficiency of > 90 % that was set as an objective of this study. This, along with a number of other method limitations, impacted greatly on the data obtained from sample analysis, and made it difficult to draw any robust conclusions with regards to microplastic contamination in Jersey intertidal sites. A microplastic profile was outlined for the particles observed on filters, however, due to the numerous method limitations and variance in the data, this is not likely to be realistic to the microplastic profile found in the environment and is thus not considered an accurate representation of microplastic contamination in Jersey intertidal sediments. Estimates for microplastic concentrations in Jersey intertidal sediments, even taking contamination levels into account, were at least one order of magnitude in excess of recorded concentrations for sediments in the literature, which further supported the theory that this data was unrealistic.

Other method limitations identified following sample analysis included microplastic contamination in the laboratory, likely in the form of airbourne fibres, microplastic particles on surfaces and equipment, and fragments leached from plastic equipment. This was quantified using the results of tap water blanks. The characteristics of microplastics > 1 mm were remarkably similar between sample and blank filters, suggesting that airbourne filters had contaminated the sample filters to some degree. There were also a number of potential sample loss steps throughout the method for sediments and microplastics, which could have impacted on the final counts. Finally, the visual sorting method used to identify and quantify microplastics on filters was highly subjective. The use of an analytical method to confirm the synthetic polymer origin of particles (i.e. FT-IR or Raman spectroscopy) would have greatly increased the reliability of the data.

These results highlight the implicit variance in microplastics data and substantiate the importance of clean laboratory spaces and standardised methods for the analysis of environmental samples.

## 6 FUTURE DIRECTIONS

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The field of microplastics research is a relatively recent development, and thus the majority of techniques for the monitoring and quantification of microplastic contamination in marine sediments are still in the preliminary stages of testing and validation. This study demonstrates the complexities of microplastics research, with multiple variables and limitations impacting the validity of environmental data, and attests to the importance in working towards standardised techniques for microplastic quantification in sediments.

To further improve the specific method outlined in this study, it is recommended that:

- A forensic approach is adopted (i.e. clean air filters, cotton lab coats, monitoring)
- Non-plastic equipment be used where possible (i.e. metal elutriation column)
- 25 % extra sediment should be collected in the field to account for drying
- The elutriation column base sieve should be secured with bolts to prevent loss
- Flootation should be carried out immediately after elutriation to reduce transfer steps
- Amend the protocol for microplastics > 1 mm (sieve separation then flootation only)
- Carry out further spiked sediment tests to assess microplastics of different polymers and morphologies, and different grain size sediments
- Optimise the aeration and water flow rate to maximise extraction efficiency
- Treat samples with enzymatic purification protocol to remove biological material
- Use FT-IR and/or Raman spectroscopy to confirm synthetic polymer origin

Alternatively, other methods for microplastics extraction from sediments could be considered, such as the use of a Sediment-Microplastic Isolation (SMI) unit, proposed by Coppock et al. (2017), which involves a single step flootation using  $ZnCl_2$  solution.

Overall, priority should be given to standardising the general protocol for microplastics research to keep the impacts of contamination to a consistent minimum across studies. The forensic approach outlined by Woodall et al. (2015) is recommended for future work to quantify microplastics in marine sediments and other environmental samples.

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# APPENDIX

## COSHH AND RISK ASSESSMENT FORMS

Risk Assessment											
Risk Assessment for the activity of:					'Extraction of microplastics from sediment' HAUTON 037						
Unit / Faculty / Directorate:			OES/ FNES		Assessor:		Date				
Line Manager / Supervisor:			[REDACTED]		Primary Site / Location:		19/06/2018 Hannah Brittain				
Brief Details / Additional notes or Comments:			Samples of sediment are fluidised within a large vertical column using an upward flow of filtered seawater. Solids which float to the surface flow out with the supernatant and are retained on filters, which are then processed using 7M zinc chloride solution within a centrifuge.							NOCs Research Aquarium and 454/01	
PART A: RISK ASSESSMENT											
(1) RISK IDENTIFICATION											
Ref No.	Activity.	Hazard.	Potential consequences: <i>(What are the potential consequences from that hazard and how could that harm arise?)</i>	Who might be harmed? <i>(e.g. worker, those nearby, other staff, the public, groups with special considerations)</i>	(2) RISK ASSESSMENT			(3) RISK MANAGEMENT			
					Inherent Risk	Control measures <i>(use the risk hierarchy and indicate what control measures are in place)</i>	Residual Risk	Further controls? <i>(use the risk hierarchy)</i>			
					LIKELIHOOD	RISK / SCORE	LIKELIHOOD	RISK / SCORE			
1	HANDLING ZNCL <sub>2</sub> POWDER TO MAKE UP 7M SOLUTION	ZNCL <sub>2</sub> POWDER IS AN IRRITANT AND POTENTIAL MUTAGEN	SEVERE ACUTE AND CHRONIC HEALTH EFFECTS, I.E. INFLAMMATION AND MUTAGENESIS, CAUSED BY CONTACT OR INHALATION	USER, OTHER LAB USERS IN THE VICINITY IF SPILLS ARE NOT CLEARED UP	4	4 HIGH 100% COTTON LAB COATS ARE WORN AT ALL TIMES WHEN WORKING IN THE LABORATORY. EYE PROTECTION AND NITRILE GAUNTLETS SHOULD BE USED AT ALL TIMES WHEN HANDLING ZNCL <sub>2</sub> POWDER. MASKS SHOULD BE WORN TO MINIMISE RISK OF INHALATION.	2	4 MED 100% COTTON LAB COATS ARE WORN AT ALL TIMES WHEN WORKING IN THE LABORATORY. EYE PROTECTION SHOULD BE USED AT ALL TIMES WHEN HANDLING 7M ZNCL <sub>2</sub> SOLUTIONS. NITRILE GAUNTLETS SHOULD BE WORN WHEN HANDLING 7M ZNCL <sub>2</sub> SOLUTIONS.	NO		
2	HANDLING 7M ZNCL <sub>2</sub> SOLUTION	ZNCL <sub>2</sub> SOLUTION IS AN IRRITANT AND POTENTIAL MUTAGEN	ACUTE AND CHRONIC HEALTH EFFECTS, I.E. INFLAMMATION AND MUTAGENESIS IF CONTACT MADE WITH SKIN OR EYES	USER, OTHER LAB USERS IN THE VICINITY IF SPILLS ARE NOT CLEARED UP	4	3 MED 100% COTTON LAB COATS ARE WORN AT ALL TIMES WHEN WORKING IN THE LABORATORY. EYE PROTECTION SHOULD BE USED AT ALL TIMES WHEN HANDLING 7M ZNCL <sub>2</sub> SOLUTIONS. NITRILE GAUNTLETS SHOULD BE WORN WHEN HANDLING 7M ZNCL <sub>2</sub> SOLUTIONS.	2	3 MED 100% COTTON LAB COATS ARE WORN AT ALL TIMES WHEN WORKING IN THE LABORATORY. EYE PROTECTION SHOULD BE USED AT ALL TIMES WHEN HANDLING 10% HCL SOLUTIONS. NITRILE GAUNTLETS SHOULD BE WORN WHEN HANDLING 10% HCL SOLUTIONS.	NO		
3	ACID WASHING GLASSWARE IN 10% HYDROCHLORIC ACID	HCL IS AN IRRITANT AND SLIGHTLY CORROSIVE TO SKIN, CLOTHING AND EYES	BURNS OR IRRITATION TO THE SKIN OR EYES	USER, OTHER LAB USERS IN THE VICINITY IF SPILLS ARE NOT CLEARED UP	4	3 MED 100% COTTON LAB COATS ARE WORN AT ALL TIMES WHEN WORKING IN THE LABORATORY. EYE PROTECTION SHOULD BE USED AT ALL TIMES WHEN HANDLING 10% HCL SOLUTIONS. NITRILE GAUNTLETS SHOULD BE WORN WHEN HANDLING 10% HCL SOLUTIONS.	2	3 MED 100% COTTON LAB COATS ARE WORN AT ALL TIMES WHEN WORKING IN THE LABORATORY. EYE PROTECTION SHOULD BE USED AT ALL TIMES WHEN HANDLING 10% HCL SOLUTIONS.	NO		
4	EXTRACTING MICROPLASTICS USING THE ELUTRIATION COLUMN	COLUMN WILL BE HEAVY WHILST IN USE AND HAS A HIGH CENTRE OF GRAVITY	A TOPPLED COLUMN OR FALLING FILTER HOLDER COULD CAUSE CRUSHING INJURIES OR DAMAGE TO OTHER EXPERIMENTS IN THE VICINITY	USER, OTHER AQUARIUM USERS IN THE VICINITY	4	3 MED SECURE THE COLUMN USING ROPE AND CONSISTENTLY MONITOR WHILST IN USE TO ENSURE COLUMN DOES NOT TOPPLE AND FILTERS ARE SECURELY PLACED; HOLD IN PLACE IF NECESSARY.	2	3 LOW SECURE THE COLUMN USING ROPE AND CONSISTENTLY MONITOR WHILST IN USE TO ENSURE COLUMN DOES NOT TOPPLE AND FILTERS ARE SECURELY PLACED; HOLD IN PLACE IF NECESSARY.	NO		

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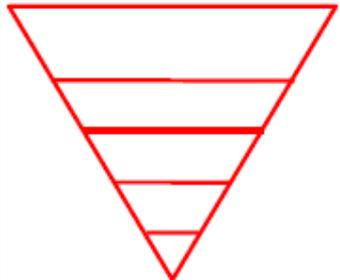
## Health & Safety risk assessment: Risk Process.

- (1) List the individual tasks associated with the work being undertaken.
- (2) Identify the hazards and any potential consequences associated with each of the tasks that you've identified. (A 'hazard' is anything with the potential to cause an adverse consequence, such as an injury or illness. Adverse consequences could also involve, damage to equipment or property)
- (3) Identify the **IMPACT** and **LIKELIHOOD** terms from the table on the next page.  
(i.e. Write down the number used with the Impact & Likelihood terms: e.g. a 'minor' injury = 2 and a 'likely' event = 4).
- (4) Estimate the **INHERENT RISK** (i.e. the risk with no controls applied) by multiplying the **IMPACT** by the **LIKELIHOOD** on the coloured **MATRIX**. (Indicate **HIGH**, **MEDIUM (Med)** or **LOW** on the form and give the overall risk score. e.g. 'minor' impact x 'likely' occurrence is  $2 \times 4 = 8 = \text{MED risk}$ ).

**Risk:** Is likelihood of the hazard event occurring and the potential consequences combined. In estimating risk, consider factors that could exacerbate risk, such as reasonably foreseeable emergencies, lone work, inexperience, new & expectant mothers, waste disposal, potential effects on others such as contractors or visitors, etc.

- (5) If the **INHERENT RISK** is **HIGH (RED)** or **MEDIUM (AMBER)** – identify **CONTROL MEASURES** to reduce the risk to as low as is reasonably practicable.
- (6) **CONTROL MEASURES** should follow the **RISK HIERARCHY**, where appropriate (as below).

Risk Hierarchy of Control		
1. Eliminate	Remove the hazard wherever possible which negates the need for further controls.	If this is not possible then explain why.
2. Substitute	Replace the hazard with one less hazardous.	If not possible then explain why.
3. Physical controls	Examples: enclosure, fume cupboard, glove box	Likely to still require admin controls as well.
4. Admin controls	Examples: Training, supervision, signage.	
5. Personal protection	Examples: respirators, safety specs, gloves.	Last resort as it only protects the individual.



- (7) Estimate the **RESIDUAL RISK** for each hazard. ('Residual' risk is that with controls applied.)
- (8) If the **RESIDUAL RISK** is **MEDIUM (AMBER or score 5 -12)** the activity may proceed or can continue, but you must identify and implement further controls to reduce the risk to as low as reasonably practicable.
- (9) If the **RESIDUAL RISK** is **HIGH (RED or score  $\geq 15$ )** **DO NOT START OR CONTINUE** with the activity until **ADDITIONAL CONTROLS** have been implemented with the risk reduced.
- (10) The cost of implementing control measures can be taken into account but should be proportional to the risk (i.e. a control to reduce a lower risk (i.e. Medium) many not need to be carried out if the cost is high but a control to manage **HIGH** risk means that even at high cost the control would be necessary).
- (11) If the **INHERENT** or **RESIDUAL RISK** is **LOW (GREEN or score 1-4)** then **ADDITIONAL CONTROLS** are not necessary and the activity can proceed.

# Health & Safety: risk estimation matrix

<b>HIGH RISK</b>	If the residual risk is HIGH (red or score >15) <u>do NOT start or continue</u> the activity until additional controls have been implemented and the risk is reduced.
<b>MEDIUM RISK</b>	If the residual risk is MEDIUM (amber or 5-12 score) the activity may proceed or continue but you must identify and implement further controls to reduce the risk to as low as reasonably practicable.
<b>LOW RISK</b>	If the risk is LOW (green or score 1-4), additional controls are not necessary.

IMPACT: Reasonably foreseeable worst-case consequence	Trivial / Insignificant	Minor	Moderate	Major	Severe
	very minor injury (e.g. slight bruising)	Injury or illness: e.g. small cut or abrasion which requires basic first aid treatment even if self-administered.	Injury or illness: e.g. strain or sprain requiring first aid or medical support.	Injuries or illness (e.g. broken bones) requiring medical support >24hr and time off work >4 weeks	Extremely significant, fatal or multiple serious injuries or illness requiring hospital admission or significant time off work.
Likelihood <sup>†</sup> of Hazard Event:	(1)	(2)	(3)	(4)	(5)
<b>Very Likely</b> high probability, 1 in 10 chance or higher, once in two weeks or longer for activities on a daily basis (5)	medium risk 5	medium risk 10	high risk 15	high risk 20	high risk 25
<b>Likely</b> significant probability, 1 in 100 chance or higher, once in six months or longer for activities on a daily basis (4)	low risk 4	medium risk 8	medium risk 12	high risk 16	high risk 20
<b>Possible</b> low probability, 1 in 1,000 chance or higher, once in four years or longer for activities on a daily basis (3)	low risk 3	medium risk 6	medium risk 9	medium risk 12	high risk 15
<b>Unlikely</b> very low probability, 1 in 10,000 chance or higher, once in a decade or longer for activities on a daily basis (2)	low risk 2	low risk 4	medium risk 6	medium risk 8	medium risk 10
<b>Rare</b> extremely low probability, 1 in 100,000 chance. Once in a century or longer for activities on a daily basis (1)	low risk 1	low risk 2	low risk 3	low risk 4	medium risk 5
<b>Risk scores are Impact x Likelihood</b>					

<sup>†</sup> For likelihoods in between the listed values, use the higher likelihood to estimate risk. These probability definitions are only a guide.

# CHEMICAL RISK ASSESSMENT FORM

Version Sept 2013



You will need the most recent MSDS (available from supplier) and the Guidance Notes (available on NOC H&S Website) to fill out this form. Contact the NOC Safety Adviser for further guidance. This assessment only addresses the risk of harm to health from the substances listed. Additional risk assessment may be required to control the risk from other hazards associated with this work/the procedure used

<b>Department:</b>	OES/FNES	<b>Location of use:</b>	NOCs Research aquarium hatchery and 454/01	<b>Persons involved:</b>	Assessor and other signatories to Risk Assessment, COSHH etc
<b>Lab procedure ref:</b>	[REDACTED]			<b>MSDS supplier and revision date:</b>	Sigma Aldrich SAFETY DATA SHEET according to Regulation (EC) No. 453/2010 Version 5.5 Revision Date 28.12.2015 Print Date 01.07.2018
<b>Describe the task:</b>	Extraction of microplastics from sediment using 7M zinc chloride				

## HAZARD IDENTIFICATION AND CONTROL

Chemical(s) or Product Name (As listed in the chemical catalogue or in the MSDS. If mixing chemicals creates a dangerous mixture please note and complete a separate line for this mixture)	Risk Phrases/Hazard Statements (Numbers and wording - full list available on H&S website). If more than one R-phrases [H-statements] choose one that gives rise to most severe classification.	Hazard Group (A,B,C,D,E) Select from Appendix 1	Exposure Potential (High / Med / Low) Assess using Appendix 2	Exposure Control Approach (ECA) Select from Appendix 3
Zinc chloride	Acute toxicity, Oral (Category 4), H302 Skin corrosion (Category 1B), H314 Acute aquatic toxicity (Category 1), H400 Chronic aquatic toxicity (Category 1), H410	A, C	Medium	ECA3

TAB TO THE END OF TABLE TO INSERT NEW ROWS

<b>For multiply chemicals what is the highest ECA required for this task?</b>	
<b>Will you be using a lower level ECA (only allowed for those denoted by*)? If yes, list the ECA and justify why?</b>	No

SPECIAL CONSIDERATIONS	
Could a less hazardous substance be used instead? <i>If yes, then detail why this cannot be used.</i>	No
Does the substance present additional risks to certain groups or individuals? (e.g. young people, expectant mothers)	No
Do your chemicals have risk phrases or hazard statements that require a DSEAR assessment? <i>See appendix 1. If yes, complete and attach a DSEAR Checklist (available on the H&amp;S website)</i>	No

PERSONAL PROTECTIVE EQUIPMENT (PPE)			
<i>State any PPE required for this task/method. Include which type and when they are to be worn. Note: PPE is to be used as the "last resort".</i>			
Eye protection:	Safety goggles should be worn when handling zinc chloride as powder or solution	Hand protection:	Wear nitrile gauntlets when handling zinc chloride.
Face protection:	No, work with open solutions in a fume cupboard	Special clothing:	None
Respiratory protection: <i>(Requires specialist training &amp; monitoring)</i>	No, as chemicals are handled in a fume cupboard	Any others:	Labcoat, closed shoes and long trousers in all cases

EMERGENCY PROCEDURES	
Eye contact:	Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.
Inhalation:	If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician
Skin contact:	Take off contaminated clothing and shoes immediately. Wash off with soap and plenty of water. Consult a physician.
Ingestion:	Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.
Spill procedure:	<p><b>Personal precautions, protective equipment and emergency procedures</b> Use personal protective equipment. Avoid dust formation. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Avoid breathing dust. For personal protection see section 8.</p> <p><b>Environmental precautions</b> Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.</p> <p><b>Methods and materials for containment and cleaning up</b> Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.</p>

HEALTH MONITORING
<p><b>Is health surveillance required for the protection of the health of employees?</b> <i>This is required when: (a) there is a disease associated with the substance in use (eg Asthma, Dermatitis, Cancers); (b) it is possible to detect the disease or adverse change and reduce the risk of further harm; (c) the conditions in the workplace make it likely that the disease will appear. Please refer to Guidance for COSHH Health Surveillance on the H&amp;S Website.</i></p>
No

<p><b>SPECIAL TRAINING REQUIREMENTS</b>  <i>Decide whether any special training is required to carry out the task safely. In most cases, on the job training will be sufficient.</i></p>
<p>None</p>

<p><b>INSTRUCTIONS FOR SAFE STORAGE</b>  <i>How should the substance be stored? (e.g. locked cupboard which is correctly labelled, away from other substances, etc.)          Is there any other substance that this substance must not come into contact with?</i></p>
<p>7M zinc chloride solutions should be stored as 50ml aliquots in screw topped falcon tubes once produced.</p>

<p><b>DISPOSAL PROCEDURES</b> <i>Detail fully how the chemical waste is to be disposed of (down sink, by specialist contractor, etc)</i></p>
<p><i>Are chemicals with Risk Phrases R50-R59 (environmental hazards) involved?</i></p>
<p>Waster zinc chloride should be stored in appropriate hazardous waste containers. Disposal should be arranged through NOCS Estates.</p>

<p><b>ASSESSMENT OF RISK USING CONTROLS DETAILED ABOVE</b></p>
<p><i>Are the hazards/risks suitably controlled, using the control measures detailed above? If not, state the further actions required, e.g. Requirement for a standard operating procedure (SOP), restricting access, prohibiting lone working, specifying supervision, etc in the box below.</i></p>
<p>Yes, although zinc chloride solutions should only be prepared during normal working hours (0900-1700).</p>

<p><b>ACCREDITATION, VERIFICATION AND REVIEW</b>  <i>I am satisfied that the control measures outlined above are adequate to control the risks to health from the hazardous substances used in the work activity described to the lowest level reasonably practicable.</i></p>					
Assessor:	HANNAH BRITTAIN	Signature:		Date:	12-7-18
Approved by:		Signature:		Date:	4-7-18

<p><b>Verification by users</b> <i>(Continue on a separate sheet if necessary)</i>  <i>I have read and understood the information contained in this Assessment and agree to abide with all safety controls.</i></p>		
Name	Signature	Date

Appendix 2: Exposure Potential

EXPOSURE POTENTIAL			
	LOW	MEDIUM	HIGH
Quantity used	<1g or ml	1 to 100g or ml	>100g or ml
Duration	≤ 1 min per day	> 1- 15 min per day	> 15 min per day
No. of persons involved	1 to 2	3 to 4	5 or more
Volatility (liquids)	BP ≥ 150°C or VP ≤ 500 Pa / 3.75 mmHg	BP 50 – 150°C or VP 500 – 25000 Pa/ 3.75 – 187.5 mmHg	BP ≤ 50°C or VP ≥ 25000 Pa/ 187.5 mmHg
Dustiness (particulates)	Pellets and non-dusty solids	Granular or crystalline (coarse dusts)	Fine solids and light powder
Nature of operation	Low energy eg careful handling	Medium energy e.g. pouring from low heights or stirring, use of hand tools	High energy, e.g. spraying, grinding, high speed stirring, sonication etc
<b>Overall Exposure Potential:</b> The more boxes for individual factors that are ticked on the right hand side of the form, the higher the overall exposure potential should be. However, the assessment cannot be based on a simple count of high or low factors, but must rely on the judgement and experience of the assessor.			
			Low
			Medium
			High

Appendix 3: Exposure Control Approach

EXPOSURE CONTROL APPROACH			
<i>Note: NERC Guidance on 20 Standard Controls must be observed at all time (available on the H&amp;S website)</i>			
ECA1: Work in a well constructed laboratory with good general ventilation (an air change rate in excess of 3x per hour) using good working practices to minimise spread / generation of high airborne concentrations of hazardous contaminants.			
ECA2: Work undertaken as above but with the application of engineering controls using LEV devices such as extract grilles, captor hoods or nozzles, partial enclosures with extraction and re-circulating, single HEPA filtered enclosures.			
ECA3: As ECA 1 plus use of high efficiency partial containment devices such as NERC Class 1 fume cupboards which are ducted to external atmosphere or, for solids or aerosols, double HEPA filtered powder handling enclosures / safety cabinets.			
ECA4: Specially devised precautions applied after seeking specialist advice and writing a detailed risk assessment. The precautions applied will involve the highest levels of engineered controls and, although fume cupboards may be appropriate, consideration should be given to using total enclosure devices such as a dedicated laboratory or containment suite may also be appropriate.			
Hazard Group of Substance	E	ECA3*	ECA4*
	D	ECA3*	ECA4*
	C	ECA2*	ECA3
	B	ECA1	ECA2
A	ECA1	ECA1	ECA2*
* These approaches may be varied or relaxed (e.g. the next lower ECA used) as justified by risk assessment		Low	Medium
Overall Exposure Potential			

Appendix 1: Hazard Group A – E

Hazard Group	Type	Concentration range	Units	R-phrases	H-statements
A	Dust	>1 to 10	mg/m <sup>3</sup>	R36, R38 and all R-numbers not otherwise listed	H303, H304, H305, H313, H315, H316, H318, H319, H320, H333, H336 and all H-numbers not otherwise listed
	Vapour	>50 to 500	ppm		
B	Dust	>0.1 to 1	mg/m <sup>3</sup>	R20/21/22 and R68/20/21/22	H302, H312, H332, H371
	Vapour	>5 to 50	ppm		
C	Dust	>0.01 to 0.1	mg/m <sup>3</sup>	R23/24/25, R34, R35, R37, R38/23/24/25, R41, R43, R48/20/21/22, R68/23/24/25	H301, H311, H314, H317, H318, H331, H335, H370, H373
	Vapour	>0.5 to 5	ppm		
D	Dust	<0.01	mg/m <sup>3</sup>	R26/27/28, R30/26/27/28, R40, R48/23/24/25, R60, R61, R62, R63, R64	H300, H310, H330, H351, H360, H361, H362, H372
	Vapour	<0.5	ppm		
E	Dust	-	-	R42, R45, R46, R49, R68	H334, H340, H341, H350
	Vapour	-	-		
<b>Risk Phrases/Hazard Statements requiring a DSEAR Assessment</b>					
R1, R2, R3, R4, R6, R17, R32					
R5, R7, R8, R9, R12, R14, R15, R16, R18, R19, R29, R30, R31, R44					



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Version Sept 2013

You will need the most recent MSDS (available from supplier) and the Guidance Notes (available on NOC H&S Website) to fill out this form. Contact the NOC Safety Adviser for further guidance. This assessment only addresses the risk of harm to health from the substances listed. Additional risk assessment may be required to control the risk from other hazards associated with this work/the procedure used.

<b>Department:</b>	OES/FNES	<b>Location of use:</b>	NOCS 454/01	<b>Persons involved:</b>	Assessor and other signatories to Risk Assessment, COSHH etc
<b>Lab procedure ref:</b>				<b>MSDS supplier and revision date:</b>	LabChem SAFETY DATA SHEET according to Federal Register Vol. 77, No. 58 Date of issue: 03.07.2013 Revision Date 24.10.2017 Print Date 12.07.2018
<b>Describe the task:</b>	Cleaning glassware for the extraction of microplastics from sediment using 10% hydrochloric acid solution				

## HAZARD IDENTIFICATION AND CONTROL

<b>Chemical(s) or Product Name</b> (As listed in the chemical catalogue or in the MSDS. If mixing chemicals creates a dangerous mixture please note and complete a separate line for this mixture)	<b>Risk Phrases/Hazard Statements</b> (Numbers and wording - full list available on H&S website) If more than one P-phrases [H-statements] choose one that gives rise to most severe classification.	<b>Hazard Group</b> (A,B,C,D,E) Select from Appendix 1	<b>Exposure Potential</b> (High / Med / Low) Assess using Appendix 2	<b>Exposure Control Approach (ECA)</b> Select from Appendix 3
Hydrochloric acid	Causes severe skin burns and eye damage (H314)	C	Medium	ECA1

TAB TO THE END OF TABLE TO INSERT NEW ROWS

For multiply chemicals what is the highest ECA required for this task?

No

Will you be using a lower level ECA (only allowed for those denoted by\*)?  
If yes, list the ECA and justify why?

SPECIAL CONSIDERATIONS	
Could a less hazardous substance be used instead? <i>If yes, then detail why this cannot be used.</i>	No
Does the substance present additional risks to certain groups or individuals? (e.g. young people, expectant mothers)	No
Do your chemicals have risk phrases or hazard statements that require a DSEAR assessment? <i>See appendix 1. If yes, complete and attach a DSEAR Checklist (available on the H&amp;S website)</i>	No

PERSONAL PROTECTIVE EQUIPMENT (PPE)			
<i>State any PPE required for this task/method. Include which type and when they are to be worn. Note: PPE is to be used as the "last resort".</i>			
Eye protection:	Safety goggles should be worn when handling hydrochloric acid solution	Hand protection:	Wear nitrile gauntlets and/or tongs when handling hydrochloric acid solution
Face protection:	No	Special clothing:	None
Respiratory protection: <i>(Requires specialist training &amp; monitoring)</i>	No	Any others:	Labcoat, closed shoes and long trousers in all cases

EMERGENCY PROCEDURES	
Eye contact:	Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing and consult a physician.
Inhalation:	Move person into fresh air and keep comfortable for breathing. Consult a physician
Skin contact:	Take off contaminated clothing and shoes immediately. Wash off with soap and plenty of water. Consult a physician.
Ingestion:	Rinse mouth with water. Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Consult a physician.
Spill procedure:	<p><b>Personal precautions, protective equipment and emergency procedures</b> Use personal protective equipment. Ensure adequate ventilation. Do not breathe mist/vapours/spray. Evacuate personnel to safe areas.</p> <p><b>Environmental precautions</b> Prevent further leakage or spillage if safe to do so. Dispose of contents/container to comply with local, state and federal regulations.</p> <p><b>Methods and materials for containment and cleaning up</b> Pick up and arrange disposal without creating vapours or mist. Keep in suitable, closed containers for disposal.</p>

HEALTH MONITORING
<p><b>Is health surveillance required for the protection of the health of employees?</b> <i>This is required when: (a) there is a disease associated with the substance in use (eg Asthma, Dermatitis, Cancers); (b) it is possible to detect the disease or adverse change and reduce the risk of further harm; (c) the conditions in the workplace make it likely that the disease will appear. Please refer to Guidance for COSHH Health Surveillance on the H&amp;S Website.</i></p>
No

<p><b>SPECIAL TRAINING REQUIREMENTS</b>  <i>Decide whether any special training is required to carry out the task safely. In most cases, on the job training will be sufficient.</i></p> <p>None</p>
--

<p><b>INSTRUCTIONS FOR SAFE STORAGE</b>  <i>How should the substance be stored? (e.g. locked cupboard which is correctly labelled, away from other substances, etc.)          Is there any other substance that this substance must not come into contact with?</i></p> <p>Stored in plastic containers. Waste HCl can be diluted with copious amounts of water and run to waste.</p>
---

<p><b>DISPOSAL PROCEDURES</b> <i>Detail fully how the chemical waste is to be disposed of (down sink, by specialist contractor, etc)</i>  <i>Are chemicals with Risk Phrases R50-R59 (environmental hazards) involved?</i></p> <p>Dilute HCl solution with large volume of water and run to waste.</p>
--

<p><b>ASSESSMENT OF RISK USING CONTROLS DETAILED ABOVE</b>  <i>Are the hazards/risks suitably controlled, using the control measures detailed above? If not, state the further actions required, e.g. Requirement for a standard operating procedure (SOP), restricting access, prohibiting lone working, specifying supervision, etc in the box below.</i></p> <p>Yes, although hydrochloric acid solution should only be used during normal working hours (0900-1700).</p>
--

<p><b>ACCREDITATION, VERIFICATION AND REVIEW</b>  <i>I am satisfied that the control measures outlined above are adequate to control the risks to health from the hazardous substances used in the work activity described to the lowest level reasonably practicable.</i></p>					
Assessor:	HANNAH BRITTAIN	Signature:		Date:	11-7-18
Approved by:		Signature:		Date:	
<p><i>Verification by users (Continue on a separate sheet if necessary)          I have read and understood the information contained in this Assessment and agree to abide with all safety controls.</i></p>					
Name		Signature		Date	

Appendix 2: Exposure Potential

EXPOSURE POTENTIAL			
	LOW	MEDIUM	HIGH
Quantity used	<1g or ml	1 to 100g or ml	>100g or ml
Duration	≤ 1 min per day	> 1- 15 min per day	> 15 min per day
No. of persons involved	1 to 2	3 to 4	5 or more
Volatility (liquids)	BP ≥ 150°C or VP ≤ 500 Pa / 3.75 mmHg	BP 50 – 150°C or VP 500 – 25000 Pa/ 3.75 – 187.5 mmHg	BP ≤ 50°C or VP ≥ 25,000 Pa/ 187.5 mmHg
Dustiness (particulates)	Pellets and non-dusty solids	Granular or crystalline (coarse dusts)	Fine solids and light powder
Nature of operation	Low energy eg careful handling	Medium energy e.g. pouring from low heights or stirring, use of hand tools	High energy, e.g. spraying, grinding, high speed stirring, sonication etc
<b>Overall Exposure Potential:</b> The more boxes for individual factors that are ticked on the right hand side of the form, the higher the overall exposure potential should be. However, the assessment cannot be based on a simple count of high or low factors, but must rely on the judgement and experience of the assessor.			
			Low
			Medium
			High

Appendix 3: Exposure Control Approach

EXPOSURE CONTROL APPROACH			
<i>Note: NERC Guidance on 20 Standard Controls must be observed at all time (available on the H&amp;S website)</i>			
ECA1: Work in a well constructed laboratory with good general ventilation (an air change rate in excess of 5x per hour) using good working practices to minimise spread / generation of high airborne concentrations of hazardous contaminants.			
ECA2: Work undertaken as above but with the application of engineering controls using LEV devices such as extract grilles, captor hoods or nozzles, partial enclosures with extraction and re-circulating single HEPA filtered enclosures.			
ECA3: As ECA 1 plus use of high efficiency partial containment devices such as NERC Class 1 fume cupboards which are ducted to external atmosphere or, for solids or aerosols, double HEPA filtered powder handling enclosures / safety cabinets.			
ECA4: Specially devised precautions applied after seeking specialist advice and writing a detailed risk assessment. The precautions applied will involve the highest levels of engineered controls and, although fume cupboards may be appropriate, consideration should be given to using total enclosure devices such as a dedicated laboratory or containment suite may also be appropriate.			
Hazard Group of Substance	E	ECA3*	ECA4*
	D	ECA3*	ECA4*
	C	ECA3*	ECA3
	B	ECA1	ECA2
	A	ECA1	ECA2*
* These approaches may be varied or relaxed (e.g. the next lower ECA used) as justified by risk assessment			Overall Exposure Potential
	Low	Medium	High

Appendix 1: Hazard Group A – E

Hazard Group	Type	Concentration range	Units	R-phrases	H-statements
A	Dust	>1 to 10	mg/m <sup>3</sup>	R38, R38 and all R-numbers not otherwise listed	H303, H304, H305, H313, H315, H316, H318, H319, H320, H333, H336 and all H-numbers not otherwise listed
	Vapour	>50 to 500	ppm		
B	Dust	>0.1 to 1	mg/m <sup>3</sup>	R20/21/22 and R88/20/21/22	H302, H312, H332, H371
	Vapour	>6 to 50	ppm		
C	Dust	>0.01 to 0.1	mg/m <sup>3</sup>	R23/24/25, R34, R35, R37, R38/23/24/25, R41, R43, R48/20/21/22, R68/23/24/25	H301, H311, H314, H317, H318, H331, H335, H370, H373
	Vapour	>0.5 to 5	ppm		
D	Dust	<0.01	mg/m <sup>3</sup>	R26/27/28, R39/28/27/28, R40, R48/23/24/25, R60, R61, R62, R63, R64	H300, H310, H330, H351, H360, H361, H362, H372
	Vapour	<0.5	ppm		
E	Dust	-		R42, R45, R46, R49, R68	H334, H340, H341, H350
	Vapour	-			
<b>Risk Phrases/Hazard Statements requiring a DSE/AR Assessment</b>					
R1, R2, R3, R4, R6, R17, R32	H200, H201, H202, H203, H250 (no H equiv to R32)				
R5, R7, R8, R9, R12, R14, R15, R16, R18, R19, R29, R30, R31, R44	H204, H205, H224, H240, H241, H242, H251, H260, H271 (no H equiv to R29 or R31)				

## SEDIMENT DRYING REGIME RESULTS

Sediment Sample	Repeat	Wet (g)	Oven	In	Out	# days	Dry weight (g)	Weight loss (%)
<b>Cleaned sediment</b>								
Hayling Island	1	770.3	autoclave	09-Jul	12-Jul	3	610.1	20.8
	2	1083.4	autoclave	10-Jul	13-Jul	3	868.3	19.9
	3	831.9	autoclave	20-Jul	23-Jul	3	677.8	18.5
<b>Jersey samples</b>								
St Aubins	1	851.2	autoclave	10-Jul	19-Jul	9	674.1	20.8
	2	735.0	autoclave	10-Jul	16-Jul	6	581.4	20.9
St Brelades	1	510.9	autoclave	10-Jul	16-Jul	6	414.2	18.9
	2	581.7	autoclave	10-Jul	17-Jul	7	464.7	20.1
	3	494.5	autoclave	18-Jul	23-Jul	5	403.7	18.4
La Pulante	1	497.4	autoclave	10-Jul	12-Jul	2	486.9	2.1
	2	572.0	autoclave	10-Jul	12-Jul	2	558.1	2.4
L'Etacq	1	1001.9	autoclave	10-Jul	16-Jul	6	804.6	19.7
	2	857.8	autoclave	10-Jul	18-Jul	8	686.0	20.0
Greve	1	504.4	lab oven	09-Jul	16-Jul	7	487.5	3.4
	2	599.1	lab oven	09-Jul	16-Jul	7	582.6	2.8
St Catherines	1	471.4	lab oven	09-Jul	19-Jul	10	377.6	19.9
	2	554.4	lab oven	09-Jul	18-Jul	9	474.1	14.5
	3	506.5	autoclave	18-Jul	25-Jul	7	454.7	10.2
Long Beach	1	549.0	lab oven	09-Jul	16-Jul	7	519.4	5.4
	2	523.8	lab oven	09-Jul	16-Jul	7	490.4	6.4
Harve des Pas	1	516.6	lab oven	09-Jul	18-Jul	9	448.5	13.2
	2	512.1	lab oven	09-Jul	19-Jul	10	417.2	18.5
	3	501.9	autoclave	18-Jul	25-Jul	7	410.6	18.2
<b>Mean average sediment weight loss from drying (%):</b>								14.3
<b>Maximum sediment weight loss from drying (%):</b>								20.9
<b>Oven temperatures</b>								
Lab oven: 60°C								
Autoclave: 120°C								
<b>Dry weight</b>								
< 500 g after drying								
> 500 g after drying								

**Table i** Sediment samples drying regime, including before and after weights.

## CATALOGUE DETAILS FOR MICROPLASTIC COLOUR EXAMPLES

Full details of the photographs included in Figure 3.2, depicting microplastics of different colours under the SCS System extracted from Jersey sediments.

- (i) Beige (BG): MMP/MFR/BG from LB2 C1 (photo #135, 23/08/18).
- (ii) Black (BK): MMP/MBD/BK from LB1 C1 (photo #17, 28/08/18).
- (iii) Blue (BL): MMP/MFB/BL from LE2 C2 (photo #93, 27/08/18).
- (iv) Brown (BN): MMP/MFR/BN from LE2 C2 (photo #89, 27/08/18).
- (v) Clear (CL): MMP/MFR/CL from LB1 C2 (photo #27, 23/08/18).
- (vi) Green (GN): MMP/MFB/GN from LB2 C1 (photo #142, 23/08/18).
- (vii) Grey (GY): MMP/MFR/GY from LB1 C1 (photo #21, 28/08/18).
- (viii) Metallic (MT): MMP/MFR/MT from LE1 C1 (photo #72, 25/08/18).
- (ix) Olive (OL): MMP/MFR/OL from LE1 C1 (photo #47, 25/08/18).
- (x) Orange (OR): MMP/MFR/OR from LE1 C1 (photo #66, 25/08/18).
- (xi) Pink (PK): MMP/MFB/PK from LE2 C2 (photo #7, 27/08/18).
- (xii) Red (RD): MMP/MFB/RD from LB1 C2 (photo #17, 23/08/18).
- (xiii) Speckled (SP): MMP/MFR/SP from LB1 C1 (photo #42, 28/08/18).
- (xiv) Transparent (TP): MMP/MFR/TP from LB1 C1 (photo #56, 28/08/18).
- (xv) Turquoise (TQ): MMP/MFB/TQ from LE2 C2 (photo #92, 27/08/18).
- (xvi) Violet (VT): MP/FB/VT from LE1 C1 (photo #42, 25/08/18).
- (xvii) White (WT): MMP/MFM/WT from LE1 C1 (photo #77, 25/08/18).
- (xviii) Yellow (YL): MMP/MFR/YL from LE2 C1 (photo #92, 26/08/18).

## GRAIN SIZE ANALYSIS DATA AND DISTRIBUTION GRAPHS

Sample name:	Class Weight Retained (g) in Different Samples								
	St Aubins 1	St Aubins 2	L'Etacq 1	L'Etacq 2	Long Beach 1	Long Beach 2	Spiked (PA)	Spiked (PS)	Spiked (PVC)
Initial Sample Weight (g):	500.00	500.00	500.00	500.00	500.00	500.00	500.00	500.00	500.00
Post-Analysis Sample Weight (g)	449.66	420.67	329.76	486.28	493.38	488.26	491.04	492.75	484.89
Aperture (µm)									
38.00	8.24	6.17	0.00	0.00	0.01	0.00	0.23	0.28	0.29
63.00	71.85	60.27	0.04	0.03	0.06	0.07	1.37	1.98	1.79
90.00	348.04	336.87	3.38	4.03	8.32	12.53	42.60	45.36	45.07
125.00	19.34	15.56	86.98	105.18	52.38	66.50	165.67	153.42	174.58
180.00	1.76	1.42	225.17	354.93	143.43	145.65	223.19	239.54	208.73
250.00	0.35	0.25	12.59	19.32	126.10	108.74	22.70	21.40	20.88
355.00	0.07	0.07	1.30	2.21	83.36	61.70	6.60	5.90	5.99
500.00	0.01	0.03	0.29	0.44	59.87	56.42	5.07	4.65	4.70
710.00	0.00	0.01	0.01	0.08	14.73	22.98	3.78	3.37	3.53
1000.00	0.00	0.02	0.00	0.06	5.12	13.67	19.83	16.85	19.33

**Table ii** Grain size analysis raw data for all sediment samples

An explanation of how the grain size data was obtained through GRADISTAT Version 8.0 (Blott and Pye, 2001) using the above raw data is given below:

“The sample statistics are calculated using the Method of Moments in Microsoft Visual Basic programming language: mean, mode(s), sorting (standard deviation), skewness, kurtosis,  $D_{10}$ ,  $D_{50}$ ,  $D_{90}$ ,  $D_{90}/D_{10}$ ,  $D_{90}-D_{10}$ ,  $D_{75}/D_{25}$  and  $D_{75}-D_{25}$ . Grain size parameters are calculated arithmetically and geometrically (in microns –  $\mu\text{m}$ ) and logarithmically (using the phi scale –  $\phi$ ) (Krumbein and Pettijohn, 1938). Linear interpolation is also used to calculate statistical parameters by the Folk and Ward (1957) graphical method and derive physical descriptions (such as “very coarse sand” and “moderately sorted”). The program also provides a physical description of the textural group which the sample belongs to and the sediment name (such as “fine gravelly coarse sand”) after Folk (1954). Also included is a table giving the percentage of grains falling into each size fraction, modified from Udden (1914) and Wentworth (1922). In terms of graphical output, the program provides graphs of the grain size distribution and cumulative distribution of the data in both metric and phi units, and displays the sample grain size on triangular diagrams.” (Blott and Pye, 2001).

SIEVING ERROR:	11.2%	<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY:	SA1	ANALYST & DATE: Hannah Brittain, 8/31/2018				
SAMPLE TYPE:	Unimodal, Very Well Sorted	TEXTURAL GROUP: Sand				
SEDIMENT NAME:	Very Well Sorted Very Fine Sand					
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	107.5	3.237	GRAVEL:	0.0%	COARSE SAND:	0.0%
MODE 2:			SAND:	98.2%	MEDIUM SAND:	0.1%
MODE 3:			MUD:	1.8%	FINE SAND:	4.7%
D <sub>10</sub> :	75.60	3.032			V FINE SAND:	93.4%
MEDIAN or D <sub>50</sub> :	103.2	3.277	V COARSE GRAVEL:	0.0%	V COARSE SILT:	1.8%
D <sub>90</sub> :	122.3	3.725	COARSE GRAVEL:	0.0%	COARSE SILT:	0.0%
(D <sub>90</sub> / D <sub>10</sub> ):	1.617	1.229	MEDIUM GRAVEL:	0.0%	MEDIUM SILT:	0.0%
(D <sub>90</sub> - D <sub>10</sub> ):	46.67	0.694	FINE GRAVEL:	0.0%	FINE SILT:	0.0%
(D <sub>75</sub> / D <sub>25</sub> ):	1.236	1.098	V FINE GRAVEL:	0.0%	V FINE SILT:	0.0%
(D <sub>75</sub> - D <sub>25</sub> ):	21.94	0.306	V COARSE SAND:	0.0%	CLAY:	0.0%
	METHOD OF MOMENTS			FOLK & WARD METHOD		
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN( $\bar{x}$ ):	104.1	100.9	3.309	102.1	3.293	Very Fine Sand
SORTING ( $\sigma$ ):	19.61	1.207	0.272	1.189	0.250	Very Well Sorted
SKEWNESS ( $sk$ ):	2.417	-0.745	0.745	-0.240	0.240	Fine Skewed
KURTOSIS ( $k$ ):	36.37	8.278	8.278	1.185	1.185	Leptokurtic

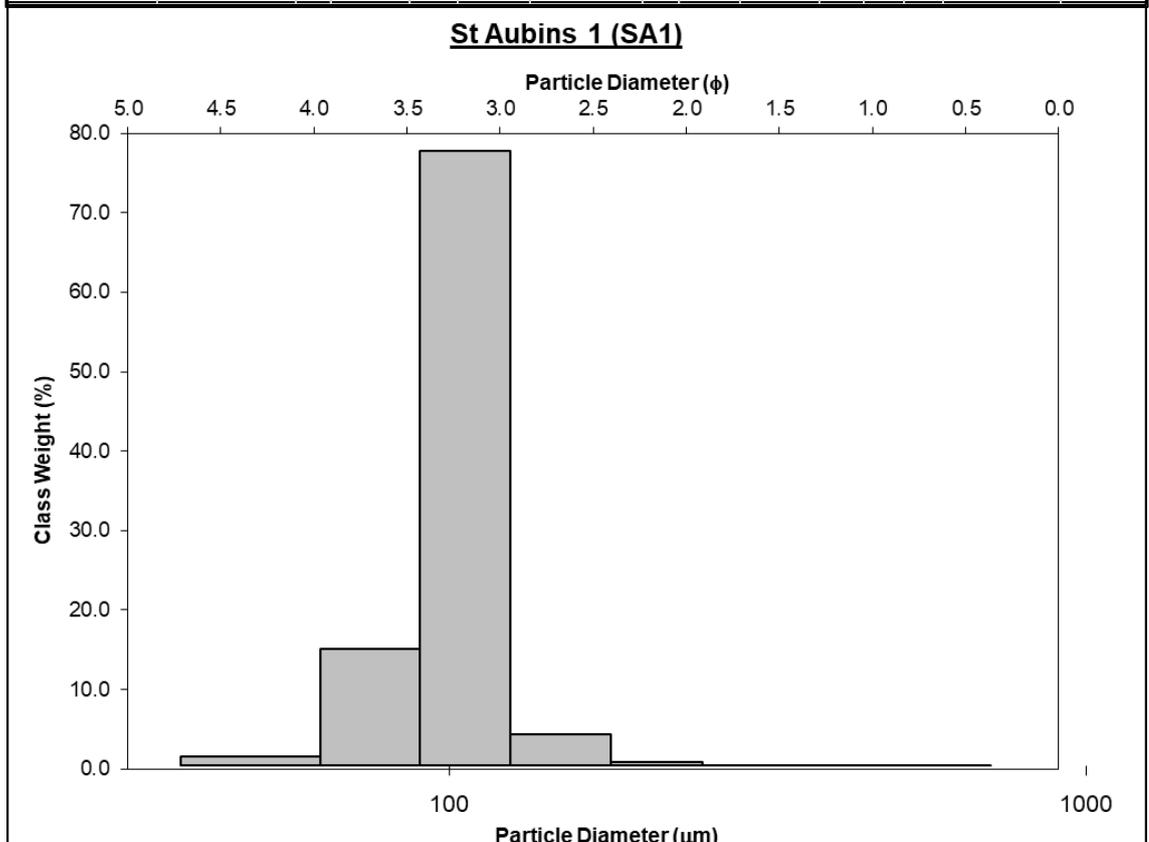


Figure i Grain size analysis data and distribution graph for SA1

SIEVING ERROR:	18.9%	<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY:	<b>SA2</b>	ANALYST & DATE: Hannah Brittain, 8/31/2018				
SAMPLE TYPE:	Unimodal, Very Well Sorted	TEXTURAL GROUP: Sand				
SEDIMENT NAME:	Very Well Sorted Very Fine Sand					
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	107.5	3.237	GRAVEL:	0.0%	COARSE SAND:	0.0%
MODE 2:			SAND:	98.6%	MEDIUM SAND:	0.1%
MODE 3:			MUD:	1.4%	FINE SAND:	4.0%
D <sub>10</sub> :	77.91	3.035			V FINE SAND:	94.4%
MEDIAN or D <sub>50</sub> :	103.6	3.271	V COARSE GRAVEL:	0.0%	V COARSE SILT:	1.4%
D <sub>90</sub> :	122.0	3.682	COARSE GRAVEL:	0.0%	COARSE SILT:	0.0%
(D <sub>90</sub> / D <sub>10</sub> ):	1.566	1.213	MEDIUM GRAVEL:	0.0%	MEDIUM SILT:	0.0%
(D <sub>90</sub> - D <sub>10</sub> ):	44.11	0.647	FINE GRAVEL:	0.0%	FINE SILT:	0.0%
(D <sub>75</sub> / D <sub>25</sub> ):	1.228	1.095	V FINE GRAVEL:	0.0%	V FINE SILT:	0.0%
(D <sub>75</sub> - D <sub>25</sub> ):	21.28	0.296	V COARSE SAND:	0.0%	CLAY:	0.0%
	METHOD OF MOMENTS			FOLK & WARD METHOD		
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN ( $\bar{x}$ ):	104.5	101.5	3.301	103.6	3.271	Very Fine Sand
SORTING ( $\sigma$ ):	19.00	1.196	0.255	1.173	0.230	Very Well Sorted
SKEWNESS ( $sk$ ):	4.721	-1.519	0.628	-0.189	0.189	Fine Skew ed
KURTOSIS ( $k$ ):	120.1	30.76	11.41	1.186	1.186	Leptokurtic

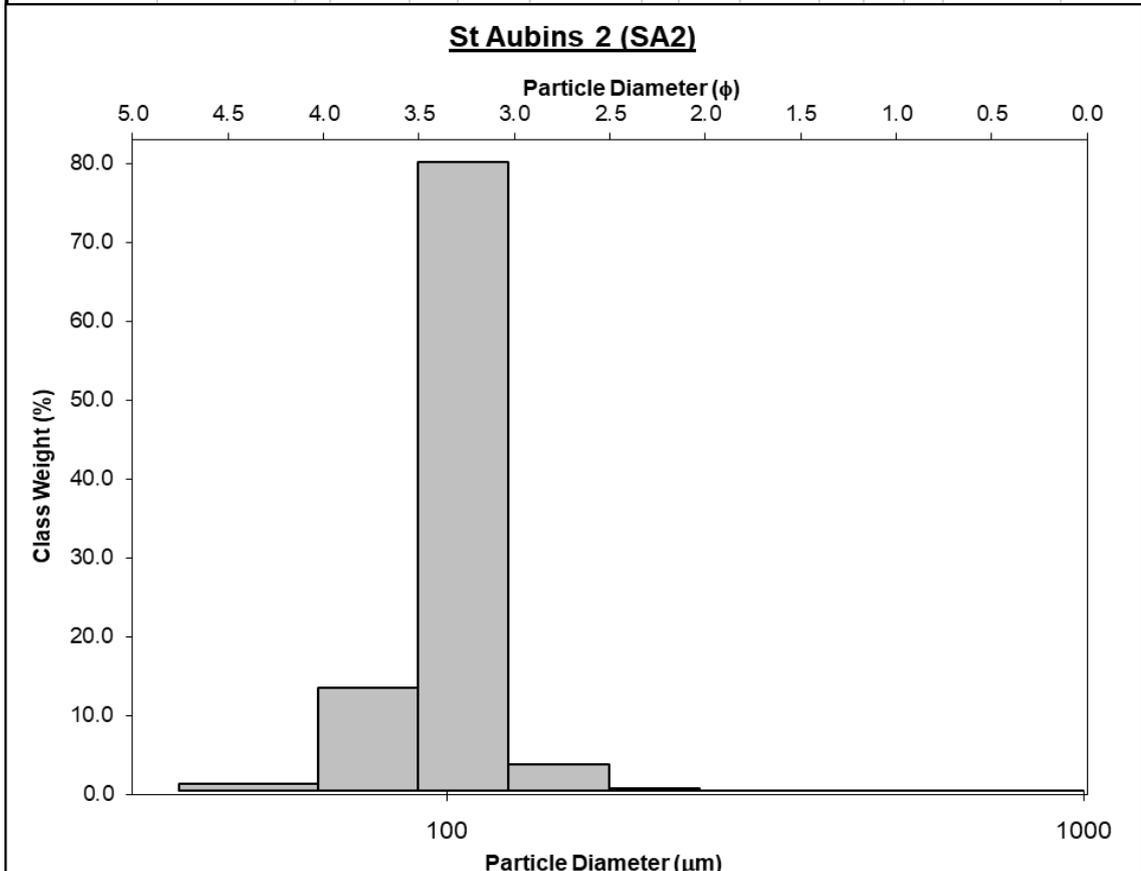


Figure ii Grain size analysis data and distribution graph for SA2

SIEVING ERROR: 51.6%		<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY: <b>LE1</b>			ANALYST & DATE: Hannah Brittain, 8/31/2018			
SAMPLE TYPE: Unimodal, Very Well Sorted			TEXTURAL GROUP: Sand			
SEDIMENT NAME: Very Well Sorted Fine Sand						
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	215.0	2.237	GRAVEL:	0.0%	COARSE SAND: 0.1%	
MODE 2:			SAND:	100.0%	MEDIUM SAND: 4.2%	
MODE 3:			MUD:	0.0%	FINE SAND: 94.7%	
D <sub>10</sub> :	141.5	2.040			V FINE SAND: 1.0%	
MEDIAN or D <sub>50</sub> :	200.7	2.317	V COARSE GRAVEL:	0.0%	V COARSE SILT: 0.0%	
D <sub>90</sub> :	243.2	2.821	COARSE GRAVEL:	0.0%	COARSE SILT: 0.0%	
(D <sub>90</sub> / D <sub>10</sub> ):	1.719	1.383	MEDIUM GRAVEL:	0.0%	MEDIUM SILT: 0.0%	
(D <sub>90</sub> - D <sub>10</sub> ):	101.8	0.782	FINE GRAVEL:	0.0%	FINE SILT: 0.0%	
(D <sub>75</sub> / D <sub>25</sub> ):	1.300	1.177	V FINE GRAVEL:	0.0%	V FINE SILT: 0.0%	
(D <sub>75</sub> - D <sub>25</sub> ):	52.21	0.378	V COARSE SAND:	0.0%	CLAY: 0.0%	
	METHOD OF MOMENTS		FOLK & WARD METHOD			
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN ( $\bar{x}$ ):	201.9	195.4	2.355	193.9	2.367	Fine Sand
SORTING ( $\sigma$ ):	39.96	1.213	0.278	1.226	0.294	Very Well Sorted
SKEWNESS ( $sk$ ):	1.737	-0.138	0.138	-0.279	0.279	Fine Skewed
KURTOSIS ( $k$ ):	17.75	4.990	4.990	0.992	0.992	Mesokurtic

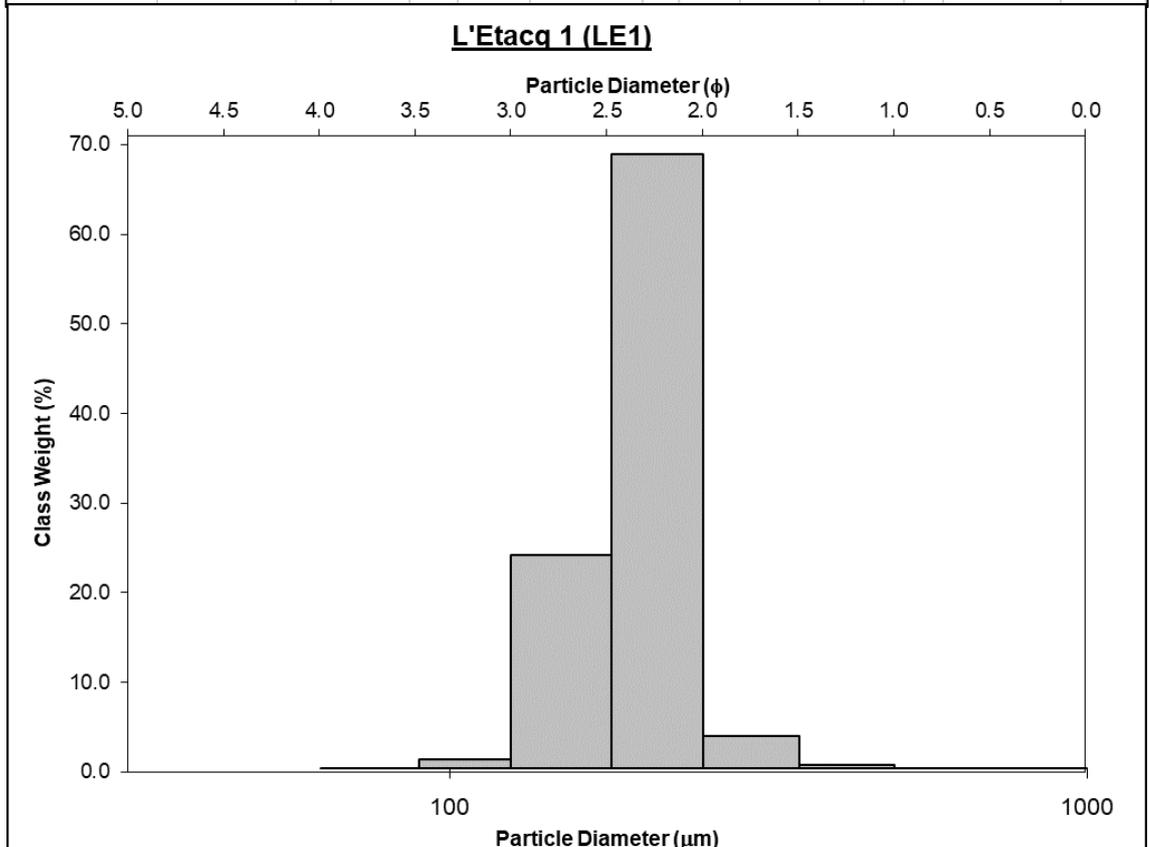


Figure iii Grain size analysis data and distribution graph for LE1

SIEVING ERROR: 2.8%		<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY: <b>LE2</b>			ANALYST & DATE: Hannah Brittain, 8/31/2018			
SAMPLE TYPE: Unimodal, Very Well Sorted			TEXTURAL GROUP: Sand			
SEDIMENT NAME: Very Well Sorted Fine Sand						
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	215.0	2.237	GRAVEL: 0.0%	COARSE SAND: 0.1%		
MODE 2:			SAND: 100.0%	MEDIUM SAND: 4.4%		
MODE 3:			MUD: 0.0%	FINE SAND: 94.6%		
D <sub>10</sub> :	145.9	2.035		V FINE SAND: 0.8%		
MEDIAN or D <sub>50</sub> :	203.7	2.295	V COARSE GRAVEL: 0.0%	V COARSE SILT: 0.0%		
D <sub>90</sub> :	243.9	2.777	COARSE GRAVEL: 0.0%	COARSE SILT: 0.0%		
(D <sub>90</sub> / D <sub>10</sub> ):	1.672	1.364	MEDIUM GRAVEL: 0.0%	MEDIUM SILT: 0.0%		
(D <sub>90</sub> - D <sub>10</sub> ):	98.05	0.742	FINE GRAVEL: 0.0%	FINE SILT: 0.0%		
(D <sub>75</sub> / D <sub>25</sub> ):	1.252	1.152	V FINE GRAVEL: 0.0%	V FINE SILT: 0.0%		
(D <sub>75</sub> - D <sub>25</sub> ):	45.95	0.325	V COARSE SAND: 0.0%	CLAY: 0.0%		
	METHOD OF MOMENTS		FOLK & WARD METHOD			
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN ( $\bar{x}$ ):	205.5	199.0	2.328	198.4	2.334	Fine Sand
SORTING ( $\sigma$ ):	39.63	1.214	0.267	1.210	0.275	Very Well Sorted
SKEWNESS ( $sk$ ):	2.383	-2.605	0.031	-0.277	0.277	Fine Skew ed
KURTOSIS ( $k$ ):	28.21	74.00	6.853	1.131	1.131	Leptokurtic

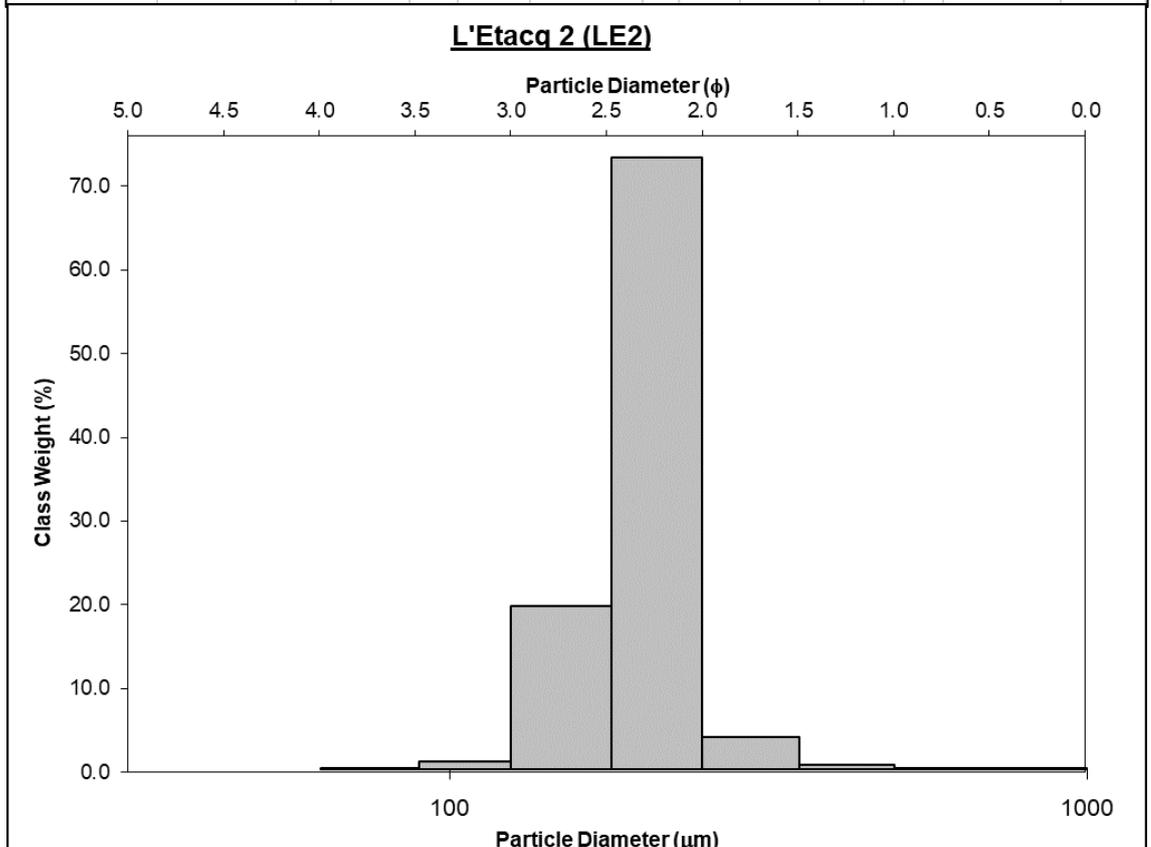


Figure iv Grain size analysis data and distribution graph for LE2

SIEVING ERROR:	1.3%	<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY:	<b>LB1</b>	ANALYST & DATE: Hannah Brittain, 8/31/2018				
SAMPLE TYPE:	Unimodal, Moderately Sorted	TEXTURAL GROUP: Sand				
SEDIMENT NAME:	Moderately Sorted Medium Sand					
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	215.0	2.237	GRAVEL:	0.0%	COARSE SAND:	16.2%
MODE 2:			SAND:	100.0%	MEDIUM SAND:	42.5%
MODE 3:			MUD:	0.0%	FINE SAND:	39.7%
D <sub>10</sub> :	166.2	0.743			V FINE SAND:	1.7%
MEDIAN or D <sub>50</sub> :	281.4	1.830	V COARSE GRAVEL:	0.0%	V COARSE SILT:	0.0%
D <sub>90</sub> :	597.4	2.589	COARSE GRAVEL:	0.0%	COARSE SILT:	0.0%
(D <sub>90</sub> / D <sub>10</sub> ):	3.594	3.483	MEDIUM GRAVEL:	0.0%	MEDIUM SILT:	0.0%
(D <sub>90</sub> - D <sub>10</sub> ):	431.2	1.845	FINE GRAVEL:	0.0%	FINE SILT:	0.0%
(D <sub>75</sub> / D <sub>25</sub> ):	2.012	1.801	V FINE GRAVEL:	0.0%	V FINE SILT:	0.0%
(D <sub>75</sub> - D <sub>25</sub> ):	210.2	1.009	V COARSE SAND:	0.0%	CLAY:	0.0%
	METHOD OF MOMENTS			FOLK & WARD METHOD		
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN ( $\bar{x}$ ):	329.0	276.5	1.751	298.2	1.746	Medium Sand
SORTING ( $\sigma$ ):	168.4	2.091	0.689	1.629	0.704	Moderately Sorted
SKEWNESS ( $sk$ ):	1.148	-4.438	-0.338	0.151	-0.151	Coarse Skewed
KURTOSIS ( $k$ ):	4.126	35.47	2.606	0.935	0.935	Mesokurtic

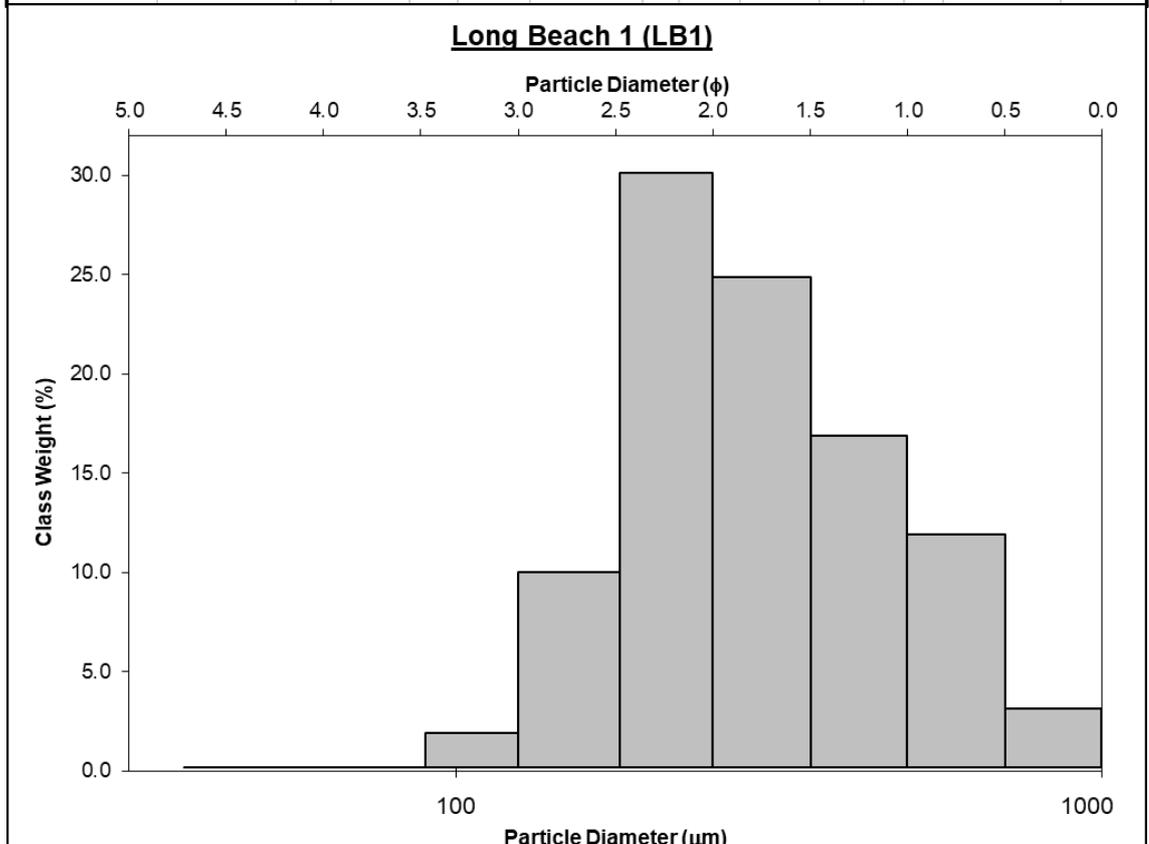


Figure v Grain size analysis data and distribution graph for LB1

SIEVING ERROR: 2.4%		<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY: <b>LB2</b>			ANALYST & DATE: Hannah Brittain, 8/31/2018			
SAMPLE TYPE: Unimodal, Moderately Sorted			TEXTURAL GROUP: Sand			
SEDIMENT NAME: Moderately Sorted Fine Sand						
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	215.0	2.237	GRAVEL:	0.0%	COARSE SAND: 19.1%	
MODE 2:			SAND:	100.0%	MEDIUM SAND: 34.9%	
MODE 3:			MUD:	0.0%	FINE SAND: 43.5%	
D <sub>10</sub> :	152.5	0.603			V FINE SAND: 2.6%	
MEDIAN or D <sub>50</sub> :	266.1	1.910	V COARSE GRAVEL:	0.0%	V COARSE SILT: 0.0%	
D <sub>90</sub> :	658.3	2.713	COARSE GRAVEL:	0.0%	COARSE SILT: 0.0%	
(D <sub>90</sub> / D <sub>10</sub> ):	4.317	4.498	MEDIUM GRAVEL:	0.0%	MEDIUM SILT: 0.0%	
(D <sub>90</sub> - D <sub>10</sub> ):	505.8	2.110	FINE GRAVEL:	0.0%	FINE SILT: 0.0%	
(D <sub>75</sub> / D <sub>25</sub> ):	2.146	1.894	V FINE GRAVEL:	0.0%	V FINE SILT: 0.0%	
(D <sub>75</sub> - D <sub>25</sub> ):	227.4	1.102	V COARSE SAND:	0.0%	CLAY: 0.0%	
	METHOD OF MOMENTS			FOLK & WARD METHOD		
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN( $\bar{x}$ ):	319.2	241.9	1.768	296.8	1.752	Medium Sand
SORTING ( $\sigma$ ):	188.5	2.872	0.775	1.752	0.809	Moderately Sorted
SKEWNESS ( $sk$ ):	1.190	-3.798	-0.482	0.274	-0.274	Coarse Skewed
KURTOSIS ( $k$ ):	4.080	20.69	2.541	0.995	0.995	Mesokurtic

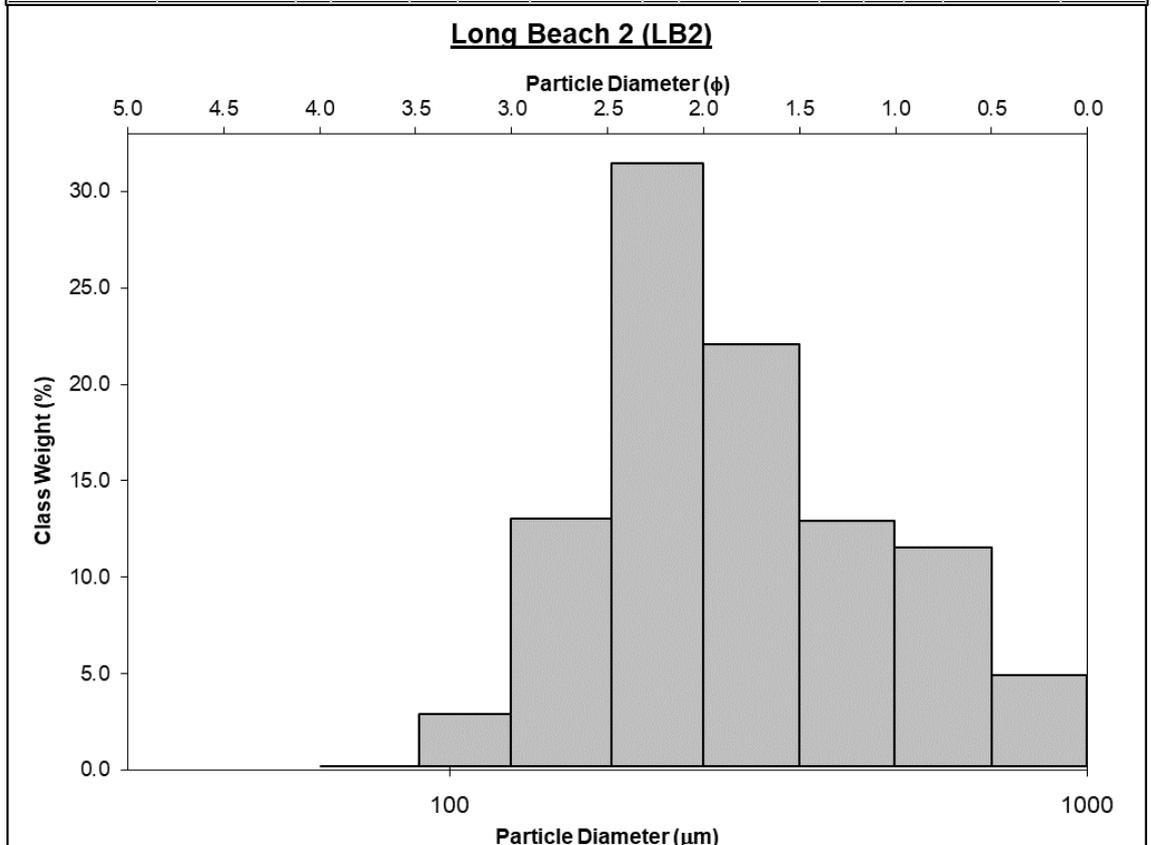


Figure vi Grain size analysis data and distribution graph for LB2

SIEVING ERROR: 1.8%		<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY: PA		ANALYST & DATE: Hannah Brittain, 8/31/2018				
SAMPLE TYPE: Unimodal, Moderately Well Sorted		TEXTURAL GROUP: Sand				
SEDIMENT NAME: Moderately Well Sorted Fine Sand						
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	215.0	2.237	GRAVEL: 0.0%		COARSE SAND: 5.8%	
MODE 2:			SAND: 100.0%		MEDIUM SAND: 6.0%	
MODE 3:			MUD: 0.0%		FINE SAND: 79.2%	
D <sub>10</sub> :	126.4	1.802			V FINE SAND: 9.0%	
MEDIAN or D <sub>50</sub> :	189.7	2.398	V COARSE GRAVEL: 0.0%		V COARSE SILT: 0.0%	
D <sub>90</sub> :	286.7	2.984	COARSE GRAVEL: 0.0%		COARSE SILT: 0.0%	
(D <sub>90</sub> / D <sub>10</sub> ):	2.269	1.656	MEDIUM GRAVEL: 0.0%		MEDIUM SILT: 0.0%	
(D <sub>90</sub> - D <sub>10</sub> ):	160.4	1.182	FINE GRAVEL: 0.0%		FINE SILT: 0.0%	
(D <sub>75</sub> / D <sub>25</sub> ):	1.529	1.287	V FINE GRAVEL: 0.0%		V FINE SILT: 0.0%	
(D <sub>75</sub> - D <sub>25</sub> ):	78.67	0.613	V COARSE SAND: 0.0%		CLAY: 0.0%	
	METHOD OF MOMENTS			FOLK & WARD METHOD		
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN ( $\bar{x}$ ):	191.3	149.4	2.341	183.7	2.444	Fine Sand
SORTING ( $\sigma$ ):	96.83	2.935	0.670	1.527	0.610	Moderately Well Sorted
SKEWNESS ( $sk$ ):	3.161	-3.969	-1.875	0.107	-0.107	Coarse Skewed
KURTOSIS ( $k$ ):	21.67	18.98	7.628	1.759	1.759	Very Leptokurtic

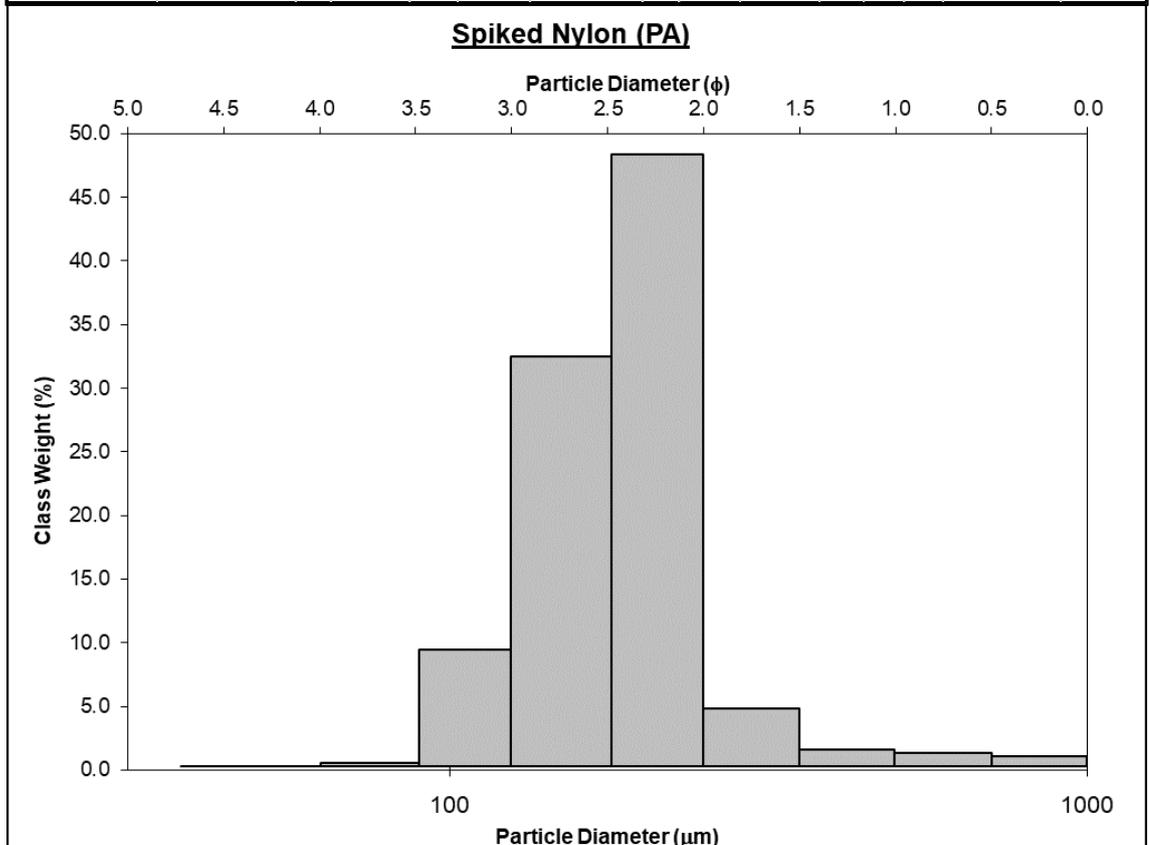


Figure vii Grain size analysis data and distribution graph for PA

SIEVING ERROR:	1.5%	<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY:	PS	ANALYST & DATE: Hannah Brittain, 8/31/2018				
SAMPLE TYPE:	Unimodal, Moderately Well Sorted	TEXTURAL GROUP: Sand				
SEDIMENT NAME:	Moderately Well Sorted Fine Sand					
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	215.0	2.237	GRAVEL: 0.0%		COARSE SAND: 5.0%	
MODE 2:			SAND: 99.9%		MEDIUM SAND: 5.5%	
MODE 3:			MUD: 0.1%		FINE SAND: 79.7%	
D <sub>10</sub> :	125.5	1.932			V FINE SAND: 9.6%	
MEDIAN or D <sub>50</sub> :	191.5	2.384	V COARSE GRAVEL: 0.0%		V COARSE SILT: 0.1%	
D <sub>90</sub> :	262.1	2.994	COARSE GRAVEL: 0.0%		COARSE SILT: 0.0%	
(D <sub>90</sub> / D <sub>10</sub> ):	2.089	1.550	MEDIUM GRAVEL: 0.0%		MEDIUM SILT: 0.0%	
(D <sub>90</sub> - D <sub>10</sub> ):	136.7	1.063	FINE GRAVEL: 0.0%		FINE SILT: 0.0%	
(D <sub>75</sub> / D <sub>25</sub> ):	1.516	1.280	V FINE GRAVEL: 0.0%		V FINE SILT: 0.0%	
(D <sub>75</sub> - D <sub>25</sub> ):	77.21	0.600	V COARSE SAND: 0.0%		CLAY: 0.0%	
	METHOD OF MOMENTS			FOLK & WARD METHOD		
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN( $\bar{x}$ ):	192.0	154.4	2.355	183.9	2.443	Fine Sand
SORTING ( $\sigma$ ):	92.39	2.720	0.639	1.467	0.553	Moderately Well Sorted
SKEWNESS ( $sk$ ):	3.237	-4.278	-1.839	0.017	-0.017	Symmetrical
KURTOSIS ( $k$ ):	23.22	22.11	8.129	1.546	1.546	Very Leptokurtic

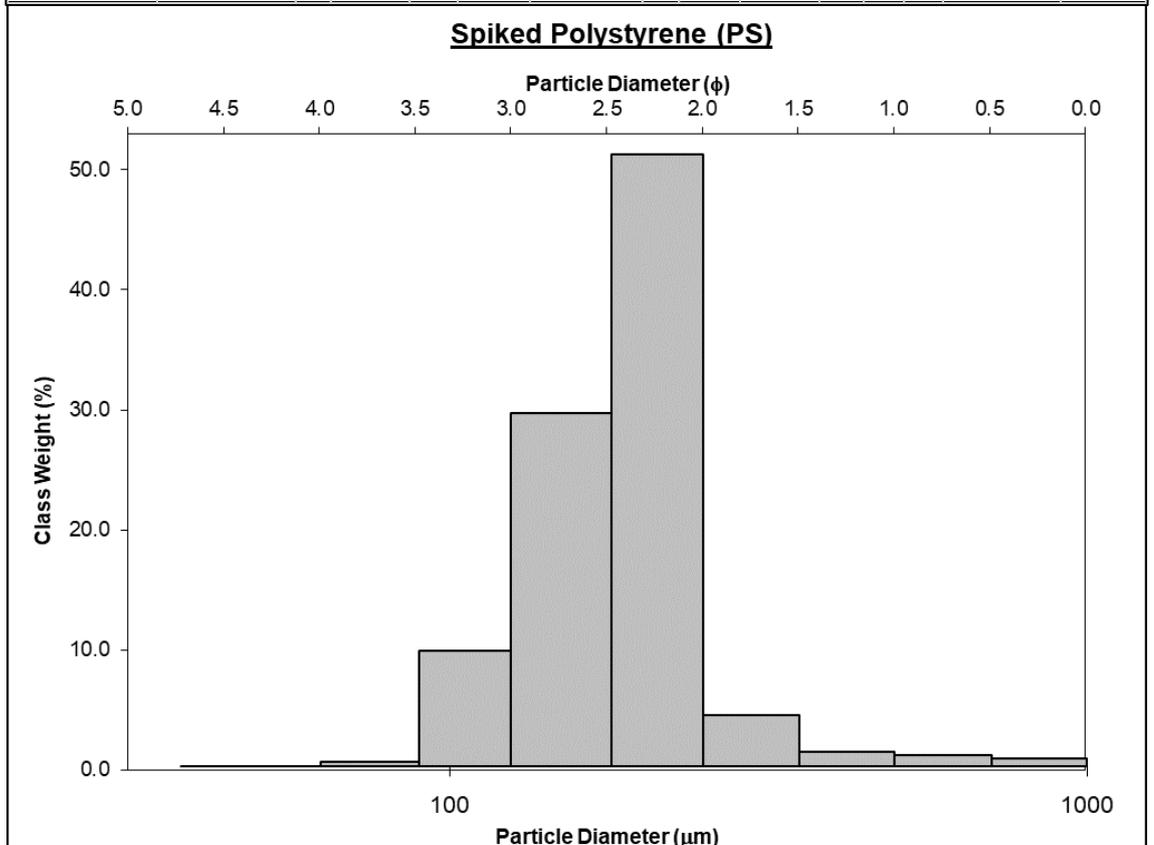


Figure viii Grain size analysis data and distribution graph for PS

SIEVING ERROR: 3.1%		<b>SAMPLE STATISTICS</b>				
SAMPLE IDENTITY: <b>PVC</b>			ANALYST & DATE: Hannah Brittain, 8/31/2018			
SAMPLE TYPE: Unimodal, Moderately Well Sorted			TEXTURAL GROUP: Sand			
SEDIMENT NAME: Moderately Well Sorted Fine Sand						
	$\mu\text{m}$	$\phi$	GRAIN SIZE DISTRIBUTION			
MODE 1:	215.0	2.237	GRAVEL:	0.0%	COARSE SAND: 5.7%	
MODE 2:			SAND:	99.9%	MEDIUM SAND: 5.5%	
MODE 3:			MUD:	0.1%	FINE SAND: 79.1%	
$D_{10}$ :	125.4	1.856			V FINE SAND: 9.7%	
MEDIAN or $D_{50}$ :	186.0	2.427	V COARSE GRAVEL:	0.0%	V COARSE SILT: 0.1%	
$D_{90}$ :	276.2	2.996	COARSE GRAVEL:	0.0%	COARSE SILT: 0.0%	
$(D_{90} / D_{10})$ :	2.204	1.614	MEDIUM GRAVEL:	0.0%	MEDIUM SILT: 0.0%	
$(D_{90} - D_{10})$ :	150.9	1.140	FINE GRAVEL:	0.0%	FINE SILT: 0.0%	
$(D_{75} / D_{25})$ :	1.542	1.291	V FINE GRAVEL:	0.0%	V FINE SILT: 0.0%	
$(D_{75} - D_{25})$ :	79.14	0.625	V COARSE SAND:	0.0%	CLAY: 0.0%	
	METHOD OF MOMENTS			FOLK & WARD METHOD		
	Arithmetic	Geometric	Logarithmic	Geometric	Logarithmic	Description
	$\mu\text{m}$	$\mu\text{m}$	$\phi$	$\mu\text{m}$	$\phi$	
MEAN ( $\bar{x}$ ):	188.2	147.4	2.365	181.4	2.463	Fine Sand
SORTING ( $\sigma$ ):	95.04	2.908	0.671	1.524	0.608	Moderately Well Sorted
SKEWNESS ( $Sk$ ):	3.221	-3.989	-1.897	0.124	-0.124	Coarse Skewed
KURTOSIS ( $K$ ):	22.49	19.19	7.760	1.703	1.703	Very Leptokurtic

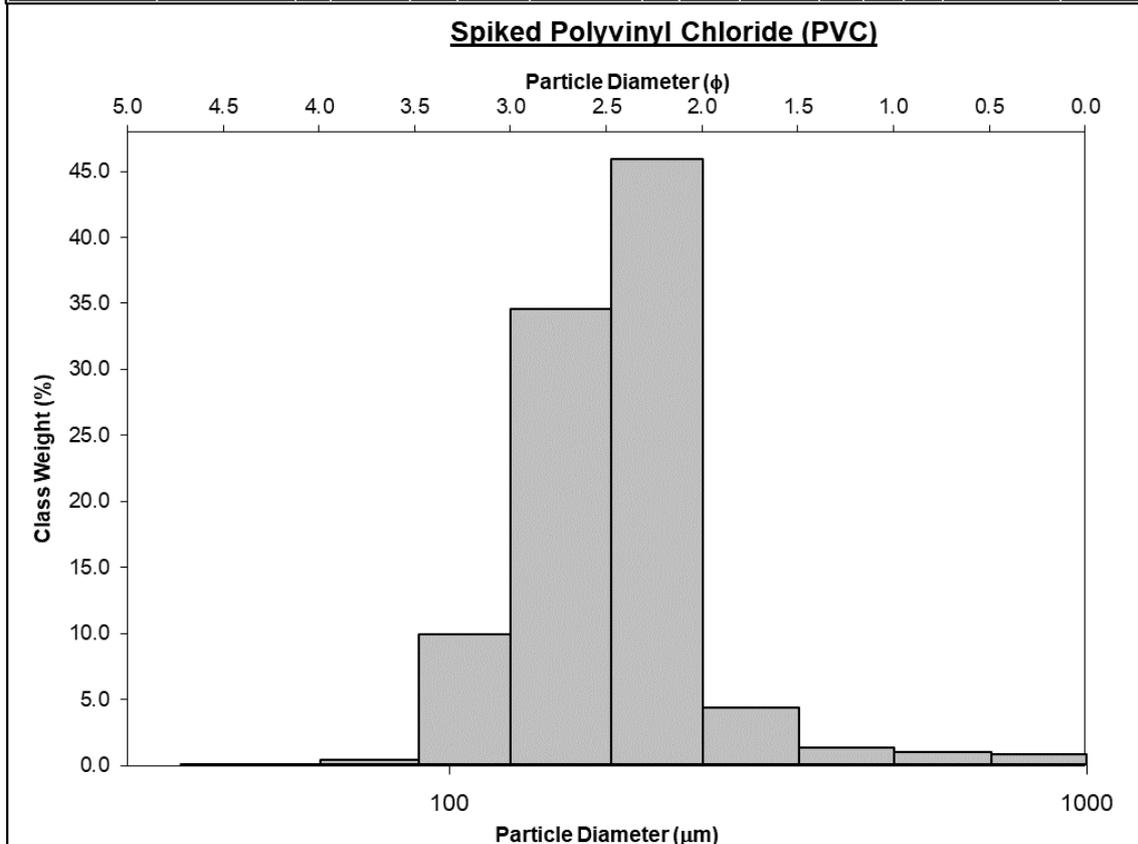


Figure ix Grain size analysis data and distribution graph for PVC