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Prioritizing Suitable Quality Assurance and Control Standards to Reduce Laboratory Airborne Microfibre Contamination in Sediment Samples

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Abstract: The ubiquity and distribution of microplastics, particularly microfibres, in outdoor and indoor environments makes it challenging when assessing and controlling background contamination, as atmospheric particles can be unintentionally introduced into a sample during laboratory analysis. As such, an intra-laboratory examination and literature review was completed to quantify background contamination in sediment samples, in addition to comparing reported quality assurance and control (QA/QC) protocols in 50 studies examining microplastics in sediment from 2010 to 2021. The intra-lab analysis prioritizes negative controls, placing procedural blanks in various working labs designed to prepare, process, and microscopically analyse microplastics in sediment. All four labs are subject to microfibre contamination; however, following the addition of alternative clean-air devices (microscope enclosure and HEPA air purifiers), contamination decreased by 66% in laboratory B, and 70% in laboratory C. A review of microplastic studies suggests that 82% are not including or reporting alternative clean-air devices in their QA/QC approaches. These studies are found to be at greater risk of secondary contamination, as 72% of them ranked as medium to high contamination risk. It is imperative that laboratories incorporate matrix-specific QA/QC approaches to minimize false positives and improve transparency and harmonization across studies.

Keywords: microplastics; quality assurance; quality control; microfibres; sediment; airborne contamination

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1. Introduction

Commercialized microfibres of synthetic and natural origin have become a topic of concern, as numerous studies have documented their prevalence in biota [1,2], river and lake bottom, and seafloor sediment [3–6], and in the water column [7–10]. Microfibres are easily dispersed in all environmental matrices because of their size (<5 mm), shape and composition. Vegetal (natural) microfibres are often recognized as cellulosic and proteinaceous, and synthetic microfibres are chemically synthesized (human-made or anthropogenic) polymers, including nylon, acrylonitrile, and polyester [11]. Anthropogenic microfibres, which may be entirely synthetic or human-altered natural fibres, degrade from textiles into secondary sources. The domestic washing of textiles has been reported as a significant source of microfibre pollution, indirectly discharging particles into wastewater and into the environment. For example, [12] approximated 1900 microfibres are released per wash, whereas [13] reported greater counts ranging from 2.1×10^5 to 1.3×10^7, with subsequent washes shedding fewer polyester and cotton fibres. The latter study examined fibres from 100 to 1000 µm long and 10 to 20 µm wide, suggesting that microfibres could be passing through filter pore sizes >20 µm. Moreover, accelerated laundering experiments determined that polyester fabrics shed less in comparison to cellulose-based varieties, with 0.1–1 mg/g and 0.2–4 mg/g of fabric, respectively [14]. Microfibres have been detected in wastewater treatment plant (WWTP) discharge and sludge [15–17]. The sludge, which is often spread on agricultural fields, has great potential to release microfibres into the environment during runoff events [18]. Microfibres have also been found in atmospheric

fallout [19,20], ranging from 29 to 180 particles/m^2/day [21], and have even been identified in remote Arctic locations [22,23]. Microfibre pollution has been identified in remote areas such as the Pyrenean Mountains, thus implying that atmospheric transport acts as an essential pathway for the deposition of microfibres [24].

Microplastic (MP) abundances determined in indoor environments show that humans are being exposed to indoor MP concentrations (33% synthetic) ranging between 1.0 and 60.0 fibres/m^3 [5,25]. Deposition of airborne microfibres in laboratory settings has also been recognized as a source of MP contamination [26–30]. Considering that microfibres are often recorded as the dominant MP form in many investigations, it is possible that airborne laboratory contamination is accounting for the majority of the microfibres found in environmental samples. For example, [6] determined that the total number of microfibres in benthic samples from the Thames River, Canada decreased by 16% following laboratory and field blank subtraction. The issue of microfibre shedding, fallout and transport in environmental samples is concerning, as overestimates of microfibre counts could occur and potentially result in research error. Applying adequate quality assurance and controls (QA/QC) is not only necessary in every working laboratory environments, but is also essential during every methodological stage when investigating MPs in sediment. Negative controls, in the form of blanks, are necessary when designing a scientific experiment and determining its validity [30].

This study prioritizes and applies laboratory blank samples and clean-air devices as precautionary tools to minimize anthropogenic microfibre contamination. Airborne particles were counted and compared between different working labs and the preliminary results indicate whether airborne microfibre transport is reduced when employing suitable QA/QC. The objectives of this study were to: (1) identify, isolate and document microfibre contamination in various research labs within one building and throughout all experimental phases (processing and imaging) by examining 14 replicate procedural blank samples; (2) apply ideal quality standards and precautionary measures in the form of clean-air devices to help control and reduce microfibre fallout and transport; (3) qualitatively assess and rank 50 studies that investigate MPs in sediment according to desired criteria, and (4) recognize trends, gaps, and provide recommendations on how to mitigate background contamination and avoid research error.

2. Materials and Methods

2.1. Intralab

The research that was carried out involved many methodological stages and took place in different laboratory spaces within one building at the University of Western Ontario, Canada. As a result, several precautionary measures were taken to reduce background contamination. Cotton lab coats were worn and washed regularly by researchers working and entering the labs. Glass jars and containers were meticulously rinsed with reverse osmosis (RO) water, air dried, and covered with aluminium foil to reduce air exposure. Equipment was wiped down before and after every use, and linoleum floors were vacuumed before exiting the labs using a cordless vacuum. Work liquids were also pre-filtered prior to sample processing. These measures are encouraged because indoor contaminants, such as microfibers, can be re-suspended from the bare floor by human movement [31].

To compare the number of microfibres under different conditions, clean-air devices were employed during sample processing and imaging. A metal microscope enclosure was also constructed to fit around a Nikon SMZ1500 microscope to act as a barrier to free falling particles (Figure 1A). Finally, HEPA air purifiers (Winix FreshHome P300 HEPA Air Purifier) were placed in labs B and C (Figure 1B), along with glass-fibre filters positioned on air exhaust systems to reduce the entry of particulate matter (Figure 1C).

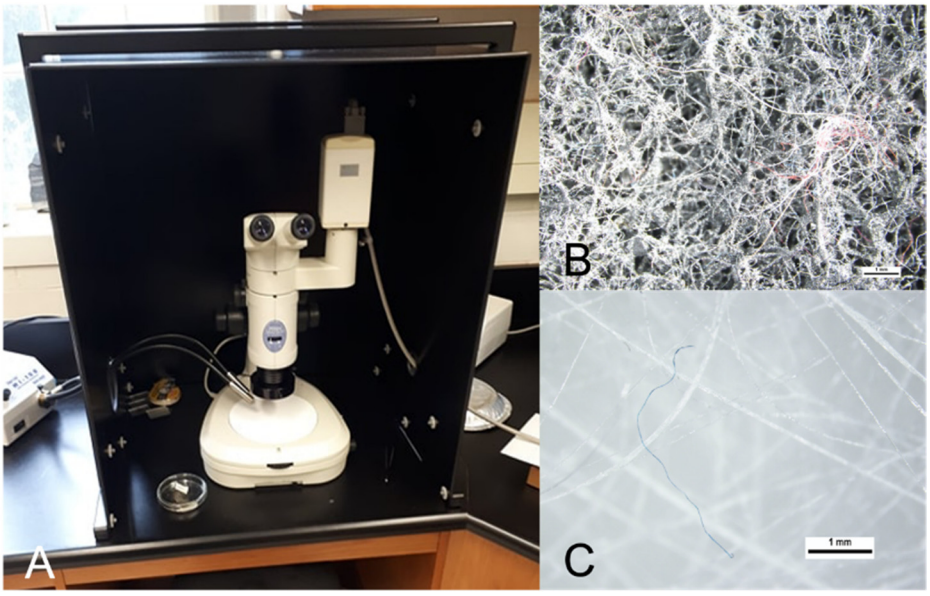

Figure 1. Images of laboratory control measures. (**A**) Metal microscope enclosure designed to reduce background contamination. (**B**) Microfibres entangled within a black carbon filter examined from a HEPA air purifier. (**C**) A blue fibre identified in a glass-fibre filter from an air exhaust unit.

Two samples from each of seven experimental set-ups were examined for anthropogenic microfibres (Table 1). Acronym terminology is as follows: A1, B1, D1 and A2, B2, and D2 are two negative control set-ups in three different labs (Labs A, B and D); B.F1 and B.F2 are controls taken in lab B with filters in place; C.NME1 and C.NME2 are controls taken in lab C without a microscope enclosure; C.ME1 and C.ME2 are controls taken with the inclusion of a microscope enclosure; and lastly, C.ME.F1 and C.ME.F2 are controls taken with the microscope enclosure and filters.

Each sample contained approximately 8 g of clean, previously inspected sediment cleared of anthropogenic particles before examination took place, in an uncovered glass Petri dish (10 cm diameter × 1 cm high). The methodology for sample processing and analysis mirrored the standard methodology used for Lakes Huron, Erie, Ontario, and the Thames River, London [4,6,32,33]. All sediment samples inspected contained zero MPs prior to quality control analysis. Laboratory blanks were exposed to the air during sediment-microplastic density separation and during visual identification of MPs under a stereomicroscope. Blanks were placed on surfaces and under equipment where continual work took place. The time of exposure depended on the average time required to process and examine sediment samples in each lab (Table 1).

Labs A and B are primarily used for sediment-microplastic preparation and processing using a magnetic stirrer, separatory funnels, polycarbonate sieve for rinsing, and a standard oven. Labs C and D are used to visually examine, characterize, collect and photograph anthropogenic particles (Table 1). Lab D was utilized during the imaging methodological stage to continue experimental momentum while the original microscope was being repaired in lab C.

Particles were transferred to a diamond compression cell and analysed using attenuated total reflection Fourier-transform infrared spectroscopy (ATR-FTIR) using a Bruker Tensor II spectrometer at Surface Science Western, Canada. The spectra were collected from 4000–600 cm^{-1}, with a resolution of 4 cm^{-1}.

Table 1. Various precautionary measures and negative control tests taken in laboratories A, B, C and D to asses microfibre contamination during all methodological stages when analysing microplastics in sediment. [1] Control with HEPA air purifier and glass-fibre filters; [2] Control with no microscope enclosure; [3] Control with microscope enclosure; [4] Control with microscope enclosure, HEPA air purifier and glass-fibre filters.

Laboratory Space	Methodological Stage and Purpose	Exposure Time	Precautionary Measures	Blank	Blue	Black	Pink	Total
Lab A	Preparation and Processing Sediment-microplastic density separation using a magnetic stirrer, glass separatory funnels, polycarbonate sieve for rinsing, drying oven	1 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Glassware covered with aluminum foil	A1 A2	2 0	1 0	0 0	3 0
Lab B	Preparation and Processing Sediment-microplastic density separation using a magnetic stirrer, glass separatory funnels, polycarbonate sieve for rinsing, drying oven	1 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Glassware covered with aluminum foil	B1	4	2	2	8
			1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Glassware covered with aluminum foil	B2	4	2	1	7
			1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Glassware covered with aluminum foil 4. HEPA air purifier 5. Glass-fibre filter	B.F1 [1]	1	1	1	3
			1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Glassware covered with aluminum foil 4. HEPA air purifier 5. Glass-fibre filter	B.F2 [1]	1	0	1	2

Table 1. *Cont.*

Laboratory Space	Methodological Stage and Purpose	Exposure Time	Precautionary Measures	Blank	Blue	Black	Pink	Total
Lab C	Imaging Visual examination, characterization and collection of anthropogenic particles using a Nikon SMZ1500 microscope	2 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use	C.NME1 [2]	3	3	1	7
		2 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use	C.NME2 [2]	7	5	1	13
		2 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Microscope enclosure	C.ME1 [3]	4	2	2	8
		2 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Microscope enclosure	C.ME2 [3]	2	1	1	4
		2 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Microscope enclosure 4. HEPA air purifier 5. Glass-fibre filter	C.ME.F1 [4]	2	0	0	2
		2 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use 3. Microscope enclosure 4. HEPA air purifier 5. Glass-fibre filter	C.ME.F2 [4]	3	1	0	4
Lab D	Imaging Visual examination, characterization and collection of anthropogenic particles using a Nikon SMZ1500 microscope	2 h	1. Cotton laboratory coats worn during preparation and processing 2. Equipment rinsed and wiped down before and after use	D1 D2	4 2	1 1	0 0	5 3

2.2. Review of Previous Studies

We compared 50 studies reporting MPs in sediment with respect to reporting and usage of quality control and assurance (QA/QC) methods. We devised a system similar to those developed by [34,35], which prioritized improving quality standards when reporting MPs in water and biota. These authors accomplished their goals by evaluating numerous records and providing a score based on each quality assurance and control practiced.

We gave scores to the 50 sediment studies by critically examining experimental methodologies and each was given a checkmark if they satisfied the criteria of the following five sections, Lab Preparation and Processing, Clean-Air Devices, Inclusion of Procedural Blank(s), Reported Blank Totals, and Negative Control Corrections (Table 2). Criteria were chosen based on a QA/QC methodological framework constructed by [36]. Developed from agreed-upon methodologies used by numerous laboratories, [36] provides a standardized approach for sediment sampling of microplastics and appropriate QA/QC protocols.

In order to receive a checkmark, a study needed to include a minimum of two precautionary measures for *Lab Preparation and Processing*. A checkmark was allotted if a researcher rinsed all glassware thoroughly and kept it covered with non-plastic material during all procedural processes, if surfaces were regularly wiped down, and if cotton lab coats were worn. A study did not receive a checkmark if the contamination prevention was solely dependent on wearing cotton lab coats.

A checkmark was given to the *Clean-Air Devices* criteria if additional actions were taken to reduce poor air conditions in the laboratory, including the use of a microscope enclosure, a laminar flow cabinet, and/or HEPA air purifiers. A checkmark was not provided in the *Clean-Air Devices* criteria if environmental samples were analysed in an operating fume hood. A recent study states that because fume hoods pull air from the room into the hood, it is not an efficient clean-air condition, as it increases the risk of airborne contaminants [34]. One investigation tested the efficiency of fume hoods when examining microplastic pollution samples and recognized that the laboratory fume hood reduced contamination by 50%, but did not fully prevent it, and could not protect a sample from secondary contamination [37]. The prior study also reported a 97% decrease in fibre contamination when working in a laminar flow hood, in comparison to exposed laboratory air. Conducting laboratory steps in a controlled air environment such as a laminar flow hood and cleanroom is recommended to help minimize air contamination [38], however, it may not eliminate it entirely as both require regular cleaning and maintenance.

A checkmark was given to the *Inclusion of Procedural Blank(s)* column if it accounted for negative controls, specifically procedural blanks. Blank samples were characterized as clean Petri dishes or jars containing previously examined, unpolluted sediment or water. Void Petri dishes and filter papers were also considered, although not the favourable option. In the *Reported Blank Totals* section, if the total number of airborne contaminants was recorded in the paper, it would receive a checkmark. Lastly, if researchers corrected for the microfibre counts in their overall totals and/or results, a checkmark was given to *Negative Control Corrections*. If a study did not satisfy these standards, it was given a 0. Checkmarks were tallied and organized according to risk: high risk (0–1), medium risk (2–3) and low risk (4–5).

Information regarding positive controls were not recorded in Table 2, although they represent an important methodological step when understanding airborne microfibre contamination. Positive controls are not a common practice among MP researchers, especially those specializing in sediment. However, particle recovery tests are being adapted in protocols, which involves spiking a known number of MPs (different size classes and polymer types) during sample processing to determine possible interferences and time-related losses [39,40]. Finally, it is possible that QA/QC measures were taken by the researchers in the examined publications, but these measures were not reported, and therefore, could not be considered in the ranking.

3. Results

3.1. Number, Composition and Intra-Lab Comparisons

The blank samples were examined under a stereomicroscope within a microscope enclosure, and anthropogenic microfibres were the only MP morphology. Microfibers have been documented as one of the most prevalent types of anthropogenic (human-made) particles [10,41]. Ref. [42] characterized microfibres as the dominant anthropogenic debris type recovered from fish digestive tracts. Similarly, [23] characterized cotton textiles as anthropogenic particles (down to ~100 mm) when examining sediment samples from the Arctic. The microfibres in the present study were counted and categorized according to color. A total of 69 microfibres (average ± SD: 5 ± 3.36) were recorded. Three colors were identified in all 14 blank samples, which were blue (39 microfibres; average ± SD: 2.79 ± 1.76), black (20 microfibres; average ± SD: 1.43 ± 1.34), and pink (10 microfibres; average ± SD: 0.71 ± 0.73). The average microfibre length was 2.0 mm, with a minimum and maximum length of 0.87 mm and 4.32 mm, respectively. Average microfibre sizes were comparable amongst all colors: 1.86 mm for blue, 1.69 mm for black, and 2.50 mm for pink microfibres. A 53-micron sieve was used to process sediment, and a magnification limit of 50 microns was used when microscopically identifying particles. All microfibres identified were therefore >53 microns in length. The low number of microfibres in each blank made identification relatively simple. A total of 15 particles (8 blue, 4 black, and 3 pink) were randomly selected (~20%) for ATR-FTIR analysis. Cellulosic fibres dominated polymer counts with 93%, along with one pink polyethylene terephthalate (PET) particle. Preliminary results indicate that the majority of secondary lab contamination is of natural origin. In studies with high numbers of natural microfibres, as determined by FTIR, those microfibres should be removed from the total microplastic contamination count. This will avoid overestimation of the number of synthetic polymers inferred to contaminate samples during processing and examination.

The preliminary results suggest that set-ups B1 and B2 produced greater amounts of airborne contaminants, with an average 7.5 microfibres, in comparison to set-ups A1 and A2, with an average of 1.5 microfibres (Table 1; Figure 2A). However, when comparing average abundances between B1 + B2 and B.F1 + B.F2, contamination decreased by 66%, from 7.5 microfibres to 2.5 microfibres. For lab C, a total of 20 microfibres were identified in samples without a microscope enclosure (C.NME1 and C.NME2), 12 microfibres with the inclusion of a microscope enclosure (C.ME1 and C.ME2), and 6 microfibres with the microscope enclosure, HEPA air purifier and glass-fibre filter (C.ME.F1 and C.ME.F2). When comparing microfibre abundances between C.NME and C.ME samples, the number decreased by only 30%. When comparing blank set-ups between lab D (D1 + D2) and lab C (NME1 + C.NME2) over a two-hour sampling period, lab D exhibited 75% fewer microfibres than lab C, before a microscope enclosure was introduced. An average of 4 microfibres were observed in D1 + D2, in comparison to 10 in set-ups C.NME1 and C.NME2.

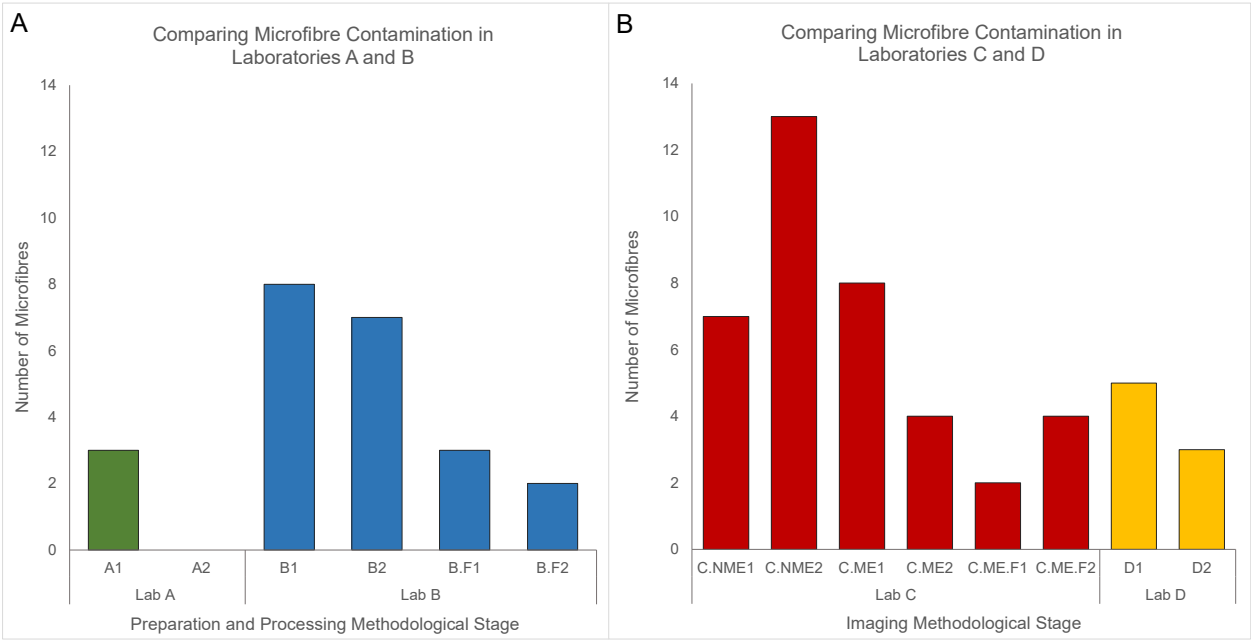

Figure 2. Atmospheric microfibres detected before and after the addition of clean-air devices. (**A**) Comparing controls between labs A and B, during the preparation and processing methodological stage, (**B**) Comparing controls between labs C and D, during the imaging methodological stage.

To test the null hypothesis of no difference in means in microfibre counts between the negative control set-ups in lab B with no clean-air devices and the set-ups with the HEPA air purifier and fibre-glass filter, a t-test was run resulting in a *p*-value of 0.0194. However, due to the low sample size the statistical power of the test is low, and thus there is a lower probability of finding an effect. Additionally, due to the low sample size we cannot test the normality assumptions required for the two-sample t-test. To this end, a non-parametric Mann–Whitney U test was also conducted resulting in a *p*-value of 0.121. This tests the null hypothesis that the medians of these two groups are the same.

Moreover, because three experiments were run in lab C (C.NME, C.ME, C.ME.F), a one-way ANOVA test of the null hypothesis that the means in the groups are the same was conducted, resulting in a *p*-value of 0.218. As the issues of low sample size persist with ANOVA as they do with the t-test, a non-parametric Kruskal–Wallis test was also completed, resulting in a *p*-value of 0.2058. This tests the null hypothesis that the medians of the groups are the same.

Therefore, based on the preliminary results, there is not enough data in this study to infer concrete conclusions. However, the differences in means are promising as is the result of the t-test of means in microfibre count between the negative control set-ups in lab B, which could support a full experimental design in the future. A two-way factorial design is recommended for this type of experiment, which would allow analysis of the impacts of several factors on microfibre counts with a relatively low number of blanks required.

The first would be a 2^3 factorial design with lab (A/B), HEPA purifier (yes/no) and filter (yes/no) as factors. The second would be a 2^4 factorial design with lab (C/D), microscope enclosure (yes/no), HEPA purifier (yes/no) and filter (yes/no) as factors. An ANOVA on the results of these designs would determine the significance of the effects of these factors on microfibre counts.

3.2. Study Comparisons

Table 2 suggests that scientists are not prioritizing quality assurance and controls (QA/QC) in their procedural designs or are not properly reporting their QA/QC data in their papers. The maximum count of 5 was achieved by four sediment studies, and 14 studies (28%) were at a lower risk of airborne contamination, receiving a total of 4

to 5 checkmarks. Only 9 studies recorded the inclusion of clean-air devices, many of which included HEPA air filters or laminar flow hoods. This suggests that 82% of studies are not employing quality controls that could help minimize laboratory contamination. Moreover, 72% of sediment samples fall within medium (0.40) to high risk (0.34). Hence, QA/QC within laboratories may have been repeatedly disregarded, as the mean among the 50 studies is 2.24 (medium risk), with a mode of 0 (high risk) (Figure 3A). This average in addition to the average of each criterion (<1), is consistent with the results identified by [35] for water and biota. The averages determined between the five quality assurance criteria suggest major room for improvement, especially *Clean-Air Precautions* (ave. 0.18) and *Negative Control Corrections* (ave. 0.34). The other criteria averages include *Lab Preparation and Processing* (0.50), *Reported Blank Totals* (0.50), and *Inclusion of Procedural Blank(s)* (0.72) (Figure 3B). All of these criteria are of similar importance, and together they represent a standardized QA/QC approach to minimizing secondary contamination in a laboratory environment when analysing microplastics in sediment.

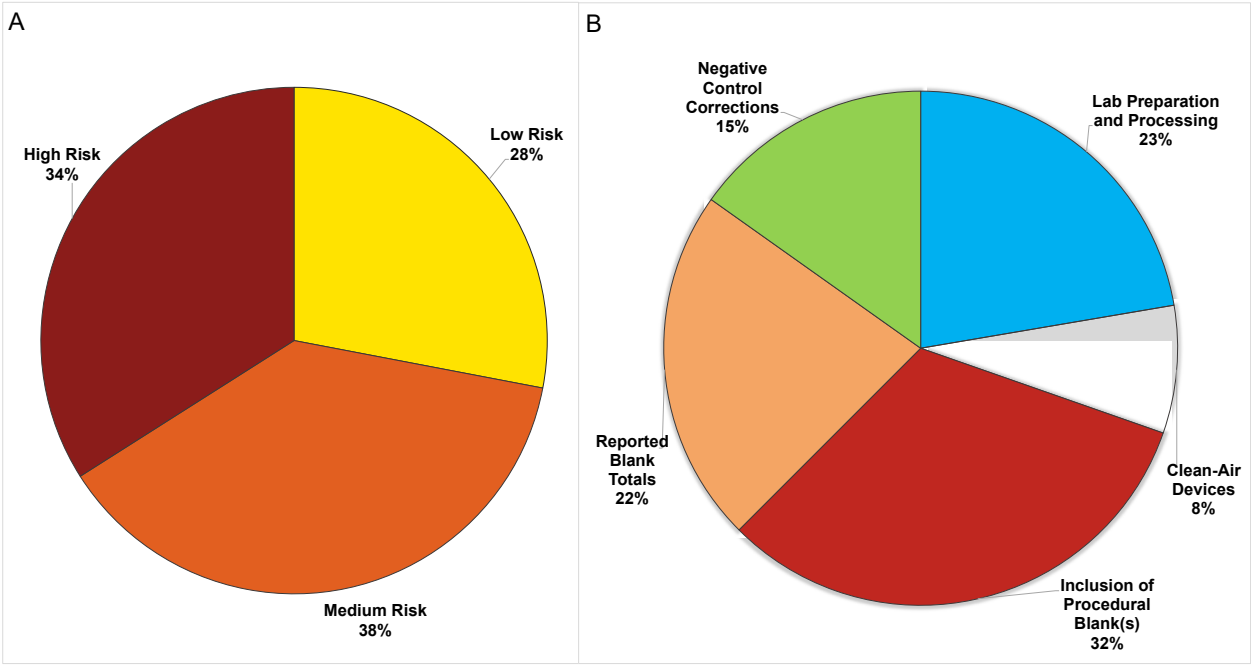

Figure 3. Illustrations of intra-study preliminary results. (**A**) Relative proportion of studies ranking low, medium and high contamination risk. (**B**) Percentage of each QA/QC criterion.

Despite contamination measures being inconsistent across studies, protocols are being adapted to reduce airborne laboratory contamination and cross-contamination between samples. For example, designated cleanrooms are recommended when processing and visually examining microplastics in environmental samples, as precautions are taken to monitor and test cleanliness [38]. Additionally, some ultra-clean room entrances incorporate air showers to remove pollutants from the outside ambient air [43]. Precautions can also be taken to minimize contamination by wearing low-shedding suits and rubber shoes, often used in forensic analysis [44]. As of late, standardized devices such as HEPA filters are being integrated into laboratory methodologies to improve air quality and minimize airborne particulate matter [45,46].

Table 2. Comparing quality assurance and controls in studies investigating microplastics in sediment. A checkmark was allocated to a study if it satisfied the desired criteria, then tallied (0–5) and assigned a risk ranking (Low, Medium, High).

Study # in Ref. List	Type	Lab Prep. and Process	Clean-Air Devices	Inclusion of Blanks	Reported Blank Totals	Negative Control Corrections	Total (Max = 5)	Risk Scale
[47]	Sed.	0	0	1	0	1	2	Medium
[48]	Sed.	0	0	1	0	0	1	High
[49]	Sed., Wat.	1	1	1	1	0	4	Low
[50]	Sed.	1	0	1	1	1	4	Low
[51]	Sed.	0	0	0	0	0	0	High
[52]	Sed.	0	0	1	1	0	2	Medium
[5]	Sed., Wat.	1	0	1	0	0	2	Medium
[53]	Sed.	0	0	1	1	1	3	Medium
[54]	Sed.	0	0	0	0	0	0	High
[55]	Sed., Biota	0	0	0	0	0	0	High
[56]	Sed., Wat.,Biota	1	0	1	1	1	4	Low
[57]	Sed.	1	0	1	1	0	3	Medium
[33]	Sed.	1	0	1	1	0	3	Medium
[58]	Sed.	0	0	0	0	0	0	High
[4]	Sed.	1	0	1	1	0	3	Medium
[59]	Sed., Wat.	0	0	0	0	0	0	High
[60]	Sed.	1	1	1	0	0	3	Medium
[27]	Sed.	1	0	1	1	0	3	Medium
[61]	Sed.	0	0	0	0	0	0	High
[62]	Sed.	0	0	0	0	0	0	High
[63]	Sed.	0	0	1	1	1	3	Medium
[64]	Sed., Wat.,Biota	0	0	0	0	0	0	High
[65]	Sed.	0	0	0	0	0	0	High
[66]	Sed.	0	0	1	1	0	2	Medium
[67]	Sed., Biota	1	0	1	1	1	4	Low
[68]	Sed.	0	0	0	0	0	0	High
[69]	Sed., Wat.	1	0	1	0	0	2	Medium
[70]	Sed.	0	0	0	0	0	0	High
[71]	Sed.	1	0	1	0	0	2	Medium
[2]	Sed., Wat.,Biota	1	0	1	1	0	3	Medium
[12]	Sed., Sewage	0	0	0	0	0	0	High
[6]	Sed.	1	0	1	0	1	3	Medium
[32]	Sed.	0	0	0	0	0	0	High
[72]	Sed., Wat.	1	1	1	1	1	5	Low
[73]	Sed., Wat.	0	0	1	1	0	2	Medium
[22]	Sed.	1	1	1	1	1	5	Low
[74]	Sed.	0	0	1	1	0	2	Medium
[75]	Sed.	1	0	1	1	1	4	Low
[76]	Sed.	0	0	1	0	0	1	High
[23]	Sed., Wat., Biota, Snow	1	1	1	1	1	5	Low
[77]	Sed., Biota, WWTP Effl.	1	1	1	1	1	5	Low
[9]	Sed., Wat.	1	0	1	1	1	4	Low
[78]	Sed.	1	0	1	1	1	4	Low

Table 2. *Cont.*

Study # in Ref. List	Type	Lab Prep. and Process	Clean-Air Devices	Inclusion of Blanks	Reported Blank Totals	Negative Control Corrections	Total (Max = 5)	Risk Scale
[79]	Sed., Wat., Biota, Snow	0	1	1	1	0	3	Medium
[80]	Sed.	1	1	1	0	1	4	Low
[81]	Sed., Biota	0	0	1	0	0	1	High
[82]	Sed.	0	0	0	0	0	0	High
[83]	Sed.	1	1	1	0	0	3	Medium
[84]	Sed.	1	0	1	1	1	4	Low
[39]	Sed.	1	0	1	1	1	4	Low
Average		0.50	0.18	0.72	0.50	0.34	2.24	

None of the analysed studies mentioned the application of *Clean-Air Devices* during all methodological phases, thereby presenting a methodological gap when running experiments, especially when investigating MPs in sediment. According to the *Inclusion of Procedural Blank(s)* average, the issue of background contamination in sediment samples is growing. However, when comparing sediment records, only half of the evaluated studies (n = 25) had stated having taken necessary precautions when preparing and processing samples. This implies that 50% of sediment studies are not embracing an essential QA/QC standard when in the laboratory.

When comparing reports that detail MPs in sediment, the columns concerning negative controls within the lab (*Inclusion of Procedural Blank(s), Reported Blank Totals* and *Negative Control Corrections*), are inconsistent. Notably, 50% of sediment reports did not record total microfibre counts from blank samples, and 66% did not correct for negative controls identified. Neglecting to adopt strict QA/QC measures for perceived airborne laboratory contamination can lead to risks associated with microfibres, potentially falsifying total counts, resulting in an overestimation of environmental concentrations.

Many of the studies exhibited inconsistencies and ambiguity during sample preparation and processing. For example, some studies expressed that the researchers had only worn cotton clothing during experimental processes, which is not a preventative measure against microfibre shedding. However, any cotton garment could be worn when quantifying microfibres and/or microplastics in samples because indistinguishable cotton polymers can be eliminated from the screening process after chemical analysis. Further actions are required, such as covering glassware with aluminium foil, and rinsing equipment and supplies thoroughly after use. Alternatively, some studies stated that lab coats were worn, but failed to state if the coat was composed of cotton vs. polyester. Cotton lab coats are often absent in MP studies, and had not become consistent laboratory staples until 2014 [85]. Microfibres could also settle out from the air onto a lab coat worn in the laboratory. To test this theory, a simple tape test was completed on a white cotton lab coat. The taping procedure was modified from [86], a study investigating the "sheddability" of textiles. A small piece of double-sided tape (30 × 12 mm) was placed on a transparent microscope slide and examined for potential microfibre contamination under the microscope. The slide with the tape was then applied gently to an area on the coat. This test was completed twice, on the coat collar (Test 1) and the right sleeve (Test 2). The coat collar was selected because it would theoretically be a hotspot for airborne microfibre contamination when worn or placed aside, and the right sleeve was selected because it was the examiner's dominant picking hand. Test 1 on the coat collar, revealed a total of 12 microfibres; 3 black, 6 blue, 2 pink, and 1 green. Test 2 on the right sleeve, revealed a total of 4 microfibres; 2 pink, 1 blue, and 1 black.

A checkmark was justified for the *Inclusion of Procedural Blank(s)* criteria, even if the method to collect microfibre fallout varied. For instance, a checkmark was provided if a study used a jar of water, filter paper or an empty Petri dish.

4. Discussion

Our experimental study indicates that clean-air devices, such as a HEPA air purifier and microscope enclosure, are relatively simple and cost-effective laboratory applications that are highly recommended when investigating microfibres in sediment, water and biota samples. The 66% decrease in microfibre contamination from B1 + B2 to B.F1 + B.F2 is interpreted to be the result of adding the HEPA air purifier. Microfibre contamination was examined in a white and black carbon filter positioned within one of the HEPA air purifiers. A 5 × 5 cm filter dimension exhibited a total of 4 fibres (pink, blue, black) in the white filter, and 104 in the black carbon filter (pink, blue, black, yellow, and green). After extrapolating abundances to scale (43 × 7 cm), ~1296 of microfibres were estimated to have accumulated in the HEPA air purifier since installation (approx. 1 year), indicating that ~3.55 microfibres are being caught per day. Ensuring these filters are frequently replaced will result in optimal functionality and the reduction in atmospheric particles. However, between the two installed fibre-glass filters (C.ME.F and B.F) negligible counts were observed, with only 3 microfibres (2 blue, 1 black). This could infer that the shedding of clothing and the transport of microfibres into a working environment is a significant contributor to secondary laboratory contamination, rather than the input of air-exhaust systems.

The 30% reduction in microfibre contamination from C.NME and C.ME samples indicates that the microscope enclosure slightly decreased the amount of microfibre fallout and transport to the stereomicroscope. However, blank samples exhibited a 70% decline in microfibre contamination with the use of all clean-air devices (Figure 2B). These preliminary results suggest that the actions taken to improve air quality have been positive, showing a decline in airborne microfibre counts with the addition of a HEPA air purifier and microscope enclosure.

Fewer microfibre totals in lab D compared to C could be linked to lab D's larger operating space and microscope location. Lab D is approximately 2× larger than lab C, and is frequented less by users, which can significantly decrease microfibre shedding. The location of the stereomicroscope, during the imaging methodological stage, could also contribute to a sample containing fewer or greater microfibre airborne contaminants. For instance, if a sample is being examined under a microscope (without a clean-air device) and is proximal to a window or door, or possibly an air-exhaust system, microfibres could be easily transported into the room and settle on adjacent surfaces. The size and layout of lab C makes it a suitable place for microfibres to persist. The microscope and enclosure are positioned in front of a large window (due to accessibility and lack of available space) and are approximately 1 m away from an air exhaust system, and roughly 2–3 m from the entrance. Lab D is a larger lab with the microscope (without an enclosure) located in the center of the room, away from all windows, doors, and exhaust units, and is approximately 5–6 m away from the entrance.

Despite precautions taken to avoid background contamination in lab B with the glass-fibre filter (B.F) and lab C with the microscope enclosure and filter (C.ME.F), preliminary results suggest that microfibres can unintentionally be transferred into the microscope enclosure during examination. This implies that these low-density microfibres can be easily transported by air in any working environment, even when wearing a cotton laboratory coat, wiping down surfaces and equipment, and covering glassware with aluminium foil. Moreover, if cotton laboratory coats are not washed, or if a lint remover is not applied to the garment regularly, airborne dust particles carrying microfibres could settle onto the coat and potentially shed into environmental samples. Secondary contamination could also be inadvertently introduced into an environmental sample from RO water used to rinse equipment, and from purchased salt-based solutions used for gravity separation.

Anthropogenic microfibres could be carried through the glass-fibre air filters in laboratories B and C, as the filtration efficiency is only 80–85%. The accessibility of our processing laboratory may also come into play when determining fibre source contamination. The entrance of lab B is from the inside of an adjoining lab. Although the door connecting the two rooms is closed at all times, this design could increase the chance of contamination

if researchers are entering and leaving lab B frequently. For instance, when examining particulate matter (dust), researchers revealed that even light activity, such as walking, can impact airborne particles greater than 5 mm, causing resuspension [37]. Notably, [25] determined that deposition rates between 1586 and 11,130 microfibres/day/m^2 occur in an indoor environment, implying that particles ranging from 190 to 670 microfibres/mg are accumulating in settled dust. If researchers were to prepare, process and visually examine environmental samples in one space, with prioritization given to clean-air devices, the risk of background contamination would decline, as the movement of microfibres in dust would be better controlled [34].

Table 2 was created to better understand if researchers are taking the necessary actions to prevent background contamination within their laboratories and sediment samples. The comparison preliminary results can be used to identify trends and emphasize areas that need to be improved. For example, the year in which the study was published has an impact on the number of precautions taken to eliminate background contamination. Many MP researchers a decade ago were unaware that microfibre pollution would become a significant problem for environmental samples. As a result, QA/QC controls were often neglected. This is evident when comparing the 50 sediment studies from 2010 to 2021. Seven studies from the years 2010 to 2014 did not receive a checkmark for any of the five criteria, implying their laboratories were at a higher risk of contamination or that the authors failed to report any of their QA/QC controls. Since 2014, 72% of the publications examined have mentioned blank samples. In the past five years (2016 to 2021), 12 records demonstrate low risk, 15 medium risk, and 7 are of high contamination risk. Therefore, despite knowledge of microfibre contamination growing in scientific popularity, sediment studies continue to lack necessary controls to minimize background contamination in laboratories.

5. Conclusions

This study acknowledges that the described criteria are not an absolute judgement of the value of studies, as not all reports investigating MPs in sediment were evaluated. In addition, the QA/QC methods involved in each study may not have been presented in each publication. Nonetheless, improvements are required and recommended for all examined criteria. Preliminary results suggest that studies are at a higher risk of airborne contamination if they do not include precautionary measures when preparing, processing and microscopically examining samples, and that alternative devices should be prioritized when advancing indoor air quality. Working in an ultra-clean room and employing the mentioned QA/QC controls, emphasizing the inclusion of procedural blanks and clean-air devices, are possibly the best methods to minimize and prevent unwanted laboratory contamination, improve data quality and reliability, and the standardization of methods. Therefore, standardized QA/QC approaches and protocols are recommended to mitigate laboratory background contamination, to evade research error, and to encourage reproducible and comparable results across studies.

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