Tracing the sources and microbial degradation of PCBs in field sediments by a multiple-line-of-evidence approach including compound-specific stable isotope analysis

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Microplastics as pollutants in agricultural soils

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Dissemination of MPs in the environment
(Pharmaceuticals, Transportation,
Agriculture, Compost)

Effect of MPs on soil environment
(Plants, Animals and Microbes)

Isolation of MPs from the soil
(Sieving, Density separation,
Filtration and Extraction)

- Identification and characterization of MPs
- Assessment of ecological potential risks

Abstract

Microplastics (MPs) as emerging persistent pollutants have been a growing global concern. Although MPs are extensively studied in aquatic systems, their presence and fate in agricultural systems are not fully understood. In the agricultural soils, major causes of MPs pollution include application of biosolids and compost, wastewater irrigation, mulching film, polymer-based fertilizers and pesticides, and atmospheric deposition. The fate and dispersion of MPs in the soil environment are mainly associated with the soil characteristics, cultivation practices, and diversity of soil biota. Although there is emerging pollution of MPs in the soil environment, no standardized detection and quantification techniques are available. This study comprehensively reviews the sources, fate, and dispersion of MPs in the soil environment, discusses the interactions and effects of MPs on soil biota, and highlights the recent advancements in detection and quantification methods of MPs. The prospects for future research include biomagnification potency, cytotoxic effects on human/animals, nonlinear behavior in the soil environment, standardized analytical methods, best management practices, and global policies in the agricultural industry for the sake of sustainable development.

Keywords: Environmental pollution; Microplastics; Analytical techniques; Agricultural systems; Soil health; Sustainable development.

1. Introduction

In recent years, anthropogenic activities are considered as the key drivers of biodiversity loss and ecosystem functions (de Souza Machado et al., 2018a). A typical marker of human activity is the overwhelming amount of plastics produced and used. Plastics are chemically miscellaneous groups of synthetic polymeric materials with multiple applications in modern lifestyle (Galloway

et al., 2017). It has been estimated that the majority of the produced plastics (>80%) are thermoplastics, which are industrialized via polymerization to form high-molecular-weight polymers from low-molecular-weight monomers (de Souza Machado et al., 2018a). Their physical and chemical characteristics can be altered and strengthened by physical methods (e.g., extrusion, melting, and palletization) and chemical methods (e.g., mixing with antioxidants, copolymer polycarbonate, plasticizers, and colorants) (Bittner et al., 2014). As a result, plastic materials have a strong physical structure along with complex chemical properties. Owing to low cost, efficient malleability and durability, the demand and usage of plastics have been increasing continuously in the past decades (PlasticsEurope, 2015), and 8300 of plastics are generated worldwide (Geyer et al., 2017).

In 2016, 27.1 million metric tons (Mt) of plastic wastes were collected in the European Union (EU), of which 31.1% were recycled, 41.6% were used for energy recovery, and the remaining 27.3% were disposed of at landfill sites (PlasticsEurope, 2018). It was estimated that the contemporary manufacturing and waste management strategies would lead to 12,000 Mt of plastic wastes in landfills/natural setting by 2050 worldwide (Geyer et al., 2017). As shown in Table 1, the European plastic demand was ~47.8 Mt in 2014; however, only 54% and 16% (approximately) of plastics underwent the waste management process and recycling system, respectively (PlasticsEurope, 2015). More recently, 64.4 Mt of plastics production was reported in the EU, while 8.4 Mt of plastic wastes were collected and recycled inside/outside the EU (PlasticsEurope, 2018). The worldwide plastic recovery and recycling rates might be even lower. It was found that approximately 32% of plastic wastes might be present in the soil environment (de Souza Machado et al., 2018b; Jambeck et al., 2015). Plastic wastes exposed to the natural environment can

undergo weathering processes such as degradation and disintegration due to the collective actions of physio-chemical and biological factors (de Souza Machado et al., 2018a; Whitacre, 2014).

Plastics waste including biodegradable plastics are actually more susceptible to physical disintegration (fragmentation) than degradation (mineralization), which result in smaller sizes of plastics (Whitacre et al., 2014). The generated plastics with particle size <5 mm are generally considered as microplastics (MPs) (Li et al., 2018). The global presence of MPs is probably due to the extensive production of microplastic particles (e.g., microbeads) for diverse applications. Natural disintegration and degradation of MPs can also generate plastic particles <0.1 µm in size, which is known as nanoplastics (NPs) (de Souza Machado et al., 2018a). Owing to the deleterious effect of MPs on the marine and shoreline environment, MPs have attracted a lot of attention of marine scientists (Galloway et al., 2017; Li et al., 2018; Wang et al., 2019). Similar effects of MPs are also observed in freshwater and estuarine ecosystems (Horton et al., 2017a), and there also are emerging concerns over MPs as pollutants in the aquatic environments (Li et al., 2018).

MPs pollution in the soil environment has received nominal scientific attention in comparison to that of marine environment, whereas the former might be 4-23 times larger than the latter in terms of mass (Horton et al., 2017b). Terrestrial soil tends to accumulate more MPs than aquatic ecosystems (Nizzetto et al., 2016a). The United Nations Environment Programme (UNEP) identified that large quantities of particulate plastics found within the marine environment globally result from the land-based sources (UNEP, 2016). According to Jambeck et al. (2015), 4.8 to 12.7 Mt of terrestrial plastic wastes enter the ocean annually, equivalent to 1.7 to 4.6% of the total plastic wastes generated worldwide. Sediment transfer during soil erosion is a process that allows the transport of particulate plastics from terrestrial to aquatic ecosystems. Despite this linkage to

terrestrial sources, most scientific investigations on plastic particles have neglected their effects (Bolan and Bradney, 2019; Bradney et al., 2019).

MPs pollution threats to the aquatic environments are frequently associated with the living organisms in the aquatic environments, i.e., MPs can serve as particulate matter for ingestion (Rehse et al., 2016), solid supports for pollutant transport (Zhan et al., 2016), and significant chances of physical injury during the movement (de Souza Machado et al., 2018a). The environmental impacts of MPs pollution in the agricultural soils may be underestimated in comparison to aquatic systems. Investigations are urgently required to draw attention to MPs in the agricultural systems. However, only a few recent studies have examined the pollution levels and the possible sources of MPs in the agricultural environment and their harmful effects on the soil biota (Boots et al., 2019; Chae and An, 2018; Mai et al., 2018; Zhang & Liu, 2018; Zhang et al., 2018b). Recent results revealed the pervasive and persistent nature of MPs in the soil environment (Zhang & Liu, 2018), and adverse effects of MPs on growth, reproduction, feeding, survival, and immunity level of the soil biota (animals, plants, and microorganisms) (Zhu et al., 2018a; Zhu et al., 2019; Zhang et al., 2019). The latest reviews came to describe the mechanisms and behavior of MPs in the soil environment (Qi et al., 2020), their biomagnification tendency via food chains, toxicological effects on soil microorganisms, and analytical methods (Guo et al., 2020; Li et al., 2020). Nevertheless, overall research on MPs pollution in agricultural soils is still in an embryonic stage and there are many knowledge gaps in this research areas. Several key questions such as pollution levels, ecological threats, dispersion mechanisms, and development in analytical and quantification technologies still require further studies in view of the high complexity and heterogeneity of the soil environment in different agricultural systems.

Therefore, this review aims to offer in-depth current knowledge about sources, fate, and dispersion mechanisms of MPs in the agricultural soils. The adverse effects of MPs of the soil flora and fauna are elucidated. This review also elaborates the analytical technologies involved in detection and quantification of MPs in soil and suggests the framework for future research.

2. Possible sources and dispersion mechanism of MPs in soil and agricultural environment

An enormous variety and range of MPs particles are available in the soil environment due to over exploitation of plastics and their unplanned management practices (**Figure 1**). Typically, the existing literature reported that terrestrial environments are the only component of the natural systems that can act as a source and distribution pathway of MPs to the aquatic environments (**Horton et al. 2017a; Karbalaie et al., 2018; Lechner et al., 2014**). For example, the release of significant quantities of illegal commercial MPs from manufacturing plants to Reverse Danube has been reported (**Lechner et al., 2014**). Plastics near coastal zones or in surface water disintegrate due to direct physical abrasion and UV-sunlight. Nevertheless, both processes are marginal in the soil environment in which plastic disintegration and degradation could be comparatively slow (**Karbalaie et al., 2018**).

Previous investigations reported limited degradation of commercial polymers in the soil environment. **Arkatkar et al. (2009)** reported only 0.4% degradation of polypropylene (PP) after one-year soil incubation, while no weight loss was observed in the case of polyvinyl chloride (PVC) after soil incubation for 10-35 years. Soil texture and composition play vital roles in the degradation of synthetic polymers in the soil environment. It was reported that clayey soils showed a greater degradation of polymers in comparison to sandy soils, possibly attributed to a higher soil organic matter (SOM) (**César et al., 2009**). There were also significant impacts of MPs on soil

and soil-water relation including water holding capacity, bulk density of soil, microbial activities, and soil structure (**de Souza Machado et al., 2018a; 2019**). The alteration in soil structure may lead to changes in the microbial composition of the soil, although it can be difficult to predict such changes (**Rillig et al., 2019**). The decrease in bulk density of the soil due to MPs pollution may increase the rate of evaporation, thus significantly affecting plants growth (**Wan et al., 2019**). Nevertheless, MPs pollution may also have some positive impacts on the soil properties. For instance, MPs facilitated soil aeration and root penetration by reducing the bulk density of the soil (**de Souza Machado et al., 2018a, Rillig et al., 2019**) and promoted the growth of onion bulbs and roots resulting in the increase in total crop biomass (**de Souza Machado et al., 2019**).

There are several factors affecting the quantity of MPs deposition, retention, and transport in the soil environment, such as anthropogenic activities (e.g., inefficient waste management practices and littering), physical characteristics of plastic particles (e.g., form, size, and density), climatic conditions (e.g., rainfall intensity and wind speed), and topography (**He et al., 2018b; Karbalaei et al., 2018; O'Connor et al., 2019**). A substantial direct contribution of primary MPs to terrestrial systems has been introduced via areal deposition of MPs. Synthetic textile fibers, wear and tear from synthetic rubber tires, and city dust are the major atmospheric sources of particulate plastics (**Kole et al., 2017; Löhr et al., 2017**). These particulate plastics are dispersed by wind in the form of atmospheric pollution. For example, 3–7% of the particulate matter (PM_{2.5}) in the atmosphere is estimated to consist of plastic derived from tire wear and tear (**Kole et al., 2017**).

Other sources of MPs in terrestrial environments include domestic wastes, personal care products, mismanaged solid waste landfills, and land application of biosolids (**Horton et al. 2017b; Steinmetz et al. 2016; Rillig et al., 2017**). In developed countries, landfill sites are enclosed by fences and dumped wastes are typically shielded with soil cover or synthetic materials,

which help to restrict the run-off of MPs from the site. Nevertheless, proper waste management practices are in their infancy in developing or underdeveloped countries (Duis & Coors, 2016). A substantial accumulation of plastic wastes in the agricultural soils has been reported in several tropical and subtropical countries, with MPs from municipal wastes dumped in open agricultural fields, parks, or landfill sites. Lwanga et al. (2016) reported that approximately 1000-4000 MPs particles per kg of dry weight of biosolids were found in agrarian fields and dumping sites in Europe; Fuller & Gautam (2016) reported MPs found in different types of soil close to a commercial zone in Australia (Table 2). According to Nizzetto et al. (2016b), due to the application of biosolids as fertilizer, up to 700,000 tons of MPs may enter agricultural fields annually in North America and Europe.

Properly designed and operated wastewater treatment plants (WWTPs) are able to effectively remove MPs debris up to 99.9% from the wastewater streams, but a substantial quantity of MPs remained in the biosolids (Gies et al., 2018; Mintenig et al., 2017; Prata, 2018). According to Alvarenga et al. (2016), more than 87% of the generated biosolids from wastewater treatment was applied in the agriculture fields of Portugal in the form of composts or raw biosolids. Also, 4 to 5 Mt of sludge solids are used as fertilizer in the European Union (EU) every year in agricultural lands. In some sites, commercial polymeric fibers were detected after five years of land application of biosolids. Compost application in agriculture field is also considered a significant contributor of plastics in soils. Although large- and medium-sized plastic fragments are mostly separated from the composts (Figure 2), minor quantity of smaller fragments formed during the process of composting (milling process) remains in the form of secondary MPs or NPs (Bolan and Bradney, 2019). Polymer-based slow-release fertilizers (Weithmann et al., 2018) and pesticides (Wang et al., 2019) along with the weathering of plastic film mulch used over agricultural fields also result

in the MPs pollution in the soil environment (**Huang et al., 2020; Ramos et al., 2015; Rillig, 2012**).

Personal care products (PCPs) such as gels, hand wash, shampoos, and facial cleaners are considered as the potential contributors of MPs and reach terrestrial environments via the application of biosolids and composts (**Alvarenga et al., 2016; Duis & Coors, 2016**). Polyester (PES), polystyrene (PS), and melamine are applied in different industries as abrasive materials, which are the potential sources of MPs. Other contributors of primary MPs including plastic powder and plastic resin pellets can enter the terrestrial environments due to improper handling and inefficient waste management practices (**He et al., 2018a; Karbalaie et al., 2018**). Likewise, plastic processing and recycling plants produce residues that are discharged into the environment and further transformed into MPs (**Karbalaie et al., 2018**). For example, larger quantities of raw materials utilized for manufacturing of plastics products were observed on the beaches closer to the plastic-processing and recycling plants (**Duis & Coors, 2016**).

Several groups of organisms such as earthworms can facilitate the conversion of primary MPs to secondary MPs and NPs by taking actions in their gizzard (**Rillig, 2012; Zhu et al., 2019**). Scraping or chewing mechanisms of Collembola or mites may lead to the generation of MPs and NPs from larger plastic debris. Likewise, burrowing mammals can also facilitate the incorporation of MPs in the soil via abrasion mechanism (**Rillig, 2012**). **Rillig et al. (2017)** reported that the activities of earthworms lead to the incorporation of PS into the soil profile from the top surface. There are various probable implications to the migration of MPs downward into deeper soil profile from the surface via existing organisms: (a) decomposition by the native microorganisms was very slow in the deeper part of the soil profile due to less microbial population (because of limited oxygen diffusion/availability), thus leading to more retention of MPs ; (b) entry of MPs in the soil

profile may also increase the chance of groundwater contamination with MPs and associated chemicals; (c) conversion of MPs into NPs in the soil environment via disintegration, decomposition, and abrasion may lead to further potential environmental menaces including uptake of NPs by plants (Rillig et al., 2017).

3. Adverse effects of MPs in the soil environment

Although MPs are defined as pure polymers of physical particles, they are often mixed with other chemical entities such as heavy metals, dioxins, and polycyclic aromatic hydrocarbons (Hong et al., 2017). Deliberate addition of extensive chemicals such flame retardants and plasticizers to the plastic products, which subsequently become MPs, can cause hazards to soil flora and fauna. A few studies demonstrated the possible pollutants transfer from MPs to beneficial soil organisms such as earthworms while others indicated the role of MPs in causing toxicity to the sludge digestive microbial flora or marine microbiota (Gaylor et al., 2013; Oliviero et al., 2019; Wei et al., 2019). During the manufacturing and processing of plastics, various chemicals and additives are used to improve the properties and the applications of the final products (Bolan et al., 2020) (Table 3). After longer exposure to the natural environment, these chemicals and additives are leached into the soil environment via slow release/desorption and photochemical degradation, causing adverse effects on soil microbial diversity and function (Bolan et al., 2020; Bolan & Bradney, 2019). Therefore, the toxicity of these chemicals associated with MPs pollution should be taken into account and carefully evaluated. A spectrum of pollutants such as pesticides (Nie et al., 2020), dyes (Kumar et al., 2019), heavy metals (Kumar et al., 2020) were found in the MPs-polluted environment. Large surface area of MPs enhances the adsorption of co-existing

pollutants, thus facilitating the transport and spreading of the laden contaminants that require prudent consideration and future investigations (Zhang et al., 2019).

3.1 Effects on plants

There are two major questions to be addressed with plants and MPs: (a) whether plants can accumulate MPs and (b) how absorbed MPs affect the plant growth and subsequently reach the food chain (Zhu et al., 2019). It is difficult to distinguish the various types of MPs in a plant tissue, which requires further studies and detailed investigations. There are different sizes of MPs identified so far including the nanoscale and microscale ones, which can get across plant's membranes and cell wall barriers and can be detected using fluorescent microbeads. Bandmann et al. (2012) observed that endocytosis assisted the entry of nano-sized (less than 100 nm) fluorescent PS beads inside a tobacco BY-2 cells, whereas edible plants were capable of incorporating micro-sized (0.2 µm) fluorescent PS beads from the environment as indicated in the whole plant culture study (Li et al. 2019). The pollution of MPs poses an additional risk to humans via trophic food chain transfer. Only few recent studies reported the adverse effects of MPs on plants (Qi et al., 2018; Rillig et al., 2019). For example, by spiking a soil sample with 1% PE plastics and biodegradable particles in the soil, both types of MPs induced negative effects on wheat plant and grain biomass, i.e., inhibition of the number and weight of the grain biomass. Although earthworms used in the same study alleviated the influence of MPs to wheat, the study did not include the examination of plant tissue containing PE particles.

3.2 Effects on soil microorganisms

Soil microorganisms, such as bacteria and fungi, can be affected by the exposure to overwhelming quantity of MPs (Bradney et al., 2019; Wijesekara et al., 2019). The effects of

MPs on soil microorganisms were investigated, including their effects on bacterial transport, spread of antibiotic resistant genes (ARGs), and overall microbial metabolisms. There was a threshold for plastic particles to bring positive or negative impacts to the microbial activity. For example, the addition of 0.05–0.4% polyester, 1 mg kg⁻¹ PS, and 0.05–0.4% polyacrylic particles stimulated negative impacts on the microbial activities (Awet et al., 2018; de Souza Machado et al., 2018a), while 7% and 28% of PP particles led to positive impacts on the microbial activities (Liu et al., 2017). Many parameters including polymer shape, type, concentration, and size of the MPs were variable in these studies, so it was difficult to generalize the toxic effects of MPs on the microbial activities with respect to individual variables. Regarding the structure of microbial community and soil structure, there was no direct evidence for deriving a generic conclusion on the MPs toxicity based on these studies. More importantly, the high concentrations of MPs under artificial spiking may be not representative of the field-relevant conditions.

The effects of MPs on microbial transport, metabolism, and genetic exchange are not scrutinized but a few recent studies may provide some insights. He et al. (2018b) observed that with increasing ionic strength, bacterial transport (*Escherichia coli*) in quartz sand was stimulated by the PS particles, while no noticeable difference was reported under a low ionic strength where both bacteria and PS particles displayed high mobility. The MPs were also found accountable for the exchange of genes between phylogenetic non-related microorganisms as they introduced additional exchange surface for genes and other metabolic products (Huang et al., 2019; Sun et al., 2018). Along with the array of potentially beneficial genes, there were many harmful genes such as ARGs that resulted in deleterious effects on human health upon transfer by MPs (Arias-Andres et al., 2018; Huang et al., 2019; Imran et al., 2019). Sun et al. (2018) observed that the retention times for ARGs and antibiotics were increased by the addition of 0.1% PS MPs to the

soil. More research should be conducted to provide additional and direct evidence on the ARGs transmission by MPs.

3.3 Effects on soil animals

There are a few reports focusing on the effects of MPs on aquatic animals, but very limited data are available for soil animals. A small subset of invertebrates was studied (including isopods, nematodes, collembolan, and oligochaeta) to examine the effects of MPs on the growth, survival, metabolism, gut microbiome, feeding pattern, and inflammatory reaction of soil animals (Kim et al., 2019; Lei et al., 2018). Lei et al. (2018) experimented with 1 mg L⁻¹ PS particles and exposed a terrestrial nematode (*Caenorhabditis elegans*) to different sizes of PS particles (0.1, 0.5, 1.0, 2.0, and 5.0 µm) for 72 h. The nematode with the 1.0 µm group demonstrated the shortest body length, low survival rate, short lifespan, and even downregulation of unc-17 and unc-47 genes expression, leading to irreversible damage of GABAergic and cholinergic neurons. These effects were attributed to the immediate uptake and high accumulation of 1.0 µm particles by nematodes. The concentration of MPs was considered as a significant variable affecting the organisms' activities. Zhu et al. (2018a) conducted a concentration-dependent experiment with oligochaete *Enchytraeus crypticus*. Small concentrations of PS (0.025 wt% in oatmeal) in soil showed a positive impact on the weight of *Enchytraeus crypticus* while 0.5 wt% did not cause any significant changes. With 10 wt%, a negative shift was observed in the weight of gut microbiome. Similar findings were validated by Lwanga et al. (2016), who observed that the survival of *Lumbricus terrestris* was adversely influenced by the addition of PE MPs in 28–60% litter (corresponding to 0.8–1.7 wt% in soil), while PE MPs in 7% litter (0.2 wt% in soil) brought no significant change in the growth and survival. Cytotoxicity analysis of MPs and biodegradable plastics on oligochaeta can be traced through histological gut assessment. The addition of 0.0625–1 wt% PE MPs in soil did not cause

any deleterious effects on earthworm *Eisenia Andrei*, but induced immune response and tissue damage under the exposure (Rodriguez-Seijo et al., 2017). The starch-based biodegradable PE films led to more negative effects on the growth of an earthworm in comparison to conventional low-density PE. This might be because biodegradable plastics consisted of more toxic monomer units, including polybutylene terephthalate (PBT) and polyethylene terephthalate (PET), than the conventional PE plastics (Rodriguez-Seijo et al., 2017).

A wide range of soil species is sensitive to MPs including collembolan strains such as *Folsomia candida*. Experiments using $\delta^{15}\text{N}$ and $\delta^{13}\text{C}$ isotopes revealed that there was a substantial modification in the metabolic turnover, i.e., 28.8% reproduction impairment and 16.8% growth inhibition upon the addition of 0.1 wt% PVC MPs in soil (56-day exposure) (Zhu et al., 2018b). The impairment in reproduction and growth may be partly due to a shift in Collembolan oviposition sites and feeding habits due to MPs (Zhu et al., 2018b). Ju et al. (2019) observed similar results in the reproduction of *Folsomia* sp. when exposed to 0.1–1 wt% PE MPs in soil. In both studies, modified animal gut microbiome was observed upon the exposure. These results suggested that Collembolan species could be a potent indicator of MPs pollution and disturbance. Owing to the significant role in litter decomposition, isopods are also employed as testing organisms. Their energy reserve and feeding behavior were observed in the presence of 0.4 wt% PE MPs with food pellets. Various end points were investigated including body mass index (BMI), food assimilation, food ingestion, defecation rate, energy storage, and mortality. No effects were observed after 14 days of exposure, indicating low deleterious effect of MPs to isopod *Porcellio scaber* (Kokalj et al., 2018).

Recent research evaluated how MPs affected the bioaccumulation of organic pollutants, but bioaccumulation tendency of MPs was still not much explored. Better understanding is required

regarding the transfer of MPs from one trophic level to another and the bioaccumulation tendency of MPs in the soil environment. For instance, **Lwanga et al. (2017)** studied the increase in concentration of MPs in home garden soil by analyzing earthworm casts and chicken feces. The concentration of MPs increases throughout the trophic level with maximum concentration (129.8 ± 82.3 particles g^{-1}) in chicken feces with 10.2 ± 13.8 MPs particles per gizzard of the chicken. This study predicted that the consumption of the chicken gizzard by humans can lead to the accumulation of 840 plastic particles/person/year. The potential long-term effects of MPs on soil organisms and soil animals should be further examined with future bioaccumulation and biomagnification studies.

4. Methods of extraction, detection, and characterization of MPs in agricultural systems

The ubiquitous presence of MPs was studied in aquatic ecosystems (freshwater and marine) using a variety of analytical methods (**Mai et al., 2018; Zhang et al., 2018a**). However, the research maturity is still insufficient to develop the standardized methods for MPs quantification in the soil and sediment ecosystems (**He et al., 2018a**). Soil is the uppermost layer of earth and home to a wide array of organisms, where MPs get accumulated along with impurities and organic matter. The soil composition plays an important role in the detection of MPs via floatation, separation, and interference with infrared signaling (**von Sperber et al., 2017**). The development of cost-effective, time-efficient, and accurate methods to analyze and quantify MPs in the agricultural soils is an immediate need of the hour.

In order to assess the MPs pollution, proper selection of the sampling sites is required before analysis and quantification can help us assess the actual status of the site. Collection of the MPs

samples is a crucial step in the analytical procedures. Different layers of top and bottom soil should be collected depending on the soil characteristics (Liu et al., 2018; Zhou et al., 2018). Then, density separation should be performed depending on the percentage of organic matter and clay on the dried, sieved, dispersed, filtered, and separated soil sample (He et al., 2018a). Density extraction and SOM digestion are carried out after which the extracted MPs can be visualized with an optical microscope. Raman and micro-Fourier transformed infrared (μ -FT-IR) spectroscopy are used for fingerprinting the types and distribution of MPs (Liu et al., 2018; Peng et al., 2017). The above protocol involving repetitive sieving and density separation is appropriate for analyzing the soil samples but requires future standardization of each procedure.

4.1 Extraction procedure

Variable sizes of sieves are used in consideration of the sample type and requirement. According to NOAA (2015) procedures, dry samples are initially sieved through a 5 mm size sieve, followed by stacked 5 mm and 0.3 mm sieves to segregate the disaggregated sediments as shown in Figure 3. Unlike sediment and water column samples, the soil samples are primarily segregated through a 2 mm size sieve after density separation of MPs (Zhang & Liu, 2018). MPs elements can be separated from the soil sample matrix using salt solutions with known concentrations as plastic particles float over high-density solution. The concentration of salt solution should be based on the density of the MPs as the concentration of 1.18 g cm^{-3} NaCl solutions is not enough to separate high-density plastics such as PVC and PET (NOAA, 2015).

A feasible and low-cost method for the extraction of light-density plastic particles including PP and PE from the soil was developed using distilled water with plastic recovery rates of almost 90% (Zhang et al., 2018b). The soil sample underwent heat treatment (3–5 s at 130°C), which made the MPs present in the sample melt and converted into circular transparent particles that can

float on the water surface and be separated easily while other components, such as organic matter and minerals, remain in their native form. **Liu et al. (2018)** used NaCl for MPs separation from the agriculture soil sample with the ultrasonic treatment over prolonged floatation time. Seven out of nine spiked MPs types were successfully extracted, including polymethyl methacrylate (PMMA), acrylonitrile butadiene styrene (ABS), polycarbonate (PC), polyamide (PA), PE, PP, and PS. The method was designed for PVC and PET but was found to be ineffective (**Liu et al., 2018**). Different solutions were then proposed such as CaCl₂, which was efficient in the extraction but led to agglomeration of organic matter and hindered the MPs identification (**Scheurer & Bigalke, 2018**).

Van Cauwenberghe et al. (2015) suggested that the extraction solution would be efficient for density range of 1.6–1.8 g cm⁻³, which was attainable through the usage of NaI or ZnCl₂ along with the addition of acid solution. However, these solutions are not environmentally friendly. Also, acid solution can alter the characteristics of MPs in the soil samples. Other methods utilizing oleophilic properties and electrostatic behavior of MPs were proposed. The oil extraction protocol was efficient for achieving 90% recovery of MPs (**Crichton et al., 2017**), while the electrostatic behavior was 100% efficient in recovering plastics from multiple environmental matrices including beach sand, water, and sediments (**Felsing et al., 2018**). However, their applicability for a large-scale extraction of MPs from the soil samples is uncertain.

4.2 Removal of SOM

Density fractionization was not efficient enough to separate SOM from the soil samples because they have a similar density (1.0 and 1.4 g cm⁻³) to several plastics such as nylon and PET (**Bläsing & Amelung, 2018**). Therefore, earlier studies utilized acids, alkalis, enzymatic digestion, or oxidizing treatment (**Dehaut et al., 2016; Jabeen et al., 2017**). **Hurley et al. (2018)** tested the

effectiveness of different reagents, including 30% H₂O₂ solution, 10% KOH, 10 M NaOH, and Fenton's reagent, of which Fenton's reagent in combination with density separation was found to be the most suitable for SOM removal in the soil MPs assessment. However, these reagents were likely to damage or modify the MPs. For instance, HNO₃ solution removed organic matter in a short span but also damaged MPs of PET, PA, and ABS (Dehaut et al., 2016; Rillig et al., 2017), while alkali treatment led to plastic abrasion (Hurley et al., 2018). Meanwhile, H₂O₂ would change the structural morphology of PP and PE plastic but the change was minimal when it was used for digestion at 70 °C. Therefore, H₂O₂ was considered as the most favorable oxidizing agent for the removal of organic matter from the environment matrices (He et al., 2018a; Hurley et al., 2018; Jabeen et al., 2017; Zhang et al., 2018b). Liu et al. (2018) confirmed that the oxidation by H₂O₂ was efficient for SOM removal from the agricultural soils.

4.3 Identification and characterization

Subsequent to the isolation of MPs from environmental samples, several techniques are applied for the identification, quantification, and characterization of MPs as listed in **Table 4**. The samples are first visually identified for their surface texture after the classification based on their shape, size, and color (He et al., 2018a). Chemical classification is performed using gas chromatography mass spectroscopy, Raman, or infrared spectroscopy (Li et al., 2018). MPs are sorted and grouped according to their sizes. In addition, MPs are sorted in terms of their shapes, namely fragment, fiber, foam, bead, and film. Although visual identification is a feasible step to start classifying an enormous variety of MPs, it possesses its own limitations with human errors. Eriksen et al. (2013) reported 20% of aluminum silicate particles being visually misinterpreted as MPs through a confirmation by SEM. Lenz et al. (2015) reported that nearly 70% of the particles were erroneously labeled as MPs using FTIR and 32% of visually labelled MPs were not identified

by μ -Raman spectroscopy. There was a need for combining visual identification with other confirmatory physical and chemical techniques in the classification of MPs. SEM provides high magnification image of the sample but requires longer time and higher costs (sample preparation, coating, analytical cost). The sample coatings may disturb the surface texture and result in inaccuracies in the detection of MPs (Bläsing & Amelung, 2018; Shim et al., 2017; Zhao et al., 2018).

One of the most popular non-destructive chemical techniques to identify MPs is infrared microscopy along with μ -Raman, attenuated total reflectance (ATR), and μ -FT-IR spectroscopy, which offer an advantage of microspectrometry coupled with automated scanning (Bläsing & Amelung, 2018). μ -Raman is more sensitive than μ -FTIR as it can detect MPs size as small as 1 μ m while μ -FTIR is effective for particles size >10-20 μ m (Cai et al., 2017). The presence of organic matter interferes with the signaling, which can be avoided using a fluorescent background in the case of Raman spectroscopy (Silva et al., 2018; von Sperber et al., 2017). Both techniques are time-consuming and costly but provide reliable information on MPs. There are a few alternative techniques available such as hyperspectral imaging which can visualize 0.5 to 5 μ m particles on the soil surface, and macroscopic dimensioned near-infrared spectroscopy combined with chemometrics which is rapid and does not require any chemical pretreatment (Paul et al., 2019; Shan et al., 2018). Another technique of thermal extraction desorption-gas chromatography-mass spectrometry (TED-GC-MS) can achieve accurate and specific quantification of PP, PE, PS, and PET (Dümichen et al., 2017).

Many parameters remain largely variable even after classifications by means of environmental factors, temporal and spatial variability patterns, reporting units, etc. It is difficult to characterize MPs in the organic matter-rich agricultural soils, and there are disparities to compare the data and

generate reproducible results owing to the use of different techniques. Therefore, standardization of methodological protocols is essential for effective comparison and monitoring.

5. Challenges and future directions

The pollution of MPs is worsening globally and the related hazards in agricultural soils need proper attention. This review describes the analytical techniques, ecological risks, and pollution characteristics of MPs in the soil environment. Scientific knowledge of MPs encompassing the sources, fate, environmental concentrations, analytical techniques, and ecological consequence are reviewed. There are noticeable knowledge gaps that demand more concerted efforts in future research, and the following challenges are of high priority:

- There is no standard protocol to isolate, quantify and characterize MPs from soil environment. It is vital to develop a precise, feasible, and efficient assay for multiple MPs identification and characterization. Future research should focus on devising a testing protocol that takes into account the variable environmental soil conditions and the heterogeneity of MPs. Depending on the origin, shape, size, and composition of MPs, it is necessary to standardize specific methods for sample collection, isolation, identification, and analysis of MPs in the organic matter-rich agricultural soils.
- There is only a limited size of database concerning the sources and fate of MPs in the soil environment and their interactions with microbes, food crops, and soil animals. We need to evaluate the distribution, transport, and degradation of MPs to holistically reveal their environmental effects and implications in the agricultural field. With time MPs are degraded partially by a range of physicochemical and microbiological drivers. It is crucial

to identify the individual roles of relevant natural and anthropogenic activities to elucidate the long-term fate of MPs in the soil ecosystem.

- Currently, the global and regional data inventory (types, concentration, composition, and types of MPs) for the pollution status of MPs in the agricultural systems and soil environment are very limited and require substantial expansion. Future research should be extended to the qualitative characterization and quantitative assessment of the MPs in different types of agricultural soils with different cropping systems under variable climates, as well as their interactions and transformations in the rhizosphere.
- MPs are recognized as emerging persistent pollutants that may transfer across different trophic levels along a food web. It is crucial to determine its potential cytotoxic effects on soil flora and animals/humans and evaluate the apparent transgenerational effects. It is also necessary to investigate both natural and engineered ecosystems to study the behavioral responses of plants, animals, and other microbial assemblages to widespread MPs pollution in the agricultural soils.
- MPs as vectors of a broad range of environmental pollutants may facilitate or inhibit the mobility and bioavailability of these environmentally persistent and potentially hazardous pollutants in the agricultural soils. Although this area has been extensively examined in the aquatic ecosystems, limited knowledge is available for different terrestrial ecosystems and needs to be properly addressed by future research studies.
- With the increasing recognition of the hazardous effects of MPs, behavioral change of consumers and plastics manufacturers would be necessary for attaining sustainable plastic waste management. Well-aligned initiatives, best management practices, more stringent policies, and joint efforts of citizens and government officials are urgently needed to reduce

illegal disposal of plastic wastes and improper use of plastic products in the agricultural industry for the sake of food safety and sustainable development.

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Figure.1: Generation and dispersion of microplastics in terrestrial environments. (adapted and modified from **Karbalaei et al., 2018**)

Figure.2: Compost is one of the major sources of MPs and NPs input to agricultural soils (picture taken in Australia by the authors).

Figure.3: Schematic representation of microplastics extraction from soil sample (adapted and modified from **He et al., 2018a**)

Figure.1:

Figure.2:

Figure.3:

Table.1: Representation of waste management statistics and plastic waste estimates released to freshwater (continental) and terrestrial environments [based on figures for European Union (EU)].

Table.2: Occurrence and characteristics of microplastics in terrestrial environments.

Table 3. Additives/chemicals used in manufacturing and processing of the plastics.

Table.4: Analytical techniques for Identification and characterization of MPs and its advantages and disadvantages.

Table.1:

Plastic (handling/disposal)	Plastic (million metric tons/year)	Reference
Plastic production (EU total, 2014)	59	(PlasticsEurope, 2015)
Plastic waste (EU total, 2014)	25.8	(PlasticsEurope, 2015)
Managed plastic waste (−2% mismanaged waste)	25.28	(Nizzetto et al., 2016b)
Mismanaged plastic waste (2% of plastic waste in the EU)	0.52	(Horton et al., 2017b; Jambeck et al., 2015)
Total mismanaged plastic waste outstanding in continental environments (EU)	0.47–0.91	(Horton et al., 2017b)
Landfill (EU total)	8	(PlasticsEurope, 2015)
Recycling (EU total)	7.6	(PlasticsEurope, 2015)
Energy recovery (EU total)	10.2	(PlasticsEurope, 2015)
Plastic in sewage sludge (EU total)	0.063–0.43	(Nizzetto et al., 2016b)
Ocean input (EU total)	0.04–0.11	(Jambeck et al., 2015)

Table.2:

Soil source/Soil type	Country and location	Abundance	Composition	Size range (mm)	Shape	Reference
Rice-fish coculture ecosystems	China (Shanghai)	10.3 ± 2.2 item kg ⁻¹	PE, PP	< 1	Mainly fibers	Lv et al., 2019
		78.00 ±				
Vegetable fields	China (Shanghai)	12.91 62.50 ± 12.97 item kg ⁻¹	PE, PP, Polyethersulfone (PES)	0.03-16	Fiber, film, fragment	Liu et al., 2018
Agricultural field	China (Loess plateau)	<0.54 mg kg ⁻¹	PE, PP	> 0.1	-	Zhang et al., 2018b
Tree planted soils	China (Yunnan)	7100-42,960 item kg ⁻¹	-	0.05-10	Mainly fibers	Zhang and Liu, (2018)

Beach soil	China (Hebei)	317 item/500 g (average)	-	1.56 ± 0.63	Fragments, granules, fibers and films	Zhou et al., 2016
Coastline soil	China (Shandong)	1.3- 14,712.5 item kg ⁻¹	PP, PE, PES, polyether urethane	60% in size of <1 mm	Foams, fibers, pellets, flakes, fragments, films and sponges	Zhou et al., 2018
Industrial soil	Australia (Sydney)	300-67,500 mg kg ⁻¹	PE, PS, PVC	-	-	Fuller and Gautam, (2016)
Floodplain soil	Switzerland	up to 55.5 mg kg ⁻¹ or 593 item kg ⁻¹	PE, PS, PVC	< 0.5	-	Scheurer and Bigalke, (2018)

Table.3:

Types of additives/chemicals	Example	Function	Reference
Lubricants	Molybdenum disulfide and graphite	Flexible plastic manufacturing used in squeeze bottles and fiber	(Biron, 2003)
Flame retardants	Decabromodiphenyl ether	Improve the safety index of cultured marble and cable coverings	(Strååt & Nilsson, 2018)
Antioxidants	Tris(2,4-di-tert-butylphenyl) phosphite, Pentaerythritol tetrakis (3,5-di-tert-butyl-4-hydroxyhydrocinnamate)	Deal with resistance against weathering and useful in plastic processing	(Hansen et al., 2013; Hahladakis et al., 2018)
Anti-statics	Glycerol monostearate, Indium tin oxide	Generate static electricity attraction (reduce dust collection)	(Gächter et al., 1993)

Foaming agents	Isocyanate, Chlorofluorocarbons	Useful in the production of building board, polystyrene cups and polyurethane carpet underlayment	(Gächter et al., 1993)
Antimicrobials	2,4-dichloro- 6-(3,5-dichloro- 2-hydroxyphenyl) sulfanylphenol	Production of wall coverings (Bithionol) and shower curtains; control formation of biofilm	(Gächter et al., 1993)
Plasticizers	Bis(2-ethylhexyl) phthalate	Utilized in gutters, wire insulation and flooring owing to slow decomposition from light	Bhunja et al., 2013; Sablani & Rahman, 2007)
Colorants	Sudan stain, Diarylide pigment	Useful in coloring plastic products	(Biron, 2003; Hahladakis et al., 2018)

Table.4:

Technique	Particle size range	Methodology	Advantages	Limitations	Reference
Spectroscopic					
μ-FTIR	Particles > 500 μm and smaller to 20 μm can be investigated by ATR-FTIR and microscopy coupled FTIR respectively.	Depending on the molecular structure and composition of the substance, samples are exposed to defined range of IR-radiation.	Emerging, quick, and reliable nondestructive method.	Only for IR active sample, difficult to analyze nontransparent particle and particles below 20 μm, pretreatment required expensive.	(Li et al., 2018; He et al., 2018a)
μ-Raman	Particles size > 1 μm but also works for 1 to 20 μm	The shift in Raman spectra can be measured, that provided substance specific spectra.	Analyzed particle size 1-20 μm. Nontransparent, dark particles can be analyzed.	Samples required refinements before analysis, time intensive procedure.	(Li et al., 2018; He et al., 2018a)

SEM	Even microscale particles can be analyzed	Generated electron interacts with the sample which eventually measures the secondary ions.	Generate high resolution images	Samples required coating, less informative.	(Bläsing & Amelung, 2018; Zhou et al., 2018; de Souza Machado et al., 2018a)
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Chromatographic

Pyrolysis GC-MS	Works well with particles size > 500 µm.	The GC column coupled to a quadrupole – MS. the generated spectra are identified comparing with available common plastic database.	It is quite sensitive, easier and reliable method which avoids possible background contamination	The plastics database is limited, single sample at a time with certain weight. single sample at a time with certain weight.	(Dümichen et al., 2017; Ivleva et al., 2017)
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Liquid Chromatography (LC)	Applied for large sample large sample size.	Samples are prepared in selective for analysis.	Selected polymers displayed better recovery.	Restricted to specific polymers (PE and PET), not recommended for environmental samples.	(Hintersteiner et al., 2015; Elert et al., 2017)
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Visual					
Microscopic Counting	Stereomicroscope	The particles are counted directly and identified.	Cost effective, faster and easier.,	It cannot determine the nature of the sample.	(He et al., 2018a)
Tagging	Microscale MPs	MPs are irradiated with blue light and adsorption of hydrophobic dye is done which renders them fluorescent.	It is a quick, inexpensive, and easier.	Impurities in the samples may leads to overestimation of MPs.	(Shim et al., 2016; He et al., 2018a)