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Investigating microplastic dynamics in soils: Orientation for sampling strategies and sample pre-processing

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Abstract

Studies on microplastics in soils is currently being established as a new research field. So far, mainly 'explorative studies' have been carried out to detect microplastics in different soil environments. To generate a deeper understanding of microplastics dynamics, 'systematic studies' are required. Such research must built on a targeted sampling strategy and considerate fieldwork and sample handling. From literature enquiry, a five-stage methodological workflow was deduced for studies on microplastics in soils. In the present review, the spatial representation of soils/soilscapes with microplastics in soils research is conceptually and practically assessed. We discuss judgmental, randomized, and metric soil sampling strategies. Then, we explain sample pre-processing and give a brief overview of methods for microplastics identification and quantification. We conclude that the establishment of the novel field of research 'microplastic dynamics in soils' requires more intensive consideration of soil sampling strategies. As soil is a complex medium and the soilcape is spatially heterogeneous, we highlight systematic sampling strategies as the best possible options for sophisticated research. However, no overall optimum methodology can be defined because the specific strategy must be in line with the particular research question. For all studies on microplastics in soils, practical improvement is needed to prevent contamination of soil samples with plastics during sampling and sample pre-processing.

KEYWORDS

field study, geospatial approach, pre-processing, soil sampling, spatial resolution

1 | AN EMERGING RESEARCH FIELD AND ITS CHALLENGES

Plastics are used in all areas of societal life. Due to the properties of plastic polymers, they are valued for diverse purposes (e.g., in industry, the economy, everyday life). Plastic production and processing are simple and cost-efficient. Hence, plastic production has increased rapidly

in the past decades (Andrady, 2017). Worldwide, 348 million tons of plastic are produced annually (PlasticsEurope, 2017). The major commonly produced polymers are shown in Table 1. In spite of increased recycling or reprocessing of plastic waste and declining landfill deposition, a large proportion of plastic waste is daily disposed into the environment (de Souza Machado, Kloas, Zarfl, Hempel, & Rillig, 2018; Karbalaeei, Hanachi, Walker, & Cole, 2018; PlasticsEurope, 2017).

Plastic particles with a size >5 mm are termed macroplastics, mesoplastics, or plastic litter. Particles of 0.01–5 mm are called microplastics. Particles <1 µm are termed nanoplastics (Hüffer et al., 2019;

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TABLE 1 Plastic polymers in the focus of past studies on microplastics in soils

Ranking by production ^a	Common plastic polymer types ^b	Corradini et al. (2019)	M. Liu et al. (2018)	Piehl et al. (2018)	G. S. Zhang and Liu(2018)	Scheurer and Bigalke (2018)	Huerta Lwanga et al. (2017)	Fuller and Gautam (2016)
1	PP (polypropylene)	Polymer type not reported	x	x		x		
2	PE (polyethylene)		x	x	Polymer type not reported	x	x	x
3	PVC (polyvinyl chloride)			(x)		x		x
4	PUR (polyurethane)							
5	PET (polyethylene terephthalate)							
6	PS (polystyrene)			x		x	x	x
7	ABS (acrylonitrile butadiene styrene)							
7	SAN (styrene acrylonitrile resin)							
8	PA (polyamide)					x		
9	PC (polycarbonates)							
10	PMMA (polymethyl methacrylate)			(x)				
11	POM (polymethylene)							
12	PES (polyethersulfones)		x					
13	SBR (styrene-butadiene)					x		
14	Latex					x		

Bold entries indicate common polymer types.

Note: (x) = detected only as macroplastic (>5 mm).

^aU28 + NO/CH plastic converter demand according to PlasticEurope (2018).

^bFrequently produced and consumed plastic polymer types according to PlasticEurope (2018).

Hüffer, Praetorius, Wagner, von der Kammer, & Hofmann, 2017; Mausra, Baker, Foster, & Arthur, 2015; S. Zhang et al., 2018). Despite various attempts at forming a definition, this terminology has been most widely adopted (Möller, Löder, & Laforsch, 2020; Rillig, Lehmann, de Souza Machado, & Yang, 2019; Silva et al., 2018; Stock, Kochleus, Baensch-Baltruschat, Brennholt, & Reifferscheid, 2019; B. Zhang et al., 2020).

Genetically, we distinguish between two types of microplastics: (a) primary microplastic manufactured at the <5 mm size range often used in cosmetic and cleaning products, and (b) secondary microplastic resulting from physical comminution and/or chemical degradation of originally larger plastic particles (Andrady, 2017; Barnes, Galgani, Thompson, & Barlaz, 2009; Napper & Thompson, 2019).

Environmental research into microplastics was first carried out in coastal waters (Carpenter, Harvey, Miklas, & Peck, 1972; Carpenter & Smith, 1972), later in the oceans worldwide (Karlsson et al., 2017; Martin, Lusher, Thompson, & Morley, 2017; Nuelle, Dekiff, Remy, & Fries, 2014; Phuong, Poirier, Lagarde, Kamari, & Zalouk-Vergnoux, 2018; Stock et al., 2019; Taylor, Gwinnett, Robinson, & Woodall, 2016; Wright, Thompson, & Galloway, 2013). A detailed database on microplastic abundance and behaviour in marine ecosystems, and the endangerment of aquatic organisms was established (Cole, Lindeque, Halsband, & Galloway, 2011; Martin et al., 2017;

Taylor et al., 2016; Wright et al., 2013). However, microplastics are not only present in the oceans. Because plastics are produced, processed, and used on land as man-made materials, microplastics are transported from land to sea. Rivers are the main transport corridors for microplastics as water moves from land to ocean (Alimi, Farner Budarz, Hernandez, & Tufenkji, 2018; Blettler, Ulla, Rabuffetti, & Garello, 2017; H. Liu et al., 2019; Siegfried, Koelmans, Besseling, & Kroeze, 2017; Xiong, Wu, Elser, Mei, & Hao, 2018). Therefore, not only marine/aquatic but also semi-terrestrial ecosystems are affected by microplastics (H. Liu et al., 2019). In addition to translocation and transport by water, microplastics also appear to be transportable by wind (Abbasi et al., 2017; Rezaei, Riksen, Sirjani, Sameni, & Geissen, 2019). These findings enable us to hypothesize that microplastics can spread farther in the landscape than previously assumed. Especially, since they are preserved for a long time in various environmental media (Chamas et al., 2020). After almost five decades of research, the environmental effects of microplastics for terrestrial ecosystems are now increasingly investigated (Engdahl, 2018; Rillig et al., 2019; Rillig, Ingraffia, & de Souza Machado, 2017; Rillig, Ziersch, & Hempel, 2017; Selonon et al., 2020; Verla, Enyoh, Verla, & Nwarnorh, 2019; Yu et al., 2019).

Surprisingly, it is a relatively novel finding that plastic occurs or is deposited in soils (Huerta Lwanga et al., 2017). Research into

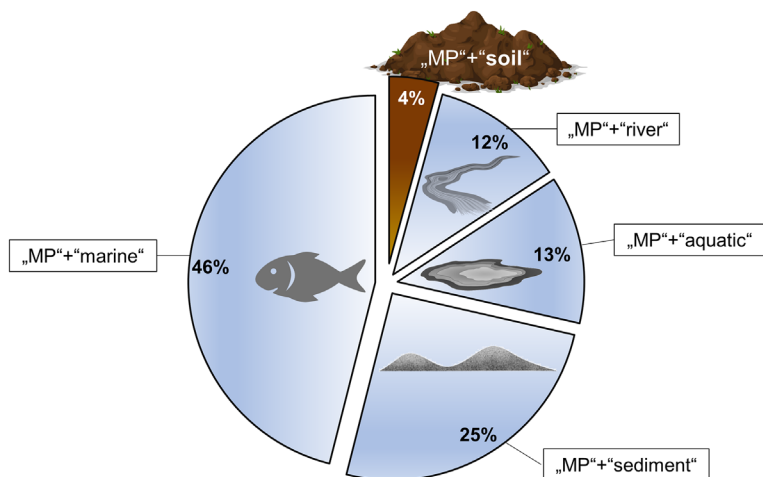


FIGURE 1 Results of a quantitative literature enquiry on microplastics-related publications on the Web of Science between 2014 and 03/2020. Specification and percentages of microplastic-related studies according to different search terms and combinations in the titles (MP = microplastic; $n = 639$) [Colour figure can be viewed at wileyonlinelibrary.com]

microplastics in soils is young and studies are still scarce. A quantitative review of the recent publications on microplastics reveals a strong increase in articles since 2014. However, only 4% of the papers are on microplastics in soils (Figure 1). These are largely 'explorative studies' seeking microplastics in certain soil environments, with a focus on microplastics occurrence and abundance. Studies have shown that microplastics occur in agricultural and strongly anthropogenically influenced soils, as well as in floodplain soils (Corradini et al., 2019; Huerta Lwanga et al., 2017; Piehl et al., 2018; Scheurer & Bigalke, 2018; G. S. Zhang & Liu, 2018). For topsoils under agricultural use, microplastic contents between 0.34 and 42.960 microplastic particles per kg of soil (mpp/kg) were reported (Piehl et al., 2018; G. S. Zhang & Liu, 2018). With regard to polymer types, studies were only able to identify some of the commonly produced plastics, because methods for the analysis of microplastics in soils are in their infancy (Table 1; M. Liu et al., 2018; Piehl et al., 2018; Scheurer & Bigalke, 2018).

Methodological advances are needed to evaluate the presently unknown environmental effects of microplastics in soils. Impacts on soil organisms are possible, including nanoplastics-uptake by plants resulting in entry into the food chain (Huerta Lwanga et al., 2017; Rillig et al., 2019). To evaluate and restrict negative impacts of microplastics in soils, a better understanding of the microplastic-related processes is required (e.g., transport routes and vectors). This can hardly be achieved by 'explorative studies' but requires 'systematic studies' focusing on microplastics' integration in and interaction with their spatial surroundings (i.e., landscape/soilscape). To conduct such studies, we still need to develop methodological foundations for precise and internationally comparable sampling, microplastic detection, and quantification.

Several authors have already dealt with microplastics in soils through reviews (Table 2). However, these publications largely focused on background information on environmental microplastics pollution or on the procedures of microplastics identification/quantification. By contrast, the establishment of adequate sampling strategies, soil sampling, and sample pre-processing has largely been neglected. These aspects are different for soil-related studies than for

studies on microplastics in waters. Furthermore, they are crucial if one wants to conduct systematic research on microplastics dynamics in soils. Hence, the present review aims to make three contributions: (a) to differentiate conceptually between explorative and systematic studies to enable the establishment of research on microplastic dynamics in soils; (b) to elaborate on strategies for creating adequate spatial representation in the empirical designs of studies on microplastics in soils; and (c) to critically discuss related sample handling and pre-processing.

From our literature enquiry, a five-stage workflow was deduced for studies on microplastics in soils, which is reflected in the present review's structure (Figure 2). With regard to the focus of the other topic-related reviews, we deal in particular with the so far underrepresented Stages 1–3 of the Workflow. Still, a short overview of analytical and quantification procedures is given, in combination with a reference list, which might lead the interested readers to further information.

2 | A GEOSPATIAL APPROACH TO MICROPLASTIC DYNAMICS IN SOILS

To understand microplastic dynamics in soils from a system perspective, we must consider the spatial contexts of microplastics in soils. First of all, this requires developing a suitable strategy for study site selection and the sampling procedure, in line with the respective research question. Such spatial considerations are significant for investigating possible displacement, transport routes, or environmental risks of microplastics in soils.

The dynamics of microplastics in soils theoretically encompass different interdependent process types (e.g., physical translocation by soil water, chemical reactions with the soil matrix, biochemical processes during mineralization). Due to their complexity, microplastic dynamics are not well understood. Thus, microplastic dispersion and potential negative effects cannot be prevented or limited effectively. Presently, the dynamics of microplastics in soils are also too technically demanding to investigate under field conditions.

TABLE 2 Text shares informing about the methodological stages distinguished in the present paper as represented in reviews on microplastics in soils (December 2017 until March 2020)

Review	Stage 1 Developing a sampling strategy	Stage 2 Sampling and sample handling	Stage 3 Sample pre-processing	Stage 4 Sample matrix separation	Stage 5 MP identification/ quantification	Background on environmental MP pollution
Möller et al. (2020)	3%	10%	1%	46%	27%	13%
Ruggero, Gori, and Lubello (2020)	0%	0%	0%	41%	59%	0%
B. Zhang et al. (2020)	0%	0%	1%	11%	13%	75%
Pinto da Costa, Paço, Santos, Duarte, and Rocha-Santos (2019)	0%	8%	0%	9%	24%	59%
Qi, Jones, Li, Liu, and Yan (2020)	0%	2%	0%	14%	2%	82%
J. Wang et al. (2019)	0%	0%	0%	0%	0%	100%
W. Wang et al. (2020)	0%	0%	0%	18%	15%	67%
Xu et al. (2019)	0%	0%	0%	1%	2%	97%
Bläsing and Amelung (2018)	0%	0%	2%	13%	8%	77%
He et al. (2018)	0%	1%	1%	17%	12%	69%
Silva et al. (2018)	0%	5% ^a	0%	5%	67%	23%
De Souza Machado et al. (2018)	0%	0%	0%	5%	8%	87%
Total	0.5%	2%	0.5%	15%	20%	62%

Note: Calculated on the basis of word count of thematic text (excluding titles, abstract, introduction, conclusion, figures, and tables).

Abbreviation: MP, microplastics.

^aConsidering sampling of water and sediments.

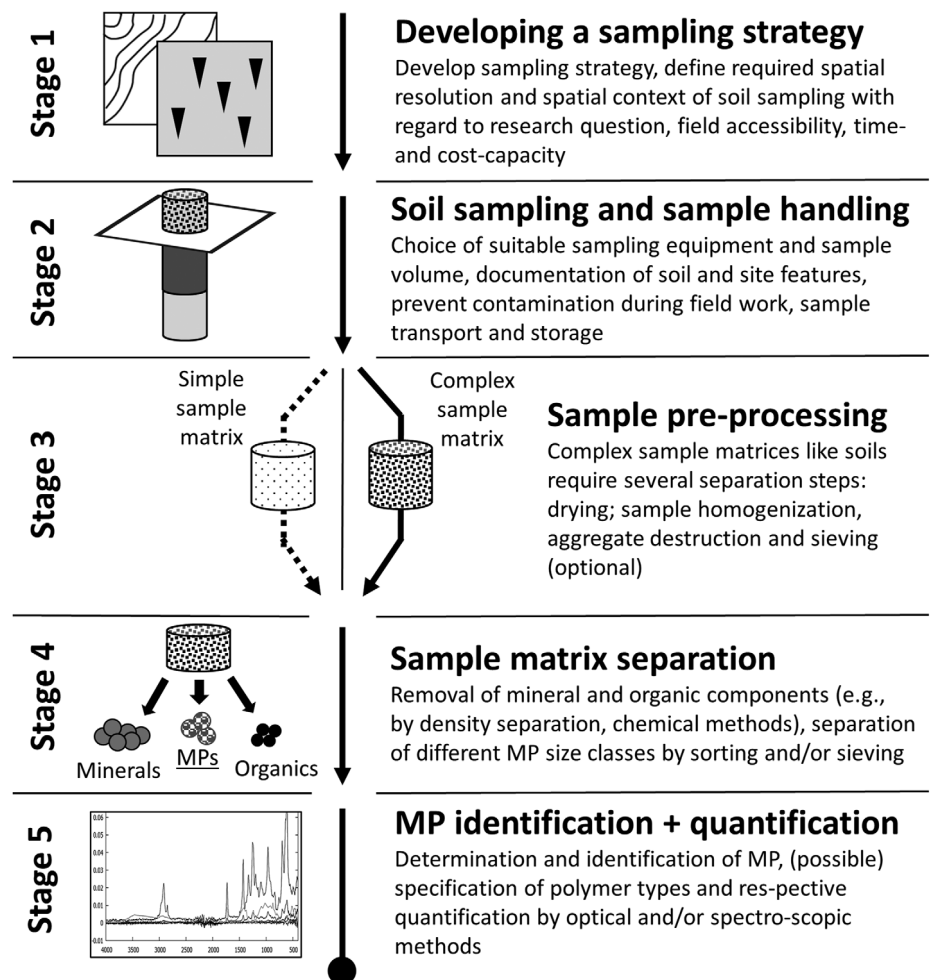


FIGURE 2 Five-stage methodological workflow for studies on microplastics dynamics in soils, as derived from literature enquiry (MP = microplastics). (Stages 1–3 are detailed further in the respective sections of the present paper. For Stages 4 and 5, an overview is given)

Hence, it is a challenge for microplastic research to generate a process for understanding microplastic dynamics in soils. As a possible solution, we propose a geospatial approach to microplastic dynamics in soils. As microplastic dynamics lead to a certain spatial distribution of microplastics in soil profiles and soilscapes, we can study this spatial distribution in the field and deduce on the processes that formed this distribution. This was proposed by Weihrauch (2019) to investigate soil phosphorus dynamics. In the present paper, we transfer this 'geospatial approach' to microplastic research.

2.1 | Developing a sampling strategy

A systematic investigation of the spatial microplastic distribution in soils requires an adequate spatial resolution. Such is achieved when the soil samples sufficiently represent the investigated two- or three-

dimensional spatial unit (e.g., a surface area or a soil profile). Spatial resolution encompasses three aspects (Figure 3a): (a) the positioning of sampling sites; (b) the number of soil samples; and (c) the spatial distribution of the samples over the investigated area/volume they shall represent.

(1) Sampling sites might be positioned in the landscape according to (a) subjective interpretation (judgmental sampling), (b) spatial randomization, or (c) metric criteria. (a) If sampling sites are chosen on the basis of interpretation (e.g., background knowledge, visual evaluation of the landscape), they often occupy particular locations related to specific research questions or hypotheses (e.g., depressions, roadside areas as potential zones of microplastic accumulation; Möller et al., 2020; Wells, 2010). The correctness of the results generated at these locations thus strongly depends on the correctness of the underlying hypotheses plus the validity of the spatial interpretation. Due to the complexity of the soilscape, the latter might introduce significant bias into interpretative soil sampling. Moreover, on the basis

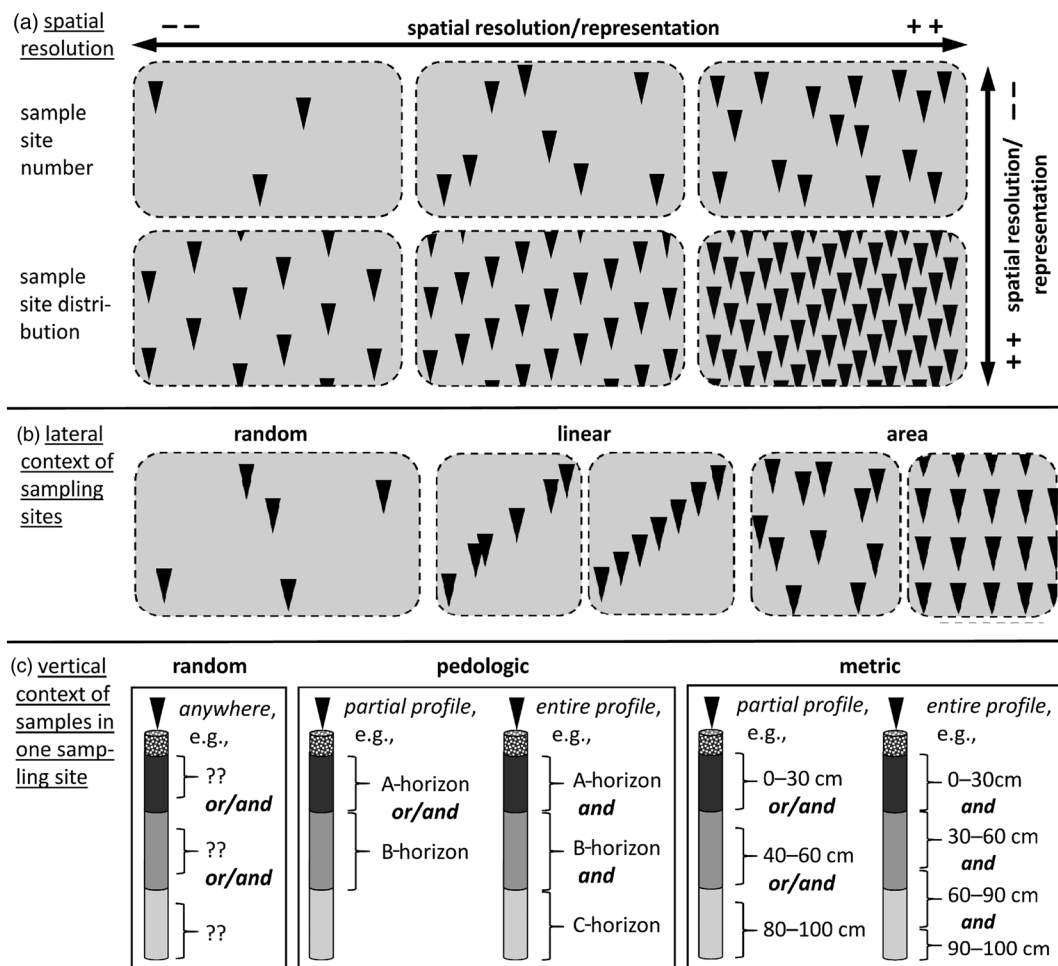


FIGURE 3 Aspects to be considered for developing a suitable sampling strategy for studies on microplastics dynamics in soils. (a) Soil sampling-based spatial resolution/representation of study areas as a function of sample site number and sample site distribution (triangles mark sample sites). (b) Possible lateral contexts of sampling sites. (c) Possible vertical contexts of samples within one sampling site. Vertical sampling resolution significantly depends on whether one or several samples are taken per sampling site. (Here only shown for mixed sampling from a larger vertical soil section combined into one sample; e.g., 0–20 cm). Alternatively, local samples can be extracted, which relate to a specific soil depth; e.g., 25 cm)

of spatially specific hypotheses, one might probably only be able to support or reject the assumptions but not to make systematic unexpected findings.

(b) Randomized sampling means the distribution of several sampling sites within a defined (i.e., limited) area under the premise that all sites have equal opportunity to be selected and that they are selected independently from each other (Möller et al., 2020; Wells, 2010). The positions of the sampling sites depend on a chosen area (mostly on the basis of interpretation or landscape evaluation, e.g., land use) but not on site or soil features of the concrete sites. The respective study area is thus treated as homogeneous with regard to soil and site features. This is conceptually critical especially because microplastics distribution is unlikely to be homogeneous (Möller et al., 2020). Anyways, this form of spatial generalization might be sufficient for studies, which aim at results representative for certain areas or landscape sections (e.g., comparison of microplastic pollution of two agricultural fields). It might also be plausible when certain statistical tests are planned (e.g., correlation analyses) as the resulting statistical sample will consist of independent data (Wells, 2010). However, randomized sampling is rather inadequate for studies on microplastic dynamics in soils as it ignores the highly relevant landscape and soilscape particularities.

(c) Metric sampling means the positioning of sampling sites on the basis of distances. This type of sampling also ignores particular site and landscape features. It might be useful to generate a good representation of an area or landscape section unbiased by interpretation or subjective landscape evaluation. By contrast to randomized sampling, it is also favourable for the comparison of different areas or landscape sections unbiased by divergent metric dimensions. It is thus favourable for studying and comparing gradients or spatial patterns (e.g., of increasing microplastic accumulation, microplastic hotspots). However, metric sampling might lead to the integration of uninteresting sites. Moreover, the resulting statistical sample would not consist of independent data. Thus, certain statistical procedures would not be available for data evaluation (Wells, 2010). In consequence, it strongly depends on the research question and the desired form of data evaluation which type of empirical design should be chosen for positioning the sampling sites.

(2) Another important aspect is the number of soil samples. Generally, larger sample numbers lead to higher spatial resolution (i.e., better spatial representation). The best possible spatial representation is achieved when all sampling sites are at an equal distance from each other, that is, when the not investigated spaces in between are smallest. Hence, a sampling strategy must be designed not only just according to the research question but also according to the size of the investigated spatial unit. One option to achieve adequate spatial representation of a particular study area could be the prior calculation of the sample number required for the specific research question and the planned statistical tests on the basis of pilot-sampling and geostatistical analyses (Li, 2019; Li et al., 2020). However, to date, such strategies have not been transferred to research on microplastic dynamics in soils, probably because analytical methods are still very costly. Thus, logistical aspects (e.g., costs, site accessibility) also have

to be considered as they would, in most cases, probably decrease spatial resolution.

The first studies on microplastics in soils (Fuller & Gautam, 2016; Huerta Lwanga et al., 2017) did not report the spatial context of the investigated soils. The more recent studies document the spatial sample contexts but not systematically (Corradini et al., 2019; Pielh et al., 2018; Scheurer & Bigalke, 2018; G. S. Zhang & Liu, 2018; Table 2). Hence, as a reader, it is difficult to evaluate if and how well the examined soils represent the respective study areas. Furthermore, it is evident from publications on microplastics in soils that often relatively small sample numbers are processed because the respective analyses are rather cumbersome (see following chapters). For instance, Scheurer and Bigalke (2018) used 87 samples to represent the whole floodplain soils in Switzerland. By contrast, Corradini et al. (2019) processed 90 soil samples to represent an area of 10 km². Hence, spatial resolution is different in studies on microplastics in soils and requires reflection in the present review.

(3) To develop a suitable sampling strategy, two dimensions of spatial resolution should be considered, the lateral (i.e., soilscape) and the vertical (i.e., soil profile; Weihrauch, 2019). Soils result from and are shaped by pedogenic and environmental processes, which do not just affect one location (i.e., one spot in coordinate space) but larger spatial areas (e.g., landscape sections). Hence, soils are no isolated phenomena but are parts of soilscapes and must be understood in their landscape context (e.g., slope position). This can hardly be achieved, when one single soil is investigated. For instance, the effect of erosion on soil formation cannot be elucidated from one soil on the topslope alone. Several soil profiles along the slope would be required that have a lateral context with each other.

Weihrauch (2019) proposes three options for the lateral context of soil sampling sites: (a) no lateral context (i.e., samples are taken randomly and not interpreted in a genetic context with each other), (b) a linear context (e.g., in transects/catenae), and (c) a two-dimensional context (e.g., mapping of a surface area; Figure 3b). Of the seven published field studies on microplastics in soils, five are based on random sampling, one on transects, and one on area mapping (Table 3). A trend shows for researchers to favour sampling without lateral context. This might suffice for explorative studies. However, systematic studies should rather be based on a linear or two-dimensional lateral sampling site context (according to the research question).

To depict vertically oriented processes (e.g., related to soil water movement or soil horizons), sampling sites need to be investigated by more than just one sample per site. The vertical resolution increases with the vertical sample number per site. The first study on microplastics in soils did not report the vertical sampling context (Fuller & Gautam, 2016). From the other studies, three are based on one sample and three studies are based on two samples per sampling site (Table 2). Hence, the vertical representation of soil profiles has generally been rather poor so far.

There are three options for the vertical context of soil samples within one site (Figure 3c). (a) Samples can be taken randomly, without regard of their depth or pedogenic background (e.g., no attribution to a certain soil horizon). (b) Samples can be taken according to

TABLE 3 Overview of the seven studies on microplastics in soils: sampling site properties and soil sampling methods

Investigated soils	Land use	Sampling context lateral / vertical	Spatial resolution of sampling	Sampling tools	Sample integrity	Sampling depth (volume)	Sample storage	Reference
Agricultural soils (Entic Hapoxerolls)	Cropland, treated with sludge over the last 10 years	Random /metric	Three randomly placed sampling sites, three samples on each field (30 fields in 10 km ² area)	Metallic auger	Disturbed (i.e., not in initial stratification)	0–25 cm (Not reported)	PP bags, PET jars	Corradini et al. (2019)
Agricultural soils (soil type not specified)	Suburbs and country -sides with vegetable cropland	Random/metric	20 sampling sites, three samples at 0.5 m ² large randomly placed sample plots	Not reported	Disturbed	0–3 and 3–6 cm (1 kg)	Aluminum box	M. Liu et al. (2018)
Agricultural soils (Entisols, Vertisols)	Cropland (barley and regularly ploughed, without agricultural plastics)	Linear /metric	14 sample plots (32 × 32 cm) located on two transects in the center area of farmland	Metal spatula	Disturbed	0–5 cm (5,120 cm ²)	PE barrels	Piehl et al. (2018)
Agricultural soils (Nitisols, Gleysols)	Cropland with plasticgreenhouse vegetable production	Random/metric	Two study sites, six randomly placed samples within five 150 m ² large plots at each sampling site	Metal spatula	Disturbed	0–5, 5–10 cm (Not reported)	Not reported	G. S. Zhang and Liu (2018)
Floodplain soils (soil type not specified)	River floodplains (grassland and wetlands)	Area/metric	29 Study sites, three mixed samples from three river-parallel transects (length: 16 m, distance: 1 m) at each site	Steel tools	Disturbed	0–5 cm (320 cm ³)	Aluminum box	Scheurer and Bigalke (2018)
Garden soils (soil type not specified)	Home gardens	Random/metric	Not reported	Not reported	Not reported	0–10, 10–20 cm (50 g)	Not reported	Huerta Lwanga et al. (2017)
Municipal soils (Technosols)	Waste facility and industry surroundings	Not reported	Not reported	Not reported	Not reported	Not reported (Not reported)	Not reported	Fuller and Gautam (2016)

pedogenic characteristics (e.g., soil horizons), either from selected soil sections or from the entire profile (i.e., all soil horizons). (c) Samples can be taken from defined metric depths or depth sections, either for some selected depth sections or for all sections of a profile.

No study on microplastics in soils applied a pedogenic vertical sampling strategy (Table 2). Hence, questions regarding soil stratification and effects on vertical microplastic distribution could not be evaluated in past research. The six studies, which report their vertical sampling context, took soil samples according to soil depth. Two studies focused on the upper topsoil (0–5 cm), one on the plough layer (0–25 cm). Three studies took samples from two depth sections (0–3, 3–6 cm; 0–5, 5–10 cm; 0–10, 10–20 cm). These authors do not explain the rationale behind their choice of sampling depths.

For the few studies on microplastics in soils, a clear trend shows for favoring investigations of topsoils. Topsoils have specific features and represent a soil profile neither quantitatively nor qualitatively adequately (Weihrauch, 2019). Topsoils are particularly critical to study due to a multitude of land use-based alterations (e.g., on agricultural fields), which might distort data comparison between sites, depths, or even across a plot (Li, Dd, Mendoza, & Heine, 2010). Hence, results generated from topsoil studies are specific and do not draw a holistic picture of microplastics distribution and dynamics in soils. For instance, a vertical translocation of microplastics (e.g., to the groundwater) can presently not be excluded as there are no studies yet that examine subsoil microplastic pollution. Hence, even for explorative studies, a topsoil focus does not seem favorable in all cases.

2.2 | Soil sampling and sample handling

For understanding soils in the landscape/soilscape context, a systematic documentation is required for: (a) sampling site features (e.g., relief, vegetation, land use history) and (b) specific soil features (e.g., soil type, horizons, respective standard features; Möller et al., 2020). In the examined studies on microplastics in soils, this information is not reported and probably was not obtained during sampling. Hence, no information on microplastics dynamics can be deduced from the studies' results, only the occurrence and abundance of microplastics.

Soil and site feature documentation is, for example, explained and standardized in the "Guidelines for soil description" (FAO, 2006) and "World reference base for soil resources" (WRB) for the international context (IUSS Working Group, 2015). Conventional soil sampling (e.g., for standard parameter analyses) is largely standardized by national standards, environmental laws, and administrative proceedings. However, it is known from marine research that artificial sample contamination with plastics during sampling, sample transport, and storage is a problem (Cole et al., 2011; Fischer, Paglialonga, Czech, & Tamminga, 2016; Horton, Svendsen, Williams, Spurgeon, & Lahive, 2017). Thus, artificial plastic contamination must be considered and excluded for soil sampling in microplastics research.

In all previous studies, topsoil samples were taken using steel sampling equipment (Table 3). All authors document the equipment used, as it is clear that plastic equipment should not be applied to avoid contaminations. Because samples were taken up to a maximum depth of only 25 cm, no drilling methods were necessary and samples could be taken out of a shallow pit or directly with a spade.

Furthermore, the type of soil samples must be considered. Soil can be sampled locally, that is, at a defined position in three-dimensional coordinate space. The respective results then relate to a certain geographical location, possibly even to a certain soil depth at this location. Alternatively, composite samples could be created by mixing soil material from several sampling sites (Möller et al., 2020). It is only plausible to mix samples from sites relatively close to each other, which are characterized by comparable site and soil factors. The respective results are regionalized and inform about a certain area in the soilscape. Which type of soil samples should be chosen strongly depends on the research question. Localized samples are suitable for investigating spatial patterns and dynamics of microplastics in soils, where spatial heterogeneity is informative. Instead, composite samples are plausible for studies where representative regional information is wanted (i.e., without small-scale spatial heterogeneity), for example, for research related to land use practices or for studies based on experimental designs with plotting. Hence, composite sampling was often used in explorative studies on microplastics in soils (Corradini et al., 2019; M. Liu et al., 2018; Scheurer & Bigalke, 2018; G. S. Zhang & Liu, 2018). Currently, no recommendation can be given regarding how many samples should be combined into one composite sample (Möller et al., 2020).

The type of soil samples taken relates to the resulting mass or volume of soil material available for further analysis. In the field studies, different sample amounts are documented. The documented sample masses reach from 50 g (Huerta Lwanga et al., 2017) to 1,000 g (M. Liu et al., 2018). The sample volumes reach from 320 cm³ (Scheurer & Bigalke, 2018) to 5,120 cm³ (Piehl et al., 2018) of moist unprepared soil. The relatively large amount of sample material is logistically needed due to the further analysis steps, especially with respect to the separation of microplastics from the soil matrix (Figure 2). Probably, larger amounts of soil material were so far also processed, because it has originally not been known if there were any microplastics in the investigated soils and how they were spatially distributed within the material. Hence, large samples could enlarge the probability of finding microplastic particles at all or in significant number.

If large sample quantities are required, these can only be gathered easily for topsoils (as they are logistically convenient to reach). To get samples from deeper soil sections, a more intensive interference with the soil is necessary (e.g., drilling). In microplastic studies, drilling methods have already been used for sediment sampling but not in studies with a soil focus (Ballent, Corcoran, Helm, & Longstaffe, 2016). Many drilling devices (e.g., augers of Pürckhauer type) only extract small soil quantities. Hence, to result in sufficiently large soil samples, several drilling cores would have to be gathered adjacent to each other and the respective samples would have to be mixed (e.g., the B-horizon material from adjoining drill cores).

Alternatively, pile-driving probes with a larger diameter could be used, which would make sampling more time-consuming and cost-intensive. It is recommended to pay attention to the contamination by plastic components (e.g., plastic caps on pile-driving probes, splinters of plastic hammers) when using drilling equipment. Many of these components can be removed before sampling. Certain textiles might also pose a risk of contamination (e.g., clothing fibers). Contamination can be avoided by reducing contact (quick storage after sampling) or by wearing cotton clothes.

Plastic particles may not just detach from our equipment during sampling but also during transport, and might artificially enrich our samples with external plastics. This means that no plastic bags or other plastic materials should be used during sampling and further processing. Especially, PE bags are often applied (Corradini et al., 2019; Piehl et al., 2018). However, if plastic bags are used for soil sampling in microplastic studies, this should only be done if no determination of materials similar to the bags' plastic type is planned and the possible contamination by the bags (e.g., through abrasion) is comprehensively controlled.

Alternatives to PE bags can be metal cases or buckets made of aluminum and glass jars. When plastic jars are used for sample transport and storage, they should be handled carefully and with low abrasion to minimize contamination. The use of metal vessels can be critical when the examination of microplastics is combined with the examination of other pollutants, like heavy metals. An alternative could be the use of biodegradable plastic bags, as long as the polymer types of these bags are known and are not the study's focus.

Finally, the samples have to be stored until the next methodological stage or for longer time. Samples should always be stored in closed containers to prevent contamination by the ambient air (e.g., dusts). If the samples should be stored in plastic containers such as PE bags or PET jars, they should be stored as dry, cool, and dark as possible to prevent potential degradation of the plastics (Napper & Thompson, 2019). In the case of biodegradable bags, it must be examined whether long-term storage is possible. In general, the polymer type of all plastic containers used should be known, the samples should be handled carefully and blank samples should be used as a control to verify any contaminations.

2.3 | Sample pre-processing

A main challenge in the analysis of microplastics from environmental media is the separation of microplastics from their respective medium (e.g., water, soil material). The analysis of water samples can usually be carried out by sieving and filtration methods. For soils and sediments, the separation of microplastics from other matter is required. Various methodological approaches for the analysis of marine, aquatic, and limnic sediments were recently transferred to extract microplastics from soil samples (Bläsing & Amelung, 2018; He et al., 2018; M. Liu et al., 2018). However, it should be considered if a simple (e.g., beach sands) or a complex sample matrix (e.g., soil material) is at hand (Figure 2). As a result of pedogenesis, the biogeochemical

properties of soil samples differ from, for example, beach sediments and form a more heterogeneous sample matrix comprised of several different components (e.g., mineral, organic, and microplastic particles).

In soil science, samples are usually dried before analysis because the results (e.g., heavy metal contents) are mostly reported in relation to soil weight (e.g., in SI of soil). For many questions, it is plausible to use the soil's dry weight. The weight of moist soil is largely influenced by weather conditions (e.g., precipitation). Thus, it gives different results with regard to the timing of soil sampling. Such results would be relative: For instance, element concentrations would appear higher in dry times (because it is related to lower dry weight) than in moist times (because it is related to higher moist weight). To come to absolute (i.e., generalizable) results, the soil moisture is thus eliminated by drying. This is done by air-drying at room temperature or in drying furnaces at temperatures between 50 and 70°C (Corradini et al., 2019; Piehl et al., 2018; Pinto da Costa et al., 2019). The application of drying furnaces significantly accelerates the drying process. However, excessively high temperatures can negatively influence plastics. Polymer melting temperatures range between 20 and 60°C for PE, 20 and 30°C for PET, 30 and 80°C for PP and PS, and 85 and 120°C for PC, depending on the production properties of each polymer (PlastikCity Ltd, 2019). The decision to perform either low- or high-temperature drying depends on the respective research question and the further scope of the investigation. Drying temperatures within the ranges of the melting temperatures of different polymers could alter the polymer surfaces or could cause unwanted reactions between polymers and the soil matrix.

After drying, the soil aggregates should be crushed to prevent microplastics from adhering to mineral components or from being enclosed in soil aggregates. Without this treatment, microplastics within soil aggregates could not be extracted and would be neglected in further analyses. Hence, it is important to disjoint soil aggregates as they are shown to contain microplastic particles and fibres (G. S. Zhang, Zhang, & Li, 2019).

In addition to manual sample homogenization by manually crushing the aggregates with pestle and mortar, it is also possible to use ultrasound techniques, which might be necessary for strongly aggregated soils with high clay contents (Piehl et al., 2018). The manual method cannot destroy microaggregates satisfactorily (Pinto da Costa et al., 2019). Treating samples with ultrasound enables to reliably disjoint both macro and microaggregates. However, at too high ultrasound or attrition energies, plastic particles could be fragmented during crushing. This secondary fragmentation of microplastic particles must be reflected in studies concerning the size, shape, and surface texture of microplastics.

2.4 | Sample matrix separation

In contrast to microplastic analyses in water and sediment samples, processing soil material faces the challenge of separating several components. Presently, no satisfactory methods exist for this purpose. As

sample separation can hardly be achieved in one preparation step, a suitable and standardized workflow is required. Three steps of microplastic extraction can be differentiated, which can be applied individually, partially, or consecutively: (a) the removal of the mineral phase, (b) the removal of organics, and (c) the size classification of microplastic particles by sorting and/or sieving. It is crucial to make sure that soil samples are not contaminated with external plastic (clothing fibres, plastic equipment) at any stage of this workflow.

2.4.1 | Removal of the mineral phase

In most studies on microplastics in soils, the principle of density separation is used to remove the mineral phase from the pre-processed samples.

The separation of the heavier mineral components (i.e., sand, silt, finally clay), that sink to the bottom, from the lighter components (i.e., microplastics, organics), that float up, depends on the density (ρ) of the separating solution and the density of the assessed plastic polymers (Durner, Iden, & von Unold, 2017). Different separation solutions are currently applied (e.g., NaCl, demineralized water, NaI, ZnCl₂, CaCl₂; Claessens, van Cauwenberghe, Vandegheuchte, & Janssen, 2013; Huerta Lwanga et al., 2017; Imhof, Ivleva, Schmid, Niessner, & Laforsch, 2013; M. Liu et al., 2018; Scheurer & Bigalke, 2018; G. S. Zhang & Liu, 2018).

A new approach is the application of castor oil for separation. The method was tested with recovery rates of $99 \pm 4\%$ for PP, PS, PMMA, and PET, but samples with a high content of organic material require additional treatment for organic matter decomposition (Mani, Frehland, Kalberer, & Burkhardt-Holm, 2019). Independent of the sealing solution, the verification, and correct adjustment of the desired density must be ensured during laboratory analyses because it depends on the room temperature and chemical processes (e.g., solubility of chemicals; Crichton, Noël, Gies, & Ross, 2017).

Density separation can be conducted in different vessels. Beakers, separation cylinders, and centrifuges made of glass or plastic are used. Because the duration of sedimentation is 25–48 hr, the use of a centrifuge, additionally coupled with a rubber disc, can significantly accelerate the process and make it much more time-efficient (Pinto da Costa et al., 2019; Scheurer & Bigalke, 2018). An alternative to open vessels or centrifuges is the application of closed sedimentation cylinders with a separation chamber. Devices like the Munich Plastic Sediment Separator (MPSS) (Imhof, Schmid, Niessner, Ivleva, & Laforsch, 2012) or the Sediment-Microplastic-Isolation (SMI) unit (Coppock, Cole, Lindeque, Queirós, & Galloway, 2017) enable the separation of the floating microplastic particles from the sunk soil particles.

Because the sedimentation process takes a long time, partly more than 24 hr, an acceleration by centrifugation is possible (Scheurer & Bigalke, 2018; G. S. Zhang & Liu, 2018). In this case, the use of lower sample amounts (e.g., 5–20 g) is required depending on the centrifuge size. However, several repetitions are needed to minimize the number of particles lost in the instruments (e.g., attached to inner vessel walls; Corradini et al., 2019; Scheurer & Bigalke, 2018).

A direct separation of microplastics from the mineral and organic soil particles presently only exists for the smallest microplastic particles (<30 μm). For this purpose, a pressurized fluid extraction with methanol and dichloromethane is used (Fuller & Gautam, 2016). However, this method can only be applied for specific research questions.

2.4.2 | Removal of organics

Organics (e.g., humus, peat particles, and plant/root fragments) mostly have a density comparable to microplastics. Thus, density separation usually results in organic components being extracted along with microplastics (Corradini et al., 2019; Felsing et al., 2018; G. S. Zhang et al., 2019). In order to isolate the microplastic particles, they can be removed manually using a stereomicroscope with respect to the detection limits/magnification of the microscope (Crawford & Quinn, 2017; Song et al., 2015). Alternatively, organics and microplastics can be separated technically.

As in marine and aquatic research, enzymatic digestion may be applied using a variety of enzymes in combination with a subsequent H₂O₂ treatment (Löder, Kuczera, Mintenig, Lorenz, & Gerdt, 2015; Mintenig, Int-Veen, Löder, Primpke, & Gerdt, 2017). During this procedure, plastic is not degraded. However, the method is time-consuming and still has to be tested for the successful application to soil organic matter (Bläsing & Amelung, 2018; Pinto da Costa et al., 2019; Prata, da Costa, Duarte, & Rocha-Santos, 2019).

Furthermore, different acid and alkaline solution treatments exist [e.g., with 65% HNO₃, 96% H₂SO₄, mix of 69% HNO₃ + 70% HClO₄ (4:1)]. With all acid treatments, a rapid removal of the organic components is observed (Enders, Lenz, Beer, & Stedmon, 2016). However, structural degradation of plastic particles was often observed.

In contrast to acid treatments, alkaline treatments influence neither the microplastic particles shape nor surface properties (Enders et al., 2016). The treatments apply NaOH, KOH, or both in combination. Both chemicals are suitable for biological samples but have not been applied to soil samples. Although these treatments do not degrade plastics, the methods are unable to remove alkali-insoluble organic matter from soil (Bläsing & Amelung, 2018). Therefore, humins probably remain in the samples after the alkaline treatment, which complicates the later identification of the microplastic particles (Dehaut et al., 2016).

Furthermore, the oxidation of humus is sometimes applied to remove organics (e.g., 30% H₂O₂; He et al., 2018; M. Liu et al., 2018; Scheurer & Bigalke, 2018). The oxidation can be improved by adding a Fe(II)-containing solution (e.g., FeSO₄) as a catalyst for the reaction (Fenton reagent). Restrictions may arise here for calcareous soils, because the Fenton reaction could be impeded by carbonates (N. Liu, Ding, Weng, Hwang, & Lin, 2016). Removing organics with just H₂O₂ causes a digestion of PE and PP (Silva et al., 2018). No negative effects are reported for the application of the Fenton reagent.

Next to the enzymatic and chemical treatments, a separation based on the different electrostatic behaviour of organic and plastic particles was developed (Felsing et al., 2018; Hidalgo-Ruz, Gutow,

Thompson, & Thiel, 2012). This method has no negative effect on microplastic particles. However, it was tested only for various sands and sediments, where it gave reliable results in the separation of organic material. The method should be validated for the application to soil samples (Felsing et al., 2018).

2.4.3 | Sieving and sorting of separated microplastic

After microplastics have been separated from the mineral and organic matrix, it is useful to characterize the plastic particles further according to size by sieving and/or sorting. According to the above-mentioned definition, microplastics range between 0.01 and 5.0 mm in diameter (see first chapter). Considering this wide range, the question arises if these thresholds are also plausible in soil science. Instead, microplastics could further be separated into larger and smaller microplastics (>2 and <2 mm, respectively) according to the soil scientific differentiation between fine and coarse soil components. The determination of microplastic size classes can provide important insights into possible transport processes, as well as the physical and chemical degradation in soils.

Generally, macro- and microplastics can be separated with sieves with a mesh size of 5 mm. Particles <5 mm are often further subdivided using specific series of sieve mesh sizes (e.g., <1.0, 1.0–3.0, 3.0–5.0 mm; 1.0–0.25, 0.25–0.05 mm; 5–1 mm; M. Liu et al., 2018; Piehl et al., 2018; G. S. Zhang & Liu, 2018). For particles <1 mm, a filtration with various pump systems (e.g., vacuum) and glass fibre filters is often used (Klein, Worch, & Knepper, 2015; Scheurer & Bigalke, 2018).

Sieving can be conducted after drying and crushing, or intermediately during analysis (i.e., after density separation as dry or wet sieving). For soil samples with a high clay content, a pre-treatment might be needed to disaggregate the soil material (e.g., with H₂O₂, Na₄P₂O₇; Piehl et al., 2018). The effective size separation during sieving also depends on whether the soil aggregates have been crushed properly (G. S. Zhang & Liu, 2018).

To enable a quantification and identification of the separated microplastics, the manual sorting and counting of the detected particles is established. This process can be facilitated by staining the sample material with Nile Red tracer (Thomas, Rebecca, Nikolaus, Karsten, & Andrew, 2017).

Depending on the size class, microplastic particles are counted and documented by eye using a binocular or stereomicroscope. In addition to taking photographs, the documentation includes the classification of the particles according to different features, which were mostly adopted from marine research (Baldwin, Corsi, & Mason, 2016; Fischer et al., 2016; Nor & Obbard, 2014). Shape, surface texture, colour, and luster are documented (Horton et al., 2017; Nor & Obbard, 2014). Furthermore, the surface area and length of the particles are determined in some studies using various imaging software (Lorenzo-Navarro, Castrillon-Santana, Gomez, Herrera, & Marin-Reyes, 2018).

Pre-treatments of soil samples for studies on microplastic dynamics could be remedied by automating the sorting and classification. Because most studies produce images of the microplastic particles, microplastics placed on filters after separation can be photographed at the appropriate resolution. Based on different colour and shape features, machine learning could then be used for automatic counting and classification (Lorenzo-Navarro et al., 2018). In addition, automatic image analyses from Fourier-transform infrared spectroscopy (FTIR) microscope images combined with an automatic database analysis have already been performed (Primpke, Lorenz, Rascher-Friesenhausen, & Gerdtts, 2017; Primpke, Wirth, Lorenz, & Gerdtts, 2018). Because these are also suitable for complex sample matrixes, an application to soil samples would be useful and should be developed in further studies.

2.5 | Microplastic quantification and identification

The final step in the analysis of microplastics in environmental samples is the quantification of the microplastic components, and, in most studies, the identification of the polymer types. Both sieving and sorting, as well as automatic sorting methods, allow us to calculate a value for the proportion of microplastic particles in soil samples. However, current studies reveal a wide range of detection limits related to the minimum size of the detected particles. Depending on the applied method, the detection limit varies from 1 to 1,000 µm, whereas in some cases, a large part of the small microplastic particles could not be detected and a comprehensive quantification was difficult (Huerta Lwanga et al., 2017; M. Liu et al., 2018; Piehl et al., 2018; Scheurer & Bigalke, 2018; G. S. Zhang & Liu, 2018).

The detected amount of microplastics can be quantified by counting the particles and putting them in relation to the mass or volume of the original soil sample. At present, the unit mpp/kg is mostly used to report results (Pinto da Costa et al., 2019; Prata et al., 2019; Silva et al., 2018). Still, the unequal size of the microplastic particles complicates the comparability when using this unit. Alternatively, by weighing the microplastic particles, it is possible to derive the mass-based unit 'mg per kg soil' (mg/kg), which increases the comparability with other soil analysis results (e.g., elemental concentrations; Möller et al., 2020; Qi et al., 2020; J. Wang et al., 2019; W. Wang, Ge, Yu, & Li, 2020). However, the exact determination of the microplastics' weight is currently difficult, because of the low density and small particle sizes (<500–300 µm). Moreover, the selection of the unit to report the results might depend on the research question. For studies on the general occurrence and abundance of microplastics in the environment, a mass-related specification (e.g., mg kg⁻¹) seems to be sufficient and plausible. Instead, when effects on soil functions, relocation processes, or modeling are in focus, informations on particle number (e.g., mpp/kg), size, shape, and type become relevant. For approaches such as simplifying the complex diversity of MP particles through a three-dimensional dimension, also size, density, and shape of each particle is required (Kooi & Koelmans, 2019).

After the visual identification of microplastics, an identification of the polymer type might be wanted in some studies, for example, to

deduce on the plastics' provenience. This can be achieved by various chemical methods whose applicability and limitations have already been reviewed in the literature (e.g., Pyrolysis-gas chromatography-mass spectrometry, ToF-SIMS, Raman spectroscopy with μ Raman, and FTIR with μ FTIR; David, Steinmetz, Kučerík, & Schaumann, 2018; Du, Wu, Gong, Liang, & Li, 2020; Dümichen et al., 2017; Hermabessiere et al., 2018; Pinto da Costa et al., 2019; Renner, Schmidt, & Schram, 2018).

The increasing number of samples and possibly high numbers of microplastic particles in soils lead to large amounts of data, which can be processed in an automated way (image analyses or automatic spectral analysis with databases; Primpke et al., 2017; Primpke et al., 2018). The application of spectroscopic methods for the analysis of plastics has already been discussed in other soil specific reviews as well as reviews of the material sciences and in marine research (Elert et al., 2017; He et al., 2018; Pinto da Costa et al., 2019; Prata et al., 2019; Renner et al., 2018; Ruggero et al., 2020; Silva et al., 2018; B. Zhang et al., 2020) and is not further elaborated in the present review.

The methods commonly applied for soil scientific studies have in common that the heterogeneous soil sample matrices require a more or less complex sample preparation or separation. First approaches to reduce this effort are the pre-scanning of the sample without chemical treatment, based on near-infrared spectroscopy (NIRS) detection (Paul, Wander, Becker, Goedecke, & Braun, 2019). Moreover, a direct quantification of heterogeneous sample matrices by the combination of thermogravimetric analyses (TGA) with thermal desorption system coupled with gas chromatography-mass spectrometry (TDS-GS-MS) and the application of twisters as solid-phase absorbers was demonstrated for PE particles (Dümichen et al., 2015, 2017). Despite the diversity of identification procedures and their different applicability to soil samples, there is still a large demand for research to validate, improve, and develop suitable, comparable, cost- and time-efficient methods—particularly regarding pre-scanning methods.

3 | CONCLUSIONS

The discovery of microplastic particles as new pollutants in the environment opens up a novel field of research for soil science. Potential hazards posed by microplastics and nanoplastics in soils (e.g., uptake by plants and introduction into the food chain) are theoretically plausible. However, a better understanding of microplastic dynamics is needed to systematically evaluate the effects of soil-bound microplastics pollution (e.g., on biota and the food chain) and to develop targeted mitigation strategies. Regarding the current trends in environmental microplastics research, we think that it is specifically required to transition from solely explorative microplastics studies to more systematic investigations. This would especially call for a more intensive consideration of spatially adequate sampling strategies than documented in previous studies. The proposed geospatial approach might thus enable further more sophisticated research.

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CONFLICTS OF INTEREST

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