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Review

Microplastics Extraction Techniques In Sediments – A Review

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Abstract

The contamination of micro plastics in environmental matrices has been reported worldwide, and the search for an extraction method is relatively recent. Sediments are considered its greatest receptor, posing a significant challenge among environmental matrices for the extraction of these pollutants. The physicochemical complexity of the sediments challenges the standardization of techniques for micro plastic extraction, making their studies more intricate, requiring more in-depth investigation. Currently, there are ongoing discussions regarding existing research gaps, and a lack of standardized techniques persists in the assessment of the presence of micro plastics in sediment samples. In this work, we conducted a study review to describe different extraction methods and highlight the advantages and disadvantages of each process, considering various salts and oils used in micro plastic extraction techniques from sediments. At the end of the study, we identified some promising methods to be used in micro plastic monitoring programs in sediments that can be replicated in laboratories at a low cost and with easy execution.

Keywords: Extraction Methods; Micro plastics; Monitoring; Sediments.

INTRODUCTION

Plastics are present in people's daily lives in different materials with multiple functions. They present numerous facilities to modern life, due to their practicality and resistance, which makes these materials one of the most used in the world. Its consumption has driven the expansion of the plastic industry. According to Plastics Europe (2023), in 2021 the global production of plastics increased by 4%, totaling a produced amount of 390 million tons. Its uses meet demands for packaging (44%), civil construction (18%), automotive industry (8%), electronics production (7%), domestic use, sports and leisure (7%), agriculture (4%), among others (12%) .

Plastics are materials of natural or synthetic origin, obtained from petroleum derivatives or renewable sources such as sugar cane or corn (Arikan & Ozsoy, 2015). Polymer chains are produced by combining chemical monomers, often composed of fossil fuels, into chains of repeating units (Horton & Dixon, 2018; Hale *et al.*, 2020). Polymers also occur naturally in molecules such as biodegradable deoxyribonucleic acid or starch, as well as more environmentally persistent cellulose and chitin (Arikan & Ozsoy, 2015; Abd El-Malek *et al.*, 2020). Its applications include food and beverage containers, thermal insulation, home and workplace furniture, electrical and electronic devices, vehicle interiors, toys, fabrics, surface coatings, and even medical devices (Echchakoui & Barka, 2020; Hale *et al.*, 2020).

Plastics can be homogeneous in terms of constituent polymer or contain distinct types of mixtures or even with cross reactions that make it possible to achieve the desired characteristics (Abd El-Malek *et al.*, 2020; Echchakoui & Barka, 2020). These additives improve

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their mechanical properties, becoming more resistant to heat and aging. They also present greater resistance to light manipulation and greater resistance to flames (Thompson *et al.*, 2009; Andrady, 2011). These characteristics give the product properties of stability and difficulty in handling, which when discarded conventionally become available in the ecosystems and organisms present (Parker *et al.*, 2021).

Among the different types of polymers, the most used are polypropylene (PP) with a consumption of 19.3%, low density polyethylene (LDPE) 14.4%, high density polyethylene (HDPE) 12.5%, polyvinyl chloride (PVC) 12.9%, polyethylene terephthalate (PET) 6.2%, polyurethane (PUR) 5.5%, polystyrene (PS) 5.3%, recycled 8.3%, thermosets 7.1%, bioplastics 1.5% and other types 7.1% (Plastics Europe, 2023). When discarded improperly, these polymers can reach the ecosystems and are then exposed to the elements. Factors such as ultraviolet radiation, abrasion, biological degradation and disintegration promote the breakdown of these polymers, producing small fragments (Andrady, 2011; Browne *et al.*, 2011; Imhof *et al.*, 2012). This fragment, however, due to its mechanical properties, hinder its biodegradability resulting in a longer bioavailability time for aquatic organisms.

Smaller fragments become more dangerous, as they can be ingested by a greater number of species, which increases their risk to ecosystem health (Thompson *et al.*, 2004; Fries *et al.*, 2013; Zhang *et al.*, 2020; Sridhar *et al.*, 2022). Studies indicate that these particles are present in various environmental matrices (Andrady, 2011; Browne *et al.*, 2011; Fries *et al.*, 2012; Rivoira *et al.*, 2020), as well as in water (Lechthaler *et al.*, 2020; Adomat & Grischek, 2021), soil (Jiajia *et al.*, 2021; Ya *et al.* 2021; Surendran *et al.*, 2023), sediment (Imhof *et al.*, 2012; Nuelle *et al.*, 2014; Coppock *et al.*, 2017; Zhang *et al.*, 2020; Constant *et al.*, 2021; Hata & Jiang, 2021; Trinh *et al.*, 2021) and food (Sridhar *et al.*, 2022), becoming one of the biggest environmental problems today.

With the increase in production, consumption of plastic and inadequate disposal, there is a gradual awareness of its presence in ecosystems, drawing the attention of the global scientific community (Liebezeit & Dubaish, 2012; Van Cauwenberghe *et al.*, 2015; Adomat & Grischek, 2021; Li *et al.*, 2021; Cashman *et al.*, 2022; Yuan *et al.*, 2022; Soursou *et al.*, 2023). Studies published on this topic are aimed at quantifying and extracting these particles in different ecosystems (Thompson *et al.*, 2004; Andrady, 2011; Fries *et al.*, 2012; Imhof *et al.*, 2012; Nuelle *et al.*, 2014; Lechthaler *et al.*, 2020; Hata & Jiang, 2021; Rogers *et al.*, 2022) and its effects on organisms (Ding *et al.*, 2019; Walkinshaw *et al.*, 2020; Bajt, 2021).

In this research process, plastic waste underwent a long discussion to define its categorization according to its size. We accepted the terminology of macroplastics, for particles larger than 5 mm; microplastics (MPs) for <5 mm and nanoplastics (NPs) for those smaller than 1 μm (Li *et al.*, 2020; Bellasi *et al.*, 2021; Soursou *et al.*, 2023).

However, major challenges are still faced, as there is no consensus in many aspects, standardization of protocols for extracting plastics from water and sediment (Hidalgo-Ruz *et al.*, 2012; Hanvey *et al.*, 2017; Li *et al.*, 2020; Soursou *et al.*, 2023), types of analysis and identification methodologies (Van Cauwenberghe *et al.*, 2015; Prata *et al.*, 2019; Kuznetsova & Timerbaev, 2022), removal of organic matter or not (Prata et al, 2019; Kuznetsova & Timerbaev, 2022), sample size (Hidalgo-Ruz *et al.*, 2012; Van Cauwenberghe *et al.*, 2015; Prata et al, 2019; Bellasi *et al.*, 2021), and adequate recovery rates (Hidalgo-Ruz *et al.*, 2012; Van Cauwenberghe *et al.*, 2015; Prata *et al.*, 2019; Lusher *et al.*, 2020a; Bellasi *et al.*, 2021).Aspects regarding the capacity of MPs to accumulate organic and inorganic pollutants and become vehicles of contaminants are also topics that researchers need to better evaluate (Kuznetsova & Timerbaev, 2022).

Among the environmental matrices, sediments have a growing recognition that their contamination can have significant adverse effects on aquatic ecosystems (Garcés-Ordóñez *et al.*, 2021; Moscoso *et al.*, 2023). The presence of small plastic fragments can impact the structure of benthic communities, enhancing their dissemination in the food chain (Rubio-Armendáriz *et al.*, 2019; Burrueco Subirà, 2022; Rellan *et al.*, 2023). Furthermore, the physicochemical complexity of sediments challenges the standardization of techniques for extracting microplastics in this type of matrix, making their study more complex and the need for further investigation (Kuznetsova & Timerbaev, 2022; Soursou *et al.*, 2023).

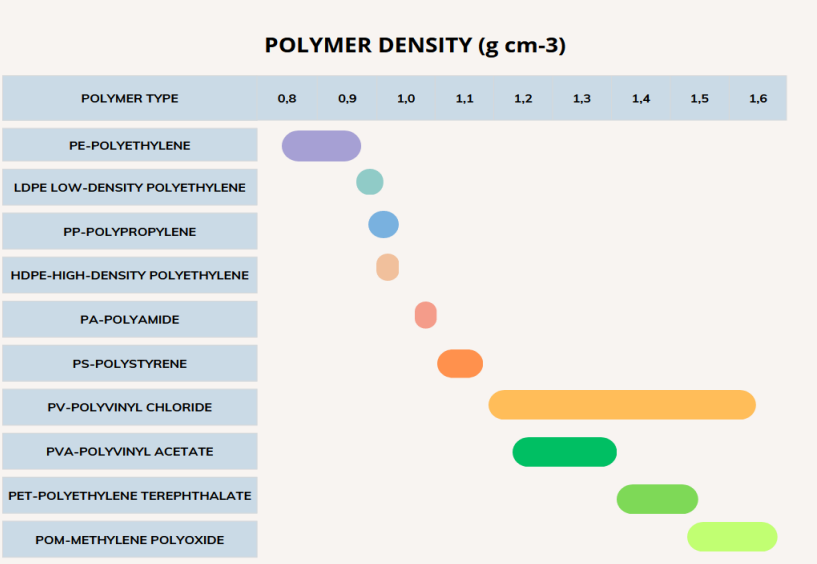
Hampton *et al.* (2023) evaluated the efficiency of microplastic extraction processes in four different matrices (drinking water, fish tissue, sediments, and surface water), enriched with a known number of MP particles of various polymers, morphologies, colors, and sizes. They also quantified the time requirements for sample processing and analysis. Their results demonstrate that more complex matrices, such as sediments, require a longer time for the extraction process (238 ± 547h), while samples from drinking water have a shorter extraction time (15 ± 22h). The authors also present the challenge of removing smaller particles (<20μm).

Most of the MP extraction techniques in sediments are based on density and electrostatic separation. Regardless of it, a great combination of methods is tested to obtain greater efficiency and practicality in the extraction of micro plastics. These extraction processes include steps of sieving wet or dry sediment samples for separation into different particle size fractions (Song *et al.*, 2015; Van Cauwenberghe *et al.*, 2015), separation with difference in density using a specific saline solution high density (Thompson *et al.*, 2004; Van Cauwenbeghe *et al.*, 2015; Rivoira *et al.*, 2020), oleophilic separation techniques using oil and water (Lechthaler *et al.*, 2020), elutriation, which is included in the injection of air in a column to help flotation or deposition of particles according to their density (Imhof *et al.*, 2012; Claessens *et al.*, 2013; Nuelle *et al.*, 2014; Kedzierski *et al.*, 2016; Coppock *et al.*, 2017); automated geology techniques (Rogers *et al.*, 2022) and centrifugation (Phuong *et al.*, 2018).

In addition to these techniques, some researchers have used dyes to aid in the visual identification of particles (Lavers *et al.*, 2016; Lusher *et al.*, 2020a; Hata & Jiang, 2021). Procedures to eliminate the presence of organic matter are also used. Acidic or alkaline agents such as potassium hydroxide (KOH) (Dehaut *et al.*, 2016; Qiu *et al.*, 2016; Bäuerlein *et al.*, 2023; Dehaut *et al.*, 2023), sodium hydroxide (NaOH) (Hurley *et al.*, 2018), nitric acid (HNO_3) (Prata *et al.*, 2019), hydrochloric acid (HCl) (Pfeiffer & Fischer, 2020) have been shown to be highly effective in the process. The use of Fenton's reagent (Tagg *et al.*, 2017; Hurley *et al.*, 2018) or even enzymatic digestion (Cole *et al.*, 2014) were also methods used to digest the organic matter present in the samples and improve the microplastic extraction.

The density of plastic is a characteristic that influences its extraction process in environmental matrices. It varies depending on the atomic composition, presence of branches or side groups, degree of crystallinity, use of fillers and the presence of additives, in addition to other properties. Specifically regarding additives, these confer particular properties to plastics, improving their mechanical performance, allowing a better surface finish of the product, increasing or reducing its hardness, in addition to reducing the cost of the material (Tecnoplastico, 2023). These additives, when added to polymers, can alter the specific density of the resins. This is an important fact to consider, as it influences the plastics extraction process using saline solutions (Hidalgo-Ruz *et al.*, 2012; Fries *et al.*, 2013). The specific density of plastic resins is described in Table 1 for some types of polymers.

Table 1. Different types of polymers and their specific densities in resins.



Environmental studies developed by Lusher *et al.* (2020a) indicated the presence of different types of micro plastics in sediments, including typically floating polymers. Possibly, the sedimentation of these floating polymers may have occurred due to the processes of biofouling (Lobelle & Cunliffe, 2011) and mineral adsorption (Corcoran *et al.*, 2015). This can reduce the buoyancy of plastics, facilitating their movement to the bottom both in environments marine as well as freshwater. Within the sediment, micro plastics become bioavailable to a wide range of benthic fauna (Coppock *et al.*, 2017) turning hazardous.

Despite all the efforts of the scientific community for the extraction, identification, and quantification of MPs, there are still gaps that need to be better defined. One of the biggest discussions on this topic is the lack of standardization of techniques, which makes it difficult to compare the presence of these pollutants in marine sediments. However, while the scientific community discusses universal standards protocols, our objective is to identify techniques that can help in the elaboration of a methodology for monitoring the presence of micro plastics in marine sediments.

In this sense, we understood that the protocol must meet the specific demands of the object of study and not necessarily collect data to be compared with other ecosystems. The good monitoring in the environment is advantageous to assess anthropogenic interference over the years in the sediments. Thus, this review aims to (i) describe different techniques for extracting micro plastics from marine and estuarine sediments, considering their advantages and disadvantages; (ii) evaluate different salts and oils used in the techniques and (iii) identify techniques used in monitoring for the presence of micro plastics in sediments that can be replicated in the laboratory with low cost and easy execution.

Different Micro plastic Extraction Techniques

Research to establish techniques for extracting and quantifying micro plastics in sediments began to intensify from 2012 onwards, with work carried out by Imhof *et al.* (2012); Fries *et al.* (2013); Nuelle *et al.* (2014) and Kedzierski *et al.* (2017), among others. Nevertheless, in 2004, Thompson *et al.* (2004) was one of the pioneers in describing the presence of these contaminants in marine sediments. The techniques have

generally been based on the difference in density between the extracting medium and the MPs, changing the salts and inserting or suppressing procedures in the techniques to achieve better extraction efficiency. In addition to techniques based on density and electrostatic separation, other interesting approaches have also been presented. Some authors innovate in the search for different techniques, such as sample staining and even automated mineralogy. In the following text, we address the different techniques, indicating the advantages and disadvantages of each one, seeking to identify a way to, in a second stage of this research, suggest a protocol for monitoring the presence of micro plastics in marine and estuarine sediments.

Techniques Using Density Difference Separation

One of the most used techniques for extracting MPs is based on the density difference principle (Hidalgo-Ruz *et al.*, 2012; Prata *et al.*, 2019; Cutroneo *et al.*, 2021), where a solution is prepared by mixing filtered or distilled water with an amount of a selected salt (Thompson *et al.*, 2004; Crichton *et al.*, 2017; Bellasi *et al.*, 2021). While the sediment particles undergo precipitation, the MP particles float on the surface layer of the dense solution and are thus separated (Bellasi *et al.*, 2021). However, the density of polymers can be changed with the addition of additives during product manufacture (Hidalgo-Ruz *et al.*, 2012) as well as the effect of biofouling. The process of separating plastic particles may require additional treatments such as centrifugation, which may or may not be accompanied by organic matter digestion and density separation steps (Phuong *et al.*, 2018; Pompêo *et al.*, 2022).

Thompson *et al.* (2004), in their sediment sample, used concentrated sodium chloride (NaCl). Nevertheless, as the density of the NaCl solution is only 1.2 g/cm3, just low-density plastics can be extracted (Thompson *et al.*, 2004; Hidalgo-Ruz *et al.*, 2012). After Thompson's work, studies have been developed to evaluate the most efficient salts for MP extraction. NaCl is the salt most used to perform density separation (Bellasi *et al.*, 2021), as it is highly available and inexpensive, does not damage ecosystems and is recommended by NOAA (National Oceanic and Atmospheric Administration) (Masura *et al.*, 2015). Sodium iodide (NaI) has high density, safety and reusability, and possibly, in combination with separation columns or the use of oil, improves recovery rates (Prata, *et al.*, 2019). Zinc chloride ($ZnCl_2$) was used by Imhof *et al.* (2013); Liebezeit & Dubaish, (2012); Coppock *et al.* (2017), among others. It is a more expensive salt, and its use is suggested for a second density separation after a NaCl step (Rivoira *et al.*, 2020). Furthermore, $ZnCl_2$ is a hazardous substance and, therefore, its use should be avoided (Bellasi *et al.*, 2021). Dibasic sodium phosphate (NaH_2PO_4) allows reliable extraction performances, is economical, non-hazardous and capable of achieving a good recovery rate (Zhang *et al.*, 2020). However, extraction with NaH_2PO_4 can be overly complex due to the need to heat the solution to increase its density (Bellasi *et al.*, 2021). Calcium chloride ($CaCl_2$) represents another good alternative due to its low cost and relatively low risks. Notwithstanding, its density is low, ranging between 1.3 and 1.35 g cm^{-3}, and does not allow the retention of PET and PVC particles (Bellasi *et al.*, 2021). Sodium tungstate and its derivatives (SPT) have been used by Corcoran *et al.* (2015), and, despite its efficiency in removing almost all types of plastic polymers (Lusher *et al.*, 2020), its cost is high and can be dangerous (Cutroneo *et al.*, 2021). From a density point of view, $ZnCl_2$, NaI, potassium iodide (KI), zinc bromide ($ZnBr_2$) and potassium fluoride (KF) salts are the best for more efficient density separation. Although, these salts are dangerous for aquatic biota and can cause serious health risks (Bellasi *et al.*, 2021).

Some studies propose the mixture of salts between NaCl and NaI to increase the recovery rate of MPs, reaching a density of 1.5 g cm^{-3} (Han *et al.*, 2019). Other researchers propose the use of tap water (Zurcher 2009; Quinn & Ewins, 2017; Phuong *et al.*, 2018) or filtered seawater (Do Sul *et al.*, 2009).

Economic issues must also be considered in the choice of salt, especially when the method is used for long-term monitoring programs. Salts such as NaI, sodium tungstatec ($Na_2WO_{4)}$, KI and KF are more expensive and are not indicated for extracting MP in large volumes of samples (Bellasi *et al.*, 2021).

Despite studies proving the efficiency of using salt for the extraction of MPs, recently its combination with oils has been sought, demonstrating a better efficiency of the process. Tests are being carried out on substances such as castor oil (Mani *et al.*, 2019), canola (Lechthaler *et al.*, 2020; Pappis *et al.*, 2021; Radford *et al.*, 2021) and olive oil (Scopetani *et al.*, 2020; Yuan *et al.*, 2022). The principle of this method is based on the oleophilic attraction between plastic polymers and oil (Crichton *et al.*, 2017). Some of these studies have shown to be very promising, with recovery rates above 90% for micro plastics of different sizes (Pompêo *et al.*, 2022).

Canola oil (Lechthaler *et al.*, 2020; Pappis *et al.*, 2021), for example, has recovery rates of over 90% and a shorter treatment time compared to saturated salt solutions and poor water retention organic matter (Crichton *et al.*, 2017). Drops of olive oil were added to saturated salt solutions to optimize MP extraction, improving recovery rates from 64% to 82% (Karlsson *et al.*, 2020). Regardless of the limitations of using oil to separate plastics and the need for a cleaning step with a detergent, the combined technique helps with recovery rates (Prata *et al.*, 2019).

Faced with the need to lower monitoring costs and seek more ecological alternatives, Bellasi *et al.* (2021) used a saturated NaCl solution (prepared with ultrapure water) with addition of 100% sucrose reaching a density of 1.30 g cm^{-3}. This mixture achieved an average flotation of 100% for PS (1.04 and 1.1 g cm^{-3}), PE (0.917 and 0.965 g cm^{-3}) and PP (0.91 g cm^{-3}), while PET, heavier as well as PVC, reached an average fluctuation of 93.30% and 73.30%, respectively. The use of sucrose with NaCl is a promising option for long-term monitoring.

Technique with Elutriation Column

Elutriation is a process that promotes a differential displacement of plastic particles subjected to a fluid flow. In the elutriation column process, the behavior of particles depends on their shape and mass, which is a function of their density and volume (Kedzierski *et al.*, 2017). Thus, micro plastics can be extracted because they have a density lower than the sediment. Claessens *et al.* (2013) developed an elutriation separation system using a 147 cm long PVC column with an internal diameter of 15 cm, equipped with a 1 mm sieve at the top and a 35 µm mesh screen at the bottom. The sediment sample is transferred to the column by washing through the 1 mm sieve to remove oversized debris. An upward flow of water is created, and the sediment becomes fluidized.

At the bottom of the column, aeration is provided to ensure plastic separation and the heavy particles settle out. The flow of water, combined with aeration, separates the lighter particles, including micro plastics, from the heavier sand particles. The supernatant water lifts the MPs to the top, where they flow over the edge and are retained in a 35-metre sieve µm. The collected solids are transferred to a centrifuge with a NaI solution of approximate density of 1.6 g cm^{-3}, where it is vigorously stirred. After centrifugation, the upper layer containing the micro plastics is vacuum filtered and the MP separated. Recovery rates are 98% of MP fibers after extraction in the elutriation tube, followed by three subsequent NaI extractions (Claessens *et al.*, 2013).

The alternative proposed by Nuelle *et al.* (2014), with the objective of decreasing the mass of the sediment sample and improving the separation, is the extraction of MPs preceding the flotation in a saturated solution of NaI with an elutriation step. First, a pre-extraction of sediments is carried out in an air-induced overflow (AIO) system with a saturated NaCl solution. The induced air creates a stochastic process forcing the lighter particles to move more frequently and faster towards the upper layer of the solution. The NaCl solution overflow and floating particles are collected in an outer glass container. The supernatant is transferred to an installed 25 mm stainless steel sieve. The sediment sample is, then, added to a volumetric flask and a NaI solution. In this step the extraction is carried out by flotation of the lower density micro plastics, in the saturated NaI solution (1.8 g cm^{-3}) (Figure 1). Then the supernatant is filtered, and the filter air-dried for further analysis.

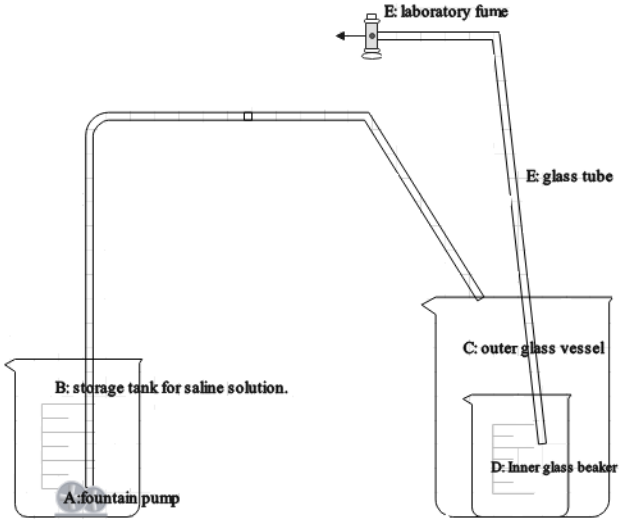


Figure 1. Simplified scheme for the AIO method, adapted from Nuelle *et al*., 2014.

Kedzierski *et al.* (2017), when evaluating the extraction speed of plastic particles in sand, conclude that these vary according to the density and granulometry in elutriation column systems. In this study, the authors propose optimal speed ranges depending on the size of the particles to be extracted.

The elutriation column presents itself as a cheap and efficient solution for removing MPs in samples of large volumes of sediments, which allows for greater environmental representation (Prata *et al.*, 2019). NaI, used in the elutriation column, has the advantage of being able to be recycled up to 10 cycles through rising and evaporation steps (Kedzierski *et al.*, 2016), with costs similar to NaCl (Kedzierski *et al.*, 2016; Cutroneo *et al.*, 2021). Thus, the use of NaI is recommended, as it is environmentally safe and can be recycled through several cycles (Prata *et al.*, 2019). Despite the attractive cost, the system requires specific equipment for extraction. However, this method takes at least 1h per sample (composed of 3 subsamples) and requires prior sieving by grain size (Kedzierski *et al.*, 2016; Prata *et al.*, 2019). The technique presents itself as a very reliable tool, as it meets the needs of extracting micro plastics exhibiting heterogeneous properties (Kedzierski *et al.*, 2017).

Munich Plastic Sediment Separator (MPSS)

The Munich Plastic Sediment Separator (MPSS) is a device developed by Imhof *et al.* in 2012 (Figure 2). It is a method based on flotation, similar to that used in the plastic recycling industry. The system uses a saline $ZnCl_2$ solution with a volume of approximately 25 L and a density of 1.6 g cm^{-3}. In this system, the sediment samples are introduced into a container with a capacity of 17 L, equipped with a rotor that adjusts and maintains constant agitation. The saline solution is inserted until its filling is almost complete, allowing a first separation of the floating materials. After agitation, a vertical tube, with a gradually reduced diameter to reach a higher concentration of particles, is installed in the system and a new $ZnCl_2$ solution is added, which lowers the fluid. With the sample chamber turned upside down, the ball valve is opened, and MP is collected using a 47 mm vacuum filter. After MP extraction, the filter must be washed with hydrogen peroxide (H_2O_2) (30%) to remove organic material and then dried at 30°C. To increase process efficiency, MPs can be anointed with a combination of pine oil and a foam conditioner (Imhof *et al.*, 2012). Despite that, the interaction of the $ZnCl_2$ solution with substrate components produces a lot of foam, which exceeds the volume of the MPSS sample chamber and makes the extraction process difficult, requiring the use of an antifoam to eliminate the problem (Imhof *et al*., 2012; Zobkov & Esiukova, 2017).

The MPSS model is designed to use large amounts of sediment (6 L). It is made of stainless steel and is approximately 1.75 m high. These characteristics make the model expensive to reproduce in the laboratory and limit its portability and viability when processing numerous replicas of small samples (Coppock *et al.*, 2017; Zobkov & Esiukova, 2017), in addition to the fact of producing contaminated waste.

The MPSS model has high recovery rates and can be used for samples with different grain sizes. Imhof *et al.* (2012) presents recovery rates of 100% for micro plastic particles of 1–5 mm and 95.5% for particles smaller than 1 mm. Zobkov & Esiukova (2017) compare the efficiency of MPSS with the modified NOAA method, using a 50/50% solution of $ZnCl_2$ and $CaCl_2$ with a density of 1.48 g cm^{-3}, as it is less aggressive to the equipment. Their extraction rates averaged above 96.8% for different sediment particle sizes. Factors such as bio fouling or adhesion of sand grains and other denser particles can decrease the MPSS extraction efficiency (Kowalski *et al.*, 2016).

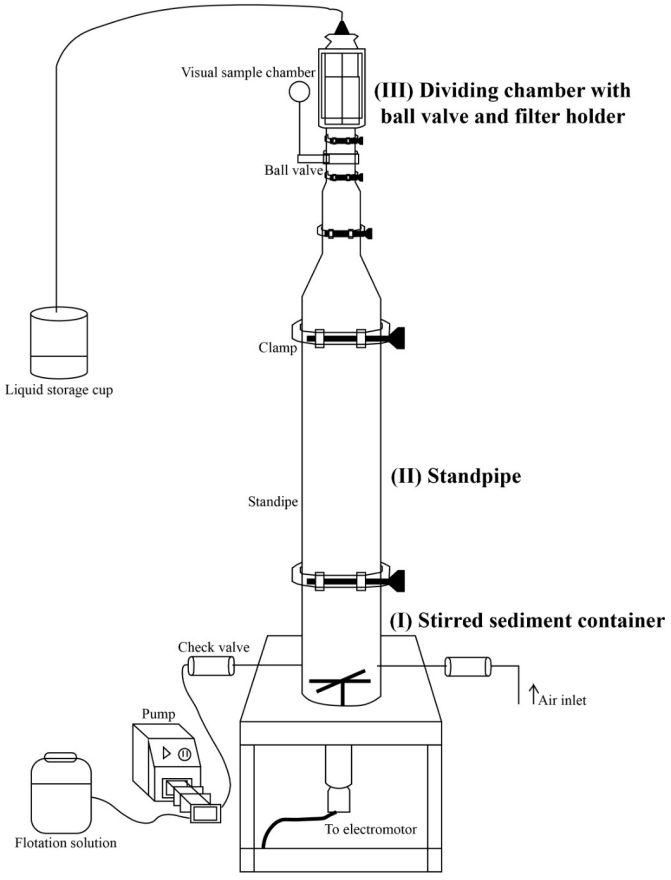


Figure 2. Simplified scheme for the technique using Munich Plastic Sediment Separator (MPSS), adapted from Imhof *et al.*,2012.

Another obstacle in the use of MPSS is salt ($ZnCl_2$) because it is expensive and toxic. An alternative found was the use of sodium polytungstate, as it has less toxicity (Bellasi *et al.*, 2021). Trinh *et al.* (2021), when evaluating the presence of MP in the Can Gio estuary in southern Vietnam, using the MPSS model, confirmed high rates of extraction of micro plastics with particle sizes greater than 0.3 mm, but indicates an uncertain efficiency for smaller particles. Another fact reported by Trinh *et al.* (2021) was that the stirrer easily sticks when hard and solid particles are introduced, a fact also reported by Zobkov & Esiukova (2017), who suggest a prior sieving of the sample.

Technique Using Electrostatic Separation

Electrostatic separation is another protocol that has been gaining ground in the scientific community. Its high recovery rates for different types of polymers demonstrate that the system is promising. Electrostatic separation is a practice used in the recycling industry and its principle is to separate samples based on their electrical conductivity properties. Organic matter and sediments have conductive charges, while micro plastics are not conductive, and these characteristics make it possible to isolate the materials (Hamos, 2023).

To carry out the protocol, the sample is introduced into an electrostatic separator where it is electrostatically charged. The electrostatic difference between conductive media (organic matter and sediments) and non-conducting media (micro plastics) enables its separation in different collectors of the equipment (Felsing *et al.*, 2018; Yao *et al.*, 2019; He & Hu, 2021; Kurzweg *et al.*, 2022; Hamos, 2023) (Figure 3). Felsing *et al.* (2018) presented a pioneering study of this methodology, in which the authors used a Korona-Walzen-Scheider (KWS) separator, manufactured by Hamos GmbH (Penzberg, Germany). Their results show high recovery rates for four different materials reaching approximately 100% in enriched samples and a reduction of the original sample volume close to 99%.

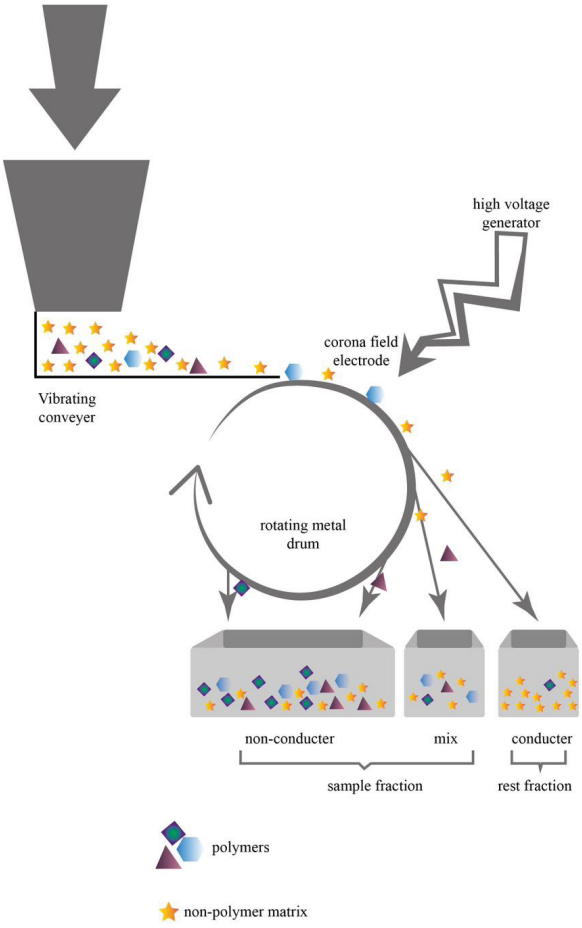


Figure 3. Simplified scheme for the technique using electrostatic separation adapted from Felsing *et al.*, 2018.

Kurzweg *et al.* (2022) modified the method and used three steps to extract and quantify MP in sediments: electrostatic separation, by density and differential scanning calorimetric. Four particulate matrices were used including commercial sands and river sediments, enriched with MPs of different types. After electrostatic separation, the mass of samples was reduced by 98% for sand and 70-78% for sediment. After density separation, the total mass reduction of the sediment samples was greater than 99%. The average recovery rates for biodegradable polymer (PCL), LDPE and PET were 74 ± 9%, 93 ± 9% and 120 ± 18%, respectively.

One of the authors' indications is the need for the samples to be carefully dried and dismembered, for the separator to completely divide the materials, eliminating possible errors (He & Hu, 2021; Hamos, 2023). Factors such as mineral composition and grain size can negatively affect recovery rates and sample mass reduction (Enders *et al.*, 2020; Kurzweg *et al.*, 2022), requiring further studies to assess the efficiency of the system in more complex matrices (Kurzweg *et al.*, 2022).

Yao *et al.* (2019) recommend the combination of density, electrostatic and organic solvent separation to improve its efficiency and to standardize the analytical process for sediment micro plastics in future studies. Jiang *et al.* (2022) evaluated and compared the method with float extraction, based on life cycle analysis, and indicated that electrostatic separation has a greater impact on the environment, mainly due to energy consumption. This fact becomes relevant when the technique is used for large volumes of samples, such as remediation actions or environmental monitoring. The electrostatic separation system has a simplified handling and automated operation with a small processing time and presents good recovery rates for different types of polymers (Felsing *et al.*, 2018; Kurzweg *et al.*, 2022), as well as do not using chemical product in the extraction process (Lusher *et al.*, 2020).

Technique Using Sample Staining

Hata & Jiang (2021) developed a method that is easy to perform and efficient and can serve as an alternative approach to study micro plastics present in sediments. In the proposed protocol, basic materials from geotechnics/ geological engineering are used, in which a sieving step, a particle coloring step and quantification through an optical microscope used in geotechnics or field geology are combined. This is a laboratory procedure, starting with the staining of the MP at a temperature of 105°C for 20 min. After the MPs are stained, they are mixed with sediment samples, followed by their extraction and quantification. This protocol helps in identifying the best dyes for later identification. The authors suggest the use of red dye, as they are easier to identify compared to yellow, green, and blue. The use of brown dye is not encouraged as it causes confusion due to its similarity to the color of organic matter.

The coloring solvent is efficient for coloring PP, PE and PS under various coloring temperatures. The method allows the classification of MP without an analytical machine and without the use of toxic reagent, being also a fast method. In the article, the authors do not present high recovery rates. It is a relatively new method and few studies after the publication of the article were developed to better evaluate its effectiveness.

Technique Using Automated Mineralogy

The approach based on automated mineralogy to identify and quantify MP using QEMSCAN® (Quantification and Evaluation of Minerals by Scanning Electron Microscopy) was tested by Rogers *et al.* (2022). The process was adapted to map carbon concentration indicating the presence of plastic in the samples. The quantification method is fast and can be used as a preliminary screening identifying MPs in relatively large geographic areas. However, this study is recent and requires the acquisition of specific equipment and training of the laboratory team.

The authors report a possible overestimation of the presence of MP in the matrix and the difficulty in differentiating MP from organic matter. The main necessary adaptations are in the sense of expanding the differentiation between synthetic and natural carbon-based materials, refining the sample grinding system, seeking other coating materials to achieve better recovery rates, and solving methodological difficulties. As the protocol was recently proposed, other studies involving this methodology have not yet been carried out.

Techniques for Quantification and Identification of MPs

As for the stage of identification and detection of micro plastics, there are several techniques used. Visual inspection for identification and quantification of plastic particles is one of the most used methods. This procedure is generally used as a preselection in chemical characterization. The segregation of the particles is performed using the physical characteristics of the MPs observed directly or through a stereoscopic microscope (Prata *et al.*, 2019). Situations such as the perception of the individual evaluator, experience and wear of materials can lead to overestimation or underestimation in identification (Lavers *et al.*, 2016). Some laboratories have resorted to the use of dyes to facilitate visual identification, as it is a low-cost method (Prata *et al.*, 2019; Hata & Jiang, 2021). To assist in the visual identification process, Lusher *et al.* (2020a) created three categories to identify micro plastics: morphology (size, shape and texture), optical properties (color, reflectivity and birefringence) and behavior (flexibility, density, among others). Some problems are reported due to the affinity of the dye for plastics and for the biogenic material in the sample, which requires a complete pre-step of digestion (Prata *et al.*, 2019; Lusher *et al.*, 2020b). Figure 4 presents the different advantages and disadvantages of each method described.

	Density Difference Separation	Elutriation Column	Munich Plastic Sediment Separator (MPSS)	Electrostatic Separation	Sample Stainings	Automated Mineralogy
Reviewed by other researchers	✓	✓	✓	✓	×	×
Low cost	✓	✓	×	✓	✓	×
Good MP recovery rate (>80%), on average	✓	✓	✓	✓	×	×
Short technique processing time	✓	×	×	✓	✓	✓
Environmental safety	×	×	×	✓	✓	✓
Factors decrease process efficiency	×	×	×	×	×	×
No specific equipment required	✓	×	×	×	✓	×
Does not require large sample volume	✓	×	×	×	✓	✓
Does not require pre-treatment of samples	×	×	×	×	×	×
No specific employee training required	✓	×	×	×	✓	×

Figure 4 - Different techniques for extracting microplastics from marine and estuarine sediments, considering their advantages and disadvantages.

Some researchers have only used the visual separation technique when spectroscopy analysis is not available (Stolte *et al.*, 2015). The use of spectroscopy gives greater credibility to the process and when it is available, the samples, which have undergone a visual selection, go on to a second stage to identify their chemical characteristics. At this stage, techniques such as SEM-EDS (Scanning electron microscopy), FTIR (Fourier-transform infrared), NIR (Near Infrared Spectroscopy), Raman (Raman spectroscopy) and NMR (Nuclear Magnetic Resonance) Spectroscopy are the most used (Fries *et al.* al., 2013; Nuelle *et al.*, 2014; Coppock *et al.*, 2017; Phuong *et al.*, 2018; Han *et al.*, 2019; Zhang *et al.*, 2020; Corami *et al.*, 2021; Kamp *et al.* , 2023; Langknecht, 2023).

The SEM-EDS is a powerful microscope that provides information about the micro plastic surface and additives present in the sample. FTIR and Raman spectroscopy has been the best technique for chemical characterization, due to its precision without damaging the sample. FTIR analysis is feasible for particles up to 20 µm, while Raman is applicable for particles up to 1 µm. Recently, NIR and NMR spectroscopy has also been used for micro plastic identification. When the NIR technique is chosen, there is no need for pretreatment, and it can detect up to 1 mm in size in an environmental sample. Analysis using NMR provides the amount of micro plastics present in the sample (Tirkey & Upadhyay, 2021). Pyr-GC pyrolysis gas chromatography in combination with mass spectrometry (MS) also helps to obtain information about the structure of macromolecules by analyzing their thermal degradation products, followed by pyrolysis (Fries *et al.*, 2013). However, Hankett *et al*. (2023) make some warnings regarding the use of Pry-CG, saying that the pH of the sample must be neutral, avoiding the presence of inorganic solids, as they can promote undesirable reactions under pyrolytic conditions. We also note the presence of chemicals that may induce hydrolysis and/or isomerization during pyrolysis, such as calcium carbonate and alumina, respectively.

Along with the spectrometry techniques, gravimetric analysis is also an option, but it is simpler. Approaches vary and may include measuring the mass of individual micro plastic particles or measuring the filter paper as a whole (Hanvey *et al.*, 2017). Gravimetric analysis has been used by NOAA (Masura *et al.*, 2015) and by Patel *et al.* (2020).

CONSIDERATIONS AND FORWARD OVERVIEW

Although the evolution of the different forms of extraction and identification of MPs in sediments is observed, there is a lack of standardization of the techniques used. Nevertheless, much has been researched in the search for reliable techniques, with low damage to ecosystems and attractive cost. Among the techniques used, the ones that stand out are those that use the difference in density for the extraction of MPs. Some methods that also seek efficiency using equipment such as the MPSS and the Elutriation Column make manipulation difficult and use more toxic salts. Electrostatic separation systems seem to be promising, but it is necessary to have the pre-treatment of samples to ensure adequate extraction and the need to purchase specific equipment. The MPSS, Elutriation Column and Electrostatic Separation techniques require a greater investment, which makes their use difficult for laboratories with limited resources.

What can be seen is that there is a great worldwide effort in the search for more sustainable solutions, efficiently and economically. Approaches such as extraction by density and use of oil have been highlighted for being very economical and effective, in addition to being non-destructive methods, allowing a characterization and quantification of samples in a second stage. However, they still need further study to be a promising alternative to be used in monitoring. The sucrose density gradient technique proposed by Bellasi *et al.* (2021) in association with other inorganic salts can represent an innovative ecological solution.

AUTHORS' CONTRIBUTION

AD – Review research, original draft writing;

CRJ – Supervision and Review;

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