Quality Assessment of Research Studies on Microplastics in Soils: A Methodological Perspective

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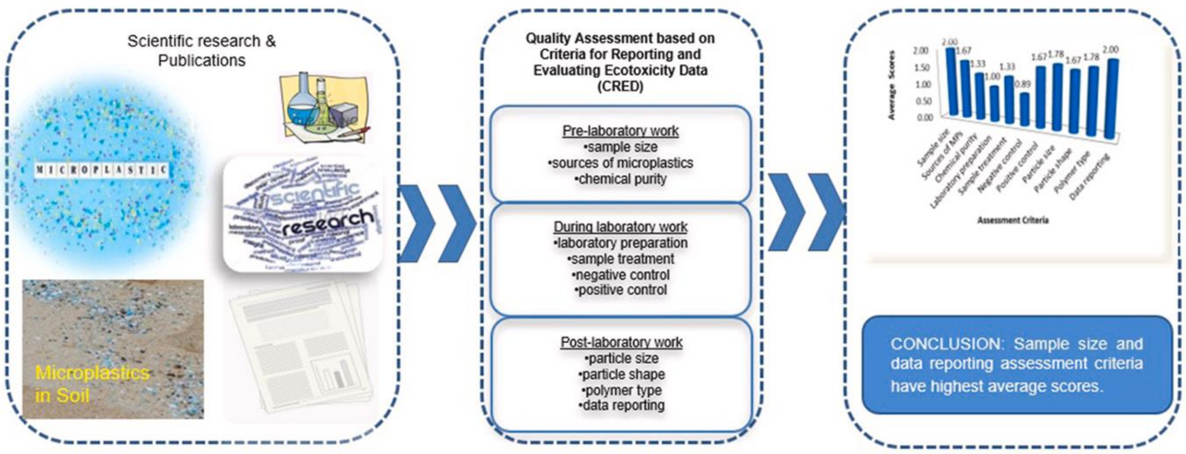
Highlights

- Quality assessment was conducted by using CRED evaluation criteria.
- A total of 11 criteria were quantitatively evaluated.
- Sample size and data reporting criteria achieved the highest average scores.
- Negative control has the lowest average score of 0.89.
- Quality assurance for soil microplastics studies can be further improved.

Graphical abstract



Abstract

Microplastics have become a global concern, and soil acts as a major sink for plastic pollution. Due to rapid development of soil microplastics research, various analysis methods have been developed, but require proper consistency and standard procedures. The objective of this study was to appraise a quality assessment concerning soil microplastics from a methodological perspective. Nine studies were selected for the quality assessment exercise based on methodological investigations on soil microplastics and were evaluated based on the adapted Criteria for Reporting and Evaluating Ecotoxicity Data (CRED) method. The highest score obtained by an individual study was 21 while the lowest was 14, leaving a wide score gap which indicated inconsistency amongst the studies. Criterion with the highest average score of 2.0 was obtained for sample size and data reporting. The lowest average score of 0.89 was for the negative control. In conclusion, the total average scores for all eleven criteria were 1.56. Current quality assessment perceived that there was room for improvement and betterment of quality assurance for studies on microplastics and a form of guideline on methodological aspects of soil microplastics studies. It was suggested that future microplastics studies should methodically include quality assurance/ quality control (QA/QC) protocols in every process to ensure that good quality data is produced and applied in the risk assessment process.

Keywords: microplastics, soil, quality assessment, CRED method

1.0 Introduction

The outspread of sewage sludge, usage of plastic mulches, and land irrigation are sources of microplastics contamination in soil (KAUR et al., 2022; Tian et al., 2022). Due to low degradation rate, microplastics have tendency to adsorb toxic chemicals and be ingested erroneously by various organisms that are habituating in soil (Campanale et al., 2020). Contaminated microplastics in soil have high possibility of being transferred to humans, causing threat to human health as microplastics are eventually passed on to animals and become bio-accumulated through the food chain (Elizalde-Velázquez and Gómez-Oliván, 2021; Zhou et al., 2021). Reports have also suggested that nanoplastics, which are potential products of microplastic weathering in soil, could be taken up by various plant species such as *Arabidopsis thaliana* (Sun et al., 2020) and wheat (Li et al., 2020). These nanoplastics will be redistributed in roots, stems, and leaves, resulting in additional risks of direct human exposure to plastic contaminants in human bodies via food (Wu et al., 2021; Xiang et al., 2022; Zhou et al., 2021).

Until now, density separation is a common method for extracting microplastics from soil as this method differentiates the density between polymers and separation (Lastovina and Budnyk, 2021; Radford et al., 2021; Sridhar et al., 2022). Although this method is reliable and rapid, it cannot separate microplastics of very high density because the heavy plastic particles along with other soil components may divide in the same separating phase (Cutroneo et al., 2021; Stile et al., 2021). The oil extraction technique exploits the oleophilic properties of microplastics, whereby the oil encapsulates the microplastics for easier extraction. However, further identification is limited as oil traces could not be eliminated (Scopetani et al., 2020). Other techniques include a separator to isolate microplastics from solid samples through electrostatic charges (Felsing et al., 2018) and the pressurised fluid extraction method by using solvents (Fuller and Gautam, 2016). However, these techniques have not been experimented further, resulting in uncertainty about reproducibility. The inconsistency amongst adopted methodologies face challenges in comparing various investigations, causing inefficient approximation of microplastics occurrence in soil (Jiao et al., 2021; Mári et al., 2021). Moreover, the methods and effects of extraction treatments on microplastics are seldom evaluated and reported for their efficiencies (Yang et al., 2021). Until now, various methodologies for extracting and examining soil microplastics were reviewed (Möller et al., 2020; Ruggero et al., 2020; Thomas et al., 2020; Zhang et al., 2020; Zhou et al., 2020). These reviews focused on recovery of microplastics based on individual experimental sampling, solutions, digestion, and extraction techniques. However, these studies lack in quality assessment of methodologies which involve soil microplastics.

Methodological aspects quality assessment of microplastics was reported in water studies (Koelmans et al., 2019), aquatic biota samples (Hermsen et al., 2018a) and bottled water (Praveena and Laohaprapanon, 2021). But reports on soil microplastic studies are not available. Various studies that involved methodology developments for microplastics analysis in soil have a high possibility to differ in the degree of quality assurance deployed, causing debate on the quality of microplastics findings (Cowger et al., 2020). Without the implementation of a proper quality assurance assessment throughout the analysis, it will result in lower quality of microplastic concentration data in the environment, leading to ineffective risk assessment and decision-making (Brander et al., 2020).

Quality assessment of methodological aspects for environmental studies comprises protocols, such as the Klimisch method, European Commission's Technical

Guidance for Deriving Environmental Quality Standards (TGD), and Criteria for Reporting and Evaluating Ecotoxicity Data (CRED) (EU, 2018; Klimisch et al., 1997; Moermond et al., 2016). The Klimisch method provides structure of procedures for assessment on reliability of studies via classifications. The Klimisch method is subjected to limitations of criteria for evaluation without specific guidance for relevant evaluation, leading to result inconsistencies. Additionally, this method does not guarantee enough reliability and relevance of the outcomes. Moreover, conflicting evaluation results influence the outcome of assessment, and thus affect the quality of studies (Ågerstrand et al., 2011; Tweedale, 2010). The European Commission's Technical Guidance for Deriving Environmental Quality Standards (TGD) lacks comprehensive information on the evaluation of reliability and relevance of studies, causing evaluations to depend on expert judgement and leading to many disagreements amongst assessors (Kase et al., 2016). The Criteria for Reporting and Evaluating Ecotoxicity Data (CRED) is an improved scientific method which provides more meticulous and transparent assessments of reliability and relevance of studies, ensuring that any data is not disregarded without clear justification (Kase et al., 2016). The CRED method provides assessors with more systematic assessment with direct and detailed instructions on method evaluation of the studies, resulting in the discovery of more weaknesses in the representation, performance, analysis and reporting of the studies (Moermond et al., 2016). Assessors are granted room for thorough discussions on the focused strengths and weaknesses of a study, as opposed to the fixed criteria of the Klimisch method (Kase et al., 2016). So far, CRED method has been well adapted for methodological aspects of microplastic studies which involve marine biota (Ruijter et al., 2020), aquatic biota (Hermsen et al., 2018a) and drinking water (Koelmans et al., 2019).

The objective of this study is to review the methodological aspects of microplastics in soil by using the adapted CRED method, focusing on 11 criteria which are divided into three main groups, namely pre-laboratory work (sample size, sources of microplastics, and chemical purity), during laboratory work (laboratory preparation, sample treatment, negative control, and positive control) and post-laboratory work (polymer size, polymer shape, polymer type, and data reporting). The current quality assessment study brings significance as it will indicate the areas of strength and weakness that need improvement, particularly in the analytical procedure aspects, such as sampling, sample treatment, use of controls, polymer identification and data reporting. This leads to steps that ensure high data quality and a foundation for standardisation of methodologies for future soil microplastics research.

2.0 Methodology

Literature was retrieved from the databases of Elsevier, Web of Science, Scopus, and Google Scholar. Extensive literature search for studies on microplastics in soil was performed until February 2021, focusing on methodology aspects for quality assessment on method development. Queries included the following search terms: “microplastics AND extraction AND soil”, “microplastics AND separation AND soil”, “microplastics AND identification AND methods”, “microplastics AND soil AND methodology”. There were 388 papers retrieved and screened. A total of 216 papers were excluded as they did not meet the study requirements. The remaining 172 full text papers were assessed for eligibility and 163 papers were excluded as they were out of scope. A total of 9 studies were selected as these studies were involved in method development for isolation and identification of soil microplastics. The nine studies were selected and included in the quality assessment from a methodological perspective by using CRED (Supplementary 2). Figure 1 provides a detailed summary on the methodology flow from literature search until data analysis.

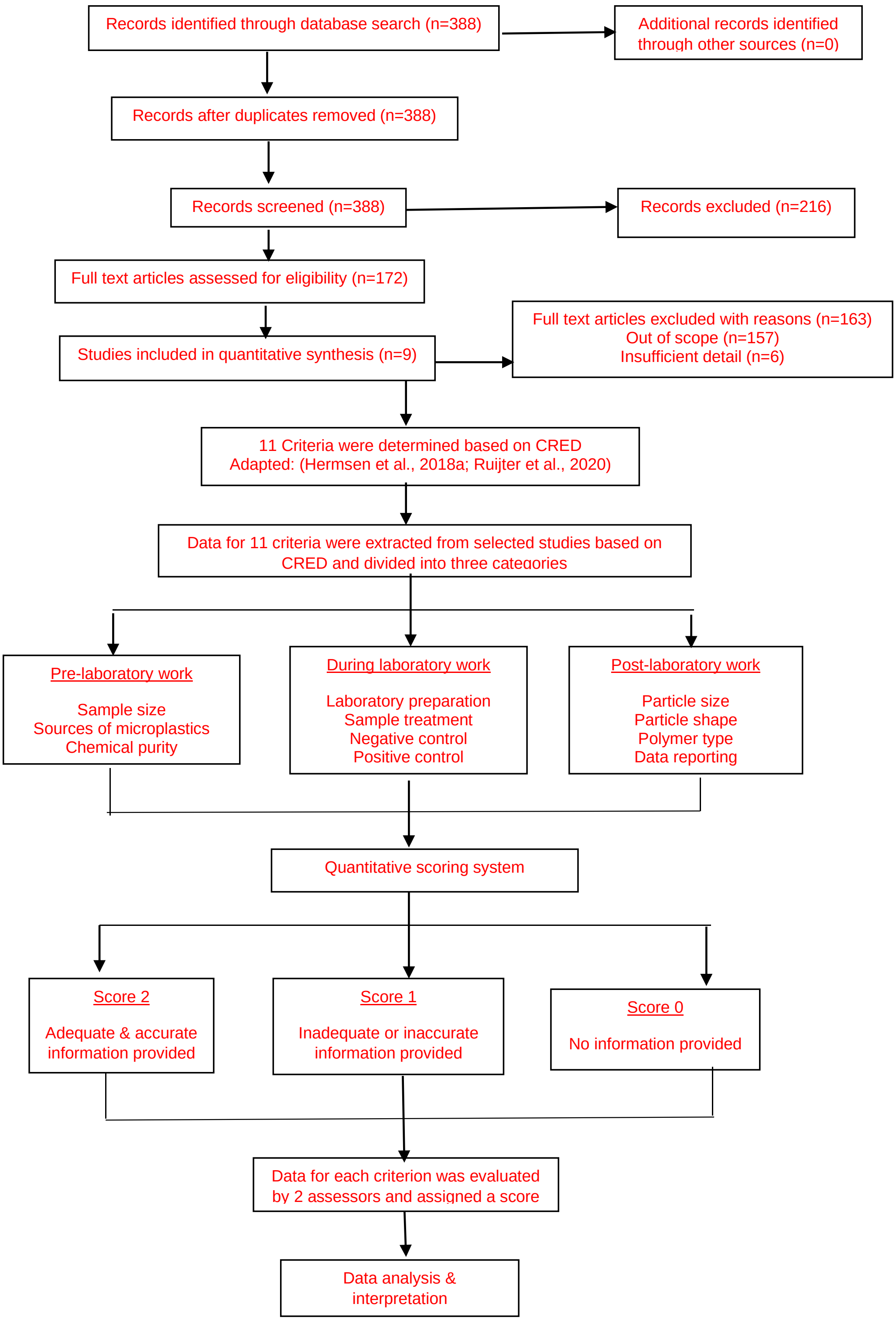


Figure 1. Flowchart of the methodology from literature search until data analysis

The selected nine studies were evaluated based on 11 quality assurance/quality control (QA/QC) criteria subjected to the CRED method and were adapted from studies by Hermsen et al. (2018a) and Ruijter et al. (2020). Quality assessment was done for the following three categories: pre-laboratory work (sample size, sources of microplastics, and chemical purity), during laboratory work (laboratory preparation, sample treatment, negative control, and positive control) and post-laboratory work (particle size, particle shape, polymer type, and data reporting). Supplementary 1 describes the proposed quantitative scoring system to evaluate the studies for extraction and identification of microplastics in soil by using the quality assessment criteria. Each study was accumulated as part of this literature review and was independently evaluated with scores by two assessors, in due course tabulated and thoroughly discussed to reduce potential ambiguities. The data on average scores for respective criteria were further analysed, paving ways to discuss the findings. The assessment criteria were mainly implemented in this study to generate a comprehension pertaining to the improvement of investigation methods for microplastics research in soil.

3.0 Results and Discussion

3.1 An overview on methodologies

A total of nine studies were selected in this review, in which three studies extracted microplastics from soil by using the density separation method (Han et al., 2019a; Li et al., 2021, 2019). Studies by Mani et al. (2019) and Scopetani et al. (2020) experimented the oil-based extraction method while Felsing et al. (2018) used the electrostatic method to extract microplastics from soil. A study by Fuller and Gautam

(2016) used the mechanical method by extracting microplastics with pressurised fluid extraction (PFE). Similarly, Liu et al. (2019) used the mechanical (circulation) method for the same purpose. The heating method was used for extraction of soil microplastics by Zhang et al. (2018).

Table 1 shows each study criterion which had assigned score of either 2 (adequate), 1 (adequate with restrictions), or 0 (inadequate) for all nine studies. The maximum total score for the 11 criteria based on each study was 22 and based on each criterion was 18. It was stressed that the scores provided for every study should not be taken as a perception indicative of the relative value of the study. A study scoring low on a certain criterion is still possible to provide valuable findings and knowledge on microplastics in soil. The detailed information per individual study is provided in Supplementary 2.

Table 1. Individual scores for each criterion based on methodological aspects of soil microplastics

References	Sample size	Source of microplastics	Chemical purity	Laboratory preparation	Sample treatment	Negative Control	Positive Control	Particle size	Particle shape	Polymer type	Data reporting
(Li et al., 2021)	2	2	2	0	1	1	2	2	1	1	2
(Scopetani et al., 2020)	2	1	1	1	2	1	2	2	2	2	2
(Liu et al., 2019)	2	2	1	0	2	1	2	2	2	2	2
(Li et al., 2019)	2	1	1	2	1	0	0	2	2	2	2
(Zhang et al., 2018)	2	2	1	1	1	0	2	2	2	1	2
(Felsing et al., 2018)	2	1	1	2	2	0	2	1	2	2	2
(Fuller and Gautam, 2016)	2	2	1	0	0	2	1	1	1	2	2
(Mani et al., 2019)	2	2	2	2	1	2	2	2	2	2	2
(Han et al., 2019a)	2	2	2	1	2	1	2	2	1	2	2

3.2 Quality assessment of selected studies

3.2.1. Pre-laboratory work criteria

All nine selected papers provided weight of soil sample size between 10 g and 200 g, and thus scoring a maximum score of 2 (Table 1). Studies by Fuller and Gautam (2016), Mani et al. (2019), Scopetani et al. (2020) and Zhang et al. (2018) reported on sampling of 10 g soil, which was the lowest sample size amongst all the selected papers, while Han et al. (2019a) reported that 200 g was the highest sample size. All selected studies obtained more than 90% recovery of microplastics, demonstrating that the range of 10 g - 200 g sampling sizes were efficient for the developed methodologies. Studies have reported that a smaller sample size such as 10 g was enough when specifically investigating particles which are meticulous to detect, suggesting that smaller particles (< 500 µm) were more ample (Cabernard et al., 2018; Hermsen et al., 2018a; Ruijter et al., 2020; Zhang et al., 2018). However, a smaller sample size (<10 g) decreased the possibility of recovering particles, leading to a reduction of the strength of study while increasing the error margin (Koelmans et al., 2019). Although extremely low sample sizes provide interesting data, it does not provide solid conclusions as statistical strength would be too low to concur a trend. A larger sample size will provide reliable findings as well as narrow the confidence intervals (Hermsen et al., 2018a). Therefore, it is advisable to provide an adequate sample size based on intent and method of study to enhance the mean result and reliability of study (Hermsen et al., 2018a).

Sources of microplastics refer to specification on the root sources of microplastics, whether bought or self-made, which maximises the reproducibility, and should be reported. Reproducibility of experiments is possible when information on microplastic materials is provided in detail, which undoubtedly influences findings of particle size, shape, and polymer type (Brander et al., 2020). A total of six studies obtained scores of 2 as detailed information on origins of microplastics, density and other particulars were provided (Fuller and Gautam, 2016; Han et al., 2019a; Li et al., 2021; Liu et al., 2019; Mani et al., 2019; Zhang et al., 2018). Three studies furnished incomplete information on the sources of microplastics, and thus scored a minimal score of 1 (Felsing et al., 2018; Li et al., 2019; Scopetani et al., 2020). Studies by Fuller and Gautam (2016), Han et al. (2019a), Li et al. (2021), Liu et al. (2019) and Zhang et al. (2018) have utilised manufactured microplastics such as polyethylene, polyvinylchloride, and polyethylene terephthalate. The outcomes of the assessment presented that most studies preferred to use manufactured microplastics over self-made ones. Manufactured microplastics such as low-density polyethylene (LDPE), high density polyethylene (HDPE), polyvinyl chloride (PVC), nylon, Teflon and thermoplastic polyurethane (TPU) decrease the possibility of non-uniformity in shape and size as the microplastics are of industrial grade and mechanically manufactured (Freile-Pelegrín and Madera-Santana, 2017). When solid polymers undergo the process of heating and moulding, polymers become hard and infusible, making them more steadfast (Bass et al., 2020). Self-made microplastics were used in studies by Felsing et al. (2018) and Scopetani et al. (2020). Self-made microplastics were prepared by manually shredding and cutting various common plastic products such as water bottles, yogurt bottles, plastic spoons, polyvinylchloride pipes, Styrofoam packaging material, and plastic strainers (Han et al., 2019a; Mani et al., 2019). The advantage of self-made microplastics is that the sources are easily available and come in various colours depending on the source, making identification easier (Hahladakis et al., 2018). However, the limitation is that there is possibility of non-uniformity in shape and size of the microplastics. A maximum of 10 types of microplastics (HDPE, LDPE, PET, PP, PS, PVC, PMMA, PLA, polyethylene fibres, and self-made tire wear) were used for soil spiking by Felsing et al. (2018) and Liu et al. (2019), and a minimum of two microplastic types (LDPE, PP) by Zhang et al. (2018).

Chemical purity is an essential criterion for quality assessment of microplastic studies. (Han et al., 2019a; Li et al., 2021; Mani et al., 2019) documented full details on chemical origin, brand, grade, and other information leading to a maximum score of 2. Assessment presented six studies received a score of 1 as insufficient detail on chemicals were reported (Felsing et al., 2018; Fuller and Gautam, 2016; Li et al., 2019; Liu et al., 2019; Scopetani et al., 2020; Zhang et al., 2018). All the selected papers utilised chemicals for sample treatment and microplastics extraction, except for study done by Zhang et al. (2018) which used distilled water for this purpose. Fuller and Gautam (2016) used solvents of high purity to extract microplastics. However, the concentrations of solvents were not reported, and thus making it difficult to be experimented further. Chemicals of high grade are known to be the purest of chemicals and contain the least number of impurities (Abdin et al., 2020). The grades of chemicals decrease as the level of impurities increase. Impurities are matters such as water and trace metals in a confined chemical stage which vary from the respective chemical composition of that phase (van Brakel, 2014). These impurities have possibilities of causing reactions that alter the property or characteristics of microplastics and affect the recovery level. Furthermore, corrosive chemicals affect the outcomes of studies as they are capable of digesting or causing surface degradation of microplastics (Chamas et al., 2020). Significant chemical alterations such as oxidation and chain scission result in reduction of molecular weight and magnitude of polymerization of the polymers (Chamas et al., 2020). Karami et al.

(2017), investigated into chemical contaminants pertaining to microplastics. Nevertheless, chemical effects still could not be eliminated from experimental findings. Unfortunately, chemical contaminants that were present in the microplastics were overlooked. Most studies were unable to differentiate between possible microplastics toxicity and chemical toxicity that caused adverse effects (Hwang et al., 2020; Ruijter et al., 2020; Wang et al., 2021).

3.2.2 During laboratory work criteria

Contamination during laboratory preparation is a prevailing phenomenon during microplastics studies, resulting in unreliability in the outcomes of many studies. Various steps were taken to prevent contamination during sampling, treatment, and analysis in microplastic investigations. A score of 2 was assigned when cotton clothes and cotton laboratory coats were worn; distilled or ultrapure water was used for cleaning and chemical preparation purposes, alongside usage of nitrile gloves, including cleaning of laboratory surfaces and equipment. A score of 1 was assigned when only a part of the measures was taken to avoid microplastics contamination or it was generally mentioned. Studies by Felsing et al. (2018), Li et al. (2019) and Mani et al. (2019) obtained a score of 2 while a score of 1 was assigned to papers by Han et al. (2019a), Scopetani et al. (2020) and Zhang et al. (2018). Three of the selected papers had limitedly mentioned the form of contamination prevention as the study focus was mainly on good extraction precision of microplastics (Fuller and Gautam, 2016; Li et al., 2021; Liu et al., 2019). Commonly, contamination from microfibre stems from clothing of researchers (Hermsen et al., 2018a). Natural fibre clothing such as 100% cotton attire and laboratory coats enables prevention of this contaminant.

Additionally, strict precautions were taken by sanitising surfaces, tools, and equipment with alcohol. However, the sanitisation method may not be rigorous to eliminate contamination, and thus meticulous washing and rinsing of laboratory tools and apparatus were considered a good alternative. Studies by Li et al. (2019), Mani et al. (2019) and Zhang et al. (2018) took further steps for contamination control by covering glassware and apparatus with aluminium foil to avoid air borne contamination from microplastics in the atmosphere. The usage of sampling apparatus and laboratory equipment should be made from glass or metal instead of plastic. When usage of plastic materials cannot be avoided, it is advisable to run procedural blanks to quantify and rectify the addition of plastics from the equipment. More so, it is of utmost importance to take appropriate storage measures of the equipment which can be possibly contaminated by atmospheric deposition. The efficacy of cleanliness and storage protocols may be periodically examined through stereoscope and procedural blanks (Brander et al., 2020). Distilled water or ultrapure water was used for cleaning and chemical preparation. Nitrile gloves were also used during microplastics investigations by Mani et al. (2019). Nitrile gloves are manufactured to be more resistant to solvents and chemicals and possess ability in breaking up electrostatic charges, which can reduce contamination in the work environment (O'Connor et al., 2020).

Sample treatment is essential as microplastics have resemblance to organic matter in soils due to similar density concentrations, which interfere in the isolation and identification of microplastics (Radford et al., 2021). Therefore, treatment of sample was required to eliminate organic matter from spiked soil samples. A score of 2 was assigned when solution details and method used was presented with reference, while a score of 1 was assigned when information was limited. Four studies received a score of 2 for reporting the method used with references (Felsing et al., 2018; Han et al., 2019a; Liu et al., 2019; Scopetani et al., 2020), while studies by Li et al. (2021, 2019), Mani et al. (2019) and Zhang et al. (2018) obtained a score of 1 due to lack of method references. Five of the selected papers had preference in using hydrogen peroxide for the digestion of organic matter through the oxidation method (Han et al., 2019a; Li et al., 2019; Liu et al., 2019; Mani et al., 2019; Scopetani et al., 2020). Hydrogen peroxide, when used at lower temperatures (up to 60 °C) proved to be a good and effective chemical agent in the digestion process due to very little polymer degradation and little effect on integrity in polymers (Al-Azzawi et al., 2020; Prata et al., 2019). The selected studies carried out the digestion method with spiked soil samples by using various types of soil such as farmland, paddy, floodplain, yellow brown, agricultural and oat field, resulting in optimum findings. Zhang et al. (2018) used the heating method to differentiate between microplastics and impurities in spiked clay soil, loess soil and sandy soil that contained organic matter of 3.23%, 4.2%, and 7.4%, respectively. Microplastics transformed into transparent, circular, and shiny particles when exposed to temperatures of 130 °C for 3 s - 5 s. The melting point of LDPE and PP was 115 ˚C – 135 °C and 130 ˚C – 171 °C, respectively. If the temperature was too high or the heating time was too long, the properties of the melted microplastics such as transparency, circular form and shine would not be observed (Zhang et al., 2018). Heating is usually part of the sample treatment process. This is carried out to speed up the treatment process especially for digestion of organic matter. Even so, the heating process can be detrimental as some microplastics can be distorted (Hermsen et al., 2018a). Additionally, Felsing et al. (2018) measured the total organic carbon (TOC) content from sediment and sand by acidifying freeze dried samples with 1M hydrochloric acid for 3 h – 4 h and analysing with a carbon analyser.

The use of negative controls in microplastics studies pertaining to method development is a growing standard practise. Possibilities of contamination by microplastics fibres and particles are high, especially during spiking the samples and handling, treatment, and analysis. Therefore, it is extremely critical to utilise controls in parallel to spiked samples (Brander et al., 1965; Hermsen et al., 2018a). Negative controls are essential to determine secondary contamination (Koelmans et al., 2019). Negative controls should not contain any microplastics. A common practice in running a negative control is to expose a wet filter paper in a petri dish at the work area during sampling, processing, and analysis in the laboratory. The moist filter paper is then analysed for microplastics by using microscopy and spectrographic methods together with environmental samples. Another method to evaluate microplastics contamination during analytical techniques such as digestion process, is to run an empty beaker with reagents used (acid, alkali, oxidants, and catalysts) parallel to digesting soil samples (Brander et al., 2020). A score of 2 was assigned when soil blanks for each batch of spiked samples with triplicates were included. Controls should be given the same full treatment as the studied spiked samples. Studies by Fuller and Gautam (2016) and Mani et al. (2019) scored a total of 2 as the studies had run blanks with triplicates. Mani et al. (2019) reported absence of microplastics in the blanks. However, Fuller and Gautam (2016) detected an average content of 0.09 mg microplastics. This could be due to possibility of the plastics being incorporated into the methanol, hexane, and dichloromethane solvents, and thus resulting in greater than 100% recovery (Fuller and Gautam, 2016). A score of 1 was given when blank soil sample was included. Nevertheless, deemed insufficient if less than three replicates. Four studies received a score of 1 (Han et al., 2019a; Li et al., 2021; Liu et al., 2019; Scopetani et al., 2020). These studies had run blanks parallel with spiked samples to check for potential source of contamination, but number of replicates was not mentioned. The rest of the selected papers obtained a score of 0 for lack of information (Felsing et al., 2018; Li et al., 2019; Zhang et al., 2018). Studies scored 0 when no form of negative control was included in the study.

Positive controls were carried out to confirm whether microplastics found in samples were accurately recovered during the isolation procedure (Dehaut et al., 2019). Positive controls, also known as spiked recovery, were artificial samples that wee spiked with known microplastics particles and given the exact treatment as unknown samples (Brander et al., 2020). The particle recoveries were calculated by tallying the numbers of retrieved particles to the amounts added. Positive controls must be run for selected microplastics, enclosing various polymer types and sizes. Polymer sizes cover a wide range, and thus it must not be taken for granted that recovered microplastics sustained a constant range of sizes and polymer types. Therefore, it was important to use significantly small microplastics as controls due to difficulty in recovering them (Koelmans et al., 2019). A score of 2 was assigned when studies included positive controls in triplicates with added known microplastics and were treated in parallel to the samples. Studies by Felsing et al. (2018), Han et al. (2019a), Li et al. (2021), Liu et al. (2019), Mani et al. (2019), Scopetani et al. (2020) and Zhang et al. (2018) had run positive controls with three or more replicates, and thus obtaining a score of 2. (Liu et al., 2019) carried out three parallel experiments with three replicates for experimental groups. Studies that report less than three control replicates were assigned a score of 1, while a score of 0 was given when no positive controls were reported. A study by Fuller and Gautam (2016) reported limited information on replicates, resulting in a score of 1. Additionally, Li et al. (2019) scored a 0 due to limited information with only a mention that control samples were used. To validate the newly developed methods, it was essential to quantify the losses by using positive controls to rectify and report insufficient recovery. For rectification purposes, the differential findings detected in the positive controls were eliminated from the findings in experimental samples. The outcome of positive controls was accounted for establishment on the performance of the laboratory-based methods.

3.2.3 Post - laboratory work criteria

Particle size is a factor which defines the effects of developed methodologies on microplastics (Hermsen et al., 2018a). The before and after comparison of particle size should be reported to ensure that the spiked microplastics were chemically unaltered during treatment and extraction processes (Mani et al., 2019). Some methods included possibility of plastic altering procedures, such as ultra-sonication and acidic or alkaline purification. Therefore, it was essential to provide a procedure which was non-destructive in nature (Bergmann et al., 2015; Claessens et al., 2013). A score of 2 was assigned to studies when before and after particle size ranges were reported with unit measurement. Studies by Han et al. (2019a), Li et al. (2021, 2019), Liu et al. (2019) Mani et al. (2019), Scopetani et al. (2020) and Zhang et al. (2018) obtained a score of 2 due to providing details of particle sizes before and after spiking. The study by Zhang et al. (2018) used LDPE particles sized <150 µm and PP particles sized <400 µm, which resulted in after-spike particle sizes being close to the original size ranges of 100 µm – 250 µm and >250 µm. Han et al. (2019a), Li et al. (2021) and Mani et al. (2019) experimented with microplastics with a size of <1 mm. However, all three studies reported no change in particle size of before and after spiking which provideed notion that the developed methodologies had not affected the sizes of microplastics. A study by Liu et al. (2019) used microplastics of 0.03 mm – 4.76 mm in size with a majority of after- spike recovery of 53.6% with <1 mm particle size. Polyethylene microplastics were classified into three size categories of 100 µm – 500 µm, 500 µm – 1000 µm and 1000 µm – 3000 µm, providing a 100% recovery for sizes >1 mm but a lower mean of 75.0% - 96.7% for sizes between 100 µm – 500 µm. The study by Scopetani et al. (2020) had experimented with microplastics of sizes 0.2 mm – 2 mm, with retrieval of after -spike particles as small as 5 µm – 300 µm, concluding that the olive oil-based extraction method works well for retrieval of smaller microplastics. A score of 1 was assigned to studies when before and after spike particle size ranges were not reported with unit measurement. Studies by Felsing et al. (2018) and Fuller and Gautam (2016), scored a 1 as the after-spike particle size was not reported. Fuller and Gautam (2016) used a much smaller size of 50 µm of powdered microplastics, facing limitations in detecting the after spiking microplastics due to inability to measure size fractions of microplastics in samples. It was perceived that the verification of smaller size microplastics from samples remained to be tough by using available equipment. The present technology such as micro-Fourier transformed infrared (µ-FTIR) spectrometry enables microplastics verification of particles < 10 µm. However, such technological systems must be improved and be readily available to researchers (O'Connor et al., 2020).

The gauging criterion pertaining to categorising of microplastics particle shape is an important factor in determining and interpreting effects of microplastics (Ruijter et al., 2020). The before and after observation of particle shape allows determination whether shapes influence the isolation and identification processes in microplastics studies. It is essential to incorporate measurements of shape by using some form of high-resolution microscope to illustrate complete microplastic characterisation (Ruijter et al., 2020). Studies that reported shapes with measurements by using high resolution microscope were assigned a score of 2. Studies by Felsing et al. (2018), Li et al. (2019), Liu et al. (2019), Mani et al. (2019), Scopetani et al. (2020) and Zhang et al. (2018) scored a 2 by documenting specific shape findings. Felsing et al. (2018) observed fragments, films, fibres, microbeads, spheres and pellets, while Scopetani et al. (2020) found PS fragments and ABS fibres with similarity in before and after spike particles. Zhang et al. (2018) used irregularly shaped particles of PP and PE before spiking, which after the heating method rolled up into circular plastic fibres, as PE and PP were light density polymers. This proved that the method developed enabled smooth identification and was efficient in extracting microplastics from soil organic matter. Additionally, Felsing et al. (2018), Liu et al. (2019) and Mani et al. (2019) reported findings of fragments, fibres, microbeads, spheres, and pellets with after spike PS of 36% microbeads and 29% foam. Liu et al. (2019) experimented with beads, spheres, pellets, films, particle, and fibre, which resulted in 65% - 98.3% recovery of particle, fibre and film. Finally, Li et al. (2019) used spiked microplastics of fragment, bulk and fibre. However, after the separation process, it was found that white fibre consisted of 38.9 % - 65.1 % was the dominating shape in soil. Particle shapes were observed by using digital microscopy and stereomicroscopy. The selected studies preferred using the stereomicroscopy technique with digital camera as the magnified images were able to identify ambiguous plastic-like particles. Nevertheless, particles of size range of <100µm without colour or definite shape faced difficulty in characterisation as microplastics (Shim et al., 2017). Studies that only reported shapes without using any form of microscopy equipment for measurement or vice versa was given a score of 1. Selected studies by Fuller and Gautam (2016), Han et al. (2019a) and Li et al. (2021) had obtained a score of 1. According to Ruijter et al. (2020), the shapes used in experimental studies and the shapes recovered from environmental samples showed a significant difference and suggested that greater refinement of shapes into specific groups would provide better mechanistic comprehension.

Polymer type is important as the potential effects of microplastics are determined by the composition of the polymer constituting the respective microplastics, which indirectly influence the density of the polymer particles (Kooi et al., 2017; O'Connor et al., 2020). This criterion determines the changes in chemical structure that occur during the isolation and extraction processes. O'Connor et al. (2020) stated that it was crucial to provide detailed information on the polymer composition, such as chemical additives, surface chemistry, degree of crystallinity and plasticisers, as they influence the outcome of microplastics in experimental design and the environment. A range of polymers from low density to high density were used for spiking experiments for the purpose of method development. A criterion score of 2 was assigned to the selected studies when polymer type was reported with instrumentation. Studies by Felsing et al. (2018), Fuller and Gautam (2016), Han et al. (2019a), Li et al. (2019), Liu et al. (2019), Mani et al. (2019) and Scopetani et al. (2020) obtained a score of 2 with complete information on polymer type including instrumentation details. Studies by Fuller and Gautam (2016), Han et al. (2019a), Li et al. (2019), Liu et al. (2019), Mani et al. (2019) and Scopetani et al. (2020) identified the spiked polymer types by using Fourier Transform Infrared Spectroscopy (FTIR). Fuller and Gautam (2016) detected similarity between the initial spiking particles (HDPE, PS and PVC) and the FTIR spectra database. This proved that chemical changes did not occur in the microplastic particles during the isolation and extraction procedures. A study by Felsing et al. (2018) reported polymer identification through Pyrolysis-Gas Chromatography Mass Spectrometry (Pyr-GCMS). This instrumentation technique presented analysis of thermally decomposed gas from polymers, whereby the pyrograms of spiked polymers were compared with known reference pyrograms. Both, FTIR and Pyr-GCMS were widely used in microplastic studies since these techniques provided accurate and reliable findings of polymer types (Shim et al., 2017). A criterion score of 1 was assigned to the selected studies when the polymer type was reported but without details on instrumentation. Studies by Li et al. (2021) and Zhang et al. (2018) received a score of 1.

Data reporting is an important criterion to be considered in the quality assessment of microplastics studies. Concentrations of microplastics are furnished as particle concentration as microplastic particles per kg soil (item/kg) or mass concentration as grams of microplastics per kg soil (g/kg) and percentage (Besseling et al., 2019). Studies that clearly reported the data regarding microplastic units, concentrations in particle number as well as in mass or percentage concentration were assigned a score of 2. All the selected studies obtained a full score of 2 due to accurate and enough information on reporting recovery of spiked microplastics with units. Studies by Felsing et al. (2018) and Fuller and Gautam (2016) recovered 100% of spiked microplastics. Five of the selected studies, (Han et al., 2019a; Li et al., 2021; Mani et al., 2019; Scopetani et al., 2020; Zhang et al., 2018) reported spiked microplastics mean recovery of more than 90%. Mean abundance of microplastics were reported in units of item/kg by Han et al. (2019a), Li et al. (2019), Liu et al. (2019) and Zhang et al. (2018) to be 5 items/kg - 295 items/kg, 200 items/kg -1290 items/kg, 136.6 items/kg – 256.7 items /kg, respectively. A score of 1 was assigned to studies that limited the reporting of microplastics recovery without any units. However, none of the studies scored 1. Inconsistency in reporting of microplastic concentrations caused difficulty in reproducibility of experiment (Connors et al., 2017; van Cauwenberghe et al., 2015). The method of reporting should be presented clearly to enable comparisons of data across numerous experimental studies (Ruijter et al., 2020). It has been previously recommended that it was better to report findings in additional units wherever possible, to enable easier comparison amongst studies until a standard for microplastics quantification was agreed upon (O'Connor et al., 2020). Consistency in unit documentation was of utmost importance as the units of microplastics concentration constitutes basic framework in quality assessment, allowing comparisons of newly developed methods (Besseling et al., 2019; Koelmans et al., 2019).

3.3 Overall Quality Assessment of Soil Microplastic Studies from a Methodological Aspect

Figure 2 shows the total scores of the selected studies and criteria of quality assessment based on methodological aspects of soil microplastics. The quality assessment was based on a total score of 22 per study. Studies by Mani et al. (2019) obtained the highest score of 21 as all information pertaining to the study criteria were documented in detail. The lowest score of 14 was obtained by Fuller and Gautam (2016) as six criteria, namely chemical purity, positive control, particle size, particle shape, laboratory preparation, and sample treatment had provided limited information. The highest score of 21 and lowest of 14 proved that there was a large gap between study scores for quality assessment of studies on soil microplastics.

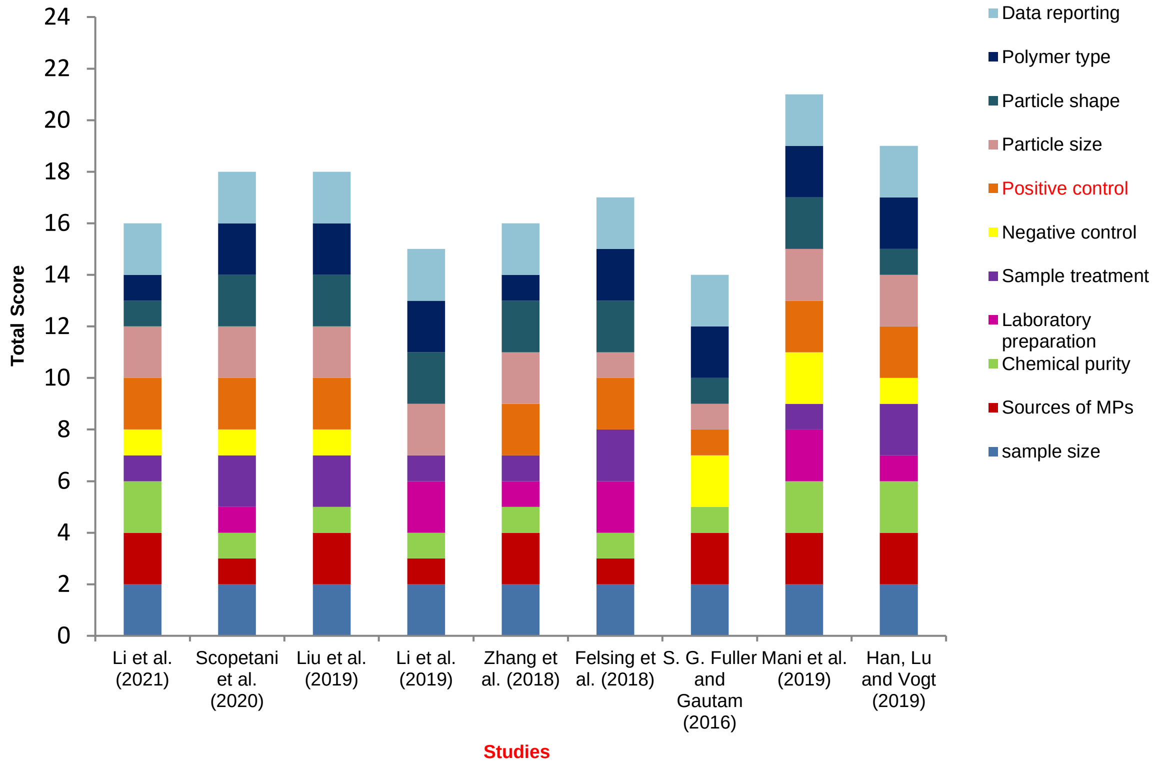


Figure 2. Total scores of the selected studies and criteria in quality assessment based on methodological aspects

Table 2 shows the average scores for each criterion based on methodological aspects of soil microplastics studies with a range between 0 and 2. The quality assessment found that sample size and data reporting criteria obtained a full average score of 2.0, concluding that these two criteria provided efficient and reliable data. Particle size and polymer type criteria scored an average of 1.78, while microplastics sources, particle shape and positive control criteria scored an average of 1.67. Chemical purity, sample treatment and laboratory preparation criteria achieved lower average scores of 1.33, 1.33 and 1.0, respectively. The criterion that required the most improvement was negative control, which obtained the lowest average score of 0.89. Although blank soil samples were included in the studies, it was deemed insufficient due to less than three replicates, or number of replicates was not mentioned and had limited information on negative control.

Table 2: Average score of each criterion for current study in comparison to previous quality assessment studies

Criteria	Soil (Current study)	Bottled water (Praveena & Laohaprapanon, 2021)	Aquatic biota (Ruijter, 2020)	Drinking water (Koelmans et.al.2019)	Biota (Hermsen et.al.2018a)
Sample size	2.00	1.1	-	1.02	1.46
Sources of microplastics	1.67	-	1.79	-	-
Chemical purity	1.33	-	0.30	-	-
Laboratory preparation	1.00	1.7	0.18	0.77	0.57
Sample treatment	1.33	-	-	0.93	0.43
Negative control	0.89	1.4	0.06	1.18	0.86
Positive control	1.67	0.2	-	0.21	0.17
Particle size	1.78	*1.3	1.30	*0.89	*0.66
Particle shape	1.67	*	1.32	*	*
Polymer type	1.78	*	1.20	*	*
Data reporting	2.00	-	1.36	-	-
Total Average score	1.56	1.14	0.94	0.83	0.69

* Average score was grouped as polymer identification (particle size, particle shape and polymer type)

The current assessment presented sample size and data reporting criteria to have the highest average scores as compared to all other previous assessments. A greater emphasis was given in providing sufficient and detailed information on sample size and reporting data with accurate unit measurement in recent studies pertaining to methodological aspects of soil microplastics. Adding on, for data reporting criteria, the study by Ruijter et al. (2020) stated that 60% of the evaluated studies did not report concentration or limited reporting to mass or number concentration, which may cause complications in cross examining data. Studies by Ruijter et al. (2020) scored higher for sources of microplastics criteria compared to the current study due to sufficient details provided. However, the current assessment focused on studies pertaining to method development that experimented with spiked soil samples. The sources of microplastics used for spiking were industrially manufactured and self-made, which require more elaborate specifications. A total of 33% of the evaluated studies had incomplete source information on type, origin, specifications of size, and shape of the microplastics. The chemical purity criteria were of utmost importance as chemical composition affects the assessment of microplastics characterisation and impurities were a source of contamination that may cause unfavourable chemical reactions (Abdin et al., 2020). The average scores for chemical purity were higher for the current study as compared to Ruijter et al. (2020). The current study provided detailed information on the chemicals used, while contrastingly, 73.3% of the evaluated studies by Ruijter et al. (2020) did not mention the possibility of chemical contaminants affecting the observed adverse effects.

Although current studies obtained higher average scores for laboratory preparation criteria as compared to studies by Hermsen et al. (2018a), Koelmans et al. (2019) and Ruijter et al. (2020), the scores were lower than studies by Praveena and Laohaprapanon (2021). The current study has focused more on contamination controls in soil microplastics analysis by using cotton lab coats, rinsing, and cleaning of laboratory apparatus and work surfaces with alcohol, and analysing with blank samples. Sample treatment criteria scored a higher average on the current study as compared to studies by Hermsen et al. (2018a) and Koelmans et al. (2019). The nature of soil as a complex and dynamic medium, rich in minerals and organic materials, required the need for heavy treatment procedures to eliminate organic matters in the current study for soils as compared to biota and water samples (He et al., 2018). Studies by Koelmans et al. (2019) had automatically assigned full scores for sample treatment criteria as their study samples (tap water and bottled water) did not require digestion steps. However, water samples from wastewater treatment plants had to meet the criteria set of treatment at 50°C to prevent polymer mass losses due to overheating.

Likewise, the current soil study achieved higher average scores for negative control criteria in comparison to biota studies by Hermsen et al. (2018a) and Ruijter et al. (2020). Studies by Ruijter et al. (2020) and Hermsen et al. (2018a) reported that 57% of the reviewed studies included blank samples for contamination control. However, details and number of blanks were not mentioned. The current assessment presented positive control criteria to have the highest average score amongst all other previous assessments. The current study obtained 77% as the reviewed studies reported three or more replicates for positive controls. Studies by Koelmans et al. (2019) achieved only 6% for providing complete data on positive controls, indicating that inclusion of positive controls was not a common practice. Similarly, Hermsen et al. (2018a) stated that 89% of the reviewed studies lacked positive control, making the studies unreliable for further investigations. Praveena and Laohaprapanon (2021) reviewed studies which provided 17% of reliable positive control samples for the analysis of bottled water, while Ruijter et al. (2020) lacked this information. The current study obtained higher average scores for particle characterisation criteria which included particle size, particle shape and polymer type as compared to other studies by Hermsen et al. (2018a), Koelmans et al. (2019), Praveena and Laohaprapanon

(2021) and Ruijter et al. (2020). Hermsen et al. (2018a) studied particle size and polymer type with ample focus on various instrumentation techniques. However, there were limitations on information regarding particle shapes. Studies by Ruijter et al. (2020) had discussed all particle characterisation criteria equally, while Praveena and Laohaprapanon (2021) focused more on particle size and shape with limitations on polymer type. Koelmans et al. (2019) had provided limited information on particle size and shape. Therefore, the current study obtained the highest average score due to provision of sufficient and efficient information on particle characterisation.

The current study involving methodology development for soil microplastic studies achieved a higher total average score of 1.56 as compared to studies by Hermsen et al. (2018a), Koelmans et al. (2019), Praveena and Laohaprapanon (2021) and Ruijter et al. (2020) with 1.14 involving bottled water samples, ensuring a trending increase in total average scores. Publications pertaining to studies on microplastics in biota and water backdate as early as 2010 and 2011 (Hermsen et al., 2018a; Koelmans et al., 2019). Studies in the early years lacked information on laboratory preparation, negative and positive control, sample treatment and polymer identification as microplastic research was still evolving (Boerger et al., 2010; Browne et al., 2011; Courtene-Jones et al., 2017; Eriksen et al., 2013; Mason et al., 2018; Murray and Cowie, 2011; Robbins, 2014; Schymanski et al., 2018). Therefore, it was perceived that the current existing literature on soil microplastics has duly adapted and improvised on methodological perspectives as research on microplastics had progressed over the years, resulting in a significant higher total average score for the quality assessment in the current study.

4.0 Conclusion

The quality assessment evaluated a total of nine studies pertaining to method development for isolation and identification of soil microplastics. The evaluated criteria groups were pre-laboratory work, during laboratory work, and post-laboratory work, with a total of 11 specific criteria. The total score for evaluation of each study is 22. The highest score obtained based on studies was 21 and the lowest score was 14. The quality criteria which achieved the highest average score of 2.0 were sample size and data reporting. The quality criterion with the lowest average score of 0.89 was negative control. Measures to improve the quality assessment of negative control criteria could be achieved when negative controls were run more regularly during experimental procedures. Possibility of microplastic contamination from chemicals that affected negative controls must also be avoided by filtering solutions. Atmospheric deposition of microplastics in the laboratory had tendency to contribute towards contamination in negative controls, and thus samples should be covered when not in use and air circulation should be limited as much as possible. These measures would enhance the purpose of utilising negative control, leading to better quality assessment of this criterion. The quality assessment method based on criteria scores was discerned to reshape as new analytical techniques became available, causing growth in methodological aspects of soil microplastic research.

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Supplementary Information

Supplementary 1: Explanation of the quantitative scoring system proposed to evaluate the studies for extraction and identification of microplastics in soil using the (QA/QC) criteria. The purpose of the quantitative scoring system criteria is to assess the quality of the papers and to give guidance for appropriate methods for microplastics particle studies in the future. The criteria (1 – 11) relates to the technical quality of extraction and identification methods. Criteria (1-3) specifically relates to pre-laboratory work, criteria (4-7) during laboratory work and criteria (8-11) post-laboratory work. For each criterion a score of either 2 (adequate), 1 (adequate with restrictions) or 0 (inadequate) points were assigned, which are explained below. (Adapted from Ruijter *et al.*, 2020 and Hermsen *et al.*, 2018).

CRITERIA RELATING TO THE TECHNICAL QUALITY OF EXTRACTION AND IDENTIFICATION METHODS (1 – 11)				
Criterion	Explanation	Score 2	Score 1	Score 0
PRE-LABORATORY WORK (Criteria 1-3)				
1. Sample size	Appropriate soil sample size (based on weight) is needed for microplastic studies.	Adequate and accurate weightage of soil sample size (eg:10-250g)	Not adequate or inaccurate information on soil sample size weightage are provided	No soil sample size
2. Source of MPs	Specification on the root sources of stock or solutions of microplastics whether bought or self-made maximizes the reproducibility and should be reported. Refers to the microplastics used in positive control	The origin and/or production of microplastics in the respective laboratory is reported in detail, used for spiked MP based study	The information given on microplastics source is incomplete and hence not fully reproducible	No information on microplastics source reported
3. Chemical purity	In order to assess the methods, all chemicals and oils used should be of pure quality and have detailed specifications	-Chemicals must be of known concentrations, purity and the specifications of the chemicals and oils should be in detail -Chemicals are analysed or studies relied on manufacturer certificate as well as literature based	Concentrations of chemicals are mentioned but no information on the chemical purity or specifications	Not form of chemical information provided
DURING LABORATORY WORK (Criteria 4-7)				

4. Laboratory preparation	Microplastics contamination arising from the laboratory (air and materials) should be minimized. All materials used (equipment, tools, work surfaces and clothing) should be free of microplastics.	- Measures are taken to prevent microplastics contamination by wiping surfaces before analysis - Cotton lab coats were used to avoid microfiber contamination -Nitrile gloves were used - Distilled or deionised water used	Only a part of the measures is taken to avoid microplastics contamination or it is generally mentioned -no cotton lab coat used	No form of contamination prevention is mentioned
5. Sample treatment	Assessment on elimination of microplastics contaminants such as organic matter through digestion technique.	-Details of solutions used for sample treatment is provided -Method used is presented with reference	-Details of solutions used for sample treatment is not provided -Method used is presented without reference	No verification on sample treatment
6. Negative control	Soil blanks should be included for each batch of samples, with at least 3 replicate blanks per batch. Controls are given the same full treatment as the studied samples.	Blank soil /distilled water/deionized sample should be included for each batch of samples, with at least three replicate blanks per batch	Blank soil /distilled water/deionized sample included, nevertheless deemed insufficient if less than 3 replicates	No form of negative control was included in the study.
7. Positive control	Field soil samples are used to test the method developed. Includes controls (3) with added microplastic particles that are treated in parallel to the samples. The particle recoveries are calculated by tallying the numbers of retrieved particles to the amounts added.	Includes 3 or more field samples and control that are run in parallel (more than 3 replicates)	Studies that report less than 3 field samples and control included	No positive controls were included
POST- LABORATORY WORK: DATA ANALYSIS (Criteria 8-11)				

8. Particle size	Size is a major factor defining effects of microplastics and should be reported based on before and after spiking BS: before spiking AS: after spiking	-If a range of sizes is used, the lower and upper limit is reported -If a single size is used, it is reported with unit measurement - Particle sizes are reported based on before (BS) and after spiking (AS)	-If a range of sizes is used, the lower and upper limit is not reported - If a single size is used but not reported with unit measurement -particle sizes are not reported based on before (BS) and after spiking (AS)	No information on particle size
9. Particle shape	Shape is a critical factor determining effects of microplastics and should be reported	Shapes are measured with high resolution microscope and reported	Particle shapes are reported but not measured using appropriate equipment	No information on particle shape is reported
10. Polymer type	Polymer type is a crucial factor explaining effects of microplastics and should be reported	Recovery of microplastics are visually identified and further quantified through appropriate instrument eg: Raman spectroscopy, FTIR, GC-MS, TGA	Polymer type is reported but without information on instrument used	No information on polymer identity is reported
11. Data reporting	Unambiguous units are required to ensure reproducibility of the experiment and to make it possible to compare data across experiments	Studies that report clearly on MPs unit, concentrations in particle number as well as in mass / percentage concentrations	Studies that limit the reporting of MPs recovery without any units	No units are presented

Supplementary 2: Score tables based on quality criterion for the selected nine studies

Chengtao Li (2021) A commonly available and easily assembled device for extraction of bio/non-degradable microplastics from soil by flotation in NaBr solution: Journal of Science of the Total Environment 759 (143482)		
Criterion	Explanation	Score
Sample size	42 samples containing 30.0 g of soil were taken	2
Sources of MPs	The plastic materials used in this study were supplied by Liangying Plastic Chemical Co., Ltd. (Guangdong, China). Sources mainly bought from supplier. Details provided	2
Chemical purity	NaBr was supplied by Tianli Chemical Reagent Co., Ltd. (Tianjin, China; purity (AR) ≧99%). Chemical used had detailed information. eg: Country, grade, density and process	2
Laboratory preparation	No information on lab or environmental condition	0
Treatment of sample	Sample pre-treatment: Collected soil samples are air-dried after removing sundries and sieving to obtain samples to be processed. No reference.	1
Negative control	An un-spiked 30.0 g soil sample was used as a control. Only one control used.	1
Positive control	42 samples containing 30.0 g of soil were taken and each sample was spiked with 0.3 g of microplastics. The original microplastics were used in controls. (PBS, PBAT, PLA) and (LDPE, PS, PP, PVC) Microplastic recovery experiments with different microplastic particle sizes and densities in spiked soil, were all performed in triplicate.	2
Particle size	The seven plastic types were mechanically crushed and grinded down into microplastic particles with particle sizes of<1mm. The microplastic particles were then separated into size classes of 100–200 µm and 200–1000 µm by sieving. (BS.) There is no significant difference in the average particle size before and after extraction with three density solutions (AS). Measurements based on SEM	2
Particle shape	A scanning electron microscope (SEM) (FEI Q45, American) was used to compare the morphology of the spiked microplastics separated and extracted with three different density solutions. The exact shape is not mentioned	1
Polymer type	3 types of biodegradable (PBS, PBAT, PLA) and 4 types of non-degradable (LDPE, PS, PP, PVC) plastics. Densities of the 7 types of plastics are tabulated. No identification with high-end instrument.	1
Data reporting	Recovery of seven microplastic polymer types (biodegradable and non-degradable types), comprising both small and large size classes, ranged from 92% to 99.6%.	2
Total score		16

Costanza Scopetani (2020) Olive oil-based method for the extraction, quantification and identification of microplastics in soil and compost samples: Journal of Science of the Total Environment 733 (139338)		
Criterion	Explanation	Score
Sample size	Five sub-samples of each soil (each 25 g) and compost (10 g) matrices (20 sub-samples in total) were spiked with all the six self-made MPs	2
Sources of MPs	Only mentioned self -made	1
Chemical purity	10ml 30% H_2O_2, 1ml of 2 mmol/L $FeSO_4$*7H_2O, 1 ml of 2 mmol/L protocatechuic acid and 5 mL of H_2O. No other specifications	1
Laboratory preparation	The possibility of self-contamination was considered, and during all steps of the sampling, treatment and analysis of the samples, fleece clothing and other plastic items, which could release MPs, were avoided. Instead, only cotton clothes were used during sampling.	1
Treatment of sample	10mL 30% H_2O_2, 1mL of 2 mmol/L $FeSO_4$*7H_2O, 1 mL of 2 mmol/L protocatechuic acid and 5 mL of H_2O (Oxidation method with reference)	2
Negative control	Procedural blanks were performed parallel with the samples to check for potential source of contamination. No microplastics were found in any of the blanks. Number of blanks not stated.	1
Positive control	To further test the method and to investigate if smaller MPs originally occurred in the collected soil and compost samples, three replicates of each matrix were prepared following the procedure described above. 2 oat field soil and 2 composted biowaste, sewage sludge samples.	2
Particle size	Six different self-made micro-polymers (range of dimension 0.2–2 mm) were used as test MPs. (BS) These findings show the method works for smaller particles (5μm-300μm) (AS)	2
Particle shape	In the sample collected from the Mäkelä field, two different polymers were found: one acrylonitrile (butadiene styrene) (ABS) fibre and one PS fragment. Visible light map of a plastic fragment and fibre through FTIR microscope	2
Polymer type	For validating the method, soil and compost samples were spiked with six different micro-polymers: PE, PS, PVC, PC, PET and PU. The MPs and car tire MPs were analysed before and after extraction using an Agilent Cary 630 FTIR Spectrometer equipped with a diamond crystal ATR (Attenuated Total Reflection)	2
Data reporting	The recovery rate also differed between the soil (mean recovery rate 73% ± 5% of added items) and compost (30% ± 18%). The method was validated using six different micro-polymers: PE, PS, PVC, PC, PET and PU and low, medium and high density polymers reached a mean recovery rate of 90%±2%, 97%± 5% and 95% ± 4%, respectively MP recovery (%)	2
Total score		18

Mengting Liu (2019) A method for extracting soil microplastics through circulation of sodium bromide solutions: Journal of Science of the Total Environment 691 (341-347)		
Criterion	Explanation	Score
Sample size	Using the separator, the mass of assayed soil can be adjusted in the range of 50 g to 200 g. 50 g of control soil and 20 repetitive Nile Red-stained MP, PMMA, PS or ABS were mixed individually.	2
Sources of MPs	Full names and other information tabulated	2
Chemical purity	Tests using three environment-friendly separation solutions: NaCl (1.19 g/ ml), $CaCl_2$ (1.42 g/ ml), NaBr (1.55 g/ ml). All reagents were purchased from Aladdin. No other specifications	1
Laboratory preparation	In the method, strict quality control was carried out to reduce plastic contamination.	0
Treatment of sample	10ml of 30%H_2O_2 were added and incubated for 3 d at 60 °C with references	2
Negative control	We used a control soil for the spiking experiments. Strict quality control was carried out to reduce plastic contamination and no MP were found in the blank control group. No mention of replicates.	1
Positive control	Three parallel experiments were performed in each group. Three replicates were set for each experimental group, as well as for corresponding control groups. 4 field samples are farmland, yellow-brown, paddy and floodplain soil	2
Particle size	Original plastic was white, except PVC and PMMA transparent, and all with the shape of bead in the size of around 3 mm. Different types of plastics were manually broken into MP by grinding and shredding and passed through a series of sieves (7-160mesh). Polyethylene MPs were divided into three size-classes: 100–500 μm, 500–1000μm, 1000–3000 μm. (BS) The size of MP ranged from 0.03mm-4.76mm, majority sizes were <1mm. (AS)	2
Particle shape	Three shape-different groups were selected as 500–1000 μm, PE MP with a particle, fibre or film. Original plastic was white, except PVC and PMMA transparent, and all with the shape of bead in the size of around 3 mm. Identified by a stereomicroscope (Nikon, SMZ25).	2
Polymer type	10 types of plastic used in this study include: PA, PP, PE, PET, POM, PVC, PC, ABS, PMMA, and PS. All types of MP were identified by (μ-FTIR, Thermo Nicolet iN10MX)	2
Data reporting	Results showed that the mean abundance of MP was 136.6–256.7 item /kg. Various MP including PP (40%), PE (35.5%), Acrylic (15.6%), PET (6.7%) and PA (2.2%) were found.	2
Total score		18

Qinglan Li (2019) Separation and identification of microplastics from soil and sewage Sludge: Journal of Environmental Pollution 254 (113076)		
Criterion	Explanation	Score
Sample size	Specifically, 50 g of soil or sludge sample was added to a 250 ml conical flask. Two litres of sludge were collected, air dried, sieved, and stored as the soil samples.	2
Sources of MPs	PE, PP, PS, PA, ABS, PET (test samples)	1
Chemical purity	30% H_2O_2, 30% H_2O_2 + H_2SO_4 (3:1, v/v) and 30% H_2O_2 + HNO_3 (3:1, v/v), separation effect of three floatation solution was compared, including saturated NaCl solution (1.2g/cm3), 5 mol/L $ZnCl_2$ solution (1.5 g/cm3) and 7.5 mol/L NaI solution (1.8 g/cm3). No other specifications.	1
Laboratory preparation	Every gravity flotation solution used in experiment was prepared by ultrapure water and the glassware after clean was rinsed three times using ultrapure water. Researchers were required to wear cotton laboratory coats during the experiment process. During flotation process, the flasks were covered by aluminium foil to prevent MP contamination from atmosphere.	2
Treatment of sample	To obtain the best oxidation efficiency and the least influence MP particle, in a preliminary experiment, we compared three treatments, i.e. 30% H_2O_2, 30% H_2O_2 + H_2SO_4 (3:1, v/v) and 30% H_2O_2 + HNO_3 (3:1, v/v) at 70 °C. No reference stated.	1
Negative ctrl	No information	0
Positive control	Only mentioned control samples. No other information. (The FTIR spectrums showed that most tested MP showed no major deviation from the control samples after three oxidation treatment)	0
Particle size	After air-dried, the soil samples were gently grounded and sieved through a 5mm and a 1mm stainless steel mesh successively. (BS) The MPs with diameter 1-5mm retained on the 1mm mesh were picked out manually and recorded numbers. The dominate morphology of MPs was white fibre with a size of 0.02-0.25 mm (AS)	2
Particle shape	The shape was divided into fibre, fragment, and bulk in µm, using stereo microscope (SteREO Discover V8, Carl Zeiss, Germany) equipped with a digital camera (AxioCom, Carl Zeiss, Germany),	2
Polymer type	6 polymers [Polyethylene (PE), Polypropylene (PP), Polystyrene (PS), Polyamide (PA), Acrylonitrile-butadiene-styrene (ABS) and Polyethylene terephthalate (PET)] were used as test samples. MPs were identified with Micro-Fourier (m-FTIR) (Nicolet iN10 MX, Thermo, USA) and Fourier transformed infrared spectroscopy (FTIR) (Tensor27, Bruker, USA).	2
Data reporting	The MP abundance separated by NaCl, $ZnCl_2$, and NaI was 200-740, 280-1180, and 420-1290 items/kg in soil and 3810-7400, 4433-10160, and 5553-13460 items/kg in sludge. Among those colours, white is the most common one (38.0-70.4%), followed by blue (16.5-35.6%) and red (3.4-24.2%).	2
Total score		15

Shaoliang Zhang (2018) A simple method for the extraction and identification of light density microplastics from soil: Journal of Science of the Total Environment 616-617 (1056-1065)		
Criterion	Explanation	Score
Sample size	10 g and 3 replicates of clay soil, sandy soil and loess soil were weighed	2
Sources of MPs	Both LDPE and PP (Riblon, Ter Hell Plastic GmbH) were white and grounded into irregularly shaped particles by the company	2
Chemical purity	Not mentioned, experimented with only distilled water	1
Laboratory preparation	The laboratory was thoroughly cleaned before the experiments and throughout the duration of our testing. Clothes made from plastic fibres were not allowed in the laboratory. In order to reduce contamination during this process, all filter papers were covered by a light aluminium specimen box during the process of filtration.	1
Treatment of sample	Microplastics and impurities were identified using a heating method (3–5 s at 130 °C). No reference stated.	1
Negative control	Just mentioned that "No microplastics were found in the control treatment". No replicates, blanks or other information mentioned.	0
Positive control	LDPE and PP were added to soil samples at five concentration gradients (0.05%, 0.1%, 0.2%, 0.5% and 1.0%, w/w) with three replicates for each plastic. 3 replicates with field samples (clay soil, loess soil and sandy soil, agricultural soil and orchard soil)	2
Particle size	The sizes of the LDPE particles were <150 μm and the PP particles were <400 μm. (BS) The densities for both kinds of plastic particles were <1 g /cm. Size distribution of LDPE and PP were determined by dry-sieving method. Size were almost similar to original proportions (AS)	2
Particle shape	To distinguish between the impurities and the microplastics, which showed transparent, circular, and shiny properties, or had a big change of shapes, e.g. plastic fibre rolled up after heating, size in μm. After putting the slide under the microscope (Leica wild M3C, Type S, simple light) (6.4 X Zoom), a photo (before and after heating) was taken using a high-resolution camera (Leica DFC 425) linked to a computer with image software (Leica Applicate Suite 4.8) in order to identify the number and size distribution of the particles.	2
Polymer type	LDPE and PP. This method cannot be used to distinguish the chemical components of microplastics, which can be detected using the method of thermal analysis, infrared spectroscopy or Raman micro-spectroscopy	1
Data reporting	The recovery rate based on the weight of LDPE ranged from 86.0% ± 0.8 to 102.7% ±4.2 in loess, 103.0% ± 4.8 to 128.0% ± 34.0 in sandy soil, 89.9% ± 0.3 to 104.0% ± 8.4 in clay soil and 87.9 ±31.1 to 112.7±22.0 in pure sand, respectively Recovery rate (%), item/kg MP (tabulated)	2
Total score		16

Stefanie Felsing (2018) A new approach in separating microplastics from environmental samples based on their electrostatic behaviour: Journal of Environmental Pollution 234 (20-28)		
Criterion	Explanation	Score
Sample size	For each sample material, 150 g was spiked with ten particles of each plastic type.	2
Sources of MPs	self-made tire wear, others manufactured. No other specifications.	1
Chemical purity	Mentioned only for TOC determination: 1 ml of hydrochloric acid (1 M). Other specifications not stated.	1
Laboratory preparation	To prevent contamination during the work, each step was carried out according to the requirements of NOAA (avoid wearing polyester-type clothing, fleece jackets, polyester lab coats, inspect all of the equipment made from plastic before use, sieves should be washed and sonicated before and after use) The device was cleaned between all recovery tests.	2
Treatment of sample	The TOC content of the four different sample materials was analysed by first acidifying 100-700 mg of freeze-dried sample material with 1 ml of hydrochloric acid (1 M) for 3-4 h. TOC measurement was conducted in Eltra Helios Carbon/Sulfur analyzer CS-580A (Eltra GmbH, Haan, Germany) (Sch€afer et al., 2015).	2
Negative control	Not mentioned	0
Positive control	Recoveries were determined by processing 3 replicates and then calculating the mean recovery. Field samples were quartz sand, beach sand, particulate matter and sediment	2
Particle size	Each material was sieved into three size fractions (63-200 um, 200-630 um, 630-2000 um), except the fibres, available only with a size in the range of 630 um to 5 mm. Five-mm particles were punched from the materials (BS) Particle sizes after spiking not mentioned, only particle recovery for size fractions	1
Particle shape	To determine whether the shape or age of the particles influenced their separation, particles of different shapes, including spheres, pellets, fibres, and fragments was observed using Keyence digital microscope to measure in µm.	2
Polymer type	7 plastic were used to produce MP standards to determine recovery: high density polyethylene (HDPE), low density polyethylene (LDPE), polyethylene terephthalate (PET), polypropylene (PP), (non-foamed) polystyrene (PS), polyvinyl chloride (PVC), and polymethyl methacrylate (PMMA). Standards were also prepared from three other plastics types: polylactic acid (PLA), polyethylene firers, and self-made tire wear. The densities of the ten plastics types covered a range from 0.85 g/cm^3 to 1.58 g/cm^3. All plastics were characterized by pyrolysis-gas chromatography-mass spectrometry (PyGCMS).	2
Data reporting	For all materials, a mass reduction of almost 99% was achieved. For example, a 150 g sample of quartz sand could be reduced after the third step by 98.4 ± 0.1% to 2.34 ± 0.17 g. MP particles from the Rhine River were also recovered at 100%.	2
Total score		17

Stephen Fuller (2016) A Procedure for Measuring Microplastics using Pressurized Fluid Extraction: Journal of Environmental Science and Technology 50 (5774-5780)		
Criterion	Explanation	Score
Sample size	The soils samples were dried at 40 °C overnight, sieved through 1 mm sieve and stored at <4 °C prior to the analysis. A sample size of 10 g was used.	2
Sources of MPs	Details from where it was bought (similar as polymer type)	2
Chemical purity	High purity solvents such as methanol, hexane and dichloromethane (Suprasolv, Merck, Germany) were evaluated. Other details not included	1
Laboratory preparation	Not mentioned	0
Treatment of sample	Not mentioned	0
Negative control	Standard laboratory quality control procedures were followed for control blanks with triplicates. Results for laboratory control blanks showed average microplastic content of 0.09 mg (SD = 0.17, n = 3)	2
Positive control	Standard laboratory quality control procedures were followed for method validation. 2 environmental samples used were composted municipal waste sample and industrial soil samples	1
Particle size	The materials were powders typically 50 µm in diameter except for PS which had a mean size of approximately 1 mm. (BS). Faced limitations in measuring size fractions after spiking	1
Particle shape	Fragments are presented with micrographs. No details of instrument used.	1
Polymer type	The plastic materials used were high density polyethylene (HDPE) (Aldrich # 434272), polystyrene block-poly (ethyleneran-butylene)-block-polystyrene (PS) (Aldrich 200557), and poly (vinyl chloride) (PVC) (Aldrich #18621−25G). The identity of the materials was confirmed by a Nicolet 6700 FTIR spectrophotometer (Thermo) prior to use	2
Data reporting	The method was initially developed by recovering 101% to 111% of spiked plastics on glass beads and was then applied to a composted municipal waste sample with spike recoveries ranging from 85% to 94%. The soil samples were found to contain 0.03% to 6.7% of microplastic. Recovery (%)	2
Total score		14

Thomas Mani (2019) Using castor oil to separate microplastics from four different environmental matrices: Journal of The Royal Society of Chemistry 11 (1788-1794)		
Criterion	Explanation	Score
Sample size	10g sample for marine beach sediments (MBS) and agricultural soil (AS).	2
Sources of MPs	Mentioned in detail (product, retailer, fragmentation methods) Appendix	2
Chemical purity	Details of chemicals provided (Origin, Brand, concentration grade etc) Appendix	2
Laboratory preparation	To prevent contamination, glassware was used whenever possible. Containers, such as petri dishes, were always covered with a lid or aluminium foil when not in use. Where the use of plastic materials for processing was unavoidable (e.g. the PTFE stopcock in the separation funnel), the item was thoroughly rinsed before use with deionised water and EtOH (70%). White lab coats (100% cotton) were worn. Nitrile gloves were worn whenever the operator's hands came into close contact with samples and glassware. To prevent cross contamination between instruments or receptacles, all used items were thoroughly washed with warm water and labware detergent.	2
Treatment of sample	A subsequent H_2O_2 treatment of these remaining residues resulted in a significantly higher final matrix reduction of 82 ± 6%. No reference stated.	1
Negative control	Procedural blanks were run during the visual sample examination phase to assess the laboratory atmosphere contamination potential. Three rinsed glass Petri dishes were placed uncovered on the laboratory bench during the entire visual sample examination phase, rinsed and drained onto cotton/cellulose filter paper and the filter paper was visually examined under a super-lighted stereomicroscope. No MP fragments were recorded in any of the blanks	2
Positive control	Each environmental matrix (Fluvial suspended surface solids (FSS) and marine suspended surface solids (MSS) and marine beach sediments (MBS) and agricultural soil (AS)) was divided into four replicates with specific target dry weights. Four replicates spiked with PP, PS, PMMA, PET-G were conducted	2
Particle size	We developed a protocol to separate microplastics (size range: 0.3–1 mm; virgin polymers: PP, PS, PMMA and PET-G). Particle sizes ranged from 0.3–1 mm (BS). 0.3-0.5mm and 0.5-1mm were found (AS)	2
Particle shape	We used fragments of four common polymer types PS opaque microbeads (33%) and PS foam (29%) were the largest contributors. Each fraction was numerically quantified using a stereomicroscope	2
Polymer type	Polymer types (PP, PS, PMMA, PET-G) with density details were reported and chemically analysed by attenuated total reflection (ATR)-FTIR.	2
Data reporting	The mean SD MP spike-recovery rate was 99 ± 4% with an average matrix reduction of 95 ± 4% MP recovery (%)	2
Total score		21

Xiaohin Han (2019) An optimized density-based approach for extracting microplastics from soil and sediment samples: Journal of Environmental Pollution 254 (113009)		
Criterion	Explanation	Score
Sample size	The 200 g clean soil and sand samples were spiked with ten pieces of the prepared microplastic particles (PP, PET, PE, PVC, PS and EPS)	2
Sources of MPs	The specific original product and sources tabulated GC vial cap, Water bottle, Yoghurt bottle, Pipe, Spoon, Styrofoam packaging	2
Chemical purity	Sodium chloride (NaCl, AR, 99.5%), sodium iodine (NaI, 99%) and hydrogen peroxide (H_2O_2, 35%) were purchased from Aladdin (Shanghai, China), Meryer (Shanghai, China) and Bohua (Tianjin China)	2
Laboratory preparation	Solutions of saturated NaCl and NaI were prepared by dissolving an excess of NaCl and NaI pellets in distilled water. No mention of laboratory conditions or elimination of contamination	1
Treatment of sample	The organic matter in the filtrate was removed by storing the filter membrane with floating particles in 30 ml of a 35% H_2O_2 solution at room temperature for 7d (Nuelle 2014). As there was little organic matter in the sandy sediment sample, only the soil sample was used to test the influence of organic matter on the recovery rates of spiked microplastics.	2
Negative control	The non-presence of microplastic particles in the blank soil and sand matrix samples were verified by extracting microplastics using the prescribed extraction device and process. No mention of replicates.	1
Positive control	Five duplicate microplastics spiked sediment samples were used in the recovery experiments, allowing the determination of standard deviation based on the amount of microplastics found in the five sediment sample replicates. One soil sample was used.	2
Particle size	For the recovery experiments, plastic particles of <1mm in size were prepared by shredding and cutting various common plastic products made from PE, PP, PVC, PET, PS and EPS. (BS) The particle sizes were similar (AS)	2
Particle shape	Fragments and fibres found, size in mm. No details of instrument used.	1
Polymer type	Samples were spiked with ten pieces of the prepared microplastic particles (PP, PET, PE, PVC, PS and EPS). The visually recognized microplastic particles were further identified by attenuated total reflection Fourier transformed infrared spectroscopy (ATR-FTIR, Bruker Tensor II, Germany).	2
Data reporting	The average recovery rates of PP, PE, PET, PVC, PS and EPS were 92 ± 11.7%, 78 ± 16%, 90 ± 11%, 100 ± 0%, 98 ± 4% and 96 ± 4.9%, respectively (average recovery MP (%) and items/kg (tabulated)	2
Total score		19