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Microplastics in soils: A comparative review on extraction, identification and quantification methods

Yoonjung Seo^{a,*}⁽⁶⁾, Venkata Chevali^b, Yunru Lai^c⁽⁶⁾, Zhezhe Zhou^{a,b}, Guangnan Chen^a, Paulomi Burey^{a,b}, Shaobin Wang^d⁽⁶⁾, Pingan Song^{a,b,**}⁽⁶⁾

^a School of Agriculture and Environmental Science, University of Southern Queensland, Springfield, Australia

^b Centre for Future Materials, University of Southern Queensland, Springfield, Australia

^c Centre for Sustainable Agricultural Systems, University of Southern Queensland, Springfield, Australia

^d School of Chemical Engineering, The University of Adelaide, Adelaide, SA, 5005, Australia

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ABSTRACT

Microplastics (MPs) have been continuously accumulating in soil, posing significant environmental concerns due to their potential toxicity and role as pollutant vectors. Effective monitoring of MPs in soils requires appropriate methods for extraction, identification and quantification. This comparative review systematically examined various methods used in MP analysis from solid matrix environments, highlighting the advantages and disadvantages of each technique. Soils are heterogeneous and complex matrices, which can interact differently with MPs, rendering the separation of MPs from soils a significant challenge. Key challenges include the interactions between MPs and soil organic matter and detection limitations of smaller size MP which can interfere with accurate extraction. Specialized techniques and devices are required for precise identification and quantification. Digestion can be effective in removing organic matter, although harsh digestion can degrade MPs. To improve the liability of MP analysis, it is critical to minimize steps that may reduce accuracy such as filtration, harsh digestion and inappropriate sample size. Conversely, adjusting or combining methods can enhance the precision of MP analysis. This review offers a forward-looking perspective by advocating extraction and detection approaches, thus providing a more accurate, reproducible and holistic framework for MP analysis in soils. These insights are anticipated to guide future research promote standardized protocols and enhance environmental monitoring efforts.

1. Introduction

Landfills are often the final destination of most plastics, which can leak pollutants into the environment. Fragmentation of plastic waste in landfills occurs as a result of a variety of processes, including mechanical friction, sunlight exposure, oxidation, and biological activity (Rafiq and Xu, 2023). Plastics, however, cannot fully decompose through these fragmentation processes; instead, they break down into smaller-sized particles, including microplastics (MPs), i.e., plastics smaller than 5 mm in size (Mitrano et al., 2021). The breakdown of plastic debris will spread throughout the hydrosphere, pedosphere, and biosphere (Barnes et al., 2009). The Pedosphere, which is situated at the interface of the hydrosphere, biosphere, and atmosphere, plays an integral role in facilitating interactions between these domains (Giri et al., 2022). Soils serve as long-term sinks for plastic waste, and they are profoundly polluted with a substantial portion of plastic waste at approximately 32 % in soils (De Souza Machado et al., 2018; Geyer et al., 2017).

Due to the persistent nature and extremely slow degradability of MPs in soil, there are serious concerns about soil properties and soil health. The physical, chemical, and biological properties of soils such as soil structure, nutrient cycling, enzyme activity, and microbial diversity and health can be significantly affected as a result of MP accumulation (Seo et al., 2024). These property changes also affect plants, but the specific effects can be indeterminate because of MP size, shape, type, and concentration (Huang et al., 2022). Some detrimental effects have potential repercussions on agricultural productivity and food security. Furthermore, MPs in soil can also contaminate freshwater bodies through surface runoff and leaching (Rillig, 2012).

* Corresponding author.

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^{**} Corresponding author. School of Agriculture and Environmental Science, University of Southern Queensland, Springfield, Australia. *E-mail addresses:* Lenny.seo@unisq.edu.au (Y. Seo), pingan.song@usq.edu.au, pingansong@gmail.com (P. Song).

Research and monitoring of MPs are the essential prerequisites to actions, including policy decisions (Amesho et al., 2023; Ng et al., 2023). To assess MP contaminations accurately, we must assess the occurrence and potential impacts of MPs to determine the magnitude of this hazard (Corradini et al., 2021). Precise identification and quantification of MP pollutants is essential for determining their impact on soils and for developing mitigation strategies. To identify and quantify MPs, we have to extract them from environmental matrices. While the study on MPs was conducted first (2004) in seawater, not in soils (2012), existing methods have been mainly focused on seawater or sediments (Rillig, 2012; Thompson et al., 2004).

Seawater is usually considered a homogeneous or low-organicmatter-containing matrix (Prata et al., 2020). Currently, several methods of extracting MPs from seawater are available, including filtering and digestion, to remove organic matter and non-target substances (Lusher et al., 2020). Filtration can be utilized for soils, albeit it cannot fully separate MPs from soil constituents. In contrast to seawater, soil is a highly complex matrix with a diverse range of textures, variable levels of organic matter and aggregate stability. During wet-dry cycles, aggregate formation is assisted by MPs for them to be trapped (Lehmann et al., 2021). Another unique challenge of MPs in soils is the presence of soil organic matter that consists of a mixture of natural litter at different stages of decomposition. These organic components can adhere to MPs or form aggregates with MPs that hinder their separation and analysis. Furthermore, stable humic fractions may also include polymeric macromolecules, which may be read as false positives (Bronick and Lal, 2005). Therefore, removing organic matter from soils is crucial for MP identification and quantification: digestion or enzymatic degradation of organic matter is often employed to break down humic substances and organic matter before the separation of MPs from environments (Steinmetz et al., 2020). These methods also help to reduce the risk of false positives, thereby improving accuracy in MP extractions from soils.

Several factors influence extraction, including soil and polymer types, experimental setup, order of analysis, and duration of the experiment. Importantly, MP extraction efficiency is significantly affected by soil type and composition of fine particles and organic matter (Zhang and Liu, 2018). Polymer types often lead to inconsistent extraction efficiency in density separation, as polymers differ in density (Radford et al., 2021). This suggests that the extraction efficiency needs to be assessed within soil-type-specific (i.e., texture and organic matter) and targeted-polymer-specific conditions. It is also important to use a standardized set of devices and methods as they can be a significant source of variation in the analysis. From the published literature, filtration devices, membranes, centrifugation, and ultrasonic devices for dispersing soil aggregates have been shown to cause variations. In soil, centrifugation has been proven efficient in MP extraction, but not at high soil loadings (Grause et al., 2022). Mitigating the obstacles in high-loading soils requires strategies such as breaking soil aggregates without altering MPs, effective removal of organic matter on the surface of MPs, balancing soil sample size to minimize interactions between soils and MPs and employing size-specific methods for accurate extraction. Ultrasonication is also useful for breaking down soil aggregates and certain organic matter (Büks et al., 2021). However, the methodology of soil-based MP extraction, identification, and quantification requires significant development.

Understandably, it is difficult to compare and extrapolate efficiency or recovery rates between different studies. To establish a precise method of extracting MPs from soils, and to find reproducible methods to identify and quantify MPs, comparison and accurate analysis are vital. To that end, this review aims to summarize and analyze the methodologies to extract MPs from soils and compare identification and quantification methods. Extraction methods were examined for their levels of widespread acceptance, reproducibility, and recovery rates. Additionally, the reported recovery rates were classified according to separation techniques, solutions, and polymer types. Identification and quantification methods were also discussed, and their advantages and limitations were presented.

2. Review of existing studies

2.1. Literature search and selection

To conduct a comparative analysis of targeted studies, we have systematically searched and selected the available literature. A Google Scholar search, a Web of Science search, and a Scopus search were conducted in the period between June to December of 2023. The search terms used were: "microplastics," "extraction", "identification", "quantification", "agricultural", and "soil". We have found that the first study was published in 2016 (Fig. 1).

After removing duplicates, the initial search results were screened and selected in the review database based on the following inclusion/ exclusion criteria: (1) published in English, (2) full text readily available, (3) MP extracts with soil, sludge, or sediment, and (4) methods of extraction, identification, or quantification. The following papers were also excluded: (1) review articles (but considered conducted studies in the review), (2) MPs extraction from aquatic environments and biota, and (3) studies in modelling or algorithm-based identification. We also included relevant studies from review papers. From the initial search, 306 published works were identified, and 69 studies met our selection criteria. It was also found that the number of papers published has also increased quite rapidly since 2016 (Fig. 1).

The selected articles were further divided into two categories: those that conducted extraction including identification and quantification of MPs (n = 55); and those which only evaluated identification or quantification of MPs (n = 14). The data were classified by source (i.e., soils, sediments, sludge, biosolid), type of salts, digestion methods, and sequence of procedure. We compared the frequency of the classified data, and the recovery rates of polymers based on salt type and particle size when comparing extraction methods. We also critically discussed the advantages and limitations associated with the identification and quantification methods of MPs.

2.2. Methods for extracting microplastics

Microplastics are analyzed differently depending on the sample type and environment (He et al., 2021). Despite the differences, the overall procedures follow similar steps such as sampling, extraction, identification, and quantification (Fig. 2). To detect and analyze MP contamination accurately, it is crucial to extract MP contaminants effectively from environmental samples.

2.2.1. Soil sampling strategies

Analysis of MPs in soils begins with soil sampling. Soil samples represent spatial resolution in the field, so the sample number needs to be sufficient. The location of the sampling site should take into account past plastic usage and direct or indirect sources of plastics (Möller et al., 2020). In agricultural fields, the application of plastic mulching film, irrigation source, the application of biosolid, and the location of the field such as nearby roads should be taken into account before sampling. For adequate comparison of soil samples, homogenous studies, such as soil type and vegetation, should also be taken into consideration.

The most common methods for investigating soil contamination are random sampling and composite sampling (Li et al., 2020; Scheurer and Bigalke, 2018; Thakur et al., 2023). Because soils are heterogeneous matrixes, a single-point sample is not appropriate since MPs could be distributed at random in the soil matrix. Thus, composite samples with random sampling or unaligned grid sampling may more appropriately represent MP contamination of the target field. To accurately represent soil contamination, a minimum of ten samples are recommended for a composite sample (Singer and Munns, 2006).

As MPs are most likely to be dispersed in shallow soil, some studies obtained soil samples from the surface to a depth of 10 cm (Li et al.,

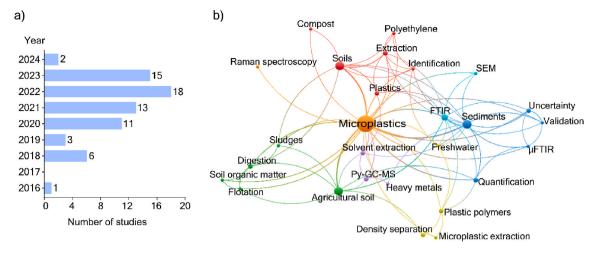


Fig. 1. a) Number of targeted MP studies in soils published in each year (searched between Jun to Dec of 2023, total n = 69); b) Keyword co-occurrence network analysis conducted using VOSviewer. The network visualizes the relationships between keywords extracted from the dataset of targeted MP studies (n = 69). Each node represents a keyword, and the size of the node is proportional to the frequency of the keyword in the dataset. The links between nodes indicate co-occurrence, with thicker lines representing stronger associations.

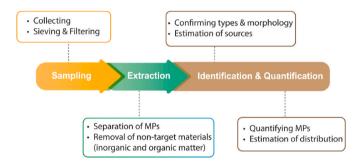


Fig. 2. General processes of microplastic analysis, MPs: microplastics.

2020; Zhang et al., 2018). It is possible to determine the depth of soil sampling depending on root depths and targeting measurements, as either shallow (i.e., 10 cm) or deeper (i.e., 30 cm). Additionally, agricultural practices such as ploughing also affect the distribution of MPs in soil (Liu et al., 2019b). MPs were detected at a depth of 100 cm after 32 years and at an 80 cm depth after 10 years in MP-contaminated fields covered with conventional mulching films under ploughing practice (Hu et al., 2021; Li et al., 2022b). Despite the bottomless vertical distribution of MPs, the highest concentrations were usually found between 0 cm and 20 cm in soil depth (Hu et al., 2021). Therefore, it is imperative that soil sampling should prioritize the 0–20 cm depth range in assessing MP contamination.

2.2.2. Microplastic separation techniques

2.2.2.1. Density separation. Density separation (DS) is widely used for MP extraction from sediments and soils (Fig. 3a). As soil minerals contact with water, they become immersed in water as they become dense and sink. Due to their hydrophobicity, most plastics do not absorb water and float on a certain density of aqueous solution. The density (specific gravity) of a solution is adjusted by adding various salts to the water. Density separation is commonly accomplished using NaCl and ZnCl₂ salts (Fig. 3b). NaCl has higher accessibility and is relatively safe in the environment (Li et al., 2019). ZnCl₂ solution has a high density, allowing it to effectively separate polymers of different densities (Prosenc et al., 2021). Mixed solutions are also often used to extract MPs in soils. Mixing NaCl and ZnCl₂ solutions can result in a higher density and lower cost than using pure salt alone. Low-density polymers (e.g., polyethylene (PE)) can be extracted with NaCl because of their low density. The

selection of a salt for density separation should consider two crucial phenomena. First, the MPs deposited are not pure polymers as they originate from commercial products where the composition and manufacturing process are the determinants of density. Second, MPs deposited in soils may have interacted with mineral and organic matter, altering their as-processed density. The use of a higher-density solution would therefore serve the twin purpose of achieving higher overall extraction performance as well as the removal of impurities on the MPs.

In the separation of soil minerals, density separation with highdensity solutions (e.g., ZnCl₂, NaI and NaBr) is an effective method. This technique is particularly useful in soils with high microplastic contamination, especially in sandy soils, where plastics are more easily separated due to the density differences. However, DS is less effective in clayey soils, where the higher mineral content can interfere with the extraction process. In clay soils, the recovery rate for microplastics is lower, with a maximum recovery of 87.5 % for particles larger than 250 μ m. In contrast, sandy soils exhibit higher recovery rates, reaching up to 98.6 %, as the mineral interference is less pronounced (Zhang et al., 2018). Additionally, the relationship between microplastic concentration and recovery rate does not appear to be linear, as recovery rates fluctuate across different concentrations and soil textures.

Centrifugation can shorten the time for DS, although MPs may be further fragmented. The centrifuge speed should be adjusted to balance effective separation while minimizing the risk of MP fragmentation. In soils with high organic matter, combining DO and DS may be an acceptable solution. However, filtration causes MPs to be trapped inside membrane pores, leading to erroneous results. Minimizing the usage of filtration is therefore desired for accurate MP extraction. To minimize filtration, alternative approaches such as direct identification and quantification techniques like Py-GC-MS or Raman spectroscopy can be used. However, if digestion is required, it is preferable to use methods that do not require filtration afterward to ensure accurate MP extraction.

2.2.2.2. Oil extraction. A second frequently employed method is oil extraction, which takes advantage of the lipophilic surface properties of plastics (Fig. 3a). Soil mineral fractions and MPs mixed with oil and water can lead to layer separation. The separation of the floated oil layer and additional steps to clean the MP surfaces can however be challenging. In sediment samples, oil extraction recovers over 90 % of MPs (Crichton et al., 2017; Scopetani et al., 2020). However, in field samples with high organic matter, the recovery rate is greatly reduced. For this reason, it is important to integrate the step of digestion of organic matter into extraction processes (Mani et al., 2019; Radford et al., 2021).

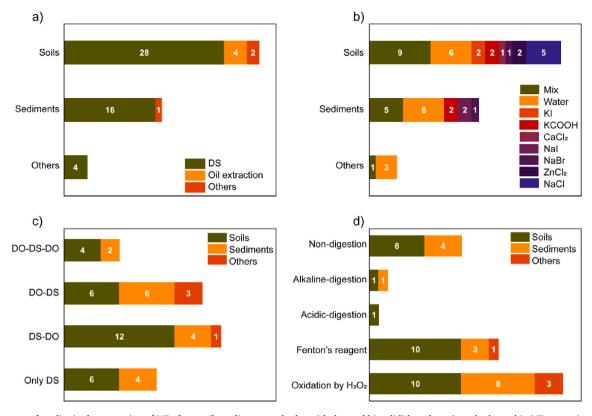


Fig. 3. Frequency of studies in the extraction of MPs from soils, sediments and others (sludge and biosolid) based on a) methods used in MP extraction studies (n = 55); b) types of salt used in density separation method (n = 48) classified by sources; c) combination of processes (within density separation method, n = 48); d) digestion methods (within density separation method, n = 48). Note: Mix-mixed salts for density separation (i.e., NaI + NaCl, NaCl + CaCl₂ and ZnCl₂ + CaCl₂). DS-density separation. DO- digestion of organic matter.

2.2.2.3. Other approaches. Microplastics can also be extracted using non-gravimetric methods. In electrostatic separation, plastics and other particles are separated based on the difference in conductivity compared to soil minerals. Electrostatic separation was shown to have a recovery rate of 90–100 % between 63 μ m and 5 mm (Felsing et al., 2018). However, soil aggregates and cohesive MPs on organic matter cannot be analyzed using this method. Using iron nanoparticles to tag MPs is another method that uses electromagnets (Ramage et al., 2022). Nevertheless, density separation still is the most preferred method as it does not require specialized equipment (Fig. 3a).

2.2.3. Removal of organic matter

As soil organic matter has a similar density to plastics, density separation may not be effective in separating MPs in soils with high organic matter. It may be difficult to separate MPs in high organic matter soils due to the lack of a clear separation level in salt solutions. However, most studies (approx. 80 %) found that digestion of organic matter (DO) was an effective method for extracting MPs using DS (Fig. 3c), underscoring the importance of DO for MP extraction. According to these studies, the order of procedures and criticality of DO is different, such as DS-DO, DO-DS, and DO-DS-DO. As their experiments use different types of salt, soil conditions, and polymer types, it is not feasible to compare the effectiveness of the combinations or the sequence used therein (Table 1).

Polymer degradation occurs during DO processes due to the effect of temperature and chemical contact. Hence, a thorough removal of organic matter is recommended from polymers without altering the structure or mass of the polymer. Fenton's reagent, or H_2O_2 oxidative digestion, was used in over 90 % of DO studies, while only a small group of studies used acidic or alkaline digestion (Fig. 3d). Acid and alkaline digestion were shown to be incompatible with sediments, and some polymers (including polyamide, polyethylene terephthalate, and

polystyrene (PS)) are partially or completely degraded by those processes (Pfeiffer and Fischer, 2020). Among the selected studies, only one study used acidic digestion: 98 % H_2SO_4 to carbonate after filtration of MPs (i.e., white PE film) from agricultural soils (Li et al., 2020). Although a spike-recovery experiment was performed for method validation, any alteration by H_2SO_4 was not mentioned in the study.

Fenton's reaction catalyzes the oxidation of organic matter with ferrous cations in H_2O_2 highlighting its efficacy. Fenton's reagent, however, leaves residues on the MP surface, increasing its mass (Pfohl et al., 2021). To avoid alteration of aged MPs, it was recommended to maintain a digestion temperature below 50 °C for longer digestion time rather than at higher temperatures and short digestion time (Savino et al., 2022). As a result of the lowered temperature, Fenton's reaction may require a much longer digestion period. A typical amount of digestion solution for Fenton's reaction is 30–50 mL per 10 g soil sample (Hurley et al., 2018; Radford et al., 2021). In any combination orders of procedure in DS and DO, additional filtration is also necessary because DS solutions are diluted in the process. Inevitably, MPs tend to be trapped within the membrane pores of a filter media and lost during filtration.

A recent study highlighted the interplay of multiple enzymatic digestions and Fenton's reagent in enhancing the digestion efficiency of soil organic matter (Möller et al., 2022). Compared to previous studies, their method was successful for relatively small-sized MPs (i.e., between 100 and 400 μ m). Nevertheless, this combined digestion was not as effective as digestion with only 2 h of Fenton's reagent for removing soil organic matter. Comparatively, enzymatic digestion takes anywhere from 7 to 22 days with continuous pH and temperature control (Mbachu et al., 2021). Enzymatic digestion is beneficial for preventing MP degradations, but it is not suitable for simple and fast MP extraction because of the lengthy protocol and long-drawn-out time requirements. Hence, it is essential to use digestion methods that are effective in removing

Table 1

Microplastic extraction methods of combination processes with the density separation and the digestion of organic matter in soils.

Total processes	Pre-processing	Pre-DO	DS	Centrifuge	Filtration	Post-DO	recovery rate	Targeting size	Reference
DO - DS	Standard soils	Fenton's reagent	Na ₆ [(PO ₃) ₆] NaI and NaCl	Yes	Glass fibre filter	-	96.67–100 %	100 and 5000 μm	Goli and Singh (2023)
	-	H_2O_2 at 60 °C (7 days for soils and 14 days for biosolids)	ZnCl ₂	Yes	Nitrocellulose 0.45 µm	-	>74 % (Size >2.6 μm), <29 % (Size <1 μm)	100, 4.8, 2.6 0.05 and 1 μm	(Wang et al., 2018)
	Oven-drying at 50 °C sieving 5 mm & 50 μm	H ₂ O ₂ (35 %) + FeSO4 (0.5 M) 4:1 (24–72 h)	NaCl	No	-	-	-	-	Rafique et al. (2020)
	Sieving 5 mm	(24-72 ll) H ₂ O ₂ (35 %)	ZnCl ₂ :CaCl ₂ (2:1.4) (1.55–1.58 g cm ⁻³)	No	Steel mesh 20 µm	-	80–90 % (97.8 ± 4.8)	${>}100~\mu m$ only	Kim et al. (2021)
	Oven drying at 60 °C Sieving 2 mm	Fenton's reagent	CaCl ₂	Yes	Glass fibre filter	-	98 - 81 %	-	Grause et al. (2022)
	-	H ₂ O ₂ at 60 °C (2 h)	NaCl	No	Stainless mesh 50 μm	-	-	$>50\ \mu m$ only	Pérez-Reverón et al. (2022)
DS - DO	Oven drying at 60 °C sieving 5 mm	-	ZnCl ₂	Yes	Steel mesh 1 mm Nitrocellulose 0.45 µm	Fenton's reagent + H_2SO_4 75 °C (24 h)	-	<1 mm, 1–5 mm	Choi et al. (2021)
	Oven drying Sieving 5 mm	-	$ZnCl_2$	No	Nitrocellulose 0.45 µm	H ₂ O ₂ 70 °C 120 rpm	-	-	Hossain et al. (2023)
	Drying at 55 °C Soil grinder Sieving 2 mm	-	CalCl ₂ NaI/ NaCl	No	Stainless mesh 5 µm	Fenton's reagent + H ₂ SO ₄ (24 h)	CaCl ₂ : 42 ± 25 % NaI/NaCl: 54 ± 10 %	5 µm to 2 mm	Chen et al. (2024)
	Drying at 110 °C Sieving 1 mm & 0.6 mm	-	NaCl	No	Nitrocellulose membrane 0.45 µm	Fenton's reagent	-	30–1000 µm	Mahesh and Gowda (2023)
	Air-drying Sieving 5 mm	-	NaCl	Yes	MCE filter membrane	Fenton's reagent + protocatechuic acid	$84.8 \pm 9.6 \%$ (olive) $95.4 \pm 6.0 \%$ (n-hexane)	>57 µm	Zhang et al. (2023)
	-	-	NaCl	-	Nitrocellulose 0.45 µm	H_2O_2	-	-	Aquino et al. (2023)
	Air-drying sieving 2 mm	-	ZnCl ₂	No	Filter paper <50 µm	H ₂ SO ₄ + H ₂ O ₂ (1:40) 70 °C (48 h)	over 95 %	154–600 μm	Yan et al. (2023)
	Air-drying sieving 2 mm	-	Water	No	Filter paper	Carbonation with H ₂ SO ₄	96, 85, 87 and 84%	$>150\ \mu m$	Li et al. (2020)
	Oven drying at 70 °C	_	NaCl:CaCl ₂	No	Nitrocellulose 0.45 μm	H ₂ O ₂ 65 °C (12 h)	_	0-0.49, 0.5-0.99, 1.00-1.99 and 2.00-5.00 mm	Ding et al. (2020)
	Sieving 5 mm	-	ZnCl ₂	No	15 µm	H ₂ O ₂ at 70 °C (24 h)	-	20–500 µm	Zhu et al. (2023)
	Oven drying 40 °C hand- milled Sieving 0.3, 0.5, 1, 2 & 5 mm	-	NaCl	No	7–9 µm	10 % KOH	-	<0.3, 0.3–0.5, 0.5–1, 1–2 and 2–5 mm	Chouchene et al. (2022)
	Standard soils (LUFA, Germany)	-	NaBr	No	4–12 μm	Fenton's reagent +5 mL HNO ₃	88 % for MP fragments and 74 % for MP fibers	PET fragments (125–250 μm), fibers (0.5–2,0 mm length)	Tophinke et al. (2022)
DO - DS -DO	Air-drying sieving 1 and 5 mm	H ₂ O ₂ (30 %) at 70 °C	NaI (1.8 g cm ⁻³) ZnCl ₂ NaCl	No	Nylon 20 µm	$H_2O_2+H_2SO_4$ at 70 $^\circ\text{C}$	-	_	Li et al. (2019)
	Oven drying at 70 °C sieving 1 mm	H ₂ O ₂ (12 h)	NaCl	No	Filter paper 11 µm	$\mathrm{H_2O_2} + \mathrm{H2SO4}$	$\begin{array}{c} 60{-}93\ \%\\ PE\ 70\ \pm\ 10\ \%\\ PP\ 93\ \pm\ 3\ \%\\ PS\ 80\ \pm\ 10\ \%\end{array}$	100 μm to 1 mm	Thakur et al. (2023)
	Air-drying sieving 5 mm	Fenton's reagent 3 h	NaCl	No	Stainless mesh 500 and 10 µm	H ₂ O ₂ (24 h)	-	10–500 μm, 500 μm to 5 mm	Jia et al. (2022
	Air-drying sieving 10 mm	Fenton's reagent at 50 °C + 30 mL 0.5 M NaOH (24 h)	NaI	Yes	Stainless mesh 1, 0.25 and 0.05 mm	H ₂ O ₂	-	10–0.05 mm	(Zhang and Liu 2018)

Note. DO: digestion of organic matter, DS: density separation, PET: polyethylene terephthalate, PE: polyethylene, PP: polypropylene, PS: polystyrene, MP: microplastic.

organic matter and relatively fast and minimize extra steps when analyzing MPs in soils.

2.3. Evaluating the recovery rate of extraction methods

Spike-recovery experiments are used to validate extraction methods for MPs with known polymer identity and quantity from soil samples. It typically includes spiking a known quantity of MP polymers to soil samples (which initially do not contain MPs) before applying a specific extraction procedure. Researchers can evaluate the efficiency of the applied extraction method by comparing the amount of polymer recovered after extraction methods. Spike-recovery tests are essential for all MP studies as a validation tool to address the intrinsic variability of soil samples, polymers, and extraction methods. Therefore, it is crucial to conduct spike-recovery experiments to develop high-quality and reproducible MP extraction methods for soils (Hurley et al., 2018). Despite this importance, only 10 studies (approx. 26 %, within combination studies of DS and DO) carried out spike-recovery experiments, as shown in Table 1.

2.3.1. Role of solutions for density separation

There was no discernible pattern in recovery rates for density solutions, regardless of solution type or polymer type (Fig. 4a). PS recovery rates ranged from 100 to 29 % when ZnCl₂ solution was used. With CaCl₂, PE recovery rates ranged from 94 to 42%. Thus, the recovery rate is not solely dependent on the density solution. In most studies, MP extraction was performed using a combination of density separation and organic matter digestion. This variability hinders the comparison of the efficacy of salt solutions across different methodologies. The efficiency of various salts has been compared in a few studies. A study used both low-density (NaCl) and high-density (NaI and ZnCl₂) solutions and reported that the use of high-density solutions led to a higher recovery rate (Li et al., 2019). MPs can vary in density when they interact with soil minerals and organic matter. In addition, plastic additives could make plastic products denser. When considering only density, high-density solutions are recommended even when analyzing low-density polymers (e.g., PE, polypropylene (PP) and PS). It is important to weigh this benefit against the economic and environmental disadvantages of high-density solutions (Kedzierski et al., 2017). A balanced approach is recommended to select salt solutions for MP extraction by considering the trade-off between density, cost, and environmental concerns.

2.3.2. Particle size effect

The correlation between MP size and recovery rate suggests that larger MP sizes usually display increased recovery rates (Fig. 4b). Larger

sizes of PE-MPs showed a higher recovery rate in density separation with the digestion of organic matter for instance (Li et al., 2020). This enhanced recovery rate can be attributed to the flocculation behavior of MPs with cohesive fine particles. MPs of smaller sizes may agglomerate with fine soil mineral particles and settle down more quickly compared to their original size (Fang et al., 2024). In addition to small-size MPs, organic matter also displayed similar adhesive behavior (Zhang et al., 2022). Additionally, limitations and errors in detection methods may also contribute to the low recovery rate for MP of sizes <10 μ m. The challenges associated with DS extraction with DO of small-sized MPs highlight the need for implementing diverse extraction methods that are tailored to a specific particle size range. It is therefore important to employ different methods such as advanced approaches and specialized equipment for less than 50 μ m to increase the efficiency and reliability of MP analysis.

2.4. Identification methods

The identification of MPs entails a determination of MP source and size distribution, the key to uncovering the potential sources of MP contamination. An MP identification process typically begins with the acquisition of microscopic images used for optical sorting (Table 2). MPs are identified using criteria such as morphology, color, shape, and surface texture (Masura et al., 2015). Furthermore, image-based automated software has now been developed to detect MPs (Primpke et al., 2017). To classify extremely heterogeneous MPs, it is essential to sort microscopic images either manually or electronically. However, both routes are prone to errors such as false positives, where non-MP particles are mistakenly classified as MPs due to color misinterpretation (Primpke et al., 2017). In manual optical sorting, around 10 % error was reported among trained team members (Fischer et al., 2016). These errors can stem from subjective interpretation or inconsistent sorting techniques, reducing the precision of MP identification.

To reduce these aforementioned errors, highly magnified images can be acquired using scanning electron microscopy (SEM). However, adopting SEM would involve a great deal of time and expense (Bläsing and Amelung, 2018; Shim et al., 2016; Zhao et al., 2018). The introduction of hyperspectral imaging technology has recently improved optical sorting techniques. Yet, hyperspectral imaging still faces limitations, such as interference from impurities and challenges with color differentiation (Table 2), which can restrict its application in MP identification.

Fourier transform infrared (FTIR) spectroscopy identifies materials based on information about molecular bonds and functional groups. FTIR analysis is most commonly used for identifying soil MPs after initial

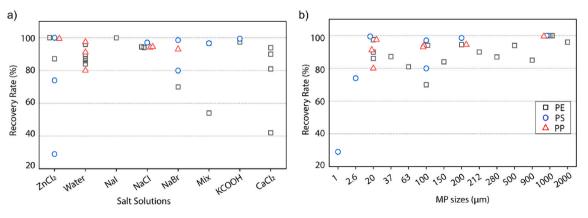


Fig. 4. The recovery rates (%) in experiments conducted in MP extraction from soils, sediments, sludge and biosolid samples based on a) density solution; and b) particle size (lowest limit of size) of polymers. Note: PE - polyethylene, PS - polystyrene, PP - polypropylene. Mix-combined solution of two different salts.

Table 2

The comparison of the methods in microplastic identification.

Identification methods	Techniques	Tested MPs	Advantages	Limitations	References
Microscopy image	-	РР	Low cost Low degree of precision and reproducibility		(Rafique et al., 2020; Zhang Liu, 2018)
	Heating or hot needle	PE, PP	Low cost	Labor and time-intensive	Zhang et al. (2018)
	Stain (Nile Red) Fluorescence	PE, PP, PS, PVC, PET	High efficiency with image processing program	Not applicable for dark-colored MPs	(Grause et al., 2022; Leonard et al., 2022)
FTIR	-	PP, PE, PS, PVC, PVA, PU, PTFE, PET, others	A quick analysis with precision	Size limitation (500 $\mu m)$ for analysis	(Choi et al., 2021;Kim et al., 2021)
μFTIR	IR-compatible filters	PE, PET, PAN	Quick bulk analysis, automated particle analysis of sizes and polymer types	The lower limit of 50 μm and the viewing time per filter was over 1 h	(Li et al., 2019; Pérez–Reverón et al., 2022)
Raman spectroscopy	Pre-treatment by H_2O_2 or alcohol	PS, PE, PMMA, PA, PP	Quick analysis	Sensitive to contaminations, limited spectral information and size (12 μ m) and low accuracy for stacked MPs	(Li et al., 2022a; Zada et al., 2018)
Hyperspectral imaging technology	Combined with three classification model algorithms	PE, PP, PVC	Improved objectivity and quick bulk analysis	Sensitive to contaminations (hyperspectral peaks may interfere with the soil)	Ai et al. (2022)
	Line-scan hyperspectral camera and modelling	PA, PE, PP, PET, PS	Rapidly detection and identification	The deficiency in predicting black or very dark particles and the size detection limit (300–5000 µm)	Vidal and Pasquini (2021)

Note. FTIR: Fourier transform infrared spectroscopy, µFTIR: Fourier transform infrared micro-spectroscopy, PA: polyamide, PAN: polyacrylonitrile, PE: polyethylene, PET: polyethylene terephthalate, PMMA: polymethyl methacrylate, PP: polypropylene, PS: polystyrene, PTFE: polytetrafluoroethylene, PU: polyurethane and PVC: polyvinyl chloride.

optical sorting with microscopic images (Gao et al., 2021; Kim et al., 2021; Li et al., 2019; Pérez-Reverón et al., 2022). In comparison to FTIR spectroscopy, Raman spectroscopy provides a better level of detection for small particles of MPs (1–20 μ m) (Maurizi et al., 2023). However, the reflectance mode of μ FTIR was more effective than Raman spectroscopy, as Raman's high laser power can cause melting (Scheurer and Bigalke, 2018). Furthermore, both FTIR and Raman spectroscopy are affected by organic matter residues which can interfere with the results (Li et al., 2022a).

Often, MPs are identified using thermoanalytical techniques such as thermogravimetric analysis (TGA) and pyrolysis-gas chromatographymass spectroscopy (Py-GC-MS). These thermoanalytical methods can identify only a subset of polymers and do not provide information about morphological features, such as sizes and shapes. Additionally, these methods are affected by organic matter, which can lead to inaccurate results.

The selection of an identification method largely depends on the MP size, and a particular method is only effective for a specific MP size range. Hence, it seems that there is no complete identification method up to now. For MP sizes larger than 500 μ m, optical methods can be used, and for sizes between 500 and 30 μ m, FTIR or Raman spectroscopy can be used. Py-GC-MS can analyze samples under 30 μ m since the MPs in this range of size are difficult to extract from soil. Furthermore, MP identification process, integrated secondary studies of plastic usage in agricultural soils can provide valuable insights into the sources of MP contamination.

2.5. Quantification methods

Quantification of MPs is vital for MP monitoring and further studies, such as assessing their impact on soils and plants. Quantification of MPs is usually conducted alongside the identification of MPs and often involves measuring the number of MP population or their mass. Optical counting is one way of quantification via analysis of microscopic images of MPs using in-built software (Corradini et al., 2019; Prume et al., 2021). MPs with small sizes (e.g., less than 20 µm) are challenging for quantification (count) since optical classification has limitations and particle numbers increase with decreasing sizes (Koutnik et al., 2021). Optical (visual) counting can only express quantity as particle numbers

per soil mass (Table 3). However, their shape, volume, and mass vary widely and it is difficult to compare these reported values for comparable studies.

Several other quantification techniques have been used to measure practical mass-based MP concentrations in the solid matrix (e.g., MP mass per soil mass: $\mu g g^{-1}$ or mg kg⁻¹) (Table 3). Thermoanalytical approaches (such as TGA and Py-GC-MS) are capable of providing simultaneous analysis of a bulk sample. The Py-GC-MS results are often affected by soil organic matter even after the two extraction steps (Liu et al., 2023). Hence, it is necessary to pretreat soil samples to reduce the interference of impurities and increase the confidence of MP detection. Polymers such as PE, PP, and PET do not yield a significant absorption peak in TGA-FTIR analysis, thus their detection is rather difficult (David et al., 2019). The quantitative analysis is further affected by the presence of mixed MPs in environmental samples as they are influencing co-pyrolysis interaction (Lou et al., 2022).

Depending on land use practices with plastic products and source environments, the representative (dominant) size of MPs varies. The dominant MP sizes were reported to vary from 1 to 1.5 to less than 0.5 mm in agricultural soils (Yu et al., 2021; Zhou et al., 2020). Because of the dominant MP sizes, quantification of total MPs requires other approaches to quantify larger-sized MPs, while thermoanalytical analysis can quantify smaller MPs (less than 150 μ m). In addition, Py-GC-MS analysis may be less representative due to its processing of a small size sample (5–200 μ g) (Mansa and Zou, 2021).

Solvent extraction analysis calculates MP concentration in a short duration with relatively simple procedures but is for analyzing the mass of pure MP fractions (Fuller and Gautam, 2016). Thus, it is often necessary to perform additional pre-analysis to obtain polymer characteristics and composition (i.e., proportion of pure polymer and additives). In addition, a matrix effect in the solvent extraction suggests their high sensitivity to soil types (Wen et al., 2021).

Microplastics of a relatively large size can be quantified by optical sorting and weighing. It is, however, difficult to quantify the mass in soils because of the distribution of inorganic and organic matter. Total organic carbon analysis (TOC) is capable of analyzing the total MP quantity after digestion and density separation from the organic matter. A strong correlation was found between TOC values and MP content in soil according to a recent study (Liao et al., 2023). It is suggested to use TOC analysis to quantify MPs of varying sizes in soil samples since TOC

Table 3

The comparison of quantification methods of microplastics.

Quantification methods	Techniques	Unit	Tested MPs	Advantages	Limitations	References
Microscopy	Optical sorting	particle kg ⁻¹	LDPE, PVC, nylon, PES, acrylic	Low cost	Low precision	Corradini et al. (2019)
	Image processing program	particle	РР	Low cost	Only for size $>300 \text{ um}$	Prume et al. (2021)
Py-GC-MS		mg g $^{-1}$	PE, PP, PS, PET, PVC	Simultaneous analysis of the whole sample	Interference of the organic and the inorganic matrix	(Bouzid et al., 2022;Lauschke et al., 2021)
	Two-step extraction	$\mu g \; g^{-1}$	PC, PS, PP, PE, PET		Only limited to soil with less than 2.51 wt % organic matter	Liu et al. (2023)
TGA	Calculate mean thermal mass losses	${ m mg~kg^{-1}}$	PET, PVC, PE, PS	simplicity, low costs, time and no pretreatments	Physical characterization such as shapes, sizes and morphologies were missing Detection limit 0.06 wt %	David et al. (2019)
UV–Vis spectrometry		%	PS	Measurement without filtration	Inability to measure floated particles	(Wang et al., 2018)
ASE	Dionex ASE 350 fast solvent extraction instrument with dichloromethane FTIR	$\mu g g^{-1}$	PA, PS, PE, PET, PVC	The total mass content analysis	Physical characterization such as shapes, sizes and morphologies were missing	Wen et al. (2021)
PLE	Combustion ion chromatography (C-IC) HCl was quantitatively released from PVC during thermal decomposition and trapped in an absorption solution	$\mu g \ g^{-1}$	PVC	Quantify down to the lower $\mu g/g$ range	Inability to measure size fractions and total mass of MPs (only pure mass of polymers)	Kamp et al. (2023)
	Methanol MPs extraction by dichloromethane	$\mu g \ g^{-1}$	HDPE, PS, PVC, PET, PP	Simplicity, cost, speed, and uniformity in reporting concentration results		Fuller and Gautam (2016)

Note. Py-GC-MS: Pyrolysis coupled to Gas Chromatography and Mass Spectrometry, TGA: Thermogravimetric analysis, ASE: Accelerated Solvent Extraction, PLE: Pressurized Liquid Extraction, HDPE: high-density polyethylene, PE: polyethylene, PET: polyethylene terephthalate, PP: polypropylene, PS: polystyrene and PVC: polyvinyl chloride.

is a relatively rapid and convenient technique.

Overall, many challenges associated with quantifying MPs in soils call for continued research and experimentation. Considering the diversity of soil composition and the complex interactions between MPs and organic matter, the need to refine and validate quantification techniques is imperative.

2.6. Quality control

The importance of quality control in MP analysis has been emphasized heavily in previous studies. Tool, water, and air contamination can contribute to inaccuracy in MP analysis. It is necessary to clean and wipe down workbenches with an isopropyl alcohol solution before use (Mbachu et al., 2021). In most studies, laboratory coats made solely of cotton can prevent contamination with synthetic fibers. An orange laboratory coat was used as a distinctive identification with frequent lint removal in one study (Pérez-Reverón et al., 2022). Some studies used labware coated with polytetrafluoroethylene (PTFE), which is usually not the subject of the analysis, while others used glass or stainless steel labware before flushing with ultrapure water. In most cases, stainless steel labware should be muffled over 500 °C to remove any impurities. However, airborne MPs may be deposited on the reagents. All reagents, including blank reagents, must therefore be filtered to ensure purity.

3. Summary: microplastic extraction, identification, and quantification approaches

3.1. Soil sample preprocessing for MP extraction

In soil handling, soil samples must be preprocessed by sieving and drying before MP extraction. Various sieving strategies can be employed to determine the size distribution of MPs as sieving is faster and more effective than optical size detections, especially for particles larger than 100 µm. However, electrostatic adhesion may cause MPs to cling to sieves. Thus, sieving alone may not separate MPs by size entirely if they are hidden within soil aggregates. Incorporating laser light diffraction techniques like Mastersizer analysis can be highly effective for size distribution after extraction (Caputo et al., 2021). However, this method has not yet been widely applied to identify MPs in soils (Fang et al., 2023; Renner et al., 2022). For MPs smaller than 100 μ m, advanced techniques such as μ FTIR, Raman spectroscopy, or Py-GC-MS may be necessary for accurate identification and quantification. To identify the major source of MPs and analysis, it is also essential to survey plastic usage in the field and collect larger plastic particles for further comparisons.

3.2. Challenges in MP extraction from soil

Removing organic matter or adhering impurities from MPs is the most challenging part of MP extraction from soils. As organic matter degradation by digestion agents can also cause MP degradation, and dispersing soil aggregates may lead to further fragmentation of MPs, careful consideration is needed to balance both processes. Implementing a pre-digestive step can help solve both issues, removing organic matter as well as dispersing soil aggregates. It is preferred to use oxidative digestion by H_2O_2 over acidic/alkaline DO. Carbonation by H_2SO_4 is a powerful digestion pathway, though certain plastics may degrade as a result. Nevertheless, soil organic matter content and soil texture determine the efficiency of DO. Testing digestion reagents on collected larger MP particles could help assess their susceptibility to selected digestions, and a prolonged digestion cycle and repeated digestion may be beneficial for eliminating organic matter under optimal conditions.

3.3. Density separation for MP extraction

In the separation of soil minerals, density separation with highdensity solutions (e.g., ZnCl₂, NaI and NaBr) is an effective method. Centrifugation can shorten the time for DS, although MPs may be further fragmented. The centrifuge speed should be adjusted to balance effective separation while minimizing the risk of MP fragmentation. In soils with high organic matter, combining DO and DS may be an acceptable solution. However, filtration causes MPs to be trapped inside membrane pores, leading to erroneous results. Minimizing the usage of filtration is therefore desired for accurate MP extraction. To minimize filtration, alternative approaches such as direct identification and quantification techniques like Py-GC-MS or Raman spectroscopy can be used. However, if digestion is required, it is preferable to use methods that do not require filtration afterward to ensure accurate MP extraction.

3.4. Identification and quantification of MPs

Among the methods for identifying and quantifying, stereomicroscopy and FTIR are widely used for identification and quantification. While this method is highly reproducible for large-sized MPs, it is less accurate for smaller MPs (e.g., 50 μ m), necessitating more suitable detection techniques such as μ FTIR or Raman spectroscopy. μ FTIR is typically effective for particles down to 50 μ m, while Raman spectroscopy can accurately identify MPs down to 20 μ m, making them more suitable for smaller MPs. Microscopy and spectroscopy only provide results in the units of particle per soil mass or number of items per soil mass. However, to enhance comparison across MP studies, it is critical to quantify MPs in terms of their mass per soil mass, providing a normalized measurement of MP contamination in soils.

3.5. Emerging methods for mass-based MP quantification

Mass-based MP quantification is currently evolving, with ongoing research focusing on precise quantifications such as Py-GC-MS, ASE and PLE. Additionally, combined approaches, like PLE with Py-GC-MS or ASE with Py-GC-MS, are being explored to improve detection accuracy. While ASE and PLE have been proven effective in extracting MPs from soils, the presence of organic matter can interfere with MP extraction efficiency. As a result, further optimization or a unique approach to these techniques is needed to enhance their reliability in MP extraction and quantification (see Fig. 5).

3.6. Uncertainty of environmental factors

Environmental factors introduce variability and uncertainties in MP analysis. High-temperature variations can affect the physical and chemical properties of MPs, adding challenges to MP analysis. High temperatures can cause thermal degradation of MPs which may alter their chemical signatures, complicating or lowering the accuracy of their identification and quantification techniques such as FTIR and Py-GC-MS (Huang et al., 2021). Rainfall and flooding affect the spatial and temporal distribution of MPs in soils, introducing uncertainties in their quantifications. Heavy rainfall or flooding can lead to the vertical and horizontal movement of MPs in soils, making the sampling strategies complicated (Han et al., 2022). Soil texture variations influence the adhesion of MPs in soils, potentially affecting extraction efficiency. High clay soils would have more interactions with MPs than sandy soils and form more aggregates, hindering the separation of MPs in soils (Guo et al., 2022). Thus, these environmental factors complicate the consistency of MP extraction and quantification. It is important to have careful consideration of soil properties and external conditions in study designs and methodologies.

4. Conclusion

Extraction, identification, and quantification of MPs from soil samples are reviewed in this work. MP identification and quantification require accurate digestion of soil organic matter. Density separation methods are more efficient with high-density solutions, especially for small-sized MPs. Since soil types and polymers differ, digestion methods and density solutions need to be carefully selected.

In terms of identification and quantification, both FTIR spectroscopy and stereomicroscopy appear to be widely used. However, the current measurement units of particle count, or particle count per unit soil mass are not adequate to determine MP concentrations for risk assessments. Mass-based quantification should therefore be developed to further assess MP occurrence, calculate removal efficiency, and estimate MP movement in the matrix.

Even though previous studies cannot be compared and analyzed in a straightforward manner, it is evident that a recovery rate test should be included in the validation process. In addition, digestion methods should also be tested to assess whether microplastics are altered or degraded. Integrated extraction and quantification methods may permit the development of standard soil MP assessment protocols.

CRediT authorship contribution statement

Yoonjung Seo: Writing – original draft, Data curation, Conceptualization. Venkata Chevali: Writing – review & editing. Yunru Lai: Writing – review & editing, Conceptualization. Zhezhe Zhou: Writing – review & editing. Guangnan Chen: Writing – review & editing, Supervision. Paulomi Burey: Writing – review & editing. Shaobin Wang: Writing – review & editing. Pingan Song: Writing – review & editing,

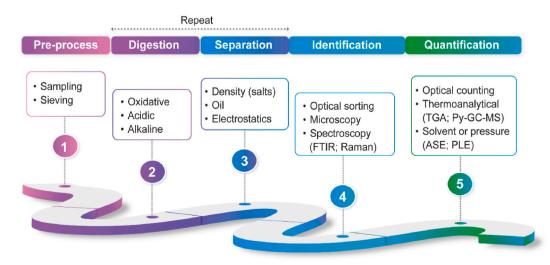


Fig. 5. Proposed procedures of MP extraction from soils, based on the literature and high recovery rates. Note: FTIR - Fourier transform infrared spectroscopy, Py-GC-MS - Pyrolysis coupled to gas chromatography and mass spectrometry, ASE - Accelerated Solvent Extraction, PLE - Pressurized Liquid Extraction.

Supervision, Resources, Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

No data was used for the research described in the article.

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