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Critical Review

Are We Underestimating Anthropogenic Microfiber Pollution? A Critical Review of Occurrence, Methods, and Reporting

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Abstract: Anthropogenic microfibers, a ubiquitous environmental contaminant, can be categorized as synthetic, semi-synthetic, or natural according to material of origin and production process. Although natural fibers, such as cotton and wool, originated from natural sources, they often contain chemical additives, including colorants (e.g., dyes, pigments) and finishes (e.g., flame retardants, antimicrobial agents, ultraviolet light stabilizers). These additives are applied to textiles during production to give textiles desired properties like enhanced durability. Anthropogenically modified "natural" and semi-synthetic fibers are sufficiently persistent to undergo long-range transport and accumulate in the environment, where they are ingested by biota. Although most research and communication on microfibers have focused on the sources, pathways, and effects of synthetic fibers in the environment, natural and semisynthetic fibers warrant further investigation because of their abundance. Because of the challenges in enumerating and identifying natural and semisynthetic fibers in environmental samples and the focus on microplastic or synthetic fibers, reports of anthropogenic microfibers in the environment may be underestimated. In this critical review, we 1) report that natural and semisynthetic microfibers are abundant, 2) highlight that some environmental compartments are relatively understudied in the microfiber literature, and 3) report which methods are suitable to enumerate and characterize the full suite of anthropogenic microfibers. We then use these findings to 4) recommend best practices to assess the abundance of anthropogenic microfibers in the environment, including natural and semisynthetic fibers. By focusing exclusively on synthetic fibers in the environment, we are neglecting a major component of anthropogenic microfiber pollution. *Environ Toxicol Chem* 2022;41:822–837. © 2021 SETAC

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INTRODUCTION

Microfibers are a widespread environmental contaminant, which originate from textiles, (e.g., clothing) and personal care products, such as wet wipes, cigarette filters, and other fibrous materials. Textiles shed fibers throughout their life cycle, from production (Chan et al., in press; Zhou et al., 2020) to normal use (De Falco et al., 2020), including during washing (Athey et al., 2020; Cai et al., 2020; De Falco et al., 2020; Napper & Thompson, 2016) and drying (Kapp & Miller, 2020; Pirc et al., 2016). Textiles then continue to release fibers after disposal (Gavigan et al., 2020; Sun et al., 2021). Although textiles are currently the best-studied source of microfibers to the environment (Carr, 2017; Gavigan et al., 2020), recent studies show personal care products (e.g., wet wipes, face masks), carpeting, and cigarette filters also release microfibers (Alipour et al., in press; Belzagui et al., 2021; Fadare & Okoffo, 2020; Ó Briain et al., 2020; Saliu et al., 2021; Shruti et al., 2020; Soltani et al., 2021). Preliminary estimates suggest that microfiber inputs from other sources, collectively, may rival those of textiles (see Belzagui et al., 2021).

The widespread contamination of microfibers in the environment is well documented and has fostered much research in recent years. Many studies, across environmental compartments, have reported microfibers as the most common anthropogenic microparticles in samples, including wastewater (Gies et al., 2018; Grbić et al., 2020), stormwater runoff (F. Liu et al., 2019; Piñon-Colin et al., 2020), rivers (Avio et al., 2020; Warrack et al., 2017), lakes (Grbić et al., 2020; Wang et al., 2017; Whitaker et al., 2019), estuaries (Bessa et al., 2018; Bikker et al., 2020; Naidoo et al., 2020), marine waters (Barrows et al., 2018; Suaria et al., 2020), and wildlife (Athey et al., 2020; Kühn

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et al., 2020; Waddell et al., 2020; Xu et al., 2020). Recent studies have found that microfibers from urbanized environments may undergo long-range atmospheric transport (Cai et al., 2017; Dris et al., 2016) to farmland (Klein & Fischer, 2019) and remote regions including the Arctic (Athey et al., 2020; Huntington et al., 2020; Ross et al., 2021) and mountain summits (Allen et al., 2019; Napper et al., 2020). Because of their prevalence in the environment and documented ingestion by a variety of biota (Avio et al., 2020; Lourenço et al., 2017; Savoca et al., 2019; Waddell et al., 2020), microfibers are a growing concern globally.

Studies report a diverse mixture of microfibers in the environment, which contain a variety of materials and chemicals. Approximately 60% of textiles are produced from synthetic materials (e.g., nylon, polyester; Carr, 2017). Semisynthetic fibers (sometimes called "artificial fibers") include regenerated cellulose fibers used in clothing (e.g., rayon, modal, lyocell; De Falco et al., 2019), personal care products (e.g., viscose; Ó Briain et al., 2020), and cigarette filters (e.g., cellulose acetate; Belzagui et al., 2021). Although derived from natural materials, semisynthetic fibers undergo chemical processing and are extruded into filaments, similar to synthetic fibers. Natural fibers, on the other hand, are not extruded and retain morphological characteristics of their source material. Natural fibers have been used for thousands of years, and today they are commonly used in textile production (De Falco et al., 2019; Zambrano, Pawlak, Daystar, Ankeny, Goller, & Venditti, 2020), as well as the production of personal care products, such as wet wipes (Ó Briain et al., 2020). Because the sources of natural fibers vary, they are often classified depending on the material of origin as cellulosic (plant-based; e.g., cotton), proteinaceous (animal-based; e.g., wool), or mineral (e.g., asbestos; Figure 1). Despite being derived from natural materials, "natural" fibers often contain a suite of chemical additives, dyes, and finishing agents added during production. Chemical additives can include toxic compounds, such as bisphenols, azo dyes, polyfluorinated alkyl compounds (PFAS), and formaldehyde (Lacasse & Baumann, 2004; Ladewig et al., 2015; Schellenberger et al., 2019; Sørensen et al., 2020; Xue et al., 2017; Zambrano, Pawlak, Daystar, Ankeny, & Venditti, 2020). Exposure to leachates from both synthetic and "natural" textiles has been shown to cause adverse effects in aquatic organisms (Belzagui et al., 2021; Carney Almroth et al., 2021). Further, the ingestion of microfibers may exacerbate the adverse effects of these toxic chemicals (Belzagui et al., 2021).

Laboratory studies show that synthetic microfibers have a range of toxicological effects (Kutralam-Muniasamy et al., 2020), from no effects to effects at the cellular level (Qiao et al., 2019; Song et al., 2019) to organism-level effects (reduced energy reserves and/or reduced feeding; Au et al., 2015; Cole et al., 2019; Jemec et al., 2016; Selonen et al., 2020; Setyorini et al., 2021; Watts et al., 2015; Welden & Cowie, 2016). Despite the documentation of ingested semisynthetic and natural fibers in a variety of biota (Athey et al., 2020; Bessa et al., 2018; Savoca et al., 2019), most microfiber toxicity studies have investigated the effects of synthetic microfibers. The effects of anthropogenically modified natural and semisynthetic fibers remain understudied. Thus, it is still generally unknown whether the toxicological effects of nonsynthetic microfibers differ from those of synthetic fibers. However, two recent studies show that nonsynthetic and synthetic microfibers cause similar impacts in freshwater and marine invertebrates (Kim et al., 2021; Mateos-Cárdenas et al., 2021).

Research on the fate of synthetic microfibers overshadows research on natural and semisynthetic microfibers. However, many recent studies report semisynthetic and anthropogenically modified natural fibers in a range of environmental compartments (Grbić et al., 2020; Sanchez-Vidal et al., 2018; Stanton, Johnson, Nathanail, MacNaughtan, et al., 2019; Suaria et al., 2020), including biota and human tissues (Athey et al., 2020; Pauly et al., 1998). Natural and semisynthetic microfibers have often been excluded from studies with the assumption that nonplastic fibers are readily biodegradable and/or harmless in the environment (Ladewig et al., 2015; Re, 2019; Wiesheu et al., 2016). Although natural and semisynthetic fibers can break down more quickly than synthetic polymers, these fibers can persist from months to decades in aquatic systems depending on the microfiber type and environmental factors (Belzagui et al., 2021; Puls et al., 2011; Turner et al., 2019; Zambrano, Pawlak, Daystar, Ankeny, Goller, Venditti, 2020). Anthropogenic modification (e.g., dyes, treatments) may also prolong fiber persistence in the environment (Park et al., 2004; Sait et al., 2021; Sørensen et al., 2020). Given their widespread occurrence, persistence in the environment, and potential impacts on biota, anthropogenically modified natural

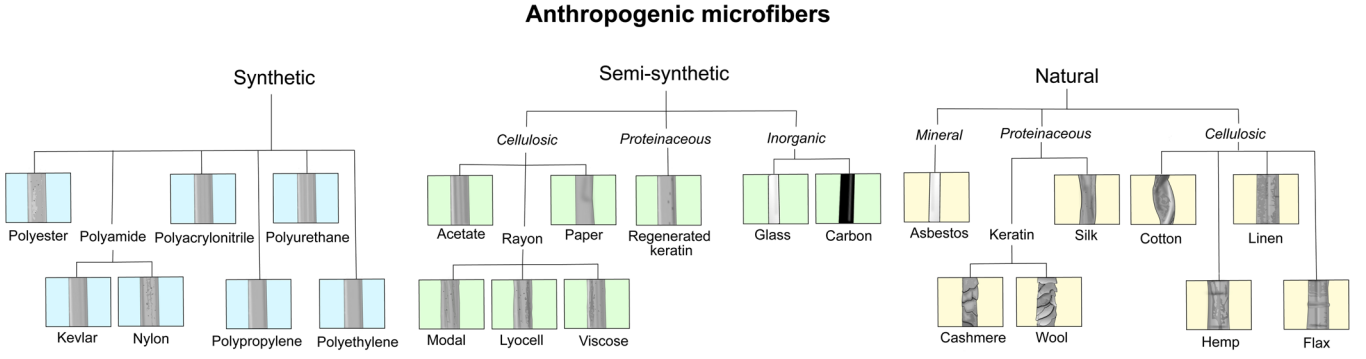


FIGURE 1: Overview of anthropogenic microfibers.

and semisynthetic fibers warrant further attention from the microfiber research community.

However, studying natural and semisynthetic microfibers in environmental matrices offers a unique set of challenges compared to synthetic fibers. Because the methods used to enumerate and characterize microfibers in the environment were originally designed for the recovery of synthetic materials, not all methods are suitable for natural and semisynthetic fibers. For example, certain chemical digestants used to isolate microfibers from organic-rich environmental matrices may result in the partial or complete degradation of nonsynthetic fibers (Dehaut et al., 2016; Treilles et al., 2020). While techniques that identify the material or chemical composition of microfibers (e.g., Raman and Fourier transform infrared [FTIR] spectroscopy) are commonly used, spectroscopic analysis of natural and semisynthetic microfibers can be more challenging compared to that of synthetic fibers. For example, one particular challenge is the low spectral signal intensities of natural materials, which make nonsynthetic fibers more prone to dye interference and reduced hit quality index values (Lenz et al., 2015; Zhu et al., 2019).

Given the historic focus on synthetic fibers and the challenges of studying natural and semisynthetic fibers, the goal of the present study was to determine if current methods for detecting and reporting anthropogenic microfibers in environmental compartments comprehensively capture and document the occurrence of semisynthetic and natural microfibers. Our objectives were to 1) identify the frequency and relative abundance of natural and semisynthetic microfibers reported in the literature compared to synthetic fibers, 2) identify environmental compartments and geographic regions that are relatively understudied in the microfiber literature, and 3) assess methods employed for capturing, enumerating, and characterizing the full suite of anthropogenic microfibers. Finally, we 4) recommend best practices to assess the abundance of anthropogenic microfibers in the environment, including natural and semisynthetic fibers.

METHODS

A systematic literature review was conducted of citations documenting microfibers in the environment published online from 2011 (the earliest publication identified) through October 2020. Our search included keyword searches with the following terms: "microfibers" AND "microplastic"; "anthropogenic fibers"; "microplastic" AND "fibers"; "microplastic" AND "threads"; "plastic microfibers"; "microplastic" AND "lines"; "microplastic fibers"; "synthetic fibers" AND "microplastic"; "textile fibers" AND "microplastic" using Web of Science (Core Collection database), SCOPUS, and Google Scholar (Figure 2). The protocol follows the guidelines set by the Preferred Reporting Items for Systematic Reviews and Meta-Analyses protocols (Moher et al., 2015). Although there is no standard definition for microfibers in an environmental context, most studies refer to textile fibers as "microfibers," "fibers," and "threads," while "lines" and "filaments" typically refer to the remnants of fishing gear (Avio et al., 2020; Murray & Cowie, 2011). Because of the lack of standardized terminology and range of sources in the literature, we included "lines," "fibers," and "threads" in our analyses. Most studies define microfibers in terms of their length, with an upper limit of either 1000 μm (Browne et al., 2011) or 5000 μm (Dris et al., 2016; Hartline et al., 2016; Kosuth et al., 2018). Inconsistencies in size classification stem from debate over the formal size bins defining microplastic pollution (Hartmann et al., 2019). Given the ongoing debate, we did not define an upper size limit. For all citations identified through keyword searching, the title and abstract were further screened to exclude those deemed irrelevant to the present study. For the citations deemed

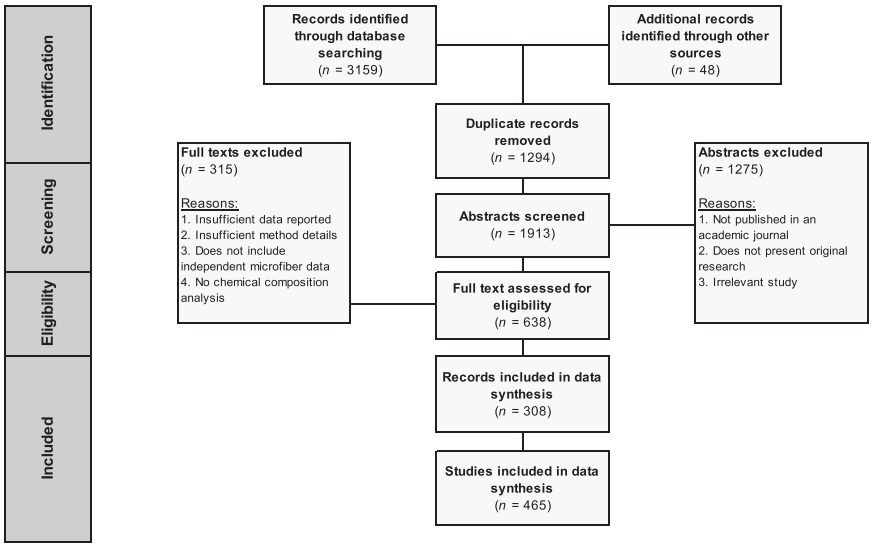


FIGURE 2: Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) diagram. The diagram presents the results and screening from the original literature searches and the return of these searches.

relevant during the first screening, the full text was reviewed in the second screening (Figure 2). We included only citations presenting original research published in peer-reviewed journals that document the abundance of microfibers in environmental compartments. Reviews, toxicity testing and laboratory studies, as well as studies documenting microfiber abundance in products sold for human consumption, aqua-cultured biota, and human tissues were not included. Further, because of our interest in natural and semisynthetic fibers, we only included citations that confirmed the material composition of fibers using confident identification methods, including Raman spectroscopy, FTIR spectroscopy, and pyrolysis gas chromatography-mass spectrometry (pyr-GC-MS).

We investigated microfiber abundance in the following environmental compartments: terrestrial soils, marine and freshwater sediments, indoor and outdoor air, indoor and outdoor dust, ice and snow, marine and fresh subsurface and surface waters, wastewater-treatment plant influent and effluent, sewage sludge and biosolids, as well as marine, freshwater, and terrestrial biota. When there were multiple environmental compartments examined for a single publication (e.g., the authors assessed microfibers in biota and sediment), we included each environmental compartment as an independent data point (or row in our database). Hereafter, we refer to each data point as a separate environmental "study," and we refer to each whole published work as a "publication." For example, Athey et al. (2020) investigated the occurrence of microfibers in freshwater sediments, marine sediments, freshwater biota, and wastewater; therefore, this publication encompassed four rows and was treated as four studies.

For each study, the following information was extracted (when available): the geographic location where samples were collected and the year(s) of collection, type of environmental media investigated, methods used for sample collection and enumeration, intermediate screening steps applied (if applicable), technique for the identification of the material composition of fibers, proportion of fibers present, and type(s) and proportions of materials identified (including synthetic, natural, and semisynthetic). These details were recorded in Microsoft Excel (Ver 16.44). In some cases, unit conversion using reported values was required to compare mean particle abundances (including microfibers) across studies. Descriptive statistics were completed in R (Ver 3.6.1).

RESULTS

Study selection and trends in the literature

A total of 1913 peer-reviewed publications were identified through keyword searches using the Web of Science (20%) and SCOPUS (79%) databases and Google Scholar (1%; Figure 2), after removing duplicate entries. Next, 1275 citations were dismissed in the first screening based on the title and abstract. These citations either 1) were not in a peer-reviewed journal, 2) did not present original research, or 3) did not meet inclusion criteria. During the second screening of the full text, 315 citations were excluded for the following reasons: 1) insufficient microfiber data (i.e., quantitative or spectral information on microfibers not included), 2) insufficient explanation of method, 3) microfibers were excluded from analysis (e.g., because of concern over airborne microfiber contamination of environmental samples; Besseling et al., 2015; Bosshart et al., 2020; Compa et al., 2020), or 4) material composition did not include a reliable technique such as Raman/FTIR spectroscopy or pyr-GC-MS. Over half of the citations ($n = 191$) included in our second screening were excluded because material composition was not confidently identified, which in part reflects a shift in expectations in the literature over time because material composition is common in current studies. A total of 308 publications were included in this systematic review (10% of papers originally identified). Because nearly one-third of the publications ($n = 97$) that we included in our final analyses assessed more than one environmental compartment and each compartment was treated as an independent data point or study (see *Methods*), we included a total of 465 data points or studies in our analysis.

Browne et al. (2011) published the first study on microfibers in environmental samples with material composition analysis (e.g., spectroscopy). Their study was also the first to apply the term "microfiber" to contaminant fibers found in environmental samples (Browne et al., 2011). While studies dating back to the early 2000s documented the presence of microfibers in environmental compartments (Habib et al., 1998; Zubris & Richards, 2005), they were not included because they did not analyze the material composition of the fibers. Since 2011, monitoring and reporting microfiber contamination in the environment has grown (see Supporting Information, Figure S1). The increase in the number of studies was observed both for studies that include natural and/or semisynthetic microfibers and for studies that only present synthetic microfibers.

Generally, the number of studies reporting nonsynthetic fibers has increased over time, with a sharp increase around 2017 (Supporting Information, Figure S1). Early studies tended to report synthetic or semisynthetic microfibers. Recent studies have more frequently reported natural and semisynthetic microfibers. Ladewig et al. (2015) highlighted the potential for natural fibers as vectors for toxic compounds in aquatic ecosystems and the need to study natural fibers in the environment, which may have initiated a shift in the literature.

Occurrence of natural and semisynthetic microfibers

Most studies (58%) report finding natural and/or semisynthetic fibers in environmental samples. The earliest reports of natural and semisynthetic microfibers in environmental compartments were of regenerated cellulose fibers (e.g., rayon) found in sea ice collected in the Arctic Ocean in 2005 and 2010 (Obbard et al., 2014), as well the gastrointestinal tracts of fish collected in 2010 in the English Channel (Lusher et al., 2013). Rayon fibers were also dominant in deep-sea sediment samples collected between 2001 and 2012 in the Atlantic Ocean, the Indian Ocean, and the Mediterranean Sea (Woodall et al., 2014). Rayon (sometimes called "cellophane,"

"modal," "lyocell," or "viscose") is made of semisynthetic fibers used in the production of clothing, cigarette filters, and personal care products (Belzagui et al., 2021; Bredereck & Hermanutz, 2008; Fadare & Okoffo, 2020; Ó Briain et al., 2020; Zambrano et al., 2019). Despite being derived from natural materials, rayon fibers have been classified as "microplastic" by some authors because of the addition of chemical additives (Peng et al., 2020; Qu et al., 2018).

Of those studies that reported the presence of natural/semisynthetic fibers, 43% ($n = 116$) reported the proportion of fibers that were natural/semisynthetic. The remaining studies ($n = 225$) generally described finding natural/semisynthetic microfibers but did not indicate the abundance of these fibers in relation to synthetic fibers. Approximately half (46%) of studies that report the proportion and/or abundance of natural/semisynthetic fibers found nonsynthetic fibers to be more abundant that synthetic fibers. The most common nonsynthetic materials were cellulosic fibers (e.g., cotton and rayon) and wool fibers. Six studies purposely stated that they excluded cellulosic and other natural materials from their analyses. Some studies included cellophane and rayon fibers as synthetic materials but excluded natural fibers such as cotton and wool (Deng et al., 2020; Neves et al., 2015; Wright et al., 2020).

Understudied environmental compartments and geographic regions

Natural and semisynthetic microfibers have been reported across the world, including in most of the locations where synthetic fibers have been reported. The 465 studies investigated in the present study were distributed between 61 different countries and seven major oceans and seas. Although the two most heavily studied continents were Asia (40%; 187 studies) and Europe (31%; 144 studies), anthropogenic microfibers, including natural and semisynthetic fibers, have been reported in every continent and ocean. As a single country, China has produced the most studies investigating the occurrence of microfibers with 121 studies, followed by England with 24 studies. Most studies in China have focused on marine and freshwater environments with samples from major rivers (e.g., Yangtze River, Qin River, Pearl River) and seas (e.g., Bohai Sea, Yellow Sea, South China Sea). We found only three studies investigating anthropogenic microfibers on the African continent, making it the most understudied continent on the planet (followed by Antarctica with four studies). Coordinated efforts and networks between research groups can be important to support microplastics and microfiber research in understudied regions and could address some of the data gaps highlighted in the present review (Nel et al., 2021; Pan-African Micro[Nano] Plastic Research Network, n.d.; Waller et al., 2017).

We found that the indoor environment is relatively understudied in the microfiber literature compared to the outdoor environment. Only four studies documenting anthropogenic microfibers in indoor compartments (e.g., air and dust) were identified, which is nearly two orders of magnitude lower than the number of studies on microfiber contamination in outdoor environmental compartments (Figure 3). Research on the indoor environment is relatively new (first published in 2016),

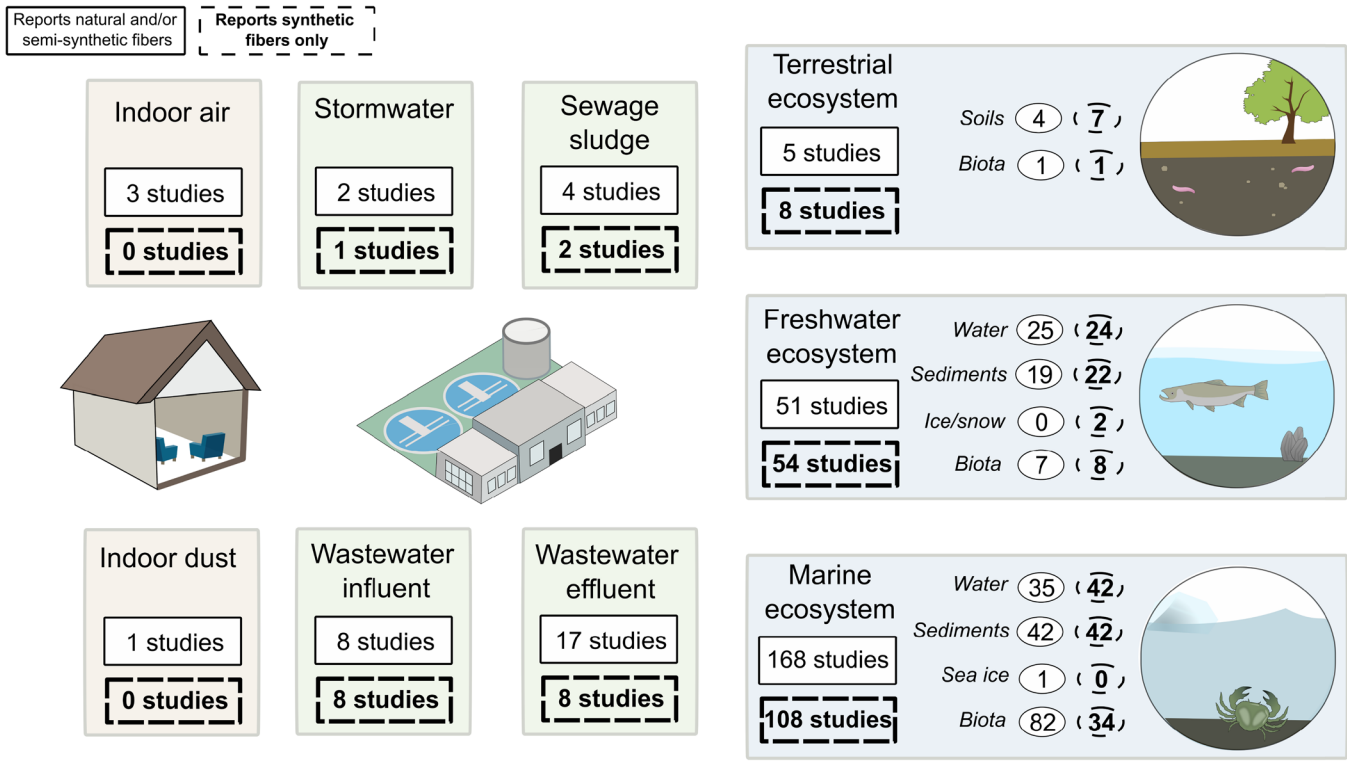


FIGURE 3: Number of studies that report only synthetic fibers (dashed outline) and number of studies that report natural and/or semisynthetic microfibers (solid outline) conducted on different environmental compartments.

which could account for this difference. Microfibers constituted 13 to 100% and 100% of the total anthropogenic particles in studies on indoor air ($n = 3$) and dust ($n = 1$), respectively. While studies were limited, data suggest that natural and semisynthetic fibers are common in indoor air and dust. All of the studies investigating indoor air and dust document natural and semisynthetic microfibers. Further, three of four studies report nonsynthetic fibers to be more abundant than synthetic fibers. Thus, we identify the indoor environment as a priority compartment for future research on microfibers. We spend >90% of our time in the indoor environment (Leech et al., 2002; Setton et al., 2013), where we may be exposed to microfibers via inhalation (Pauly et al., 1998; Vianello et al., 2019) and accidental ingestion via contaminated dust or airborne fiber fallout (Catarino et al., 2018). More research on the occurrence of microfibers in indoor environments (e.g., office vs. home) is needed to better understand whether microfiber type varies between environments, the magnitude of human exposure, as well as the acute and chronic effects of airborne microfiber exposure. Cox et al. (2019) recently estimated that annual inhalation of microplastic particles (including synthetic microfibers) ranges from 48,000 to 62,000 particles, concluding that inhalation was the greatest human health exposure pathway. While studies investigating the effects of microfiber inhalation are limited, in vivo studies conducted in industrial settings and in vitro research on human lung organoids have suggested acute and chronic health impacts following exposure to synthetic and nonsynthetic microfibers (Eschenbacher et al., 1999; Hours et al., 2007; Winkler et al., 2021). Given their abundance in indoor air and dust, future studies estimating human exposure to microfibers should incorporate natural and semisynthetic microfibers to better understand the threat that the full suite of anthropogenic microfibers poses to human health.

When looking at pathways to the environment, most studies have focused on wastewater-treatment plant effluent, whereas other important pathways remain relatively understudied. Although biosolid application to land is an important route introducing microfibers to the terrestrial environment (Gavigan et al., 2020), only six studies investigated microfiber occurrence in sewage sludge or biosolids. The mean particle abundance ranged from 53.5 to 4044 particles per gram dry weight, with microfibers constituting 15 to 100% of the total particle load found in sewage sludge and biosolid samples (Figure 4). For decades, the presence of microfibers has been used as an indicator of sewage sludge application in terrestrial soils (Habib et al., 1998; Zubris & Richards, 2005), with recent work confirming that microfiber concentration in soils indicates biosolid application (Corradini et al., 2019). Biosolid application to land is considered to be one of the largest known sources of microplastics and microfibers to terrestrial ecosystems (Weithmann et al., 2018), and this is expected to increase as the amount of treated wastewater around the world continues to grow (Gavigan et al., 2020). Future research should investigate how microfibers behave in sludge and in agricultural soils. Although there is a growing body of research on the biodegradation of different microfiber types in water, it is still unknown how natural, semisynthetic, and synthetic microfibers behave in sludge and whether some microfiber types biodegrade in these conditions. Further, organic chemical contaminants applied to textiles such as PFAS can be released to the environment via microfibers, either as the original organic chemical contaminant or in a transformed state (Schellenberger et al., 2019). Thus, research should investigate how microfibers contribute to contaminant loading in terrestrial environments and whether textile-specific chemical congeners are entering food systems.

Another understudied pathway we identified is storm water, which includes surface water from precipitation runoff. Only three studies have investigated the occurrence of microfibers in storm water. When reported, mean particle concentrations in storm water range from 15.4 to 175 particles per liter (Figure 4), and 41% of particles were microfibers. These studies suggest that untreated storm water is a major conduit for microfibers to coastal wetlands and freshwater systems and warrants further attention from the research community (Grbić et al., 2020; F. Liu et al., 2019; Piñon-Colin et al., 2020). Because microfiber emissions from storm water can far exceed emissions from other pathways, work should include investigations of storm water to look at its relative contribution to microfiber emissions. For example, in San Francisco Bay, the average concentration of anthropogenic particles in storm water was approximately 140 times greater than that in wastewater (Zhu et al., 2021). Thus, this pathway should be prioritized for future research, to understand the relative contributions of microfiber emissions from storm water in different environs. By understanding the relative contributions of microfibers from different pathways, mitigation strategies can be developed to capture microfibers before they are released to the environment (e.g., rain gardens for storm water).

Our next focus was on the environmental sinks of microfibers, including marine, freshwater, and terrestrial ecosystems. Most microplastic studies have been conducted in marine systems (Blettler et al., 2018), and we confirm that this was similar for microfiber studies. We found that 60% of studies ($n = 277$) investigated the occurrence of microfibers in marine environmental compartments, including waters, sediments, and biota. Although no studies were published on microfibers in freshwater environments prior to 2015, this number has increased to 29 and 42 studies published in 2019 and 2020, respectively. A total of 105 studies (23% of all studies analyzed) have been published on freshwater sediments, water, and biota. Concentrations of microfibers in freshwater and marine surface and subsurface water samples, although on the same order of magnitude, varied highly and are likely dependent on proximity to urban sources and collection method (Grbić et al., 2020; Hung et al., 2021; Tamminga et al., 2019; Figure 4). The concentration of anthropogenic particles (including microfibers) was similar in both marine and freshwater sediments (Figure 4). Interestingly, on average mean particle concentrations in freshwater biota were slightly higher than those in marine biota (15.6 and 9.8 particles per individual, respectively). Microfibers constituted an average of 65 and 73% of particle burdens in

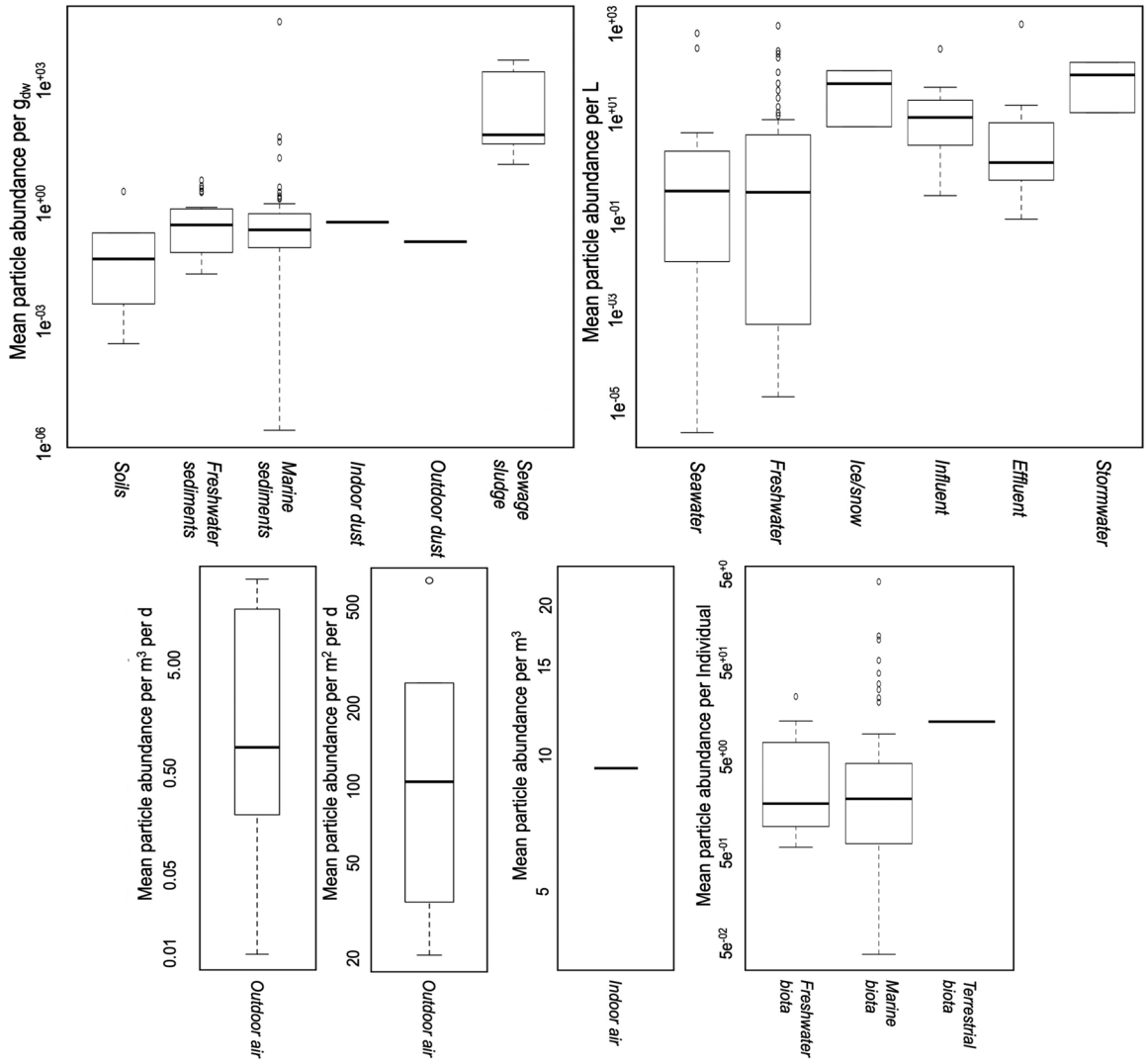


FIGURE 4: Boxplots showing mean particle abundance for various environmental compartments, grouped by common units. Mean particle abundance per gram dry weight values reported in soils ($n = 6$), freshwater ($n = 27$) and marine ($n = 60$) sediments, indoor ($n = 1$) and outdoor ($n = 1$) dust, and sewage sludge ($n = 3$) (top left). Mean particle abundance per liter reported for salt water ($n = 61$), freshwater ($n = 25$), ice and snow ($n = 2$), wastewater-treatment plant influent ($n = 12$) and effluent ($n = 16$), and storm water ($n = 2$) (top right). Outdoor air samples were reported in mean particle abundance per cubic meter per day ($n = 4$, bottom left) or mean particle abundance per square meter per day ($n = 5$, bottom middle left). Indoor air concentrations are shown in mean particle abundance per cubic meter ($n = 1$, bottom middle right). Mean particle abundance per individual values reported in marine ($n = 85$), freshwater ($n = 11$), and terrestrial ($n = 2$) biota (bottom right).

marine and freshwater biota, respectively. Investigated biota included marine mammals and marine and freshwater birds, fishes, and invertebrates.

Most microfiber studies from freshwater focused on lakes, rivers, and streams. We did not find any studies on microfiber contamination in groundwater. This is surprising given that microplastic particles, including microfibers, can move within soil strata (Engdahl, 2018; Goeppert & Goldscheider, 2021), and over one-third of the world's population relies on groundwater resources (Goeppert & Goldscheider, 2021; Re, 2019). This remains an important and needed area of study in the freshwater environment. Although it is known that contaminants can enter groundwater, it is unknown whether microfibers can also be transported through soil to this resource. In addition, we only identified two studies on freshwater ice and snow, highlighting another understudied freshwater compartment. In these studies, mean particle concentrations ranged from 7.8 to 117.1 particles/L of snow, of which fibers constituted 99 to 100% of particles identified (Figure 4). Snow presents a transport pathway for scavenging atmospheric microfibers and delivery to terrestrial and aquatic ecosystems in northern latitudes, from urban to remote regions such as the Arctic and Mount Everest (Bergmann et al., 2019; Huntington et al., 2020; Napper et al., 2020). Future research focused on these compartments will provide useful insights into microfiber transport.

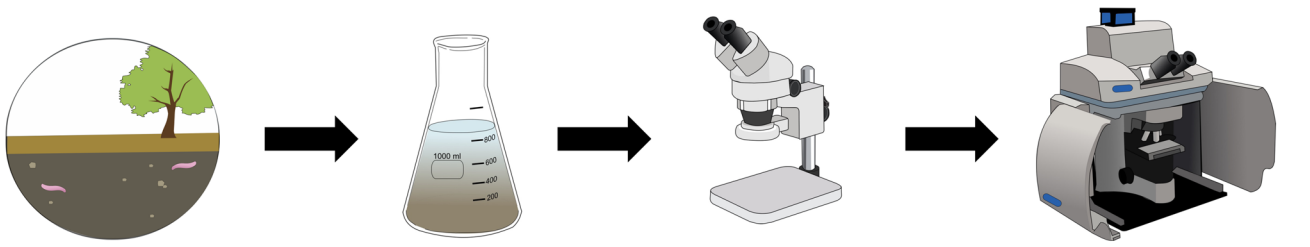
Few studies have investigated microfibers in the terrestrial environment, which includes soils and biota. Only 13 studies (3% of all studies) analyzed investigated the terrestrial environment, although the number of studies on microfibers in

the terrestrial environment has increased in recent years. Publications began with two studies in 2018, which increased to 10 studies published in 2020. Compared to studies on marine and freshwater biota, studies on microfiber ingestion by terrestrial biota reported the highest mean particle concentrations (~18 particles per individual; Figure 4), with microfibers constituting 86 to 100% of particles found (Carlin et al., 2020; Roblin & Aherne, 2020). Terrestrial environments are suspected to be a source of microfibers to aquatic environments via runoff (Crossman et al., 2020) and atmospheric transport (K. Liu et al., 2019). Our findings support the need for further investigation into the occurrence of anthropogenic microfibers in terrestrial ecosystems.

Methods for extraction and characterization of microfibers

The study of microfibers presents a number of challenges, some of which are common to all types of microfibers and others that are specific to nonsynthetic fibers. An overview of these challenges can be found in Figure 5. First, the size and shape of microfibers pose particular challenges for efficient recovery from certain environmental matrices (e.g., surface and subsurface waters, wastewater influent and effluent). Because microfibers in their smallest dimension have a diameter of approximately 10 to 20 μm, they may not be effectively captured by common microplastic sampling methods such as manta trawls or neuston and bongo nets (Lindeque et al., 2020). For example, many surveys of microplastics in surface water have employed coarse-mesh nets (e.g., manta, neuston), which are effective at monitoring larger particles (300–500 μm) but not microfibers. A comparison of methods found higher proportions of microfibers compared to other particle morphologies in bulk water versus manta sampling methods (Hung et al., 2021; Tamminga et al., 2019). Thus, in many studies that have used coarse mesh or sieves, microfibers are likely underestimated. We identified that 28% ($n = 35$) of studies investigating microfibers in marine and fresh surface waters used course-mesh nets (>300 μm mesh size). The remaining studies used finer mesh nets (<300 μm mesh size; $n = 19$) or bulk water approaches ($n = 61$).

A total of 288 studies analyzed used digestive methods to aid in the enumeration of anthropogenic particles (including microfibers) from organic-rich environmental matrices, such as sediments, biosolids, biotic tissues, and, occasionally, fresh and marine waters. Because digestive methods vary, recovery experiments use standardized materials that vary by size, shape, and polymer composition; aim to test the efficiency of particle extraction for a given enumeration method; and evaluate the effects of chemical digestants on different types of materials. Recovery experiments with microfibers are still rare, and to date, most testing has focused on synthetic fibers (Cai et al., 2019; Hurley et al., 2018; Thiele et al., 2019; Treilles et al., 2020). However, results from recovery experiments using natural and semisynthetic microfibers have suggested that commonly used digestive methods may be destructive to natural materials (Prata, Castro, da Costa, Duarte, Cerqueira, et al., 2020; Treilles et al., 2020). This may ultimately result in the underestimation of these types of anthropogenic microfibers.



Sampling strategy	Enumeration method	Intermediate screening	Material analysis
Challenges	*Challenges*	*Challenges*	*Challenges*
Low volume, grab samples can be too small to provide an accurate concentration	Oxidative digestives (e.g., H_2O_2) result in loss of natural and semi-synthetic fibers	Intermediate screening methods for microfibers have not been thoroughly tested	Hit quality index (HQI) can be affected by numerous variables
Mesh nets (e.g., Manta, Neuston) are quantitatively less effective at capturing fibers	There is limited recovery testing using natural and semi-synthetic fibers for digestion methods	Guidelines for qualitative screening methods (e.g., Hot Needle Test) are not available.	High HQI values can exclude natural materials
	Certain digestants can degrade synthetic fibers	Lipophilic dyes may not distinguish between non-synthetic and synthetic fibers	Incorrect library matches between similar materials (e.g., cotton, rayon, cellulose acetate) can occur
		Surface coatings applied to anthropogenic natural fibers may affect results of qualitative screening methods	
Best practices	*Best practices*	*Best practices*	*Best practices*
High volume, bulk water samples are best for fiber collection	Conduct recovery testing that includes non-synthetic fibers	Avoid intermediate screening for fibers	Avoid use of a high HQI in the case of natural materials
Use fine mesh (<300 um) when sampling with nets	Low temperature and shorter incubation times reduce fiber degradation	Perform and report method testing using synthetic and non-synthetic fibers	General classifications can be used (e.g., 'anthropogenic cellulose fibers')
Note sampling apparel (material color and composition)	Use minimally destructive digestion methods only when necessary		Analysis of surface morphology can aid in more confident fiber identification
Field blanks should be collected to monitor contamination	Avoid oxidative agents when digestion is necessary		

FIGURE 5: Overview of challenges and best practices for enumerating and characterizing microfibers in the environment.

Oxidative agents such as hydrogen peroxide are common digestants used in many studies, although these methods may result in significant losses of microplastics and microfibers. We found over two-thirds of studies ($n = 120$) that included a digestion step with H_2O_2, making it the most common digestant used in the microfiber literature. Hydrogen peroxide digestions have been applied to aquatic sediments (Jeyasanta et al., 2020; Yao et al., 2019; Zhang et al., 2019), biota (Ambrosini et al., 2019; Avio et al., 2020; Markic et al., 2018), water (Jiang et al., 2020; Wilkens et al., 2020; Zhao et al., 2019), wastewater (Akarsu et al., 2020; Gündoğdu et al., 2018), and sewage sludge/biosolids (Gies et al., 2018; Li et al., 2018). While the concentration, incubation temperature, and digestion time vary throughout these studies, the most common concentration (v/v) of H_2O_2 was 30% ($n = 97$), followed by <15% ($n = 10$) and >34.5% ($n = 9$). High concentrations of H_2O_2 are destructive to synthetic polymers (Nuelle et al., 2014) and natural materials (Prata, Castro, da Costa, Duarte, Cerquiera, et al., 2020; Treilles et al., 2020). Treilles et al. (2020) tested degradation of natural (cotton and flax) and semisynthetic fibers (viscose) incubated in 30% (v/v) H_2O_2 for 48 hours at 40 °C and found increased brittleness and subsequent fragmentation for viscose and cotton fibers. Chemical digestion also reduced FTIR spectral matching for viscose fibers. Further, Prata, Castro, da Costa, Duarte, Cerquiera, et al. (2020) found that lower concentrations of H_2O_2 (15% v/v for 8 days, at room temperature) caused degradation of natural (cotton and linen) and semisynthetic fibers (rayon and viscose). Other studies have found that incubation in H_2O_2 at high temperatures causes partial or complete degradation of natural, semisynthetic, and synthetic fibers (Cai et al., 2019; Helcoski et al., 2020; Treilles et al., 2020; Wiggin & Holland, 2019; Zhao et al., 2018).

The next most common digestant method was the use of alkalis such as potassium hydroxide (KOH) and sodium hydroxide (NaOH). We found that approximately one-third of studies ($n = 59$) employed KOH as a digestive step. In most cases, KOH was used to digest tissues of marine and freshwater biota. Although the KOH concentration, incubation length, and temperatures varied, nearly all studies that employ a KOH digestion method use 10% (v/v) KOH. The 10% KOH method was first applied in a study on marine fish by Foekema et al. (2013), who digested gastrointestinal tissues in 10% KOH at room temperature for 3 to 4 weeks. Since then, recovery experiments of 10% KOH methods have shown that the extent of degradation varies by polymer type of particles and may result in spectral changes, weight reduction, morphological changes, and partial or complete disintegration (Cai et al., 2019; Dehaut et al., 2016; Treilles et al., 2020). Generally, natural polymers (e.g., cellulose) do not withstand KOH digestion as well as synthetic polymers (Treilles et al., 2020). The impact of temperature in KOH digestions is significant in enhancing the degradation of natural and semisynthetic fibers. Most of the studies reported incubating samples at room temperature ($n = 6$), 40 °C ($n = 8$), 50 °C ($n = 4$), or 60 °C ($n = 18$). Some studies did not report incubation temperature ($n = 10$). Typically, KOH methods with higher temperatures require less time to digest organic matter (Dehaut et al., 2016). While higher temperatures can shorten KOH incubation times, higher temperatures can also result in reduced recoveries of microfibers (Bråte et al., 2018; Thiele et al., 2019). Studies have shown the complete loss of certain natural and semisynthetic fibers at 60 °C and no loss at 40 °C (Bråte et al., 2018; Thiele et al., 2019). Other studies show the complete degradation of wool at 40 °C (Treilles et al., 2020). Sodium hydroxide is another alkali digestant that has been used in seawater and biota studies ($n = 11$). While the effects of NaOH digestion on natural and semisynthetic fibers are mostly unknown, Dehaut et al. (2016) found that digestion in NaOH at high temperatures (65 °C for 24 hours) degraded cellulose acetate fragments.

Another common oxidative agent is the Fenton reagent employed in the catalytic wet peroxide oxidation method. Fenton reagent is a solution of H_2O_2 (30%, v/v) and ferrous iron (iron[II] sulfate; 0.05 M) used to catalyze organic matter (Hurley et al., 2018; Tagg et al., 2017). This method was originally applied to wastewater to remove organic matter and contaminants (Kuo, 1992) and has been applied in microfiber studies since 2013 (Faure et al., 2015). The few studies that have used this method have shown that it can degrade cellulose (Peller et al., 2019) or cause discoloration and breakage in cotton and rayon (Wiggin & Holland, 2019). Incubation in Fenton reagent can also cause iron deposits on viscose and cotton (Treilles et al., 2020). Further, we found that oxidative agents are sometimes used in combination with other digestants; studies combined oxidative agents with KOH ($n = 4$), acids ($n = 5$), and enzymes ($n = 2$). These combinations may have different effects. For example, oxidative agents used with KOH can degrade natural and semisynthetic fibers (Cai et al., 2019), whereas the combination of H_2O_2 and cellulase did not completely degrade rayon and cotton fibers but caused significant color loss and breakage (Wiggin & Holland, 2019).

Other digestants have included enzymes (cellulase, protease, amylase, trypsin, and lipase) and acids (NaClO, $NaClO_4$, HNO_3, HF, and HCl). Enzyme digestion was common in studies with biota ($n = 27$), although no studies have reported the effects of enzyme digestion on natural and/or semisynthetic fibers. Thus, we are unable to evaluate whether these methods are destructive to natural and/or semisynthetic fibers. Acid digestions have also been employed in the microfiber literature ($n = 21$), most of which were in combination with the other methods discussed. Method testing for acid and enzyme digestates using natural and semisynthetic fibers has been limited. Conley et al. (2019) investigated digestion in H_2O_2 and HCl, concluding that the use of H_2O_2 in combination with an acid causes loss of natural (silk and cotton) and semisynthetic (rayon) fibers. For example, Treilles et al. (2020) found that incubated flax, cotton, and viscose fibers in 25% NaClO for 15 hours at room temperature degraded flax fibers, promoted fragmentation of viscose fibers, and did not appear to affect cotton fibers.

Given the widespread use of digestion methods that may result in the destruction of natural and semisynthetic microfibers and the general pattern that these methods can often be more destructive for nonsynthetic fibers, it is likely these publications underestimate the occurrence of nonsynthetic fibers in

environmental samples. Underestimation is of highest concern when digestion methods are particularly destructive. Further, we found that 18% of studies employed a digestant where the effects on natural and semisynthetic fibers have not been investigated; thus, the impacts of those methods are still unknown. Future experiments should include recovery testing with a range of different materials (including natural and semisynthetic fibers) to determine whether these digestant methods are destructive.

Characterization poses another challenge for studying the occurrence of natural and semisynthetic fibers in the environment. Intermediate screening steps have been employed to distinguish between synthetic and natural materials in an effort to reduce the number of particles in subsequent analysis steps. We found that a few studies (6%; $n = 27$) employed an intermediate screening step prior to spectroscopic analysis, which included the use of staining and fluorescence microscopy (Nile red [NR] and Bengal rose dyes) or the "hot needle test." Nile Red is a lipophilic dye that adsorbs to the surface of particles and renders them fluorescent under particular wavelengths of light. This has been favored as a relatively cheap method for detecting synthetic fibers, and its reported use is found in seven studies in the microfiber literature. However, the NR method has also come under scrutiny in recent years given its unreliability. One challenge in using the NR method is that NR dyes all organic material present in a sample. Few studies have actually tested the accuracy of using the NR method to distinguish natural from synthetic fibers, with mixed results. Catarino et al. (2018) and Devalla et al. (2019) tested the accuracy of the NR method using cotton and synthetic fibers, and both concluded that natural fibers can be distinguished from synthetic fibers using NR, in contrast to results reported by Prata, Castro, da Costa, Duarte, Rocha-Santos, et al. (2020) and Stanton, Johnson, Nathanail, Gomes, et al. (2019). However, Stanton, Johnson, Nathanail, Gomes, et al. (2019) also found that NR significantly overestimated the number of microplastics within a sample by falsely identifying natural and semisynthetic textile fibers as synthetic. Further, the accuracy of NR when applied to microfibers depends on the solvent employed as well as the light filter used (Tamminga, 2017). Given the uncertainty and potential for misidentification of anthropogenic microfibers in environmental samples, further work is needed to assess the NR method and other staining methods.

Another popular intermediate screening method includes the "hot needle test" as a coarse test to distinguish between natural and synthetic materials. The hot needle test (also called the "hot point test") uses a heated needle to distinguish between an organic polymer that melts and an inorganic polymer that does not melt. The method, which relies on a qualitative assessment by the researcher, suggests that if the particle is plastic, the needle will melt the surface of the particle, leave a "mark" (Devriese et al., 2015; Iliff et al., 2020; Roch & Brinker, 2017; Vandermeersch et al., 2015), or cause the particle to "move when in contact with the needle" (Berglund et al., 2019). The accuracy of this test has not been thoroughly tested, and the instructions of the technique are often vague and subjective. Because natural materials degrade rather than melt at high temperature, the stickiness on the surface of a particle that results from melting would hypothetically only occur when the hot needle is applied to a synthetic material (Lusher et al., 2017). However, fibers may be the most challenging particle type to analyze using this method, given their thin diameter. Further, the effects of synthetic coatings applied to natural fibers may influence how a fiber responds to heat (Sait et al., 2021). The earliest reported use of this test comes from De Witte et al. (2014), although the original source of the method is uncertain. We identified 14 studies that used the hot needle test as an intermediate screening step prior to spectroscopic analysis.

Finally, we examined spectroscopic methods used to analyze microfiber composition. Approximately 98% of studies ($n = 455$) used FTIR or Raman spectroscopy. While pyr-GC-MS is a reliable method for material identification of microfibers, only four studies employed it, none of which included natural or semisynthetic materials in their analyses. Both FTIR and Raman spectroscopic analyses use a hit quality index (HQI) to evaluate the confidence of the library match for a particular material undergoing analysis. Many factors can impact the HQI, including instrument parameters (e.g., laser excitation, laser filter, grating), quality of the environmental sample being analyzed (e.g., environmental aging, presence of biotic film, fluorescence), the signal intensity inherent to different materials, and the presence of dyes or colorants (Lenz et al., 2015; Munno et al., 2020; Park et al., 2017; Zhu et al., 2019). Natural materials, such as cellulose, often exhibit lower signal intensities compared to synthetic polymers; and, as such, they are more prone to dye interference. Because of this characteristic of natural materials, a high HQI has been employed as a technique to exclude natural and semisynthetic fibers from analyses (Kanhai et al., 2018). We found that one-third of studies that conducted FTIR or Raman spectroscopic analysis used a high HQI value (>60%), which could exclude materials with low signal intensities (e.g., cellulose).

It is also possible to obtain incorrect library matches between similar materials, such as cellulosic materials like rayon, cellulose acetate, and cotton, which have similar spectra. This may be especially problematic in cases with limited library databases and/or low spectral signals, which can lead to misestimations. For example, mistaking cellulose acetate fibers for cotton fibers may underestimate the contribution of sources of cellulose acetate fibers, such as cigarette filters. To address the possibility of incorrect library matches, some studies refer generally to cellulose fibers as "cellulose fibers," rather than specific types of cellulose fibers (e.g., rayon, cotton, lyocell; Baechler et al., 2020). More general classifications have included "anthropogenic cellulose" and "cellulose of unknown origin," depending on evidence of anthropogenic modification (Athey et al., 2020; Adams et al., 2021).

Analysis of the surface morphology of fibers is an important step for more confident identification of natural and semisynthetic fibers (Athey et al., 2020; Zhu et al., 2019; Figure 1). We found that only 11 studies (or approximately 2% of the total studies analyzed) employed multiple lines of evidence to confirm spectroscopic identification.

Methods for reporting microfibers in environmental samples

A major challenge in understanding the abundance of nonsynthetic fibers in the environment relates to the lack of standardized methods for capturing, enumerating, and characterizing microfibers in environmental samples. There is considerable variability in methods and reporting. For the 42% of publications that did not report natural and/or semisynthetic fibers, it is unclear whether these particles were or were not present in environmental samples or if enumeration methods, such as chemical digestion, resulted in their destruction (Cai et al., 2019; Conley et al., 2019; Helcoski et al., 2020). A few studies excluded natural fibers based on their shape (e.g., fibers that were "segmented," "twisted," or contained a "cellular structure"; Jones et al., 2020; Mohamed Nor & Obbard, 2014). Other studies used methods that are known to chemically digest natural microfibers (Cai et al., 2019; Conley et al., 2019; Helcoski et al., 2020) or exclude natural materials during spectroscopic analysis (Kanhai et al., 2018). Lastly, natural and semisynthetic fibers may simply be left out of final data reporting (Deng et al., 2020; Jones et al., 2020; Martin et al., 2017).

Best practices for the future of microfiber research

As discussed in the previous sections, anthropogenic microfibers are underestimated in the literature. To better quantify and report the full suite of anthropogenic microfibers, we have recommended best practices (Figure 5). Following these best practices will enable researchers to fully assess microfibers, including natural and semisynthetic fibers.

During sample collection, steps should be taken to fully capture microfibers in samples and exclude microfibers from contamination. As described in *Methods for extraction and characterization of microfibers*, the use of course mesh or sieves can result in the underestimation of microfibers in surface and subsurface water samples (Hung et al., 2021; Tamminga et al., 2019). If methods incorporate bulk water samples and use fine-mesh sieves where applicable, researchers can better capture and enumerate microfibers. Microfibers are a widespread airborne contaminant, and therefore careful quality assurance and quality control practices are required to accurately estimate microfiber abundance in environmental samples (Song et al., 2021). Researchers rely on field and laboratory blanks to correct for this contamination (Cowger et al., 2020), which is especially important for microfibers because fibers often are the dominant particle type in laboratory and field blanks. It is possible for researchers to inadvertently introduce contaminant microfibers from their clothing into samples because microfibers are released from clothing to air. Many laboratories have employed 100% cotton, white laboratory coats in an effort to easily distinguish contaminant fibers from synthetic sample fibers. However, as the field expands to include nonsynthetic anthropogenic microfibers (including clear and white cotton fibers), this approach may not be sufficient. We suggest wearing laboratory coats in colors that are not commonly reported in environmental samples, such as bright orange or pink. Researchers should ensure that they are wearing the same materials when processing environmental samples and blanks. Subsequently, it is critically important to compare and report the color and composition of fibers found in all blank samples to those from study personnel clothing.

In the laboratory, extraction and enumeration methods should be carefully considered. Many of the common extraction methods, such as wet peroxide oxidation and digestions at high temperatures, may be destructive to natural fibers. Low-temperature digestions are less likely to destroy natural and semisynthetic microfibers and should be used in future studies. However, because there are many digestants that haven't been evaluated in recovery tests, recovery testing should be carried out to assess how a particular method may bias the recovery of synthetic, natural, and semisynthetic fibers.

To determine microfiber type, chemical identification and morphology are needed at the particle characterization step. It has become the norm in microplastics studies to use a robust method for determining material type, such as Raman or FTIR spectroscopy (Rochman et al., 2019). For microfibers, proper characterization is also important because it can distinguish between a wide array of materials. However, because microfibers often contain dyes that can cause interference and reduced HQI values, morphology may provide an additional line of evidence to confirm a spectral match.

Lastly, reporting should include quantitative information on all microfiber types (e.g., abundance, proportion of different fiber types). Because determining particle types with chemical identification falls at the final stage of laboratory analyses, often a final data set will consist of information on all particle types. Based on a study question, researchers may decide to intentionally exclude data on natural and semisynthetic microfibers. We argue that data on natural and semisynthetic microfibers provide valuable information that should be included in future studies. At the very least, studies should include raw data with chemical spectra so that data can be extracted for meta-analyses.

CONCLUSIONS

Most studies have focused on synthetic microfibers, yet natural and semisynthetic microfibers often dominate anthropogenic particles when they have been identified. In the present study, we identified both understudied compartments and geographic regions that are understudied in the microfiber literature. These included source compartments (e.g., indoor air and dust), pathways to the environment (e.g., storm water, sewage sludge, and biosolids), freshwater compartments (e.g., groundwater, ice and snow), as well as compartments in the terrestrial environment.

Some major gaps in anthropogenic microfiber research remain. Many of these gaps exist because microfiber research originated from microplastics studies, and over time, many have expanded to include the full suite of anthropogenic

microfibers. However, many of the methods that were developed for microplastics are inadequate to assess the full suite of anthropogenic microfibers. Although updated methods are needed in many cases, we recommend simple steps that can be taken to successfully enumerate and identify semisynthetic and natural fibers from environmental samples. By including natural and semisynthetic microfibers in future studies, we will be able to better understand the sources, pathways, and effects of microfibers as a whole.

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