# REVIEW

Microplastics and Nanoplastics

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# Status quo of operation procedures for soil sampling to analyze microplastics



Kristof Dorau<sup>1\*</sup><sup>®</sup>, Martin Hoppe<sup>1</sup><sup>®</sup>, Daniel Rückamp<sup>1</sup><sup>®</sup>, Jan Köser<sup>1</sup><sup>®</sup>, Georg Scheeder<sup>1</sup><sup>®</sup>, Katrin Scholz<sup>2</sup> and Elke Fries<sup>1</sup>

### Abstract

Since 2015, the determination of microplastics (MPs; < 5 mm) in soil has gained increasing attention. However, usual analytical protocols still render a comparison of results challenging. This structured review integrates an in-depth screening of scientific articles (n = 106; from 1980 to 2022) that focused on determining MPs in soils. The different studies were divided into groups regarding (i) MPs input pathways, (ii) study site information, (iii) sampling design, (iv) sample preparation, and (v) analytical methods for MPs detection. Most of the studies (63%) focused on a defined study site influenced by a known point source, whereas 37% measured MPs background contents related to delocalized MPs emission sources. Even though soil was the target compartment, only 26% classified the soil type, mostly using the World Reference Base (WRB) as a classification system. Additional information of MPs recovery tests and analytical results. In nearly all studies, the mass of the single samples equaled the final mass of the composite sample, with a mean of 1.32 kg  $\pm$  1.07 kg. However, other procedures that involve a larger sample mass seem promising but are still seldom applied. Our structured review revealed that a standard operation procedure with harmonized methods is urgently needed with a coherent and comprehensive workflow, including field sampling and sample preparation. Such a procedure would ensure the reproducibility and representativeness of analytical results, which are mandatory for evaluating and restricting MPs pollution in soils in the future.

**Keywords** Microplastics, Synthetic polymers, Soil sampling protocols, Analytical methods, Soil pollution, Method harmonization

\*Correspondence: Kristof Dorau Kristof.Dorau@bgr.de Full list of author information is available at the end of the article



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#### Introduction

The rapid evolution of plastic production since the 1930s and 1940s [3] has paved the way for microplastics (MPs; size < 5 mm, with the upper size limit of nanoplastics still under debate at 100 or 1,000 nm; [18]) to enter terrestrial ecosystems. Thus, their ubiquitous distribution has finally been recognized as a threat to soils, and even in remote areas - such as deserts [1] - MPs have entered soils. A variety of input routes for MPs in soils exist that can be either localized on a smaller scale (e.g., application of sewage sludge or compost on single fields) or diffuse and more widespread (e.g., by aeolian or fluvial transport and deposition). However, in both pathways for MPs, the vast range of polymers is likely to be persistent and, thus, will accumulate in soils [45]. MPs become part of a complex mixture of the soil organic matter (SOM) pool and associate with mineral components over time [3]. Overall, incorporation into soil aggregates limits and hinders analytical methods for identifying and quantifying MPs pollution [38], in particular, the isolation of polymers from complex organic-rich soil represents a major hindrance for adequate MPs analysis [25]. A broad size range of MPs particles and different polymer types, shapes, aging states and additives characterize the complex nature of MPs [28]. Much emphasis has been placed on improving analytical techniques and identifying bottlenecks and shortcomings during the laboratory stage (e.g., [10, 12, 20, 35, 36, 40]). However, relatively little attention has been given to how to take representative samples for MPs analysis [35, 64].

Sampling design for determining MPs in soil – a particulate material potentially inhomogeneously distributed in an inhomogeneous matrix - is arguably challenging in many ways. Generally, sampling is a random process and includes uncertainties due to (i) selection of the number and location of sampling points and sample volume and (ii) sample preparation due to operations involving homogenization, sample division, transport, and storage [21]. In most cases, the research aim determines the optimal sampling design and includes, for instance, aspects of regulatory enforcement, regulatory compliance, and routine monitoring [65]. By no means will bad sampling generate good reliable data, and the reliability of the analytical result depends on the representativeness of the sample [65]. Plastic particles have variable sizes and shapes and can be (i) uniformly distributed in the field due to deposition processes or continuous, widespread application of biosolids or (ii) heterogeneously distributed due to roadside emissions or fragmentation of larger particles into MPs [64]. What makes MPs such special

analytical target compounds with regard to sampling techniques is that polymers are generally resistant to mechanical breakdown and are insoluble in comparison to classical inorganic or organic pollutants. A homogenous distribution is therefore hampered. In addition, pedogenesis – a process that results in generally similar soil properties within a distinct horizon over thousands of years – is not relevant for MPs as a distribution process since MPs have been entering soil since less than 100 years ago. Information on the transport/distribution schemes of MPs in soils is still scarce. Information on the transformation of MPs is only available for biodegradable polymers [19], i.e., a tiny fraction of the synthetic materials released into soil.

Obviously, a reproducible and representative sampling strategy is a cornerstone of an accurate assessment of the presence and distribution of MPs in soils. The techniques applied for MPs analysis in the laboratory influence how the samples should be collected. For instance, MPs often need to be enriched by density separation (DS) to separate them from the matrix or if the MPs contents are very low to obtain a signal using pyrolysis gas chromatography-mass spectrometry (Pyr-GC-MS; [55]). At present, data on MPs contamination in soils are hardly comparable because of the lack of harmonized methods including standardized operation procedures (SOPs), particularly in terms of soil sampling strategy and sample preparation. To quantify the risk of MPs pollution, a common basis of empirical research, method development, and quality control standards is urgently needed to achieve regulatory actions [35]. To date, no guidelines for monitoring MPs in soil to provide these data have been published [4]. MPs research for soil typically lags behind method development that is already present or more established for the media air and water, as indicated by the total number of research articles within the given environmental compartment (Fig. S1A). Some reasons for this issue are the comparable ease with which MPs can be extracted from a water column contrary to a complex organo-mineral soil matrix. In addition, plastic debris particularly accumulates along shorelines with a much better visibility in comparison with MPs pollution in soils that restrict the ease in visibility due to incorporation into the soil matrix [45]. In the future, reproducible analytical pipelines (RAPs) and technological readiness levels (TRLs) appear to be flexible decision-making tools to enable global harmonization of plastic pollution monitoring methods [2].

The aim of this study is to assemble data from studies on MPs in soils to review current procedures for soil sampling to analyze MPs and to identify knowledge gaps, particularly in terms of method harmonization. Thereby, we incorporated the complete workflow in MPs research to gather information (i) about the global MPs distribution, (ii) on MPs sampling in soils, (iii) sample preparation procedures, and (iv) the broad range of analytical prerequisites to analyze MPs. Finally, all available technical information from the reviewed studies (n=106 articles) were incorporated in a comprehensive summary in order to highlight the extent to which degree a study can be reproduced from technical information that are very well described towards information, which are basically not available.

#### Methods

#### Structured review/data source

In advance of the review process, we defined different categories with distinct criteria that are important for determining MPs in soil (Table S1). The categories were differentiated into groups covering information on (i) input pathway, (ii) study site, (iii) sampling design, (iv) sample preparation, and (v) analytical method. A literature search was performed in Science Direct (n=2,278articles; https://www.sciencedirect.com/) and SCOPUS (n=1,548 articles; https://www.scopus.com/) using the keywords "Microplastics" AND "Sampling" AND "Soil". The last keyword "Soil" was replaced exclusively with "Air" and "Water" for a comparison of MPs research within these compartments. We also included studies on MPs distribution along beaches and marsh ecosystems since these settings often belong to distinct soil groups, e.g., as part of a soil-regolith toposequence or catena. Only research articles that underwent a peer-review process were included. Reviews, book chapters and conference proceedings were excluded. The data were collected on 2022–05-02, and the articles were managed by using JabRef 5.6 software (https://www.jabref.org/). All articles were filtered in a hierarchical fashion by reading each title; if the title was relevant, we read the abstract, and if the abstract verified a match, the relevant information was extracted from the main text. Duplicates or preprints and various other study types, e.g., incubation studies in the laboratory, studies that used soil only for method development, or toxicity tests of MPs on soil flora and fauna, were excluded. We included studies with comparisons among multiple study sites at larger scale, e.g., at the national level, but also studies that focused on the intrafield spatial variability of MPs. Article selection was based on the following criteria: soil must have been sampled in the field, sample preparation was conducted in the laboratory, MPs particle counts or polymer masses were derived. A target of the study must have focused to assess a potential contamination of MPs background values and studies with focus on multiple compartments beside soil, e.g., sediment or water, were additionally included in the structured review.



The values in the brackets are the mean particle counts and standard deviation ( $\pm$ ) of the processed data after outlier removal by using the Interquartile Range (IQR) multiplying by 1.5.

Fig. 1 Metrics derived from the reviewed studies (n = 106) for the group 'input pathway' (A to D) and particle counts for the diffuse and localized pathway (E). The raw data without outlier removal of particle counts is presented in Table S2 for comparison



Fig. 2 Metrics derived from the reviewed studies (n = 106) for the group 'study site'. The box in the boxplot of Panel (A) extends from the 25<sup>th</sup> to the 75<sup>th</sup> percentile, where the line inside the box is the median

Finally, from the total number of articles that were found by the literature search, only a small proportion of 4.6% (n = 106 articles) were considered relevant and included in the present analysis (Figs. 1, 2, 3, 4, 5 and 6) to contribute to a better understanding of MPs in soils. All data analysis and reporting were performed with R software version 4.2.0 and RStudio version 2022.2.3.492 [43, 47], featuring the main packages included in the Tidyverse [62]. While the presentation and visualization of the data are based upon the raw data (as histograms, boxplots, or pie charts), the calculated metrics (mean and standard deviation of the mean  $(\pm)$ ) are based upon processed data obtained by outlier removal. Outliers were identified by the interquartile range (IQR: 1.5 times



Fig. 3 Metrics derived from the reviewed studies (n = 106) for the group 'sampling design'



Fig. 4 Metrics derived from the reviewed studies (n = 106) for the group 'sample preparation' for the drying procedure (**A**), the applied mesh sizes during sieving of the soil sample (**B**) and the method to remove SOM (**C**)



Fig. 5 Metrics derived from the reviewed studies (n = 106) for the group 'analytical method' of analytical results (**A**), the method to analyze MPs (**B**) and the polymers that were found (**C**)



Study site O Sampling design O Sample preparation O Analytical method

Fig. 6 Percentages of missing data for the categories differentiated within this structured review from the reviewed studies (*n* = 106), where the colors correspond to the predetermined groups

higher than the 75th and 1.5 times lower than the 25th percentile of the data), and the calculated metrics based on the IQR are discussed in this manuscript (Table S2). The metadata of the articles revealed that studies were conducted within 35 countries spanning the globe, with the majority coming from China, followed by Germany and Spain (Fig. S2A).

#### **Results and discussion**

#### Input pathway

Over the past decade, a vast number of studies have reported on the potential hazards of MPs in soil, with a cumulative number of 2,278 articles related to soil sampling having been published (Fig. S1A). Humans are not directly threatened by the consequences of soil pollution, unlike the case for MPs pollution of air or drinking water. Not surprisingly, significantly more studies addressing the prevalence and distribution of MPs were published for air, with 5,159 articles, and water, with 9,221 articles. Thus, research on MPs in soils is indisputably in its infancy, whereby 2015 can be seen as the starting point of the systematic assessment and delineation of MPs in soils with a lag time of approximately 5 years compared with air and water (Fig. S1B). One-third of the studies considered diffuse MPs input (Fig. 1A), among which marine transport and deposition (50%) dominated over fluvial (39%) and aeolian (8%) input (Fig. 1B). Obviously, marine plastic debris is an omnipresent feature in seas and oceans worldwide and raised public awareness earlier (e.g., [9]) than windborne MPs from distant sources (e.g., [1]). The majority of studies considered a known MPs entry route (localized MPs input) into soil (63%; Fig. 1A), of which agriculture is the dominant source (81%) over urban-associated littering (13%) and traffic-related MPs pollution, which includes tire wear containing street runoff due to traffic (6%) (Fig. 1C). Most studies with an agricultural context reported the impact of plastic mulching (41%) as a source of MPs emissions, but compost (6%), fertilizers (13%), and sewage sludge (9%) were also considered (Fig. 1D). Differences in entry pathway result in significant differences between the average number of MPs particles (P) that can be found. Soils that were predominantly affected by aeolian MPs input with an average of  $0.42 \pm 0.33$  P kg<sup>-1</sup> featured significantly fewer MPs than soils in urban areas, with up to  $6,176 \pm 6,481 \text{ P kg}^{-1}$ (Fig. 1E). An elevated MPs background content is often associated with human activities, and it is rather surprising that soils have been only relatively recently recognized as a long-term sink for polymers.

#### Study site

The size of the study area defined for sampling ranged over several orders of magnitude from less than a square meter  $(m^2)$  (e.g., [63]) to several square kilometers  $(km^2)$ (e.g., [48]), with an average size of  $736 \pm 1452$  m<sup>2</sup> and a median of 25  $m^2$  (Fig. 2A). In most studies, the area for which a composite sample was representative was not clearly defined. Larger study sites require a larger number of samples, which causes the problem that the collection of a given number of samples from a small plot of one  $m^2$  rather than a plot of one  $km^2$  results in a value that is closer to the true value, e.g., the mass of a certain polymer per kg soil. While geostatistical analysis has been used to determine the spatial variability of multiple soil properties in the past [5], such analysis is lacking for MPs distribution at the field scale, which is expected to vary depending on the landscape and the input pathway. Generally, sampling of larger areas is desired because subtle differences in altitude, e.g., due to depositional processes along a river, have a tremendous impact on the MPs particle count, and these features are overlooked if only a limited number of composite samples are taken in the field from only a small compartment within a certain distance of the river [46]. A sampling scheme or a scaled map of the sampling area could be helpful to provide information about the study site and highlight the area for which the particle count or mass is representative, but a sketch of the study site was missing in 47% of the reviewed studies (Fig. 6). Even though the soil matrix and its properties are known to influence the extraction efficiency and analysis of polymers, soil classification is not adequately represented. In 76% of the reviewed studies, information about the soil type was incomplete (Fig. 2B). Only in four studies were two classification systems simultaneously referenced, the World Reference Base (WRB; [27]) and the Soil Taxonomy [53]. MPs might be entrapped within soil aggregates, clay-rich soil samples diminish the recovery of MPs [66], and SOM-rich samples might bias and interfere with polymer analysis via Pyr-GC-MS [55]. Thus, it is absolutely necessary to provide information on the soil type, parent material, and soil properties of the study site. Among soil parameters, SOM>texture>pH were most commonly measured concomitantly with the abundance of MPs, but such measurements were performed for only 20% of the screened studies (Fig. 2C). Moreover, in particular for soils developed from fluvial material, age dating of the soils from which MPs were extracted was performed to derive information on the time of MPs deposition (e.g., [31]) (Fig. 2C).

#### Sampling design

The sampling tool was not mentioned in 25% of the screened studies, but if it was mentioned, a spade was the sampler of choice (21%) for obtaining a mass-based sample (Fig. 3A). Vacuum procedures were also used to collect MPs derived from abrasion and fragmentation of

clothing from soil surfaces along recreational trails [14]. Collecting soil on a volume-based approach by augers (21%) or steel cylinders (8%) has the advantage that variable bulk densities can be accounted for (Fig. 3A), e.g., in floodplain soils [50]. In tidal freshwater marshes with sediment bulk densities ranging between 0.26 and 0.81 g cm<sup>-3</sup>, it can be even beneficial to give MPs particle counts based on area (P m<sup>-2</sup>) rather than per weight (P kg<sup>-1</sup>) or volume (P L<sup>-1</sup>) [24].

Random or stratified random sampling was the method of choice and was performed in 66% of the reviewed studies (Fig. 3B). If MPs are emitted from a longitudinal source, it is recommended to build a composite sample by applying a sampling design in the form of a transect, e.g., along rivers [50], plastic covers [54], or roadside soil [37]. However, composite sampling was only performed in 12% of the studies (Fig. 3B). The timing of sampling was evenly distributed throughout the year (Fig. S3A). However, it was recently shown that the MPs load in soil rises after each successive application of sewage sludge [7]. Thus, it makes a difference if samples are taken before or after the application of sewage sludge [7], compost [52], or fertilizer-containing plastic microcapsules [29]. In addition, if sediments deposited by a summer flood are analyzed for their MPs load, sampling should be performed in the subsequent fall or winter season [30].

Remaining open questions include the optimum number of mixed samples per study site and the optimum sample weight to achieve a satisfactory representativeness of MPs results in soils. From the reviewed studies, we calculated the following mean sample numbers:  $3.7 \pm 1.6$  single samples from a given spatial unit were obtained and mixed to form  $1.4 \pm 0.7$  composite samples per study site (Fig. S3D and E). In most of the studies (54%), only a single mixed sample was taken per study site with a mean weight of 1.3 kg $\pm$ 1.1 kg (Fig. S3F). Larger sample masses typically show smaller errors than small sample masses, but if the units are expressed in P  $kg^{-1}$  dry weight, it is recommended that at least 1 kg of soil be taken to the laboratory [35]. In some cases, the sample mass needs to be enhanced a posteriori due to a heterogeneous MP distribution, for instance, from 200 to 500 g sample mass [49]. In nearly all studies, the mass of the single samples were equivalent to that of the composite sample. However, recently, it was recommended that samples should be as large as possible (e.g., 200 L of soil from an area of one m<sup>2</sup>) and reduced afterward by the quartering method instead of taking hundreds of small soil cores [64]. Since the MPs contents in compost or sewage sludge [22, 39, 61] and in plastic residues originating from plastic mulching [17] vary, this assumption still needs to be validated for a variety of MPs sources to reliably quantify MPs. Larger representative elementary volumes (REVs) were only taken in 3% of the studies and were not routinely employed. The applied analytical technique must also be considered when choosing the sample mass.

With regard to soil depth, in the majority of the reviewed studies, sampling was performed within the upper 30 cm. Several surface soil samples [14] and samples up to even 200 cm soil depth [60] were also investigated. However, fewer than 9% of the reviewed studies focused on determining MPs contents in the subsoil at>30 cm depth. These soil depths are even less thoroughly characterized (Fig. 3C). Indeed, it remains uncertain to which extent subsoil is a sink for MPs due to translocation. Among soil characteristics, soil depth is fundamentally important and must always be investigated considering the depth to which MPs might be easily translocated. Sixty percent of the reviewed studies studied soil samples collected from a single depth (Fig. S3B), with rare examples of up to ten depths for a single soil profile [60]. The rationale behind choosing a particular depth is not obvious. Stepwise vertical sampling is currently the exception and in most cases is limited to the number of samples that can be analyzed with a given amount of time and money. We calculated that a mean number of  $43 \pm 37$  samples were analyzed in a wide range of studies that counted visible plastic mulch fragments (e.g., [16]; 444 samples) or that involved tedious sample preparation following thermal extraction and GC-MS (e.g., [37]; 12 samples) (Fig. S3C).

#### Sample preparation

In general, temperature during transportation and storage of soil samples is not relevant for MPs as target compounds since they are persistent at usual ambient and room temperatures. In terms of drying soil samples, either air drying (39%) or oven drying (60%) is the method of choice since freeze-drying (1%) limits the sample mass that can be dried in a given time (Fig. 4A). Overall, temperatures above 40 °C are not recommended because they affect the physical and structural properties of MPs by glass transition, melting or degradation [56]. Samples are physically and chemically modified in advance, and an adequate sample homogenization step is mandatory. Sample homogeneity refers to the degree to which the analyte is randomly distributed in the sample under investigation, but in reality, homogeneous materials are rare or nonexistent [26], which is particularly true for MPs in soil. Manual homogenization by "spooning", similar to the cone and quarter technique, or the use of automatic and mechanical sample dividers such as a spiral mixer, cement mixer, or the most commonly employed riffle splitter or rotary splitter can be employed. The value of sample homogenization should not be underestimated because it has an impact on the result, and the handling time in the laboratory can range between 5 and 30 min per sample [51]. Information about sample homogenization procedures in the field and in the lab prior to analysis were missing in 68% and 86% of the reviewed studies, respectively (Fig. 6).

MPs are typically classified by size fraction; however, there is no clear scientific justification for this choice based on actual evidence, but rather, the size fraction is used from a pragmatic perspective on size categories [23]. For instance, the National Oceanic and Atmospheric Administration proposed that all plastic particles < 5 mm in diameter be referred to as MPs [23], but differentiation into subgroups of large MPs (1 to 5 mm) and small MPs (< 1 mm) seems commonly accepted [28]. This classification is in line with the most commonly applied sieve mesh size being < 5 mm, but overall, a total of 30 different mesh sizes are used to physically separate distinct particle sizes (and MPs sizes) for further analysis (Fig. 4B). Sieving soil samples < 2 mm to determine MPs by particle number or mass is certainly not concomitant with this nomenclature distinguishing large MPs (from 1 to 5 mm) and small MPs (<1 mm). However, a sieve size of 2 mm is conventionally applied to determine a variety of other soil properties in soil science, such as particle size distribution or soil pH. The number of applied mesh sizes ranged from one (e.g., [44]; < 5 mm) to three (e.g., [46]; <5 mm, <1 mm, <0.5 mm) and five ([37]; >2 mm, 2-1 mm, 1-0.5 mm, 0.5-0.1 mm, <0.1 mm). Sieving a sample to a given size fraction greatly reduces the soil matrix volume to improve the following density separation (DS) steps. In addition to sieving, sequential automated image analysis enables the automatic recording of MPs sizes based on the length of MPs particles along the longest axis and their division into multiple subgroups [1]. Thus, MPs size classes do not necessarily have to be separated by sieves, but can be classified on the basis of MPs attributes by using semiautomatic image software, e.g., ImageJ, to distinguish particle abundance, shape, and size distributions [58].

In most cases, spectroscopic methods need a purified sample to minimize the signal-to-noise ratio and minimize unclassified pixels that do not belong to a distinct polymer by collecting characteristic spectrograms. This is equally true for thermogravimetric methods to minimize misinterpretation of organic marker molecules that occur in the pyrogram. Thus, for a variety of analytical methods – spectroscopic and thermogravimetric - it is valuable to incorporate a purification step by (i) H<sub>2</sub>O<sub>2</sub>, (ii) Fenton's reagent, and/or (iii) enzymatic-oxidative digestion even if no visual plant debris or lightweight particulate organic material remains on the filter where MPs are collected. After sieving, SOM is typically removed. The method typically uses hydrogen peroxide  $(H_2O_2)$  rather than Fenton's reagent  $(H_2O_2 + ferrous iron; Fig. 4C);$  an equal soil:solution ratio with a mean value of  $5.7 \pm 10$  mass volume<sup>-1</sup> (Fig. S4A); a 30% concentration of  $H_2O_2$  (Fig. S4B); and heating to a mean temperature of 60  $^{\circ}C \pm 9 ^{\circ}C$  with a range between 25 °C and 75 °C (Fig. S4C) for a mean duration of 44 h  $\pm$  26 h with a range between 1 and 192 h (Fig. S4D). Regardless, the sample should be treated with  $H_2O_2$  until no bubble formation is apparent and the purity is sufficient for the analysis. In particular, SOM present in the sample or as part of the coating of pristine MPs can hinder identification by producing overlapping spectral information, e.g., when hyperspectral imaging used to identify polymers [13]. Fifty-six percent of the reviewed studies removed SOM, but in 44% of the studies, it was not stated if SOM was removed or if SOM removal was not necessary for MPs analysis.

In most cases, analysis of MPs is only as successful as the degree to which the bulk soil can be removed efficiently from the analyte by DS. In only 13% of the studies, it was not clear if DS was performed; the majority of studies utilized DS by using a single salt solution (63%), two salt solutions (23%) or up to three salt solutions for a single study. Soil with an average sample mass of  $84\pm87$  g (Fig. S4F) was treated with salt solutions with an average density ( $\rho$ ) of 1.4±0.2 g cm<sup>-3</sup>, but as seen from the bimodal distribution of the data, the density was not standardized (Fig. S4G). The most cost-effective and extensively used salt is sodium chloride (NaCl), which has a maximum  $\rho$  value of 1.2 g cm<sup>-3</sup> at saturation. NaCl is less expensive than sodium polytungstate (typical  $\rho$  1.6 g cm<sup>-3</sup>) and less toxic than zinc bromide (typical  $\rho$  1.7 g cm<sup>-3</sup>). However, 14 solutions adjusted to various densities with a range from  $\rho$  1.0 to 1.85 g cm<sup>-3</sup> (Fig. S4G) are commonly used, which still renders it challenging to determine an appropriate and best applicable density. Low-density salt solutions, such as NaCl, may be less effective in removing high-density polymers with a higher density, such as polyethylene terephthalate (PET;  $\rho$  1.31–1.43 g cm<sup>-3</sup>), polyoxymethylene (POM;  $\rho$  1.20–1.58 g cm<sup>-3</sup>), and polyvinyl chloride (PVC;  $\rho$  $1.41-1.61 \text{ g cm}^{-3}$  [38]. Thus, solutions with higher density are more appropriate. A very high removal>99.9% of soil matrix from 250 g to  $\leq$  160 mg was achieved by using a zinc chloride solution, but a lightweight fraction of particulate organic matter remained on the filter

after decanting the supernatant, which might alter the analysis of MPs by using  $\mu$ -Fourier transform infrared spectroscopy ( $\mu$ FTIR; [36]). However, a compromise has to be made in terms of cost since the salt price usually increases with salt density. It is necessary to verify the relative density of the salt solution and, if needed, adjust it throughout laboratory analysis, and the value should be checked regularly, e.g., by using a pycnometer.

It is well known that the recovery rates of polymers from soils depend on the soil properties, e.g., aggregates, carbonate and SOM content, and texture. Thus, matrix-dependent recovery tests are mandatory and must include all sample preparation procedures. Within the reviewed studies, 37 of 106 (35%) performed recovery tests based on spiking experiments with highly variable recovery rates. The recovery rates strongly depended on the target polymer and the SOM content, as shown by Corradini et al. [7], which highlights the importance of determining soil properties in addition to assessing MPs. Careful considerations must be made if sample preparation techniques for soils are adapted from procedures developed for MPs determination in marine sediments with a sandy texture and low SOM content [8]. Soils typically have a more diverse particle size distribution, elevated SOM content and higher number of natural soil colloids.

#### **Analytical methods**

The widespread interest in investigating the abundance of MPs in the environment has led to a variety of sophisticated analytical methods that are also suitable and widely used to determine MPs in soils, with a clear trend toward visual identification to determine particle counts (87%). Visual inspection by stereomicroscope is an easily available method that might explain the bias for this method over thermogravimetric methods (13%) when determining polymer mass (Fig. 5A). A minority of the reviewed studies investigated both particle counts and polymer mass (e.g., [6, 57]). Most studies employed a stereomicroscope (70%) to identify MPs particles, sometimes in combination with Fourier transform infrared (FTIR; 40%), attenuated total reflection FTIR (ATR-FTIR; 23%), or Raman spectroscopy (14%), to identify the polymer type (Fig. 5B). However, a preselection of the suspected MPs by light-microscopy followed by IR methods typically introduces a bias by the operator due to translucent and/or very small particles present in the sample that might be overlooked [42]. Therefore, automated procedures such as FTIR microscopes in conjunction with focal plane array (FPA) detectors are on the rise for automatic detection of MPs in environmental samples [32]. High-resolution imaging via Raman spectroscopy might generally yield very high particle counts ranging between  $2.2 \cdot 10^4$  and  $6.9 \cdot 10^5$  P kg<sup>-1</sup> [67]. In the future, technological breakthroughs will enable the visual identification and quantification of even smaller particles than is possible at present [33]. However, the smaller the particles are, the more robust the cross-sensitivity of the applied methods must be to successfully discriminate between natural organic matter and synthetic polymers. On some occasions, scanning electron microscopy and energy dispersive X-ray diffraction (SEM-EDX; 7%) were used to further obtain the degree of polymer degradation (e.g., [44, 59]). Spectroscopic methods are improved by using polarized light microscopy or fluorescent dyes such as Nile Red [35], which has been applied in conjunction with FTIR [34] and Raman spectroscopy [41] for MPs research. Particles with fluorescent staining serve as a proxy in the quantification of microplastics but it must be checked for potential interference of the dye with the spectroscopic data. Methods based on Pyr-GC-MS enable the identification of the polymer type and quantification of its mass. The major drawback is the labor-intensive sample pretreatment steps of DS and SOM removal, cost-intensive equipment, the high level of analytical skill required and the very small analytical sample mass ranging from 10 to 50 mg [11]. Upscaling a very small sample mass of a few mg to one kg remains a challenge and highlights the need for appropriate sample homogenization procedures in the field and in the laboratory. Tun et al. [57], for instance, identified one sample as an outlier after they found 70 MPs particles in a sample with a mass of 10 mg. In this case, upscaling to a hypothetical unit of 1 kg (factor 100,000) would ultimately yield a very high particle count of  $7 \cdot 10^6 \,\mathrm{P \, kg^{-1}}$ .

The unit in P kg<sup>-1</sup> was used in 80% of the studies, representing a very clear trend, but sometimes this unit was applied by upscaling from a significantly smaller sample (Fig. S5). In contrast, some studies expressed their results only in units relevant to the sample mass that was analyzed, e.g., in units of P/g, P/5 g, P/30 g, or P/100 g (Fig. S5). Obviously, this makes a direct comparison of MPs content in soils challenging. The most frequent and abundant polymers found in soil were polyethylene (PE; including low and high density PE) > polypropylene (PP) > polystyrene (PS) > polyethylene terephthalate (PET) > polyvinyl chloride (PVC) (Fig. 5C). Thus, it is recommended to call these polymers the `common five' based upon their ubiquitous distribution in soil. This idea arises to some extent from the analogy of the 'dirty dozen', a group of persistent organic pollutants defined by the Stockholm Convention in 2001 based upon their toxicity and persistence

in nature. Among the reviewed studies, a total of 51 further polymers were identified in soils, which highlights the very heterogeneous polymer fingerprint and poses a challenge to future analytical quantification procedures (Fig. 5C). However, a careful decision is mandatory to prioritize upon target polymers to the studied areas while exclusion of additional polymers possess the risk to exclude polymer stressors that are not yet in the focus.

Another aspect that needs to be evaluated and optimized in the future is that soils have variable background contents of MPs. Particle counts for soils containing MPs primarily derived from aeolian processes had a significantly lower background, with  $0.42 \pm 0.33$  P kg<sup>-1</sup>, than soils from urban environments, with a value of  $6,176 \pm 6,481$  P kg<sup>-1</sup> (Fig. 1E). Low MPs masses and numbers with a µm size range will probably be below the limits of detection or proper identification, due to the resolution limit of a microscope or image analysis methods. Large projects and initiatives, such as the EUROpean quality Controlled Harmonization Assuring Reproducible Monitoring and assessment of plastic pollution (EUROqCHARM) framework, currently aim to determine reproducible RAPs [2]. This is key for making a comparison between interlaboratory results obtained from different settings more feasible. Overall, focusing on a particular group of polymers, e.g., the `common five' suggested here, will give a reasonable overview of the MPs pollution of a particular study site. These polymers occurred in more than 50% of the studies worldwide and correspond to benchmark polymers in soil (Fig. 5C); their prevalence is similar with the order of the most produced polymers in terms of market share [15].

#### **Conceptualized recommendation**

Various sampling designs as well as missing basic information and harmonized methods render it still challenging to compare the results of studies reporting on MPs occurrence in soils. In general, upscaling approaches must be applied very carefully due to the heterogeneity within soil samples, highlighting the demand for larger and more representative sample masses. A conceptualized workflow of groups proposed within this structured review and suggestions to enhance the reproducibility of sampling for MPs analysis in soil are given in Fig. 7. This can serve as a roadmap describing the important technical information and includes calculated metrics of the reviewed studies (e.g., sample mass for DS) to support a reproducible and representative sampling strategy for MPs analysis in soil. From our perspective, the key factors that should be elucidated in the future to enhance a more reproducible and representative study design are the following:

 Input pathway: MPs enter soil by various pathways, and each source features a distinct fingerprint (size class, shape and polymer composition). A coherent historic survey of possible applications of, e.g., biosolids (timeline, number of applications, and mass



Fig. 7 Proposed recommendations to determine the abundance of MPs in soil. The text in the colored boxes describes recommended items that should be elucidated in MPs studies, while text in bold denotes major findings derived from this structured review

of applied material), is mandatory to put the findings into context.

- Study site: Soil classification and properties should be stated to allow interstudy comparisons. In particular, knowledge about the spatial distribution of MPs for a given soil unit and across various scales (horizon, pedon, field, landscape) is necessary for soil-tailored guidelines on how to sample and analyze MPs. A simple and valuable tool for providing sampling site conditions is a study site map, which should optimally include GPS coordinates to allow use of the results in the future for geostatistical modeling.
- Sampling design: Knowledge of the spatial distribution of MPs is needed to evaluate the representativeness of the samples. It is necessary to determine the required representative elementary volume (REV) the smallest volume for which a measurement can be made and that is characteristic of the complete volume under consideration. Identification of REVs suitable for MPs research would minimize errors associated with erroneously upscaling particle counts or polymer masses from a small subvolume up to the pedon or landscape level to identify the "true value".
- Sample preparation: MPs must be isolated from the solid matrix by various approaches, including manual extraction, electrostatic separation, and/or the use of density solutions. Purification and removal of SOM is a mandatory step for subsequent analytical methods. While large microplastics can be manually removed from the soil for further analysis, reproducible analytical sample preparation steps are mandatory, especially with decreasing MPs particle size.
- Analytical methods: Thermogravimetric methods should at least state a limit of detection and integrate analysis of sample blanks. Spectroscopic methods should mention both the upper and lower particle size that can be properly identified as MPs. Recovery tests are important and valuable tools to validate the applicability of individual protocols, but only if supplemented certified reference materials – adapted according to the MPs characteristics of the emission source and the analytical method – become available to the public along with guidelines about the best practices for performing these tests. The reproducibility of recovery tests is a cornerstone in good scientific practice and helps to validate the accuracy of reported MPs data.

#### Conclusions

The steadily increasing number of reports and data on MPs in soils using a variety of methods highlights the need for representative sampling strategies, reproducible sample preparation techniques, and harmonized analytical methods to improve the comparability of results. A main consideration to enhance the usefulness and reliability of surveys on MP contents in soil is the extent of technical information supplied. It is obvious that some criteria were well described throughout the literature, while other information was simply not given or was even not considered in the study design at all. For instance, information about the land use at the study site, the analytical methods applied to derive MPs particle counts or polymer masses, and the sampled soil depth were missing in fewer than 10% of the reviewed studies. In contrast, information about sample homogenization procedures in the field and in the lab prior to the analysis were missing in most cases. In conclusion, extracting or concentrating a solid and non-water soluble polymer from a solid matrix such as soil remains challenging, and the recovery rate depends crucially on the applied methods and the matrix, hence the soil properties. It is even more surprising that soil classification was missing in most of the reviewed studies. In addition to information on the study site, information on soil properties would strongly benefit future method development in terms of determining how to addresses challenges in extracting MPs from the solid matrix, such as the reproducibility of recovery rates and interferents during analysis. To better understand spatial and vertical MPs distributions in soil, the integration of geostatistical and modeling approaches is needed. Currently, such integration is employed at larger scales (e.g., province or state), but a sampling design based on higher-resolution data at the field scale or at the even smaller pedon scale is urgently needed. Systematic analytical quality control and use of reference materials are mandatory to improve the scientific merit of the analysis. Overall, this structured literature review proposes a conceptualized guideline with important considerations to close knowledge gaps and thus enhance the reproducibility of MPs analysis in soils.

Abbreviations	
μFTIR	Micro Fourier transform infrared spectroscopy
ATR-FTIR	Attenuated total reflection Fourier transform infrared
	spectroscopy
DS	Density separation
EUROqCHARM	EUROpean quality Controlled Harmonization Assuring
	Reproducible Monitoring
MPs	Microplastics
Р	Particles
PE	Polyethylene
PET	Polyethylene terephthalate
POM	Polyoxymethylene
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl chloride
Pyr-GC–MS	Pyrolysis gas chromatography–mass spectrometry
RAP	Reproducible analytical pipelines

Representative elementary volume
Scanning electron microscopy and energy dispersive
X-ray diffraction
Soil organic matter
Standardized operation procedures
World Reference Base

#### **Supplementary Information**

The online version contains supplementary material available at https://doi. org/10.1186/s43591-023-00063-5.

Additional file 1: Figure S1. Articles per year addressing MPs research for the media soil, air, and water (A) and magnification of the development trend over time (B). Figure S2. Metadata of research articles that investigate MPs in soil. Figure S3. Metrics about procedures regarding sampling time (A), number of sampling depths under investigation per article (B), number of samples investigated per article (C), number of single samples (D), the number of samples merged into composite samples (E), and the final mass of each composite sample (F). Figure S4. Metrics for the density separation (DS) procedure and soil organic matter (SOM) removal by application of hydrogen peroxide. Figure S5. Most common units employed for the 106 articles within this structured review. Table S1. Overview of groups and categories of sampling strategies for MPs research. Table S2. Summary statistics of groups and categories of sampling strategies for MPs research. For each category, the data were calculated from the raw data (white background) and from processed data by outlier removal (gray background). Outliers were identified by the interquartile range (IQR: 1.5 times higher than the 75<sup>th</sup> and 1.5 times lower than the 25<sup>th</sup> percentile of the data). IQR data are discussed in this manuscript.

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#### Authors' contributions

KD performed the structured review and analyzed the data. KD wrote the initial draft of the manuscript with support from MH, DR, EF. All authors provided critical feed-back and helped to shape the manuscript in the present form.

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#### Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

#### Declarations

#### Ethics approval and consent to participate

Not applicable.

#### **Consent for publication**

All authors approve the manuscript and give their consent for submission and publication.

#### Competing interests

The authors declare no competing interests.

#### Author details

<sup>1</sup>Federal Institute for Geosciences and Natural Resources (BGR), Stilleweg 2, Hannover 30655, Germany. <sup>2</sup>German Environment Agency (UBA), Wörlitzer Platz 1, Dessau-Roßlau 06844, Germany. Received: 15 February 2023 Accepted: 27 June 2023 Published online: 13 July 2023

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