



Microplastics elutriation from sandy sediments: A granulometric approach



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ABSTRACT

Although relatively easy to extract in the marine environment, microplastics are very difficult to recover when they are trapped in sediments. The elutriation column is one of the best tools currently available for extracting plastics from sediment, but with a high sand recovery yield. This study aims to address the following questions: (i) is it possible to use a sedimentological approach to limit the sand recovery? (ii) does the extraction velocity of the sand and plastic particles vary according to density and granulometry? (iii) what is the relative recovery efficiency obtained for dense polymer particles mixed with marine sand? Based on a new granulometric classification, different plastic particle-size fractions are defined. Their extraction velocities are experimentally determined on particles of sediment and different plastics (PA, PVC). The particle recovery experiments indicate that it is possible to extract >90% of dense plastic particles in cases of negligible sand recovery.

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1. Introduction

Due to their physical and chemical properties, plastics are commonly used in a large range of industrial products. In connection with demand, the production of plastics has greatly increased during the second half of the 20th century (PasticsEurope, 2013). Plastic particle pollution of the marine environment appeared along with this increase in production (Carpenter and Smith, 1972). In 2010, it is estimated that between 4.8 and 12.7 million tons of plastics entered the oceans (Jambeck et al., 2015).

Plastics are generally considered as inert materials. However, additives, which are molecules added to the plastic to modify its properties, can become leached into the marine environment (Cadogan et Howick, 2000 in Nerland et al., 2014). Some of these additives (e.g. bisphenol A; triclosan; hydrazine) are known to be hazardous (Hansen et al., 2012). Furthermore, when dispersed into a polluted environment, plastics can adsorb other pollutants (Frias et al., 2010). An increasing number of studies reports the presence of contaminants such as metals (Ashton et al., 2010; Holmes et al., 2012, 2014), endocrine disruptors (Browne et al., 2013; Rochman et al., 2013, 2014) or persistent organic pollutants (e.g. Bakir et al., 2014; Rios et al., 2007; Van et al., 2012) on microplastic particles. Because of their small dimensions, microplastics can be ingested by marine organisms (Lusher et al., 2013; Setälä et al., 2014; Van Cauwenberghe et al., 2015a) and the pollutants can be

desorbed (Avio et al., 2015; Colabuono et al., 2010; de Sá et al., 2015). In this way, some pollutants can enter the food chain. The impact of microplastic ingestion has been tested in a large range of organisms such as *Arenicola marina* (Van Cauwenberghe et al., 2015a), *Mytilus* sp. (Browne et al., 2008; von Moos et al., 2012) and *Carcinus maenas* (Farrell and Nelson, 2013), and numerous effects have been demonstrated that are due to this ingestion, such as: (i) accumulation (Setälä et al., 2014; von Moos et al., 2012); (ii) translocation (Farrell and Nelson, 2013); (iii) inflammatory response (von Moos et al., 2012); (iv) reduction in available energy (Watts et al., 2015); (v) developmental defects (Della Torre et al., 2014 in Van Cauwenberghe et al., 2015a,b).

Plastics are the most common type of debris found on beaches (Nerland et al., 2014), and some studies show that marine sediments are contaminated by dense microplastics (i.e. $\geq 1035 \text{ kg} \cdot \text{m}^{-3}$) (Claessens et al., 2011; Thompson et al., 2004; Vianello et al., 2013). These dense microplastics are available for uptake by benthic organisms (Van Cauwenberghe et al., 2015b). Thus, several cases of plastic ingestion by commercially important benthic crustaceans are known (Van Cauwenberghe and Janssen, 2014). Through the proliferation of microplastic particles, there is an increasing risk that humans will be indirectly exposed to marine pollutants (De Witte et al., 2014; Van Cauwenberghe and Janssen, 2014). Consequently, the evaluation of contamination of marine sediments by microplastics is a major issue.

In most studies, microplastics are recovered using dense media such as NaCl solutions (Hidalgo-Ruz et al., 2012). Unfortunately, these saline solutions do not extract microplastics denser than $1200 \text{ kg} \cdot \text{m}^{-3}$

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(Claessens et al., 2013). Other solutions, such as NaI or ZnCl_2 , allow high recovery yields. However, these solutions are very expensive and cannot be applied when there are significant volumes of sediment to be treated (Claessens et al., 2013; Nuelle et al., 2014). Due to this recurrent difficulty, some methods have been proposed that are generally based on the differences in migration velocity between sand and plastic particles in a flowing fluid medium (Claessens et al., 2013; Nuelle et al., 2014; Zhu, 2015). The elutriation column developed by Claessens et al. (2013) allows high recovery yields of dense microplastics. This method is inexpensive and allows elutriation on significant volumes of sediment. However, for the extraction velocity of microplastics, the sand recovery yield is high. Some methodological approaches currently used in sedimentology may help to enhance the elutriation process and limit the sand extraction. In the present study, we aim to determine: (i) if splitting the sample into different size classes can improve the elutriation process (ii) the variation of extraction velocity of the sand (mainly quartz particles) and plastics (PA, PVC) according to their density and size class (iii) the relative recovery yield of a dense polymer (PVC) extracted from marine sand.

Based on these elements, the elutriation process will be improved and made easier to apply regardless of the column characteristics.

2. Materials and methods

2.1. Elutriation system

The elutriation column separates particles according to their terminal falling velocities which depend on their respective density, size and form. In the case of plastic particles extracted from a sediment, two different columns have been previously developed (Claessens

et al., 2013; Zhu, 2015). Building on the initial concept proposed by Claessens and colleagues, we have designed an innovative elutriation system that includes (Fig. 1): (i) an injection and flow control system; (ii) an elutriation column; (iii) a storage and filtration system; (iv) a water temperature control.

The elutriation system uses water to extract particles. To avoid overconsumption of water, the elutriation system operates as a closed circuit. The storage and filtration system comprises two superimposed polypropylene tanks. Between these tanks, a $32\text{ }\mu\text{m}$ mesh sieve prevents contamination of the reservoir tank by very fine elutriated particles ($32\text{--}63\text{ }\mu\text{m}$). The water flow is monitored and controlled by two flowmeters connected in parallel. The elutriation column is 1.86 m high and has an internal diameter of 106 mm. Water is injected at the bottom of the column. The microparticles are pulled upward and extracted at the top of the column. They are finally captured by the $63\text{ }\mu\text{m}$ mesh sieve. To avoid changes in particle dynamics due to density or viscosity modifications (Richardson et al., 2008), the water temperature is maintained constant at $20\text{ }^\circ\text{C}$ throughout the elutriation system using a Minichiller thermostat.

2.2. Tested particles

The marine sand used for these experiments is mostly composed of grains of quartz with a density is $2650\text{ kg}\cdot\text{m}^{-3}$, a value close to that measured for the sediment particles making up the sand (i.e. $2631 \pm 103\text{ kg}\cdot\text{m}^{-3}$; Lorient, France). The experiments were carried out on two different plastic powders purchased from industrial producers: polyamide (PA6) and poly(vinylchloride) (PVC). PA is a thermoplastic polymer, with an average density of $1070\text{ to }1080\text{ kg}\cdot\text{m}^{-3}$ (Nuelle

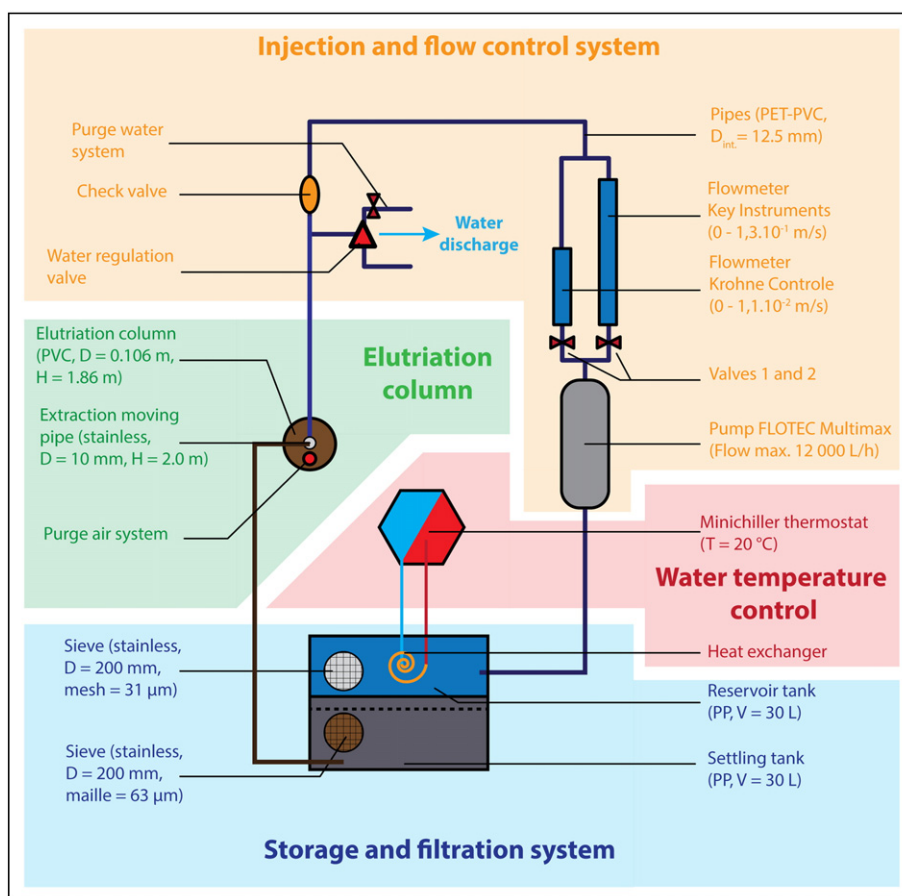


Fig. 1. Schematic diagram of the elutriation system.

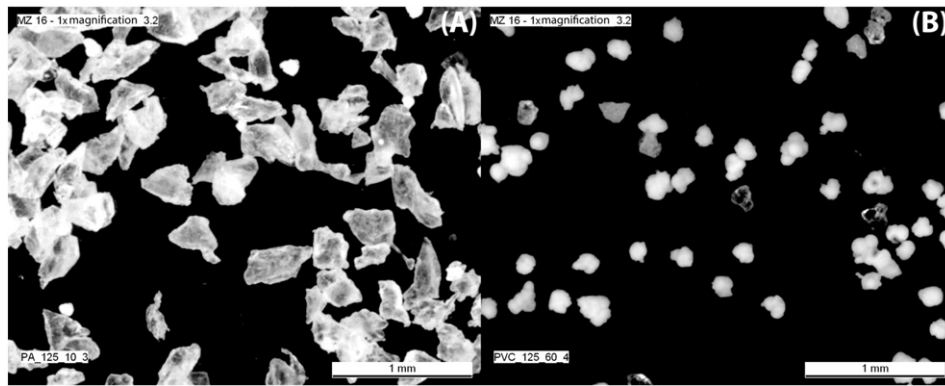


Fig. 2. Particles of polyamide (A) and polyvinyl chloride (B) used for the experiments. The PA grains have very angular shapes, whereas the PVC grains are more rounded. Observation: Leica M216 binocular magnifier, $\times 3.2$; IM500 software.

et al., 2014), which is widely used in coastal economic activities such as fishing (e.g. for fishing lines and nets). The density of the PA6 powder used in our experiments is $1080 \pm 10 \text{ kg} \cdot \text{m}^{-3}$, while their shape is usually sharply angular (Fig. 2A). PVC is a dense thermoplastic polymer generally used in industry (10.7% of the European demand in 2012; PasticsEurope, 2013). The density of the neat PVC powder (unfiled, without any additives as plasticizers for example) used here is $1388 \pm 60 \text{ kg} \cdot \text{m}^{-3}$. The grain shape is relatively spherical (Fig. 2B).

2.3. Particle-size calibration

The aim of the elutriation process is to extract the major part of the plastic particles while recovering the least possible amount of sand. The first observations show that, to extract the heavier plastics, the fluid velocity must be sufficiently low to prevent a large proportion of fine sand

to be entrained. Consequently, to achieve the most effective separation, the size parameter of the particles needs to be set. Thus, assuming that the particles are perfectly spherical, for a given particle size, density is the only particle parameter having an influence on the elutriation. The density is very different between the densest plastics (1.4) and the minerals making up the sand (2.65). For a given flow velocity of fluid, it is possible to extract plastic particles from graded sediment without entraining the sand. It is thus important to define the particle size ranges to optimize the elutriation process.

For this purpose, sub-sampling must be performed to separate particles into several size ranges. It is interesting to correlate the class limits of these sub-samples with a size-range classification of plastic particles. In this way, it is possible to set up a scale which can be easily used for very diverse particle size ranges (i.e. from a few μm to tens of mm). The currently used size classification of plastics (Galgani et al., 2013; Van Cauwenberghe et al., 2015b) is not suitable for the creation of sub-samples. In fact, these four size ranges (i.e. macroplastic, mesoplastic, large and small microplastic particles) are too broad to allow the extraction of plastic particles without also entraining a large part of the sand. Consequently, a new size classification for microplastics has been developed (Table 1), based on the Gradistat sedimentological classification, which takes into account the existing plastic classifications (Galgani et al., 2013; Van Cauwenberghe et al., 2015b). The plastic classification proposed here allows to define particle size ranges with a view to optimize the elutriation process.

Because of the fineness of PVC and PA powders, wet sieving was used to separate the size-calibrated samples of plastic particles. For the coarser and denser sand samples, dry sieving was carried out for 15 min using 200-mm diameter stainless sieves with a height of 50 mm (ISO 3310) and mesh sizes of 2 mm, 1 mm, 500 μm , 250 μm , 125 μm and 63 μm . Various size-calibrated samples were established (Table 2).

Size-calibrated samples finer than 1 mm were characterized by laser particle size analyzer (Mastersizer 2000, Malvern Instruments). Each measurement was performed three times. These measurements allow to determine the percentage of poorly classified particles.

Table 1

Particle-size classifications for sediment and plastic particles. The usual classifications of plastics are not suitable for enhancing the elutriation process. In this study, a new plastic classification is developed from the Gradistat classification (Blott and Pye, 2001) and existing plastic classification systems (Galgani et al., 2013; Van Cauwenberghe et al., 2015b). In the new classification, for example, “coarse microplastic L” ($\text{Mi}^{\text{L}}_{\text{c}}$) corresponds to “coarse sand” (0.5–1 mm). It is also possible to take account of the mesh sizes of the nets used to trap microplastics on the aquatic environment. For example, two mesh sizes are commonly used, 300 and 330 μm , and can constitute two size ranges on the medium microplastic L sub-categories ($\text{Mi}^{\text{L}}_{\text{m}}$), respectively $\text{Mi}^{\text{L}}_{\text{m1}}$ and $\text{Mi}^{\text{L}}_{\text{m2}}$.

Size (mm)	Sediment (after “Blott and Pye, 2001”)		Plastic (this study)		Plastic (after “Van Cauwenberghe and al., 2015”)
	Categories	Sub-categories	Categories	Sub-categories	
1024	Boulders	Very large	Macroplastic (Ma)	Very coarse (Ma_{vc})	Macroplastic
512		Large		Coarse (Ma_{c})	
256		Medium		Medium (Ma_{m})	
128		Small		Small (Ma_{s})	
64	Gravel	Very small	Mesoplastic (Me)	Very small (Ma_{vs})	Mesoplastic
32		Very coarse		Very coarse (Me_{vc})	
16		Coarse		Coarse (Me_{c})	
8		Medium		Medium (Me_{m})	
4	Sand	Fine	Microplastic L (Large; Mi^{L})	Fine (Me_{f})	Large microplastic
2		Very fine		Very fine (Ma_{vf})	
1		Very coarse		Crossover ($\text{Mi}^{\text{L}}_{\text{c}}$)	
0.5		Coarse		Coarse ($\text{Mi}^{\text{L}}_{\text{c}}$)	
0.25	Silt	Medium	Microplastic S (Small; Mi^{S})	Medium ($\text{Mi}^{\text{L}}_{\text{m}}$)	Small microplastic
0.125		Fine		Medium ($\text{Mi}^{\text{S}}_{\text{m}}$)	
0.063		Very fine		Fine ($\text{Mi}^{\text{L}}_{\text{f}}$)	
0.031		Very coarse		Very fine ($\text{Mi}^{\text{S}}_{\text{vf}}$)	
0.016	Clay	Coarse	Nanoplastic (Na)	Very coarse ($\text{Mi}^{\text{S}}_{\text{vc}}$)	Nanoplastic
0.008		Medium		Coarse ($\text{Mi}^{\text{S}}_{\text{c}}$)	
0.004		Fine		Medium ($\text{Mi}^{\text{S}}_{\text{m}}$)	
0.002		Very fine		Fine ($\text{Mi}^{\text{S}}_{\text{f}}$)	
0.001				Crossover (Na_{c})	

Table 2

Test samples prepared from sediment, PA and PVC powders are indicated by an asterisk (*).

Particle composition	63–125 μm	125–250 μm	250–500 μm	500–1000 μm	1–2 mm
Sand	*	*	*	*	*
PA	*	*	*	*	*
PVC	*	*	*	*	*

Table 3
Samples masses used for the experiments.

Sample (Type [Size class]; μm)	Mass (g)	Balance
Sand [≥ 125]	100 ± 0.1	Badway PS 8000/C/1
Sand [63–125]	5.0 ± 0.1	Sartorius BP 210S
PA [500–1000]	1.0 ± 0.01	Sartorius BP 210S
PA [125–250] and [250–500]	0.5 ± 0.005	Sartorius BP 210S
PVC [63–125]	1.0 ± 0.01	Sartorius BP 210S
PVC [125–250]	0.5 ± 0.005	Sartorius BP 210S

2.4. Determination of optimal extraction velocity

A test protocol was implemented to determine the extraction velocities of particles according to their density and size range.

The mass of the tested samples varied (Table 3), due to the highly variable amounts of particles recovered after sieving. The samples were weighed using a Badway PS 8000/C/1 ($d = 0.01$ g) or a Sartorius BP 210S balance ($d = 0.1$ mg).

After being poured into the column, each sample was subjected to an increasing fluid flow rate up to $1.3 \cdot 10^{-1} \text{ m} \cdot \text{s}^{-1}$. According to the flowmeter used, the fluid velocity was increased in increments of $1.9 \cdot 10^{-3} \text{ m} \cdot \text{s}^{-1}$ from 0 to $1.1 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$ (Krohne Control flowmeter) and $9.5 \cdot 10^{-3} \text{ m} \cdot \text{s}^{-1}$ from $1.9 \cdot 10^{-2}$ to $1.3 \cdot 10^{-1} \text{ m} \cdot \text{s}^{-1}$ (Key Instruments flowmeter). Each step lasted 300 s. At the end of each step, the extracted particles were collected in the 63- μm sieve and placed in an oven at 60 °C for 36 to 48 h. Once dry, the extracts were weighed (Sartorius BP 210S; $d = 0.1$ mg). Each series of tests was reproduced three times.

2.5. Extraction efficiency

The aim of the elutriation process is to maximize the microplastic extraction and minimize the sand recovery. The velocity range allowing this optimization can be graphically evaluated from the results of the experiment described in part 2.4.

The denser the polymer, the more difficult the extraction. In fact, in this case, there is a small difference in density between polymer and mineral matter. For this reason, these tests were carried out on mixtures of sand and PVC and not with PA which displays a very low density.

PVC powder (125–250 μm ; mesh size) was mixed with sand in the same size range. The mass ratio between plastic and sand is about 1/100, with a total sample mass of about 50.5 g (Sartorius BP 210S $d = 0.1$ mg). The sample was poured into the elutriation column. For this size of PVC particles, two appropriate extraction velocities were determined: $1.2 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$ and $1.9 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$. To obtain the particle extraction yields, samples were subjected to these fluid flow velocities for 300 s. The extracted particles were then placed in an oven at 60 °C for 36 to 48 h. Once dry, the extract was weighed and maintained at 850C (Volca V50e oven) for 1 h. At this temperature, all the plastic

Table 5
Values of the first decile (d_1), mean and ninth decile (D_9) for different size distributions obtained by laser granulometry.

Calibrated sample name	d_1	Mean	d_9
Sand [1000–2000]	–	–	–
Sand [500–1000]	280.8	598.9	1152.0
Sand [250–500]	283.4	444.6	687.2
Sand [125–250]	144.8	216.9	324.3
Sand [63–125]	85.4	127.1	188.6
PA [500–1000]	679.7	1002.7	1456.0
PA [250–500]	309.2	510.9	837.2
PA [125–250]	139.6	252.8	450.2
PVC [125–250]	122.6	169.8	234.4
PVC [63–125]	87.5	120.4	165.8

particles are eliminated. The inorganic residue (i.e. the extracted sand) was then weighed. The mass of initial extract minus the mass of inorganic residue gives the extracted PVC mass. The experiment was reproduced three times for each velocity.

3. Results

3.1. Sieving

The cumulative particle-size distributions (PSD) of the different samples were obtained by laser granulometry (Table 4). The results show that 90% of the calibrated sample mass is distributed over two size classes. These two classes correspond to the desired particle sizes and the upper range of the size class. For example, the PA [250–500] calibrated sample is composed of 93.7% of particles with sizes comprised between 250 and 1000 μm .

Using these results, the calibrated sample size ranges are adjusted based on the first and ninth decile of the particle-size distributions (Table 5). In the case of the sand [1000–2000] sample, the class name was not changed due to a lack of information on this size range (laser particle size analyzer cannot process particles > 1 mm).

3.2. Particle-size distribution in relation to terminal falling velocity

Particle size-distributions as determined from terminal falling velocity have a clear sigmoidal shape (Fig. 3). This is clearly marked for the curve corresponding to the extraction of sand belonging to the size range [283–687] μm . On tests carried out on the sand, the relative standard deviation of the recovery yield is low, showing an average of 1.44%.

In the case of sand between 85 and 189 μm , the recovery yield begins to increase when the fluid velocity exceeds $1.0 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$ and reaches a level $> 90\%$ at $2.9 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$. For sands in the size ranges [145–324] and [283–687] μm , the extraction begins when the fluid velocities are respectively $1.9 \cdot 10^{-2}$ and $3.9 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$, and reaches 90% recovery for a speed of $4.8 \cdot 10^{-2}$ and $9.5 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$. For size ranges [280–1152]

Table 4
Mass percentage (%) of different size classes obtained by laser particle size analyzer within each calibrated sample. The dominant size classes are noted in bold. Most of the calibrated samples are distributed according to two main size classes.

	[15.6–31] (μm)	[31–62.5] (μm)	[62.5–125] (μm)	[125–250] (μm)	[250–500] (μm)	[500–1000] (μm)	> 1000 μm
Sand [500–1000]	0	0.03	0.89	6.33	29.88	46.73	16.16
Sand [250–500]	0	0	0	4.92	57.87	37.08	0.13
Sand [125–250]	0	0	3.56	63.41	33.01	0.02	0
Sand [63–125]	0	0.59	47.29	51.32	0.79	0	0
PA [500–1000]	0	0	0	0	0.53	49.12	50.34
PA [250–500]	0	0	0	2.81	45.07	48.68	3.44
PA [125–250]	0.15	0.08	5.94	42.88	44.92	6.03	0
PVC [125–250]	0	0	11.46	82.33	6.21	0	0
PVC [63–125]	0	0.14	55.71	44.11	0.03	0	0

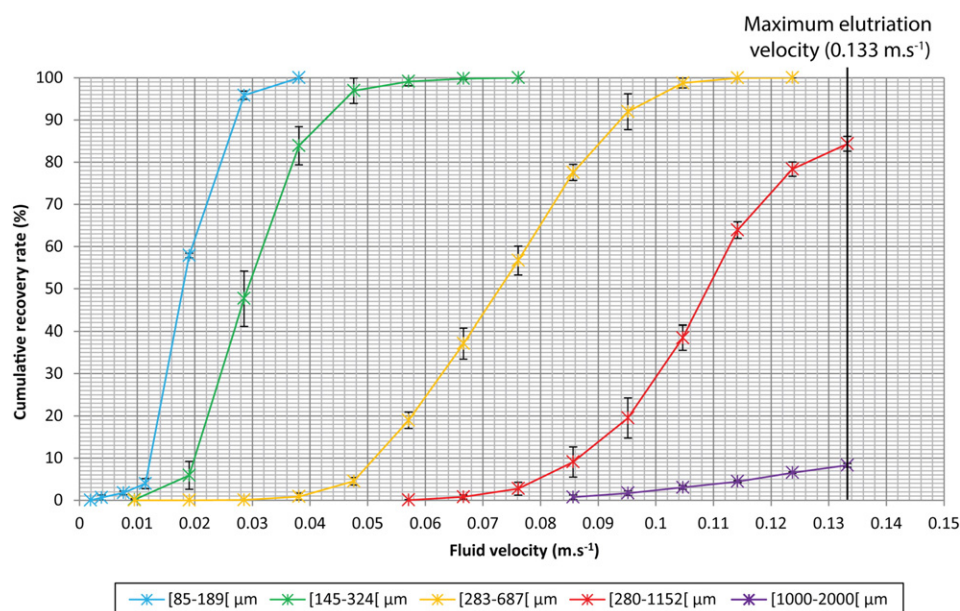


Fig. 3. Evolution of the cumulative recovery yields (%) of different size ranges of sand according to elutriation velocity ($\text{m} \cdot \text{s}^{-1}$).

and $[1000-2000] \mu\text{m}$, the starting recovery velocity is $7.6 \cdot 10^{-2}$ and $9.5 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$, respectively. The maximum velocity of the elutriation column ($1.33 \cdot 10^{-1} \text{ m} \cdot \text{s}^{-1}$) does not allow sand extraction in the case of the coarsest size ranges.

Recovery yield curves for different size ranges of polyamide (PA) also display a sigmoidal shape (Fig. 4). The standard deviations of recovery yield are greater than for the sand grains, but are still low, with an average value of 5%.

To recover PA $[140-450] \mu\text{m}$, the fluid velocity should be comprised between $1.9 \cdot 10^{-3} \text{ m} \cdot \text{s}^{-1}$ and $1.9 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$, whereas for PA $[309-837] \mu\text{m}$ and PA $[680-1456] \mu\text{m}$, the fluid velocity needs to be comprised between $7.6 \cdot 10^{-3}-3.8 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$ and $1.1 \cdot 10^{-2}-7.6 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$, respectively.

The average standard deviation on the PVC recovery yield is 4.1%. For the tested size ranges, extraction of PVC begins at $1.9 \cdot 10^{-3} \text{ m} \cdot \text{s}^{-1}$

(Fig. 5). For the size range $[88-166] \mu\text{m}$, the extraction is complete at a fluid velocity of $3.8 \cdot 10^{-3} \text{ m} \cdot \text{s}^{-1}$. For the size range $[123-234] \mu\text{m}$, the extraction yield is around 97% and was obtained with an elutriation velocity of $2.9 \cdot 10^{-3} \text{ m} \cdot \text{s}^{-1}$.

3.3. Extraction yield of PVC mixed with sand

The particle-size distributions derived from the terminal falling velocity of sand and PVC allow to determine the fluid velocity required to extract PVC $[123-234] \mu\text{m}$ from sand $[145-324] \mu\text{m}$. In the case, two fluid velocities were tested: $1.2 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$ and $1.9 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$.

For a fluid velocity of $1.2 \cdot 10^{-2} \text{ m} \cdot \text{s}^{-1}$, the PVC recovery yield is $92.0\% \pm 7.1$ (Table 6) and the sand recovery yield ($2.5 \cdot 10^{-2} \pm 1.9 \cdot 10^{-2}\%$) is negligible. Once the fluid velocity increases until

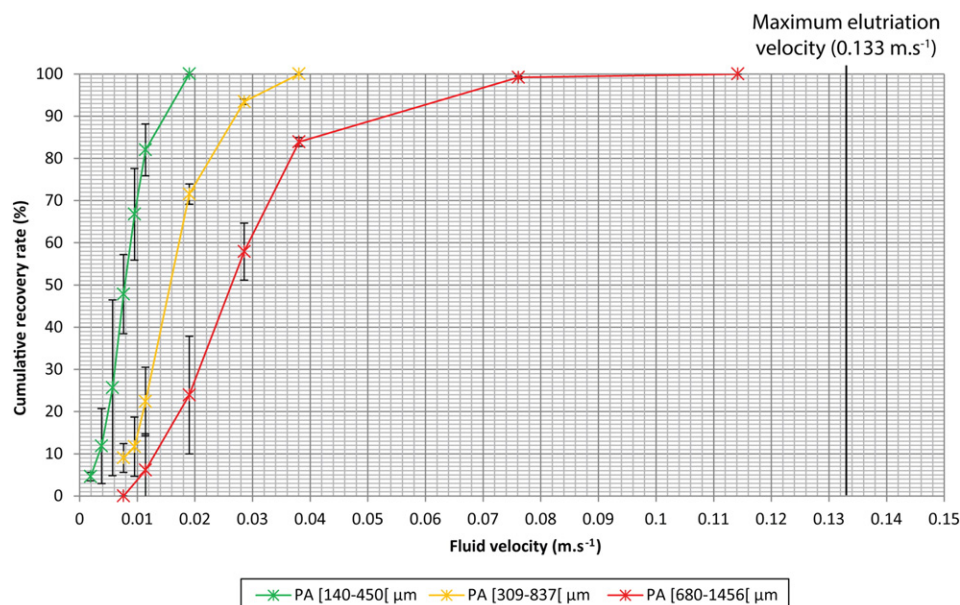


Fig. 4. Evolution of the cumulative recovery yields (%) of three different size fractions of a polyamide according to the elutriation velocity ($\text{m} \cdot \text{s}^{-1}$).

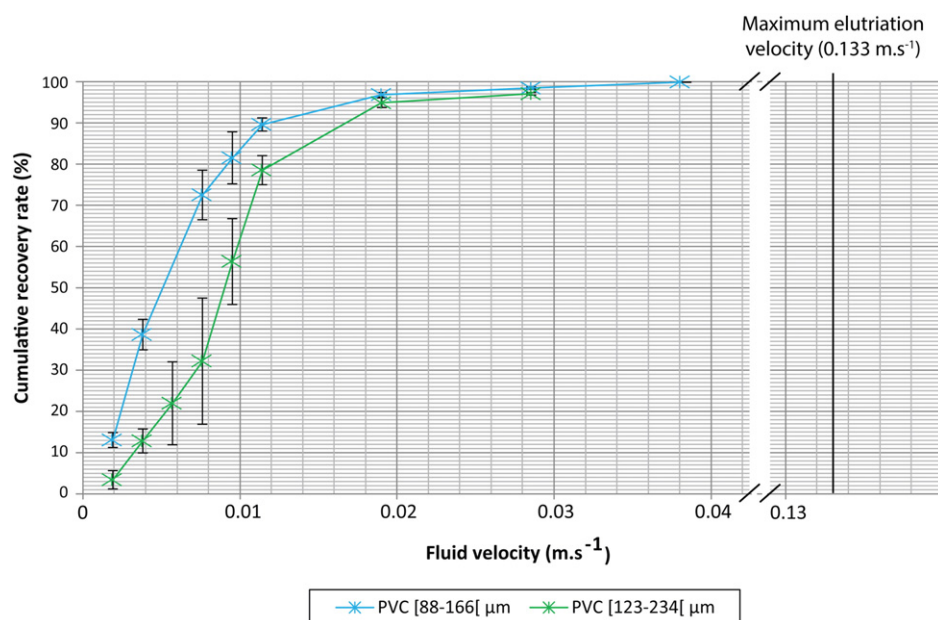


Fig. 5. Evolution of the cumulative recovery yield (%) of two different size fractions of a polyvinyl chloride according to the elutriation velocity ($\text{m}\cdot\text{s}^{-1}$).

$1.9 \cdot 10^{-2} \text{ m}\cdot\text{s}^{-1}$, the PVC recovery yield is slightly higher: $96.8\% \pm 2.8$. Simultaneously, the sand recovery yield sharply rises with an average value of $8.7\% \pm 3.6$.

4. Discussion

4.1. Sedimentological approach to improve microplastics extraction

Various approaches are used to extract plastics from marine sediments. Some of these methods are based on differential displacement of plastic particles subjected to a fluid flow (e.g. Claessens et al., 2013; Nuelle et al., 2014; Zhu, 2015). In the case of the elutriation column, the behaviour of particles (i.e. sedimentation, resuspension and extraction) depends on the shape and mass of the particles, which is a function of their density and volume. In this way, microplastics can be extracted because they have a lower density than sediment. However, if the sediment is poorly sorted, the separation will be not efficient (Fig. 6). To achieve the separation of particles according to their density, it is necessary to reduce the particle-size variability of the sample by separating it into different size classes.

In the literature, different definitions are given for the size of a “microplastic particle”. The first definition of the term “microplastic particle” refers to particles with a size of tens of μm (Thompson et al., 2004). More recently, different definitions based on the particle size have been proposed. Currently, the most commonly accepted upper limit is 5 mm (e.g. Frias et al., 2010; Galgani et al., 2013; Mohamed Nor and Obbard,

2014; Wright et al., 2013). However, an upper limit of 1 mm is also commonly used, being more consistent with the “micro” terminology, (e.g. Browne et al., 2011; Claessens et al., 2011; Van Cauwenberghe et al., 2015b; Vianello et al., 2013). Based on these limits, granulometric classifications of plastic particles have begun to emerge in the literature (e.g. Lee et al., 2013; Mohamed Nor and Obbard, 2014; Ryan et al., 2009). For example, the European MFSD technical subgroup on Marine Litter proposes a classification dividing microplastics into two subcategories: small and large microplastics (Galgani et al., 2013). This type of classification is not fine enough to improve the elutriation process. In sedimentology, various particle-size classifications have been developed. The logarithmic scale of Gradistat (Blott and Pye, 2001) is commonly used by sedimentologists. This logarithmic scale covers a range from $2 \mu\text{m}$ up to 2.05 m according to a geometric progression (common ratio = 2, scale vector = 1 mm), which allows an accurate description of fine and coarse particles at the same time. The precision of this classification could be sufficient to be used for the elutriation process. The plastic classification system developed from the Gradistat scale and the two main definitions of “microplastics” offer three advantages: (i) a precise classification of microplastics according to the particle size; (ii) a comparison between plastic and sediment grain-size distributions; (iii) sorting of particles according to their size with the aim of improving the elutriation process.

The different methods of particle-size measurement do not rely on the same aspects of size (Blott and Pye, 2001) and the comparison of results is complex (Pye, 1994 in Blott and Pye, 2001). Furthermore, in

Table 6
Results of particle extraction from a mixture of PVC–marine sand.
S.: sand; P.: PVC; \bar{x} : mean; σ : standard-deviation.

Fluid velocity ($\text{m}\cdot\text{s}^{-1}$)	Sample	Sample mass (g)		Extracted mass (g)		Extracted (%)		Average extracted sand (%)		Average extracted PVC (%)	
		S.	P.	S.	P.	S.	P.	\bar{x}	σ	\bar{x}	σ
$1.2 \cdot 10^{-2}$	S.1	50.4	0.47	$0.2 \cdot 10^{-2}$	0.44	$4.7 \cdot 10^{-3}$	94.4	$2.5 \cdot 10^{-2}$	$1.9 \cdot 10^{-2}$	92.0	7.1
	S.2	50.2	0.47	$1.3 \cdot 10^{-2}$	0.46	$2.7 \cdot 10^{-2}$	97.5				
	S.3	50	0.46	$2.2 \cdot 10^{-2}$	0.39	$4.3 \cdot 10^{-2}$	84.0				
$1.9 \cdot 10^{-2}$	S.1	50.0	0.50	5.3	0.48	10.6	94.8	8.7	3.6	96.8	2.8
	S.2	50.1	0.50	5.5	0.48	11.0	95.5				
	S.3	50.0	0.50	2.3	0.50	4.6	100.0				

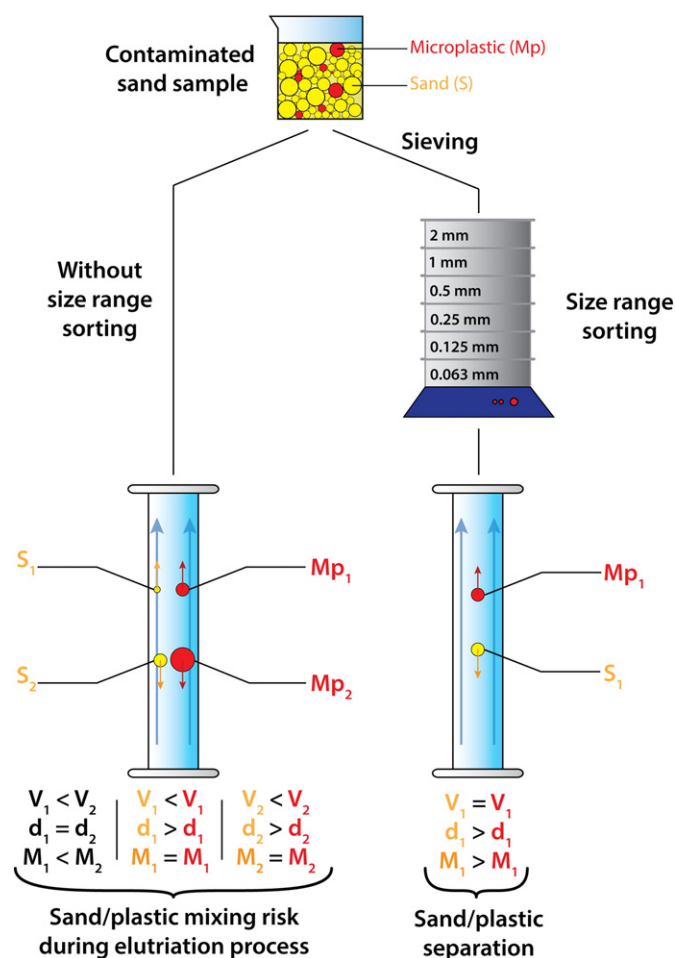


Fig. 6. Principle of the elutriation process. For a given water flow velocity, the particle behaviour is mostly controlled by the mass (M) of the particle. Without sorting of particle sizes, the elutriation process cannot separate particles as a function of their density (d). In fact, the particle volumes (V) may vary over a wide range in the sample. In this case, the sand-plastic separation is not very efficient. A sieving step can enhance the process. In fact, with a reduced range of particle volumes, the separation becomes a function of the particle density. The plastic extraction is then more efficient.

the particular case of sieving, two main parameters, the mesh size and the particle shape, can make it difficult to separate the particles size by size. Indeed, the sieve mesh is square shaped and the length of the diagonals is greater than the specified mesh value. The sieve retains particles when their two smaller dimensions are larger than the mesh diagonals. However, in the case of the elutriation process, the sieving step does not bias the elutriation. Indeed, the test carried out here on particle extraction shows that most of the plastic is collected and that the size classes defined ensure correct functioning of the elutriation column.

4.2. Determination of optimal extraction velocity ranges

For an optimal use of the elutriation column, it is important to determine, for a given size of particles, the velocity range which allows the maximum extraction of plastics and minimum sand recovery. This latter parameter has never been previously considered (Claessens et al., 2013; Zhu, 2015). Thus, to separate microplastics from the sand, the fluid velocity corresponding to the start of sand recovery can be considered as the upper limit that should not be exceeded (Fig. 7). The lower limit is given by the maximum velocity required to extract the heaviest microplastics. The experiments on the recovery yield of sand and

plastics as a function of fluid velocity show that the maximum velocity reached for extraction of the heaviest microplastics may be higher than the velocity of the onset of sand extraction.

In Table 7, we propose the optimal velocity ranges, which correspond to a compromise between the recovery yield of plastics and sand. The choice of velocity is a function of the kind of information which is required. If the velocity is high, all the microplastics can be extracted and also a part of the sand. In this case, the number and size distribution of the plastic particles can be precisely evaluated. However, the sand recovery involves an expensive post-treatment with NaI, for example. If the velocity is lower, all the microplastics cannot be extracted but the number of microplastic particles can be sufficient to be statistically representative. In this context, the presence/absence of plastic pollution and the chemical nature of the contamination can be evaluated. Without sand recovery, this evaluation does not require post-treatment and consequently is relatively inexpensive.

With an average value of 1 to 5%, the standard deviation of these recovery yield experiments is very low. This low variability demonstrates that the elutriation process is highly reproducible. Based on this adaptive methodology, the elutriation system appears as a very reliable tool since it matches the needs for extracting microplastics exhibiting heterogeneous properties.

4.3. Sand and microplastics recovery yields

For a $0.012 \text{ m} \cdot \text{s}^{-1}$ fluid velocity, the PVC recovery rate is 80% for PVC alone and 92% for PVC mixed with sand, this difference becomes negligible at $0.019 \text{ m} \cdot \text{s}^{-1}$. Two complementary explanations of this phenomenon can be proposed here. Firstly, the recovery rate depends on terminal falling velocity which is proportional to the difference in density between plastic particles and fluid. When the plastic particles are embedded within a dense pseudo-homogeneous fluid composed of water and sand, the terminal falling velocity of the PVC particles decreases and hence, easier to resuspend. Secondly, the radial fluid velocity decreasing from the center to the edge of the column due to frictions, the particles close to the limit layer are more difficult to resuspend. For sand/PVC mixtures, sand particles enhance the collisions which favor the resuspension of PVC particles trapped on the column edges.

The Air-Induced Overflow (AIO) method recovers 100% of the PVC particles, while, 8% of the sand is extracted on average (Nuelle et al., 2014). The use of the elutriation column also allows a recovery yield of 100% for PVC particles (Claessens et al., 2013). However, in this latter study, plastics were not extracted from sand and there is no information given on the sediment recovery yield associated with PVC extraction. In our study, the results obtained on the first series of tests show that the elutriation column allows up to 92% of plastic recovery yield. At the same time, the recovery yield of sand is low. Thus, PVC particles are concentrated by a factor of about 100. The volume of sediment that needs to be treated with NaI solution (e.g. Claessens et al., 2013) is strongly reduced. The PVC recovery yield can be increased up to nearly 97% by increasing the flow velocity. However, the sand recovery yield increases and becomes close to values obtained by the AIO method.

4.4. The elutriation system: economical and technical information

Our elutriation system, easy to implement, inexpensive (4000 euros) and efficient, is cost-effective, compared to iodide sodium treatment, for periodic separations. In fact, to analyze 250 g of sand, with a density of $1850 \text{ kg} \cdot \text{m}^{-3}$ and a porosity of 0.3, the separation requires 176 mL of NaI solution with a density of $1800 \text{ kg} \cdot \text{m}^{-3}$ (500 euros/L). Thus, the total cost of elutriation column is approximately the same that of 45 sediment subsamples treated by iodide sodium.

When 250 g of sediment are sieved, six subsamples are obtained whose only 2 or 3 need to be elutriated ($>20 \text{ g}$). The others can be directly treated with sodium iodide. With 3 subsamples elutriated per

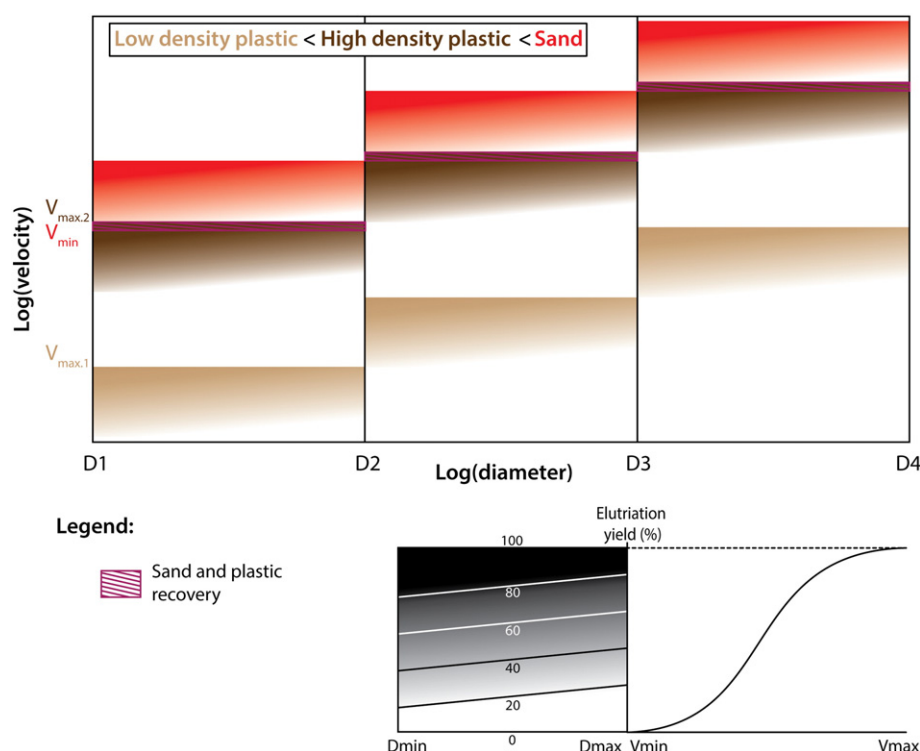


Fig. 7. The elutriation process is a compromise between sand and plastic extraction. If the plastic density is lower than the sand density, the velocity needed to extract all the plastic ($V_{\max,1}$) in a grain-size range ($[D_1-D_2]$) is lower than the minimum velocity needed to start recovery of sand particles (V_{\min}). In this case, all the plastic particles can be extracted without any sand. On the other hand, the closer the densities of sand and plastic, the greater the amount of sand that will be extracted along with plastics by the elutriation process. In fact, in this context, $V_{\max,2}$ could be higher than V_{\min} and the elutriation process would extract plastics as well as sand. In this case, the “optimal extraction velocity” is a compromise between the plastic extraction yield and the sand extraction yield.

hour, it is about 8 to 12 samples which can be treated per day. These yield values depend on the initial mass of the sample and its size distribution.

Generally, the sand is mostly composed by quartz particles ($2650 \text{ kg} \cdot \text{m}^{-3}$) associated with mica ($2850\text{--}3000 \text{ kg} \cdot \text{m}^{-3}$) and feldspar ($2600 \text{ kg} \cdot \text{m}^{-3}$). However, the sediment can be composed by other mineralogical particles. For example, for a number of tropical sand, carbonates are a major component. In such cases, the carbonates have a very similar density (e.g. calcite: $2700 \text{ kg} \cdot \text{m}^{-3}$) to quartz and the elutriation will be also similar. For carbonate shell clasts, they could have a lesser density than the carbonate minerals due to the presence of air in the particles and the elutriation shows that gasteropods and foraminifera could be extracted with microplastics. For volcanic sands, the mineralogical particles are denser than quartz (e.g. olivine: $3350 \text{ kg} \cdot \text{m}^{-3}$; pyroxene: $3300 \text{ kg} \cdot \text{m}^{-3}$; amphibole: $3200 \text{ kg} \cdot \text{m}^{-3}$) and the elutriation will be more efficient.

The design of the elutriation system could be equally adapted as a function of the granulometric ranges of the sediments. The elutriation system here developed allows to elutriate microplastics L ($63 \mu\text{m}$ – 2 mm). An elutriation system with a smaller diameter of the column and a powerful pump would permit to elutriate mesoplastics ($2\text{--}64 \text{ mm}$) and macroplastics (64 mm – 2.05 m). On the contrary, for microplastics ($63 \mu\text{m}$ – $0.002 \mu\text{m}$), a smaller and larger column might

be better. Due to these considerations, the elutriation process can be easily adapted to a broad spectrum of sediments.

To conclude, this methodology, which doesn't need chemical compounds other than water, has different advantages compared to the conventional methods. Firstly, it allows extracting dense plastics (around $1400 \text{ kg} \cdot \text{m}^{-3}$). In some sediments, plastic particles with a density of $1600 \text{ kg} \cdot \text{m}^{-3}$ have been recovered with elutriation column (unpublished data). The elutriation system is useful to take into account some new aspects of the microplastics pollution of the sediments. Secondly, the elutriation system can elutriate several hundred grams of sediment. For this quantity of sediments, it is complicate to use sodium iodide solution because of the actual cost of this salt. Finally, the separation of the sample in different range sizes increases the time required to extract the microplastics. However, the granulometric distribution of microplastics can be obtained by this mean. This information is very important to understand the pollution dynamics.

The experimental approach proposed here to determine the ranges of optimal velocity shows several limitations. The PVC used for the experiments is a dense polymer with a density of $1390 \text{ kg} \cdot \text{m}^{-3}$, which is one of the highest among the common plastics. However, some polymers containing additives can attain even higher densities (US EPA, 1992 in Nuelle et al., 2014). Furthermore, plastic powder samples cannot be used to test different size ranges. On the other hand, the characteristics of marine sands may vary from one foreshore to another in terms of grain-size distribution and mineralogy. The behaviour of particles in the column is also influenced by particle morphology. These factors imply that the optimal velocity range may vary slightly from one sample to another. All the combinations of factors cannot be experimentally tested. Nevertheless, the equations of fluid mechanics (Richardson et al., 2008; Harker et al., 2013) can be used to build a simple digital model to calculate the behaviour of particles. According to the characteristics of the column and the sedimentary and microplastic factors, this model would allow to determine suitable recovery velocities.

Table 7
Optimal velocity ranges according to particle-size classes.

Mesh size of sieve used to sort the calibrated samples (μm)	Optimal range of velocity ($\text{m} \cdot \text{s}^{-1}$)
[1000–2000]	$[8.6 \cdot 10^{-2}\text{--}9.5 \cdot 10^{-2}]$
[500–1000]	$[7.6 \cdot 10^{-2}\text{--}8.6 \cdot 10^{-2}]$
[250–500]	$[2.9 \cdot 10^{-2}\text{--}3.9 \cdot 10^{-2}]$
[125–250]	$[1.2 \cdot 10^{-2}\text{--}1.9 \cdot 10^{-2}]$

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