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Cite This: J. of. Macromolecules and Material. Sci.2021, 1, 01-09

Sciforce

Journal of Macromolecules and Material Sciences

Journal homepage: www.sciforce.org

The Impact of Microplastics On The Soil: A Mini-Review On Sources, Pre-Treatment, And Characterization

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ARTICLE INFO

Articlehistory:
Received: 20210724
Received in revisedform: 20210814
Accepted: 20210814
Available online: 20210824

Keywords:
Microplastic Soil;
Characterization;
Identification;
Analytical Methods.

ABSTRACT

Currently, polymers have been the subject of several studies because of by-products known as microplastics (particles smaller than 5 mm). Due to the complexity of the heterogeneous soil matrix, it is difficult to study microplastics in the soil, especially considering differences in methods adopted for sampling, extraction, and quantification of the particles in various studies that analyze microplastics in soil samples.Thus, the article presents a review of studies on the analysis of microplastics in the terrestrial environment, aiming to identifythe advantages and disadvantages of methods for analyzing polymeric fragments. Theresults show substantial variation in the techniques proposed, including sieving, digestion, density separation, and filtration to extract fragments in samples. On the other hand, the methods usually adopted to identify and characterize polymers in soil refer to combinations to perform classification and spectroscopies, including Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, and scanning electron microscopy (SEM). In conclusion, a combination of methodologies for the characterization of polymers in soil samples seems to be more efficient for detecting and analyzing particles and overcome analytical challenges, thus providing more effective monitoring of microplastic soil contamination.

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Introduction

Plastics are one of the most consumed materials in the world due to theirgeneral properties of plastics, including easiness in processing, 'cost-benefit, versatility, low density, and flexibility, among others. Unfortunately, the global increase in the adoption of plastics led to increase in plastic waste generation. Approximately 6.3 thousand Mt of plastic waste has been generated since 1950, mostly discarded in landfills.

Considering the maintenance of current practices, around 12,000 Mt of plastic waste should be sent to landfills or discarded in the natural environment by 2050 [1]. Plastic waste represents a massive loss of valuable material and poses a significant risk to the environment and wildlife since plastic degradation takes hundreds of years [2].Considering the maintenance of current practices, around 12,000 Mt of plastic waste should be sent to landfills or discarded in the natural environment by 2050 [1]. Plastic waste represents a massive loss of valuable material and poses a significant risk to the environment and wildlife since plastic degradation takes hundreds of years [2].

Microplastics (MPs) are usually categorized according to origin. Primary MPs are already produced in micro-dimension [3], and secondary microplastics are plastics that have been degraded by the action of weather to micro-dimension,both with high impact on the environment[4,5].Microplastics (MPs) are usually categorized according to origin. Primary MPs are already produced in micro-dimension [3], and secondary microplastics are plastics that have been degraded by the action of weather to micro-dimension,both with high impact on the environment[4,5].

Thus, recent advances in the area show that MPs are widespread in diverse environments, being most studied in aquatic environments (71% of the articles surveyed),e.g., sea, lakes, and dams[6]. However, in terrestrial environments, its presence has been generally neglected[7], is identified in only 5% of the studies'found[6]. However, evidence points to estimates of 4 to 23 times greater presence of MPs in soil than inwater [8]. Furthermore, the ingestion of MPspotentiallyleaches toxic persistent organic pollutants (POPs) absorbed from the environment.Thus, recent advances in the area show that MPs are widespread in diverse environments, being most studied in aquatic environments (71% of the articles surveyed),e.g., sea, lakes, and dams[6]. However, in terrestrial environments, its presence has been generally neglected[7], is identified in only 5% of the studies' found[6]. However, evidence points to estimates of 4 to 23 times greater presence of MPs in soil than inwater [8]. Furthermore, the ingestion of MPspotentiallyleaches toxic persistent organic pollutants (POPs) absorbed from the environment.

Therefore, the abundance of microplastics in the oceans and soil is expected to continue increasingdue to the continuous production and discard of plasticsin the environment. In addition, the ocean warming process caused by climate changestends to increase animals'' metabolism and increase feeding rates, thus increasing exposure to microplastics[9].Therefore, the abundance of microplastics in the oceans and soil is expected to continue increasingdue to the continuous production and discard of plasticsin the environment. In addition, the ocean warming process caused by climate changestends to increase animals'' metabolism and increase feeding rates, thus increasing exposure to microplastics[9].

A major concern regarding MPs in the marine environment is the impacts on the feeding and reproduction of living organisms in aquatic habitats,destabilizing the ecosystem[10]. However, terrestrial plastic pollution may be more hazardous due to its impacts on soil, watersheds, rivers, and lakes, thus contributing to aquatic pollution and increasing the effects of greenhouse gas, which leads to accelerated climate changes[11,12].A major concern regarding MPs in the marine environment is the impacts on the feeding and reproduction of living organisms in aquatic habitats, destabilizing the ecosystem[10]. However, terrestrial plastic pollution may be more hazardous due to its impacts on soil, watersheds, rivers, and lakes, thus contributing to aquatic pollution and increasing the effects of greenhouse gas, which leads to accelerated climate changes[11,12].A major concern regarding MPs in the marine environment is the impacts on the feeding and reproduction of living organisms in aquatic habitats, destabilizing the ecosystem[10]. However, terrestrial plastic pollution may be more hazardous due to its impacts on soil, watersheds, rivers, and lakes, thus contributing to aquatic pollution and increasing the effects of greenhouse gas, which leads to accelerated climate changes [11,12].

The literature review by Qi *et al*. (2020) found microplastics in plantation, handling, and cultivation of fruits, and vegetables, reaching households through food consumption.Foods with higher contamination rates among fruits and vegetables were carrots, apples, pineapples, and cabbage[13]. In the study by Barboza *et al*. (2018), MPs and other types of synthetic products were found in foods and ingredients intended for human consumption (e.g., canned sardines, salt, beer, honey, and sugar) and water distributed in plastic bottles[14].

Based on this finding, researchers investigated the potential consequences of microplastics ingested or aspirated by humans, noting that human epithelial and brain cells showedcytotoxic effects related to oxidative stress, which reinforces speculations on further impacts caused by MPs contamination in human health [14].

Studies have shown that most residues originate from terrestrial anthropogenic activities[15–17]. According to theevidence, MPs found in the environment are primarily caused by anthropogenic activities, particularlyurbanization linked to population density, even though sea cycles, storms, and floods also contribute to their dispersion [8].Studies have shown that most residues originate from terrestrial anthropogenic activities[15–17]. According to theevidence, MPs found in the environment are primarily caused by anthropogenic activities, particularlyurbanization linked to population density, even though sea cycles, storms, and floods also contribute to their dispersion[8].

According to Ren *et al*. (2020), the influence of plastic fragments in the soil tends to reduce the microbial community's diversity and richness.In addition,it seriously impactsthe terrestrial biogeochemical cycles due to changesin soil nutrients (resources necessary for microorganisms). The process alters metabolic functions in the environment, e.g., circulation of carbon dioxide[18], such as in Figure 1.

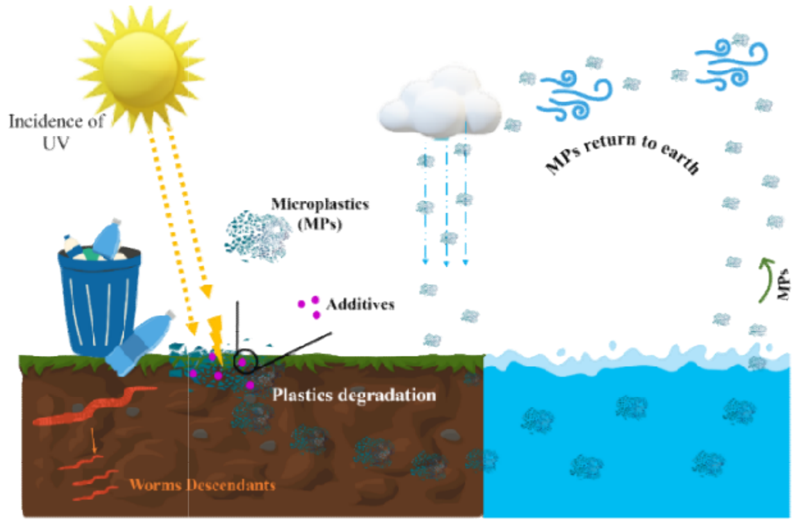

Figure 1-Carbon cycle in the presence of plastics in terrestrial and aquatic environments. Based on Dees *et al*., 2020.

Figure1 shows the microplastics dissemination, penetrating the soil and potentially releasing chemical products (environmental contaminants, additives, heavy metals, monomers, etc.) adsorbed on them to the surrounding environment[19]. The main substances usually released in the environment include bioavailable heavy metals, which may act as catalysts for undesirable reactions[20]. Thus, the adsorption/absorption properties ofplastic fragments favor the carriage of harmful substances that alter the physical properties of the soil. MPs can increase porosity, and change the aggregate structure, abruptly altering the microbial activity of the soil, for example[21].

Another concern regarding MPs refers to the actions of weather (rain, wind, atmospheric deposition, and ocean waves) in spreading to distant places, like the Arctic[22,23]. MPs were also found in 98% of wet and dry samples examined from remote protected areas in the USA[24] causing changes in living organisms' behavior and food chain interaction[6].

Furthermore, soils carry out carbon sequestration and promote biological heterogeneity, and substances that alter the fundamental properties of the soil affect the physical and biological environment, like structure, consistency, porosity, magnetic ores, carbonates, manganese, sulfides, among others. Thus, changes in alkalinity levels affect soil fertility and lead to a deficiency of several essential nutrients. The decomposition processes of organic matter in the soil are influenced by the presence of MPs due to changes in soil temperature, affectingthermal degradationthat would support the reproduction of microorganisms. In addition, humidity ensuresthe proliferation of bacteria and fungi and generates an appropriate environment for germinating microorganism spores. Finally, oxygen plays a key role in allowing cells to breathe, helping aerobic decomposers.In sum, plastic pollution generates by-products that become severe threats to the soil biota due to changes in the terrestrial habitat[25].Álvarez-Hernández *et al.* (2019) indicated that Polyethylene (PE) is the most common MP found in terrestrial systems. Other studies covered a more comprehensive array of polymers, finding significant plastic pollution in agricultural soils: PE - 62.50%, Polypropylene (PP) 52.50% and, to a lesser extent, Polyamide (PA) - with 32.50% [26–29]. Furthermore, it seems that agricultural coverings (mulching - usually made of PE) and application of sewage sludge comprises some of the main routes of entry of MPsinto the soil[30,31]. According to Bläsing and Amelung (2018), other access routes would be through landfills, flooding, bioturbation, and atmospheric deposition [32].

Yet, there are still considerable gaps in the processes and pathways of microplastics in soils. Considering the various protocols usually adopted for sampling, extraction, and analysis of plastic fragments in the soil, it is challenging to compare evidence from studies conducted on the characterization of soil pollution by MPs.After soil extraction, there are diverse techniques applicable for MPs analysis, including the main methods of polymer identification:Fourier transforms infrared spectroscopy (FTIR), Raman spectroscopy, and scanning electron microscopy (SEM).

Alternative analyzes performed to prove the existence of microplastics in soil samples include thermal desorption coupled with automated mass spectrometry for Pyrolysis-gas chromatography-mass spectrometry (pyrolysis GC-MS), which comprises a multifunctional tool for comprehensive characterization range of polymers and their degradation products[33].

There is a lack of studies synthesizing the main characteristics of studies on the identification, characterization, and estimation ofmicroplastics flows of soils, especially towards the methodological standardization that may support further analysis of its environmental impacts.Therefore, to allow an improved understanding of MPs in the soil, we propose to compile methodological characteristics of studies published within the last five years on soil microplastics.

1. Sampling, pre-treatment, and analysis

1.1. Sampling

The improper disposal of plastics generates degradation under environmental weather conditions, changing physical and chemical properties like crystallinity, sorption capacity, color, etc. The conversion into microplastics in the soil may cause further fragmentation and dissemination due to biotic and abioticfactors[34]. However, studies of characterization of soil plastic pollution are currently limited by the lack of adequate methods to quantify microplastics in soils[19]. In general, there is the absence of standard operating procedures to quantify microplastics in the environment, particularly in soil.

Soils are heterogeneous solid mixtures composed of minerals with a wide variety of particle size distributions and organic matter at various stages of decomposition[35]. The complexity inorganomineral interactions and the variability of soil media pose a challenge to soil sampling procedures for microplastic characterization, although it is recognized as an important emerging issue. Considering the complexity and the heterogeneity of soil components, the extraction and the separation of MPs may be complicated. Therefore, it is necessary to sample, measure, and quantify the amount of microplastics in the terrestrial environment at a wide range of spatial and temporal scales to determine the risk of adverse effects.

The selection of an adequate sampling method comprises an appropriate step of the process, involving considerations on the distribution of fragments in the field, their potential sources, and the site's geomorphology.Möller*et al.* (2020) recently indicated common sampling strategies and, in accordance with the ISO 18400-102 standard, the sampling depth must be defined byconsidering the soil profile and management practices. Sampling in agricultural fields,for example, is limited to 5 cm depth in most studies[29]; however, a Federal Ordinance on Soil Protection and Contaminated Sites in Germany imposes a minimum depth of 30cm for soil sampling[36].

Soil samples are usually collected using an auger in a predetermined area[37]. The variety of sampling ranges from randomly replicated samples[27,38]to selected land strips [29,38,39]and stratified random sampling[39]. About the volume of soil collected, there is wide variation in practices adopted by researchers in knowledge: some collect composite samples, whilst others carry out samples in a larger volume box, which are later reduced. The quartering method,for example, maybe performed according to the ASTM-C702 standard. The reduction increases the sampling efficiency and avoids disturbing the concentrations of MPs when achieved adequatelyin the field or the laboratory, thus preventing bias in measuring plastic concentrations[40]. The amount of samples collected fromeach location is dependent on the size of the area under analysis: some researchers collect only one sample[41] although most adopt composite samples[39].

A further difficulty relies on the choice of procedures for soil preparationto analysis:someresearchers determine 50 g of clean soil (without organic matter) for sample analysis[28,42], and others adopt 250g[36]. However, none of the studies mentions reasons,protocols, or standards that guided theadoption of the procedures described in the studies.

1.2. Soil characterization

Considering that MPs accumulate in the soil, they become part of the complex mixture with minerals and organic materials, making it difficult to remove particles for separation[35]. Thus, some authors believe that it is relevant to know in advance the soil characteristics,like pH, soil texture, and othersafter density separation when there is high organic matter in the soil, influencing sample preparation and other stages of analysis, since these elements may reduce the MPs recovery rate[27]. Other authors,like Zhang *et al.* (2018), attributed a higher rate of MPs recovery to intrinsic characteristics of sandy soilsthandifferent types of soil. However, other studies could not identify any reason for high MPs recovery rates in a given soil, considering the absence of differentiating characteristics in relation to other studies [39,43].

1.3. Sample's pre-treatment

The first step to extract MPs from the soil is to dry the samples in a greenhouse to eliminate moisture, hinderingthe separation of organic matter[7]. Studies like Thomas *et al*., 2020 used the recommendation of ISO 11464, which recommends drying the soil at 40ºC for 72 hours,whileother authors like Liu *et al.* (2018) placed samples 24 hours at a temperature of 70ºC. However, some authors urged caution in the process [43], considering that temperatures above 40ºC could change the structural and physical properties of MPs due to degradation, melting, and eventually glass transition, e.g., in polybutylene terephthalate (40°C) and polyamide (50-75°C). After the drying process, sieving is recommended since it removes excess organic matter and fractionates granulometrically, facilitating the removal of particles larger than 5 mm[7]. Then, considering that the organic matter has excess debris, one study reported the need to sift the samples to analyze MPs size-frequency distribution using several stacked sieves meshes with aperture sizes set to 2.0, 1.0, 0.5, and 0.25 mm[44]. However, there is no consensus in the studies analyzed during the review.

Following, solutions of sodium chloride (NaCl), sodium bromide (NaBr), sodium iodide (NaI), and zinc chloride ($ZnCl_2$) are used[41] to take advantage of the difference in densities of plastics and soil particles[35,36,43]for the elimination of organic matter, which interferes in the chemical analysis for the identification of MPs[45], ismost recommended the NaI[7,46]. However, the authors warn that NaI is expensive and relatively toxic to the biota;thus, cost-benefit and disposal procedures must be assessed before adopting NaI. Thus, several aqueous solutions with different densities isolate the fragments, depending on type and size[27,37].

In addition, some researchers adopted an additional step through combination with other extraction methods to increase the efficiency of organic matter removal, such as KOH[47] or Fenton Reagent[38]. Present in several publications, Fenton's reagent is composed of the oxidizer H_2O_2 and is a strong ferrous ion catalyst (Fe_2^+) used as a cleaner of complex environmental samples due to its effectiveness in removing organic composite materials[41,48]. There was a combination of flotation with NaCl and digestion in other studies, which is a widely used technique to eliminate organic matter from samples; however, the reagent or temperature can degrade the polymers partially or even totally[7].

Few studies have reported filtration, which may help retain MPs, depending on the pore size (ideal porosityreportedbetween 10µm and 20µm), although it is a slow process is[49]. In addition, some authors strongly recommend using the filter to remove particles that can contaminate the samples[22,50]. The filtersusually reported to perform filtration in studies, which present low interference with the identification of microparticles by the FTIR,are aluminum oxide and polycarbonate (PC)[43]. After filtration, the soil samples must be kept in the oven until dry, avoiding moisture interference in the analysis[7,51].

1.4. MPs characterization

Recently, studies reported the use of dyes (like Nile Red or Evans Blue) to allow the visual identification of MPs by contrast with the surrounding matrix[52,53] and the application of thecomplementary techniqueto avoid error rates in visual identification classification[35],like Fourier transform infrared (FTIR) spectroscopy (Figure 2).

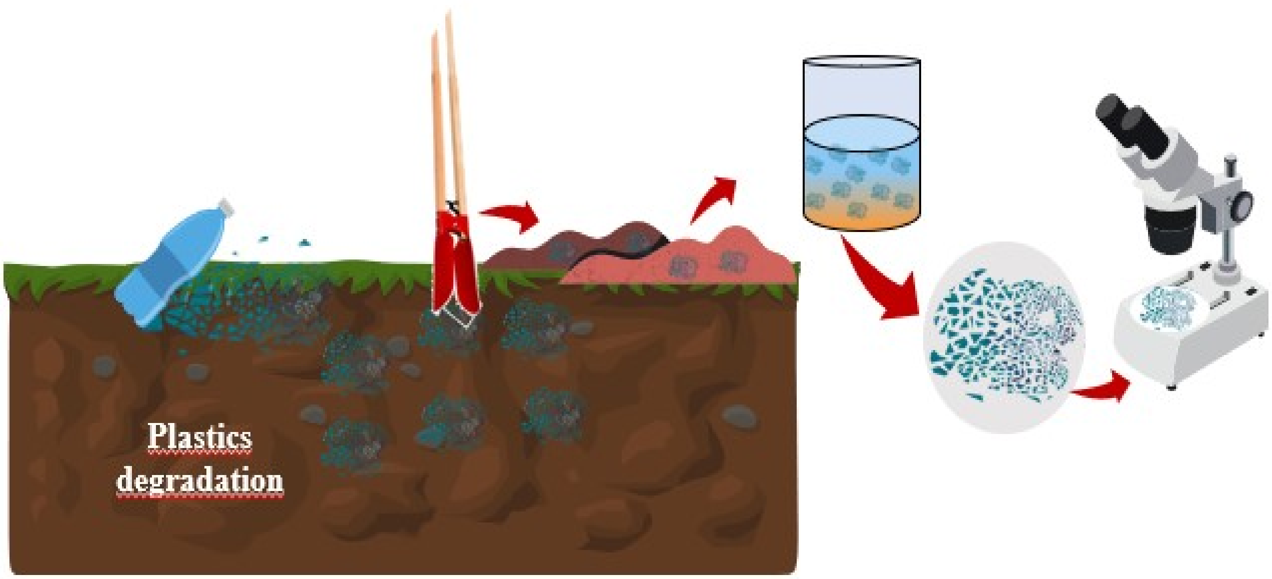

Figure 2 - Illustration showing methods used in previous studies on the detection and characterization of microplastics in soil and their impacts.

In general, the FTIR encompasses the technique usually adopted in the current MPs literature, confirming the identification, type, shape, and size of the fragments with a resolution of up to 20 µm[54]. FTIR spectroscopy qualitatively assesses MPs due to recognition of the spectra of the type of polymer in comparison to the spectra of known plastics and allows the identification of functional groups in the fragments[19], the occurrence of structural changes in MPs[55], and the presence of substances absorbed/adsorbed by the polymers that may have been released into the soil[56,57].

The detection of extra peaks witha low percentage of similarity aboutthe characteristic spectrum of a pure polymerin FTIR spectroscopy indicates the need for further investigation on the possibility of polymer degradation, supporting the identification of secondary origin fragments[7]. Furthermore, FTIR analysis is widely adopted because it does not destroy polymeric samples [58].

Raman spectroscopy is also usedfrequently, and the combination of the electron microscope and spectroscopic investigation (either Raman or FTIR) improves analytical results[59]. Raman spectroscopy coupled to microscopy may identify MPs below 1 µm.In addition,there is less interference of humidity in the analysis, surpassing the limits of the FTIR. However, it requires precise technical skills to achieve spectral images[60], and the high intense energy of the laser may destroy polymeric fragments[50]. The use of pigments and dyes introduces higher complexity in Raman analysis due to changes in the cations of the spectra in the technique, therefore, the use of Raman is not recommended in certain cases[7]. Another disadvantage of Raman is the Raman spectrum's obstruction due to additives used in plastics found in synthetic polymers[617]. Another disadvantage of Raman is the Raman spectrum's obstruction due to additives used in plastics found in synthetic polymers[7].Raman spectroscopy is also usedfrequently, and the combination of the electron microscope and spectroscopic investigation (either Raman or FTIR) improves analytical results[59]. Raman spectroscopy coupled to microscopy may identify MPs below 1 µm.In addition,there is less interference of humidity in the analysis, surpassing the limits of the FTIR. However, it requires precise technical skills to achieve spectral images[60], and the high intense energy of the laser may destroy polymeric fragments[50]. The use of pigments and dyes introduces higher complexity in Raman analysis due to changes in the cations of the spectra in the technique, therefore, the use of Raman is not recommended in certain cases. Another disadvantage of Raman is the Raman spectrum's obstruction due to additives used in plastics found in synthetic polymers [61].

According to Ruggero*et al*. (2020), an alternative technique to identify MPs would be using the hot needle test, which may be used directly in a global sample or among residues that have undergone previous treatment. The technique is based on the contact of a hot needle, handled with tweezers,with the fragments suspected of being microplastics. The needle's heat makes the plastic sticky, leaving a mark on its surface, while other particles will not present a reaction to the heat. However, theadoption of the method is highly questioned due to the absence of validation in matrices like the soil with biota diversity and the lack of tests on its reliability regarding the size of the MPs that could be detected with this technique[46].

Some researchers also use thermogravimetric analysis (TGA) to complement images acquired by FTIR or Raman, in addition to analyzing the thermal decomposition of the analyzed MPs. TGA analyzes the thermal stability of polymer samples and their fragments[44], being a technique valuable for detecting the degradation level of the particles and evaluating theprimary or secondary origin of the fragments[24]. Furthermore, considering that plastics degradation is a long process, their permanence in the environment makes them suitable “habitats” for microorganisms[62]. Thus,another method adopted for thermoanalysis of fragments would be chromatography, which can identify and quantifyseveral polymers,e.g., polyethylene (PE), polystyrene (PS), polypropylene (PP), and polyethylene terephthalate (PET), in organic sediments[63].

Photomicroscope, obtained by optical (OM), and scanning (SEM) microscopy provide high magnification images of microplastic surfaces,being useful to reveal the effects of degradations on the surfaces of microplastics since degradation by photo-oxidation occurs in the presence of light sources and air.The most common degradation process occurs through the appearance of microcracks arising from the splitting of polymer chains[55]. In addition, the analysis also helpsto determine the size of the fragments[64] and verifying the existence of microorganisms on the surface of the microplastics[65].

The SEM provides information on particle size, composition, and morphology, in addition to detecting patterns of degradation like fractures, grooves,etc.[66], based on monitoring of the MPs surface througha high-intensity electron beam, which generates high-resolution images and allows the identification of mechanical stress[59,67]. The technique has also been used to identify plastic additives of inorganic origin,like metals, since it is possible to verify the morphological characteristics of these microplastics[59,68]. In addition, optical and electron microscopy allows the identification of evidence particle morphology and size [69,70]. Nevertheless, although most researchers adopt the latest techniques, there are still studies being carried out with the use of an optical microscope and visual identification of MPs without equipment; however, errors or false positives may occur depending on the subjective attitude of the operator, the color and shape of the plastics, and the environmental matrix,considering the difficulty in differentiating MPs from the other materials within a global sample [46].

2. Conclusions

Currently, the research on the origin,characteristics, and impacts of microplastics is relatively recent. Thus, methods for extracting microplastics, especially fibers, from soil samples need further investigation. Furthermore, there is the absence of qualitative and quantitative methodssuitable for real-time monitoring to detectmicroplastics in effluent treatment plants. Techniques like FTIR are expensive, while lower-cost processes (like visual inspection) are time-consuming. Therefore, there is a need for research targeting the development of innovative,cost-effective qualitative and quantitative methods for the accurate determination of microplastics in the environment, especially soil pollution. Nowadays, methods require extensive pre-treatment methods to filter samples with moisture to facilitate the extraction of microplastics. The analytical methodsdescribed present requirements and limitations due to the complexity of heterogeneous soil matrices. The authors suggest combining methods to obtain comprehensive sampling, identification, characterization, and quantification of microplastics in different samples. On the other hand, the suggestion also highlights the disadvantages of some methods designed to encompass sample preparation until characterization, since they may generate divergent results and hinder comparison of evidence from different studies since there is no standard protocol for sample collection, treatment, or analysis. Different analytical methodologies will lead to discrepant results regarding properties, concentration, and other characteristics of MPs. The present article shows an inventory of the main methods for sample collection, identification, and quantification of MPs adopted in the literature recently published worldwide on plastic soil pollution.It is important to the point that the research of MPs in terrestrial environments is complex due to the heterogeneity of the soil samplesand the difficulties in removing organic matter, leading to the need foracombination of techniques for higher reliability in the characterization, according to the specific properties of the soil. There is high variability in the procedures for removal of the matrix to conserve the microplastics.Techniques are often adapted to try to ensure the efficient removal of organic materials. The use of vibrational spectroscopy (like FTIR) to confirm polymer characteristics enhances the reliability of the quantification process,whilst the use of Raman spectroscopy with fluorescent demarcation dyes should be avoideddue to potential bias in the analysis. Thus, there is urgent need for standardization of methodologies for investigation of MPs to ensure higher reliability of theresearch and provide consistency of comparisons of results in different environments.

ACKNOWLEDGMENTS

This research was funded by CNPq (#305819/2017-8, #306401/2013-4, and #420217/2016-9) and FAPESP (#2020/12208-9, and #2018/11277-7). The authors thank the UFABC, CAPES (Code 001), REVALORES Strategic Unit, CAPES (001), and Multiuser Central Facilities (CEM - UFABC).

Conflicts of 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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