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Risk assessment of microplastics in freshwater sediments guided by strict quality criteria and data alignment methods



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HIGHLIGHTS

G R A P H I C A L A B S T R A C T

- We provide a holistic risk assessment framework for microplastic (MP) in sediment.
- A quality screening tool was developed and applied to sediment MP exposure data.
- We assessed risks using MP volume and surface area as ecologically relevant metrics.
- These relate to effects triggered by food dilution and translocation, respectively.
- \bullet The HC_5 lower limit for volume and area was exceeded in 32% and 17% of locations.



ABSTRACT

Determining the risks of microplastics is difficult because data is of variable quality and cannot be compared. Although sediments are important sinks for microplastics, no holistic risk assessment framework is available for this compartment. Here we assess the risks of microplastics in freshwater sediments worldwide, using strict quality criteria and alignment methods. Published exposure data were screened for quality using new criteria for microplastics in sediment and were rescaled to the standard 1–5000 μ m microplastic size range. Threshold effect data were also screened for quality and were aligned to account for the polydispersity of environmental microplastics and for their bioaccessible fraction. Risks were characterized for effects triggered by food dilution or translocation, using ingested particle volume and surface area as ecologically relevant metrics, respectively. Based on species sensitivity distributions, we determined Hazardous Concentrations for 5% of the species (HC₅, with 95% CI) of 4.9 $\times 10^9$ (6.6 $\times 10^7 - 1.9 \times 10^{11}$) and 1.1 $\times 10^{10}$ (3.2 $\times 10^8 - 4.0 \times 10^{11}$) particles / kg sediment dry weight, for food dilution and translocation, respectively. For all locations considered, exposure concentrations were either below or in the margin of uncertainty of the HC₅ values. We conclude that risks from microplastics to benthic communities cannot be excluded at current concentrations in sediments worldwide.

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1. Introduction

The release of microplastics (MPs) into the environment and MPs accumulation across all habitats globally have raised concerns about their potential risks to biota (Science Advice for Policy by European Academies, 2019). Here, 'risk' relates to assessing the likelihood that observed effects are caused by past or ongoing exposure to MP. Much effort has been made over the past decade to quantify current exposure levels of MPs in the environment (Schell et al., 2020) and to assess their effects on multiple organisms (Gomes et al., 2022). With the data generated, several studies have characterized the environmental risks of MPs from exposure via water (VKM, 2019; Adam et al., 2019; Besseling et al., 2019; Everaert et al., 2020; Burns and Boxall, 2018), with some works stating that risks can occur in some hotspot locations (VKM, 2019; Adam et al., 2019; Besseling et al., 2019; Everaert et al., 2020). For instance, Besseling et al. (2019) assessed the risks of MPs by comparing the upper limits of reported ranges in aquatic systems with toxicity data from biota exposed to MPs via water, and concluded that risks could occur in coastal marine waters (Besseling et al., 2019). VKM (2019) compared measured environmental concentrations (MECs) in aquatic ecosystems with predicted no-effect concentrations (PNECs) for aquatic species exposed via the water phase and concluded that risks may arise at 6% of the sites studied (VKM, 2019). Although all works assessing the risks of MPs include toxicity data for pelagic and benthic organisms, only exposure via the aqueous phase has been considered. No systematic risk assessment has ever been performed for benthic species exposed via the sediment phase. A recent study quantifying the concentrations of MPs in the Elbe river stated that freshwater sediments contained on average 600,000 times higher MP concentrations (in particles $/ m^3$) compared to the water phase (Scherer et al., 2020a; Winkler et al., 2022). Sediments also represent time averaged sinks of MP in aquatic systems (Barrett et al., 2020; Van Cauwenberghe et al., 2015; Kooi et al., 2017), so they may represent a more important exposure route for freshwater organisms than the aqueous phase. Therefore, there is an urgent need to assess the risks of MPs to benthic organisms using realistic sediment exposure scenarios (Scherer et al., 2020a).

Thus far, published MECs for MPs are incomparable, as MP sampling and analytical methods target different MP size ranges (Lu et al., 2021). Similarly, species sensitivity distributions (SSDs), cumulative probability distributions describing the sensitivity of a group of species to a particular stressor, are built by combining toxicity data for MPs of different sizes, shapes and polymer types (VKM, 2019; Adam et al., 2019; Besseling et al., 2019; Burns and Boxall, 2018; Everaert et al., 2018). This makes the data in the SSDs difficult to compare and interpret. Furthermore, MPs used in effect studies often consist of monodisperse particles (i.e., a single polymer type of a particular shape and/or size), while MPs found in the environment are composed of diverse particles with different characteristics (Kooi et al., 2021). Also, the mechanisms behind the observed effects are not considered, while it is now plausible that the volume of the ingested MP causes energy loss due to dilution of ingested food, and that the particle surface area is relevant for toxicological responses after tissue translocation (Koelmans et al., 2022, 2020; Mehinto et al., 2022). In this way, not all MPs used in the toxicity tests are biologically relevant to the exposed species, as the ingestion and tissue translocation of MPs is size-selective (Kalinkina et al., 2021; Scherer et al., 2017). Although fully consistent with the best available knowledge at the moment, this implies that most of the current MP risk assessments for aquatic ecosystems are fundamentally flawed.

To build robust SSDs and consistently characterize MPs' risks, exposure and effect data need to be aligned (Koelmans et al., 2020). Exposure and effect data should be rescaled to the full MP size range (1–5000 μ m) and effect threshold concentrations should be aligned to account for the polydispersity of environmental MPs (size, shape and polymer type), and the bioaccessible MP fraction for each species (Kooi et al., 2021; Koelmans et al., 2020). The mechanisms driving the observed effects must also be taken into account, as these determine

which ecologically relevant dose metrics (ERMs) should be used to quantify exposure and effects (Koelmans et al., 2022, 2020, 2017). To date, two studies have performed a fully aligned risk assessment for MPs, applying rescaling methods to correct for the differences in the MECs for freshwater and marine ecosystems and the reported effects and ingestible MP ranges for aquatic species exposed via water using SSDs (Koelmans et al., 2022).

Besides the mismatch between MP exposure and effect data, the usability of many MECs and effect threshold concentrations in risk assessment is also questioned due to the limited quality of the study designs and methodologies used (De Ruijter et al., 2020; Koelmans et al., 2019). Levels of MPs are often quantified without verifying possible contamination using negative controls or without quantifying losses during analysis using positive controls (Koelmans et al., 2019). The use of poor-quality data can lead to biased results when assessing the risks of MPs if a sample is highly contaminated, or if the MP concentrations recovered after the implementation of the extraction methods are unknown. For MP effect data, No Observed Effect Concentrations (NOECs) are often derived from two concentrations only, without distinction between particulate and chemical effects, or after force-feeding the organisms with MP (De Ruijter et al., 2020). The use of too few doses, the inability to identify the mechanisms behind the effects found, and a lack of environmental realism in a study can also lead to unsubstantiated conclusions on the risks of MPs (De Ruijter et al., 2020). For this reason, data must be screened for 'fit for purpose' in the context of risk assessment. To date, Quality Assurance/Quality Control (QA/QC) criteria have been developed for studies assessing the effects of MPs on aquatic species (De Ruijter et al., 2020), toxicity studies aimed at evaluating risks to human health (Gouin et al., 2022), and studies reporting MP concentrations in air (Wright et al., 2021), surface and drinking water (Koelmans et al., 2019) and biota (Hermsen et al., 2018). However, for studies quantifying MECs in sediment samples, a QA/QC criteria screening tool is not yet available.

The aim of this study was to assess the risks of MPs in freshwater sediments, considering strict quality criteria and data alignment methods. First, an in-depth literature search was carried out to compile studies reporting MECs for MPs in freshwater sediments, and studies reporting effect threshold concentrations for freshwater biota exposed to MPs via the sediment. Thereafter, QA/QC screening criteria were defined building on Koelmans et al. (2019) (Koelmans et al., 2019), and implemented to assess the quality of studies reporting sediment MECs of MPs. Studies reporting effects of MPs on sediment-exposed benthic organisms were screened using the 20 QA/QC criteria defined by de Ruijter et al. (2020) (De Ruijter et al., 2020). Threshold effect concentrations from studies that met a series of QA/QC criteria were aligned to account for the polydispersity of environmental MPs and for the bioaccessible fraction of MPs for each species (Kooi et al., 2021; Koelmans et al., 2022, 2020). Finally, two SSDs were constructed based on established alignment methods, one for particle volume and one for particle surface area as quantitative ERMs (Kooi et al., 2021; Koelmans et al., 2020). For these SSDs, the HC₅ was calculated, which is the value at which 5% of the species would be affected. Finally, the risks of MPs to freshwater benthic species were assessed by comparing the aligned MECs to the calculated HC5 values, considering the quality of the available MECs after the implementation of the QA/QC screening tool.

2. Methods

2.1. Data collection and study characteristics

An extensive literature search (until April 2022) was performed using the Web of Science (WOS) and ProQuest databases to collect MECs of MPs in freshwater sediments and effect threshold concentrations reported for freshwater benthic species exposed to MPs via sediment. For MECs, the following strings were used: (concentrations OR levels OR occurrence) AND (microplastic(s) OR plastic particle(s)) AND (freshwater OR aquatic OR lake OR river) AND (sediment OR benthos OR riverbed OR bottom). For effect threshold concentrations, the following strings were used: (effect OR impact OR toxicity) AND (microplastic(s) OR plastic particle OR fiber) AND (freshwater OR aquatic) AND (sediment OR benthic). Every article found in this search (64 reporting MECs, 20 reporting effect threshold concentrations for freshwater benthic species) was read and for those reporting MECs for freshwater sediments or effect threshold concentrations for freshwater benthic species in particles / kg sediment dry weight (dw), study characteristics were summarized in Tables S1 and S2 of the Supporting Information (SI), respectively.

2.2. Quality Assurance/Quality Control (QA/QC) evaluation

A quantitative evaluation of the quality of studies reporting MECs and effect threshold concentrations for MP was done to ensure the use of reliable and reproducible data only in the environmental risk assessment. The quality of 60 studies reporting 103 MECs for freshwater sediments (in particles / kg dw) was assessed building up on Koelmans et al. (2019) (Koelmans et al., 2019), who developed a QA/QC tool with 9 criteria to screen the quality of studies quantifying MPs in drinking and surface water samples. These criteria were revised and adapted to sediment samples, and one additional criteria was included to address within-site variability (Koelmans et al., 2019). The 10 criteria were englobed in 4 main categories, which cover all steps from MP sampling to analysis: sampling; contamination mitigation in the laboratory; sample purification/handling; and polymer analysis (Supporting Information, Table S3). Detailed motivations for all criteria are provided as Supporting Information.

The quality of 8 studies reporting effect threshold concentrations for freshwater benthic species exposed to MP via the sediment (in particles / kg dw) was determined following de Ruijter et al. (2020), who developed a QA/QC screening tool for MP effect studies performed with aquatic biota (De Ruijter et al., 2020). No further modifications were made to this quality screening tool, which includes 20 crucial criteria englobed in 4 main categories: particle characterization; experimental design; applicability for risk assessment; and ecological relevance. Motivations for the scoring of each criterion within the QA/QC screening tool for effect studies can be found in the SI of de Ruijter et al. (2020) (De Ruijter et al., 2020). For each criterion described in both QA/QC screening tools, a score of either 2 (adequate), 1 (adequate with restrictions), or 0 (inadequate) was given for each dataset (see Table S3). A 'Total Accumulated Score' (TAS) was calculated by adding the scores for each criterion, with a maximum of 20 and 40 points for MECs and effect threshold concentrations, respectively.

With the aim of constructing high-quality SSDs, the results of the QA/QC screening were used to select threshold effect concentrations that obtained a TAS of at least 20 (out of 40) and had non-zero values for 5 criteria: 6 "chemical purity", 13 "endpoints", 14 "addition of food", 16 "quality of dose-response relationship, and 20 "exposure time". Motivations for the selection of these criteria can be found in the SI. As in previous method evaluation reports (De Ruijter et al., 2020; Koelmans et al., 2019; Gouin et al., 2022; Wright et al., 2021; Hermsen et al., 2018), we emphasize that the scores assigned to each study should not be construed as a judgment on the relative merit of a study, i.e., a work that scores low on a particular criterion may still always provide valuable and reliable information about other potential insights.

2.3. Data alignment and construction of species sensitivity distributions (SSDs) for relevant dose metrics

2.3.1. Alignment of measured environmental microplastic concentrations From the available datasets we retrieved the mean, minimum and maximum MP number concentrations in sediment (particles / kg dw). These concentrations cannot be compared directly between studies because they targeted different size ranges. Minimum sizes ranged from 1 to $1000 \ \mu\text{m}$, and maximum sizes ranged from 2000 to 5000 μm . For the same reason, they cannot be directly compared to the reported effect threshold concentrations for benthic organisms to characterize the environmental risks of MPs. To allow for consistent comparisons of exposure concentration data, all data were rescaled to a standard MP size range from 1 μ to 5000 μ m by multiplying by a correction factor (CF) (Koelmans et al., 2020):

$$CF = \frac{5000^{1-a} - 1^{1-a}}{x_2^{1-a} - x_1^{1-a}} \tag{1}$$

Here, x_1 and x_2 are the minimum and maximum values of the targeted size range in the sediment monitoring studies (µm). Alpha (α) is the exponent of the MP size distribution $y = bx^{-\alpha}$ in which y and x are the relative abundance and size (i.e., length of the longest axis), respectively. For freshwater sediment a value $\alpha = 3.25 \pm 0.19$ has been reported based on an analysis of the sizes of 19,676 MP sampled from multiple locations (Kooi et al., 2021). This value was considered as a proxy of the slope of the generic power law distribution for MPs in the global sediments studied here.

2.3.2. Alignment of laboratory effect threshold concentrations to the environmentally relevant and bioaccessible effective concentration

In order to convert an effect concentration measured for mono- or polydisperse particles in a laboratory test to the effect concentration for an environmentally relevant mixture of MP particles with a different degree of polydispersity, a correction was made that takes into account the ecologically relevant dose metric (ERM) (Koelmans et al., 2017). For a given ERM 'x', effect threshold concentrations of particles with different degrees of polydispersity can be related to each other, as long as the overall size of the ERM remains the same (Koelmans et al., 2020):

$$EC_{poly}^{env,bio} = \frac{EC_{test} \times \mu_{x,test}}{\mu_{x,poly}^{env}}$$
(2)

Here, $EC_{poly}^{env,bio}$ is the effect concentration for *bioaccessible* environmentally relevant polydisperse MPs, EC_{test} is the effect concentration reported in the laboratory toxicity test, $\mu_{x,test}$ is the average value for ERM 'x' for either the mono- or polydisperse MPs used in the laboratory toxicity test, and $\mu_{x,poly}^{env}$ is the average value for ERM 'x' for the polydisperse MPs as they occur in nature. In the present study, the ERMs particle volume and area were selected for a food dilution effect mechanism and for toxicity dependent on tissue translocation, respectively.

The ECtest values used were the NOECs reported as particles / kg dw selected after screening the compiled data. When the suspected mechanism of effect depends on the ingestion of the particle (i.e., the 'food dilution' mechanism), particles that are too large to be ingested by the organism in question (i.e., wider than the mouth opening) were considered biologically unavailable and were excluded from further alignments (Kooi et al., 2021; Koelmans et al., 2020). When the MPs used in the effect test were smaller than the maximum ingestible particle size for the test organism, no correction was required. For each of the species, bioaccessible size fractions of MPs were obtained from either MP ingestion data, food ingestion data, or mouth opening size (Table S4, SI). In the case of toxicity dependent on tissue translocation, particles that are too large to be displaced through tissue were considered biologically unavailable (Kooi et al., 2021; Mohamed Nor et al., 2021). Recently, the probability of tissue translocation via gastrointestinal uptake has been reported to be significantly correlated with particle length, and binomial logistic regression showed that the particle length corresponding to the 50th percentile probability of translocation is 83 µm (Mehinto et al., 2022). So, this length was used as maximum for translocation. In the case of organisms with mouth openings smaller than 83 μ m, their mouth opening size was used as the upper limit for bioaccessibility (Table S4, SI). Here, bioaccessibility is defined as the fraction of microplastic particles that is actually taken up from the environment and is available to cause a biological response (McLaughlin and Roman, 2014). Following Koelmans et al. (2020) (Koelmans et al., 2020), for macrophytes no particle ingestion or translocation-based alignment was applied. When the maximum size of the tested particles was larger than the bioaccessibility criterion, EC_{test} was corrected with Eq. 1, with bioaccessible size range and exposure size range covered by the numerator and denominator, respectively. This was the case for 3 studies that used the same 20–500 µm polydisperse microplastic particles; Clokey et al, unpublished; Redondo-Hasselerharm et al. (2018); van Weert et al. (2019)). For these particles a power law slope $\alpha = 2.66$ was obtained by fitting a power law distribution to size data obtained from an ImageJ image analysis of 316 particles.

Values for $\mu_{x,test}$ can be obtained as long as the distribution of the ERMs follow a power law dependency, by fitting the power law slope α on tested particle characteristics, and by calculating the mean $\mu_{x,test}$ from the power law equation (Kooi et al., 2021). However, in the present study a power law did not fit the data well (Fig. S1). Therefore, the aforementioned ImageJ analysis of tested particles was used to acquire particle length (L) and width (W) data, whereas height (H) was calculated from the approximation L/W=W/H (Kooi et al., 2021; Koelmans et al., 2020; Mintenig et al., 2020). Subsequently, volume and surface area for each individual particle were calculated by approximating the fragments as ellipsoids (Koelmans et al., 2020). Mean volume for ingestible particles were directly calculated by considering only particles smaller than the mouth opening of the tested organism. Mean surface area of translocatable particles were directly calculated by considering only particles smaller than the translocation size boundary of the tested organism. In one of the selected effect studies, lab-prepared fragments were used (Scherer et al., 2020b), for which the characteristics were assumed similar to the aforementioned selected studies, and $\mu_{x,\textit{test}}$ was calculated accordingly.

For $\mu_{x,poly}^{env}$ a power law distribution with a power law slope a_x calibrated to the aforementioned 19,676 MP sampled from multiple freshwater sediments (Kooi et al., 2021) was used, in which case $\mu_{x,poly}^{env}$ can be calculated as (in case $\alpha \ddagger \{1,2\}$) (Kooi et al., 2021):

$$\mu_{x,poly}^{env} = \frac{1 - a_x}{2 - a_x} \times \frac{X_{UL}^{2-a_x} - X_{LL}^{2-a_x}}{X_{UL}^{1-a_x} - X_{LL}^{1-a_x}}$$
(3)

Where a_x was 1.53 for volume as ERM *x*, and 1.89 for area, and UL and LL are the upper and lower limits of ERM *x* (Kooi et al., 2021).

With all known variables in Eq. (2) we calculated $EC_{poly}^{env,bio}$ for each of the species. This relates to the effect number concentration for environmentally relevant MP as found in freshwater sediments, but still only for the species-specific bioaccessible MP fractions. In order to compare with the exposure number concentrations in a risk characterization, a species-specific rescaling was performed to calculate the effect concentration in terms of 1–5000 µm particles:

$$EC_{env} = EC_{poly}^{env,bio} \times CF_{bio} \tag{4}$$

Where CF_{bio} was calculated with Eq. (1) with the bioaccessible size range covered by the denominator and a power law slope for size of a = 3.25 (Kooi et al., 2021).

The aligned effect concentrations EC_{env} (particles / kg dw) were used to construct the SSDs.

2.3.3. Construction of species sensitivity distributions for ecologically relevant metrics

Two SSDs were constructed with the selected rescaled NOECs for both volume and surface area as ERMs using the ssdtools package in Rstudio (version 4.1.3) (Thorley and Schwarz, 2018). This tool uses maximum likelihood estimation to fit 10 cumulative distribution functions to the NOECs of the different species. The Anderson-Darling, Kolmogorov-Smirnov, Cramer-von Mises tests and the Akaike's Information Criterion (aic), Akaike's Information Criterion corrected for sample size (aicc) and Bayesian Information Criterion (bic) were used to evaluate goodness of fit of all distributions. The 5% Hazardous Concentration (HC₅) and the 95% confidence limits were calculated for the best fitting distribution using parametric bootstrapping (based on 1000 bootstrap iterations).

2.4. Risk characterization

To assess the environmental risks of MP for freshwater benthic biota, the rescaled to 1 – 5000 μ m mean, minimum, and maximum MECs (Table S1) were plotted as a cumulative frequency distribution together with the HC₅ values and confidence intervals calculated in the SSDs with the rescaled effect threshold concentrations for volume and surface area as ERMs. The rescaled mean, minimum, and maximum MECs reported by each study were plotted in a specific colour depending on the TAS obtained in the QA/QC evaluation. The risk characterization ratio (RCR) was calculated by dividing each rescaled MEC by the median HC₅ values obtained for volume and surface area. If the obtained RCR for a particular MEC is < 1, no risk of MPs for the benthic species inhabiting that freshwater ecosystem is expected, while for RCR > 1, the benthic species inhabiting that freshwater ecosystem might be at risk. A summary of the methodology followed to select the data included in the risk characterization is shown in Fig. 1.

2.5. Data visualization

All graphs were made with ggplot2 in Rstudio (version 4.1.3) (Wickham, 2016).

3. Results and discussion

3.1. Study characteristics and QA/QC evaluation

3.1.1. Measured Environmental Concentrations (MECs) in freshwater sediments

A total of 103 MECs of MPs in sediments from freshwater bodies across all continents were collected from 60 studies (Table S1). Of the studies collected, 46.7% reported MECs for freshwater bodies in Asia, 35.0% in Europe, 11.7% in America, 5.0% in Africa and 1.7% in Oceania (Fig. S2). Of the 103 freshwater ecosystems for which MECs in sediments were reported, 59.2% were rivers, 36.9% were lakes, and 3.9% corresponded to other types of freshwater ecosystems (Fig. S3). Fibres, fragments, and films were the most commonly detected shapes, with 85.0%, 80.0% and 45.0% of the studies reporting the detection of these shapes among the recovered MPs (Fig. S4). These three shapes were also the most frequently reported in surface and drinking water, although fragments were more often detected than fibres (Koelmans et al., 2019). Most commonly found polymer types were polypropylene (PP), identified by 65.0% of the studies, polyethylene (PE), identified by 56.7% of the studies, and polystyrene (PS) and polyethylene terephthalate (PET), both identified by 38.3% of the studies (Fig. S5). In surface and drinking water, PE, PP, and PS were also the most frequently detected polymers, with PE being found more often than PP (Koelmans et al., 2019). This confirms that low density MPs tend to settle over time, and that PET fibres might sink faster to the sediments due to their higher density. Scores obtained in the QA/QC evaluation of reported MECs in freshwater sediments are presented in Tables S15-S64. As stated in previous QA/QC assessments (De Ruijter et al., 2020), these scores are not intended to assess the value of the studies, but to identify those exposure data that provide the most reliable and robust assessment of the ecological risks of MPs. Of the reviewed studies, only 20% obtained a TAS greater than 10 (out of 20), while 80% had a TAS lower than 10 (Fig. S6). Only three studies obtained non-zero values for all 10 criteria, which were Pan et al. (2021) (TAS = 17) (Pan et al., 2021), Jian et al. (2020) (TAS = 16) (Jian et al., 2020) and Mani et al. (2019) (Mani et al.,



Fig. 1. Summary of the methodology followed for the selection and alignment of data included in the risk characterization of microplastics in freshwater sediments.

2019) (TAS = 14). This indicates that most of the MECs for MPs in sediments might not be fully reliable and reproducible, which could affect the conclusions driven in risk assessment. The mean TAS in all studies was 7.8 (range 2 –17). Average scores per criterion ranged from 0.35 to 1.30 (Fig. S7). Criteria with the highest average scores were "sampling reporting" (1.30), followed by "polymer identification" (1.10), and "in-site variability representation" (0.97). Criteria with the lowest average scores were "clean air conditions" (0.53), "sample size" (0.48) and "positive controls" (0.35) (Fig. S7). These findings are similar to those reported by Koelmans et al. (2019) for drinking and surface water, where the highest and lowest average scores were also found for the criteria "sampling reporting" and "positive controls", respectively (Koelmans et al., 2019).

3.1.2. Effect threshold concentrations for freshwater benthic organisms

In the 8 collected studies, a total of 14 freshwater benthic species were exposed to MPs via the sediment, including 5 crustaceans, 2 insect larva, 2 worms, 2 macrophytes, one snail, one bivalve and one nematode (Table S1). One of these studies corresponds to an in preparation manuscript, and a summary of its content is provided in the SI. Of the 8 studies, 5 tested the effects of MP fragments; Redondo-Hasselerharm et al., 2018; van Weert et al., 2019; Scherer et al., 2020b; Khosrovyan and Kahru, 2020), 2 tested spherical MPs (Höss et al., 2022; Ziajahromi et al., 2018), and only one tested MP fibres (Setyorini et al., 2021). Of the studies testing MP fragments, 3 used PS; Redondo-Hasselerharm et al., 2018; van Weert et al., 2019), one used polyamide (PA) (Khosrovyan and Kahru, 2020) and one used polyvinylchloride (PVC) MPs (Scherer et al., 2020b). Of the studies testing MP spheres, one used PS (Höss et al., 2022) and the other one used PE MPs (Ziajahromi et al., 2018). Fibres were made of PET (Setyorini et al., 2021). Of the 14 species tested, only 5 were adversely affected by the presence of MPs in sediments. Effects were found on the reproduction of C. elegans after 4 days of exposure (Höss et al., 2022), the growth of Gammarus pulex (Redondo-Hasselerharm et al., 2018) and Myriophyllum spicatum (van Weert et al., 2019) after 28 and 21 days, respectively, and the growth and emergence of C. riparius (Scherer et al., 2020b) and C. tepperi (Ziajahromi et al., 2018) after 28 and 10 days, respectively. For the rest of the species, the NOEC was assumed to correspond to the highest concentration tested. For the bivalve Sphaerium corneum, the NOEC was excluded because the maximum ingestible particle and food sizes found in the literature were always $< 20 \,\mu m$, which was the smallest MP used in the effect test (Redondo-Hasselerharm et al., 2018). In addition to the 8 collected studies, 12 others were found that reported effect threshold

concentrations in mass for freshwater benthic organisms exposed to MPs via sediment. These were excluded from the risk assessment because of the additional uncertainty associated with the mass-to-number conversion required to align the MP concentrations.

Scores obtained in the QA/QC evaluation by the 8 studies reporting effect threshold concentrations for freshwater benthic species exposed to MPs via the sediment are shown in Tables S65-S72 (SI). The average TAS across studies was 25.1 (range 18 - 31). Average scores per criterion ranged from 0.13 to 2 (Fig. S8). There was no single study that received non-zero values in all criteria, which is consistent with the findings of de Ruijter et al. (2020), where all 105 effect studies evaluated obtained a value of zero in at least one criterion (De Ruijter et al., 2020). Criteria for which all studies scored a 2 were "source of MP", "homogeneity of exposure", "replication", "endpoints", and "presence of natural (food) particles". The categories that were most adequately covered by all studies were "particle characterization" and "applicability for risk assessment", as the average score was always above 1 for all criteria. In contrast, criteria within the category "experimental design" had low average scores, except for "homogeneity of exposure" and "replication". The ecological relevance was better addressed in the case of the "concentration range tested" and the "exposure duration", compared to the "aging and biofouling" and the "diversity of MPs". Of the 8 studies assessed, 5 achieved a TAS of at least 20 and met the defined subset of 5 critical criteria. For this reason, the data from these 5 studies were further used to construct high-quality SSDs. This implies that the data used in the SSDs were reported by studies that: 1) ruled out the potential influence of chemical stressors, 2) only evaluated ecologically relevant endpoints linked to individual or population effects, 3) included natural particles in the systems to avoid force feeding the organisms with MPs, 4) tested at least 5 concentrations, and 5) consisted of chronic exposures. Thus, SSDs were built with the NOECs of 12 freshwater benthic species from 7 different taxonomic classes exposed to MPs via the sediment.

3.2. Species sensitivity distributions for volume and area as ecologically relevant metrics

The SSD for MPs with volume as ERM was constructed by fitting the gamma distribution, which was the best fitting model, to the rescaled NOECs (Fig. 2). The HC₅ (95% CIs) obtained were 4.9×10^9 ($6.6 \times 10^7 - 1.9 \times 10^{11}$) particles / kg of sediment (dw). The most sensitive species in the SSD was the macrophyte *M. spicatum*, for which the growth was affected after 21 days of exposure to MPs via the sediment (van Weert et al., 2019). The second most sensitive species was the nematode



Fig. 2. Species sensitivity distribution (SSD) for microplastics in sediment corrected for bioaccessibility and polydispersity, and accounting for the ecologically relevant metric of volume. This metric relates to the effects caused by the ingestion of MPs, which trigger energy loss due to dilution of ingested food. The black solid curve corresponds to the gamma distribution and the dashed lines show the HC₅ (4.9 × 10⁹ particles / kg of sediment dw). The grey area relates to the 95% confidence interval of the SSD. The markers show the rescaled NOECs of the species, and the colour of the markers relate to the taxonomic class of each species.

C. elegans, for which the reproduction was affected after 4 days of exposure (Höss et al., 2022).

For the SSD for MPs with area as ERM, the best fitting distribution was a Log-normal/Log-normal mixed distribution model which, however, was overparameterized so that no reliable CIs could be quantified. To obtain a defensible statistical model with more robust CIs, we constructed the SSD for MPs with the second-best fitting distribution that allowed the calculation of CIs using 1000 bootstrap iterations, which was the Weibull model. This model was fitted to the rescaled NOECs (Fig. 3), and the HC₅ (95% CIs) obtained were 1.1×10^{10} ($3.2 \times 10^8 4.0 \times 10^{11}$). In this case, the most sensitive species in the SSD were the macrophytes *M. spicatum* and *Elodea* spp. As there are no other SSDs available for benthic species exposed to MPs via the sediment, and alignment methods have only been applied in one earlier environmental study for surface water, we cannot compare our results with earlier works.



Fig. 3. Species sensitivity distribution (SSD) for microplastics in sediment corrected for bioaccessibility and polydispersity, and accounting for the ecologically relevant metric of area. This metric is hypothesized to drive the effects triggered upon translocation of MPs. The black line corresponds to the gamma distribution and the dashed lines show the HC₅ (1.1×10^{10} particles / kg of sediment dw). The grey area relates to the 95% confidence interval of the SSD. The markers show the rescaled NOECs of the species, and the colour of the markers relate to the taxonomic class of each species.

3.3. Risk characterization

To characterize the risks of MPs in freshwater sediments, we plotted the rescaled mean, minimum and maximum concentrations of MPs as a cumulative frequency distribution together with the HC5 and 95% CIs obtained in the SSDs for volume and area as ERMs (Fig. 4). It appears that all rescaled MECs are lower than the HC5 values derived for volume and area (Fig. 4), indicating that there are no immediate risks of MPs for freshwater benthic species. However, the HC₅ for volume is close to the highest maximum reported MECs of MPs, and the low confidence limit for volume and area is exceeded by 16% and 1% of the mean MECs, and 32% and 17% of the maximum MECs, respectively (Fig. 4). We find that the risks in case of a food dilution effect mechanism, with volume as the ERM, would be greater than from potential effects caused by translocation. Taking the lowest limit of the HC5 for volume as ERM, 84% of the sediment sites would be considered unaffected by MP based on the mean MP concentration at the site, while this percentage would be only 68% if we used the highest reported ('hotspot') locations of each of the sites. The TAS obtained in the QA/QC by each study reporting MECs is shown with colours to identify relationships between the quality of the studies and the MP concentrations detected. In fact, all MECs that exceed the lowest confidence limit for volume and area obtained a TAS below 10. This means that our observation that risks cannot be excluded can gain in significance by improving the quality of exposure data. In the risk assessment performed by Koelmans et al. (2020) (Koelmans et al., 2020) for MPs in surface water, in which alignment methods were used for exposure and effect data, 1.5% of the locations exceeded the HC5 value, and 28% of the locations exceeded the lower limit of the 95% CI. Note that these results are for another exposure medium and thus difficult to compare. Furthermore, the study by Koelmans et al. (2020) mainly focused on the implementation of the alignment procedures and did not yet contain such a rigorous data quality screening as we apply here.

3.4. General discussion and implications

Here we provide a novel framework for assessing the risks of MPs in freshwater sediment to benthic biota, integrating tools for QA/QC screening, data alignment, and for consistency with respect to known effect mechanisms. The framework has been applied to data for aquatic sediment worldwide and shows that the occurrence of effects for significant percentages of aquatic sediment worldwide cannot be excluded.



Fig. 4. Cumulative frequency distributions of the rescaled minimum, mean and maximum Measured Exposure Concentrations (MECs) of MPs (1–5000 μ m) in global freshwater sediments reported by 60 studies, plotted together with the HC₅ (vertical solid line) and 95% CIs (dashed lines) obtained from the SSDs for volume (purple) and area (green) as ecologically relevant metrics (ERM). The colours of the MECs indicate the Total Accumulated Score (TAS) obtained in the QA/QC screening: (black: \geq 16; red: \leq 5; blue: 11 \geq 15; yellow: 6 \leq 10).

It is expected that plastic emissions and exposure to MP will only increase (Borrelle et al., 2020). This means that the number of sediment locations at risk will also increase.

The risk assessment framework follows the recent development of a similar concept for addressing risks from MP in the aquatic environment (Kooi et al., 2021; Koelmans et al., 2022, 2020, 2017). Although the basis of the framework as such is well established, there is certainly a need to further validate the alignment methods for mechanisms of effect that are relevant for exposure to MP (De Ruijter et al., 2020; Koelmans et al., 2019).

More refinement can also be added depending on nutritional characteristics as, for example, epibenthic and endo-benthic species are exposed differently to MPs. More knowledge about possible effect mechanisms can also lead to refinement, e.g., fiber entanglement, or sharp edge lesions on fragments, both related to the shape of the particles. Furthermore, there is a need to improve the quantity and quality of the exposure and effect data used within this framework. For example, only 21% of the exposure data (including mean, minimum and maximum MECs) had a TAS in the top 50% (Fig. 4, blue and black data points), while the SSDs had a limited number of data points, leading to HC₅ values with wide CIs. An important finding is that methods to analyze MPs fall short in measuring the entire MP continuum. Including empirical data for particle sizes down to 1 µm in the exposure assessment, and the use of standard materials for environmentally relevant heterogeneous MP mixtures in effect tests would minimize the need for rescaling and alignment. This would greatly improve the accuracy of the MP risk assessment.

CRediT authorship contribution statement

Albert A. Koelmans and Paula E. Redondo-Hasselerharm conceptualized the study. Albert A. Koelmans, Andreu Rico and Paula E. Redondo-Hasselerharm designed the methodology. Paula E. Redondo-Hasselerharm carried out the data collection, and Paula E. Redondo-Hasselerharm and Albert A. Koelmans did the formal analysis of the data. Paula E. Redondo-Hasselerharm and Albert A. Koelmans drafted the manuscript, which was reviewed and edited by Andreu Rico. Paula E. Redondo-Hasselerharm, Andreu Rico and Albert A. Koelmans acquired financial support for this work.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Statement

Pollution with microplastics is a pressing environmental and social problem, with adverse effects on aquatic ecosystems noted worldwide. Here we introduce a holistic risk assessment framework for freshwater sediment, a compartment for which no framework was yet available. We provide a new tool to assess the quality of exposure data from the literature, as well as new methods to resolve the mismatch between exposure and laboratory effect data. We show that risks from microplastics to benthic communities cannot be excluded in some locations.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jhazmat.2022.129814.

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