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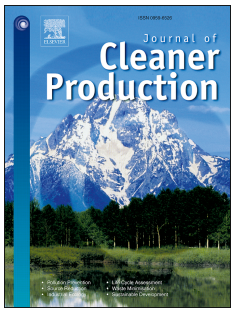

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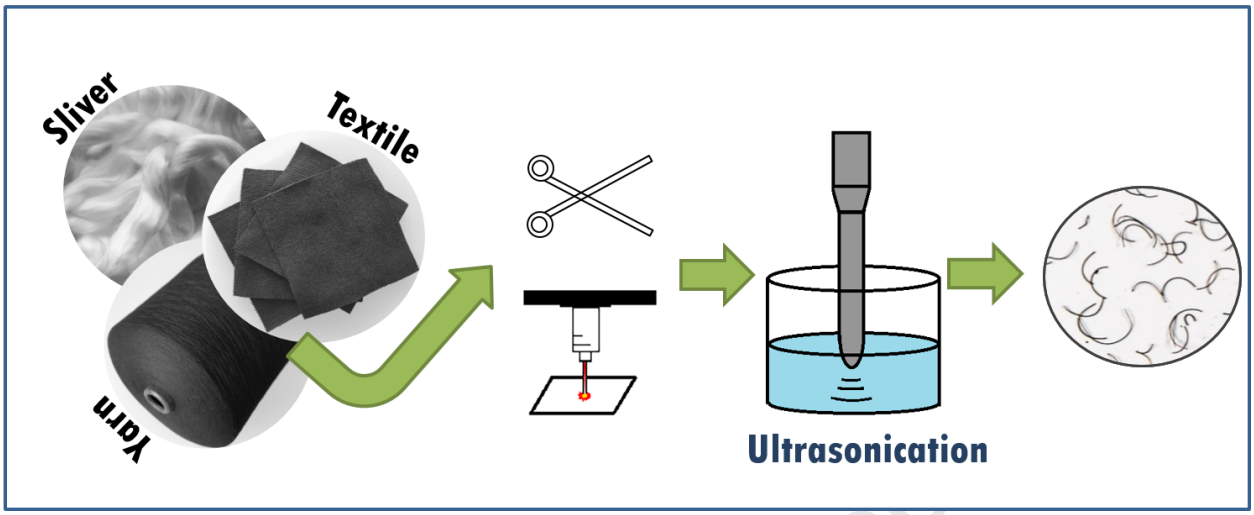

The origin of microplastic fiber in polyester textiles: the textile production process matters

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Highlights

- A sonication method was developed to extract microplastic fibers (MPFs) from textiles
- The method was applied to 18 representative products along the textile production line
- The number of MPFs extracted was influenced by the type of textiles and the cutting method
- High(er) quantities of MPFs originate from processed surfaces and cut edges

Abstract

Microplastic fibers (MPF) are often detected in waste water treatment plants and environmental samples, which implies a pathway of MPF release from domestic washing of textiles into the environment. Although there are many textile washing/release studies, it is still unclear to what extent the liberated MPFs originate from processes during washing (e.g. abrasion) or rather from processes earlier in the textile supply chain. Understanding the origin of MPFs is important since different MPF formation mechanisms would lead to different mitigation strategies. Therefore, the aim of our study was to investigate the presence of MPFs in various intermediate and finished polyester textiles products. In this study, we developed a sonication extraction method to quantify and characterize extractable MPFs already present in the textiles (i.e. manufacturing related MPFs). To identify the manufacturing process responsible for the MPF formation, this study included 18 representative products along the textile production line. The extraction dynamics of MPFs for all materials were investigated by ultrasonication. The number of extracted MPFs ranged from 15 MPF/g for a filament to 45'400 MPF/g for a scissor-cut microfiber textile. We found that a rotor yarn exhibited an elevated number of extracted MPFs (4'310 MPF/g) compared to other types of yarns (160-230 MPFs/g), suggesting that the rotor spinning may be a critical step responsible for MPF formation. On average, five times more MPFs could be extracted from textiles with processed surfaces (such as Fleece, Plain brushed and Microfiber) compared to those with unprocessed surfaces. This suggests that abrasive friction during production may be another critical factor for MPF formation. Furthermore, scissor-cut textiles demonstrated three to 31 times higher number of extracted MPFs than laser-cut textiles, enabling us to quantitatively discriminate between the contribution of MPFs from the textile surface opposed to those originating from the textile edges. The majority of the extracted MPFs were found to be between 100 to 800 µm in length. The results of this study may help to reduce the MPF release from textiles by modifications throughout the production and finishing process.

Keywords: microplastic fiber release, textile production, ultrasonic extraction, polyester, pollution

1. Introduction

The increasing global production of synthetic fibers raises the concern that microplastics released from synthetic textiles are likely to continue contaminating our environment in the future (Henry et al., 2019). Fibers are often detected as the dominant constituent of microplastics found in waste water treatment plants (WWTP) (Dris et al., 2015; Kay et al., 2018; Murphy et al., 2016) as well as in many environmental samples (Desforges et al., 2014; Frias et al., 2016). A recent modeling study has reported that fibers from textiles significantly contributes to microplastic releases into freshwater (Kawecki and Nowack, 2019). These findings imply that a pathway of microplastic fibers (MPF) from domestic washing of textiles into the environment likely exists (Browne et al., 2011). Although WWTPs exhibit a high removal efficiency (above 98%) for microplastics (Schmiedgruber et al., 2019; Sun et al., 2019), elevated microplastic concentrations were still observed downstream of WWTPs compared to control sites (Estahbanati and Fahrenfeld, 2016; Kay et al., 2018; McCormick et al., 2014). Moreover, approximately 50% of the sewage sludge is estimated to be used on agricultural soils in Europe and North America (Nizzetto et al., 2016), constituting a direct release pathway of the fibers removed during wastewater treatment to the environment. One also needs to keep in mind the disparity of wastewater connectivity ratio to WWTPs, which ranges from about 70% in high-income countries to only 8% in low income countries (WWAP, 2017). This results in the (direct) release of unprocessed wastewater. Therefore, MPFs can be released into the environment regardless of the fact that the MPFs are in the effluent or captured by the sludge.

Several studies have been performed to quantify MPF release from textiles during laundering. Browne et al. (2011) were the first to quantify the number of MPF from polyester garments. Later, additional experiments were conducted to investigate factors which may influence MPF release during washing, such as the addition of detergents, temperature, washing duration and types of textiles (Almroth et al., 2018; Belzagui et al., 2019; Henry et al., 2019; Hernandez et al., 2017; Jonsson et al., 2018; Napper and Thompson, 2016; Pirc et al., 2016; Sillanpaa and Sainio, 2017). A decrease of the MPF release with repeated wash cycles has been observed in several studies (Belzagui et al., 2019; Cai et al., 2020; Napper and Thompson, 2016; Sillanpaa and Sainio, 2017). These findings spurred us to question if there were any MPFs present in textiles before washing.

The manufacturing of synthetic textiles usually begins with polymer chips which are melted and spun into endless filaments. These filaments are cut into short staple fibers and then carded into slivers (Figure 1). In the next step, different yarn spinning methods are applied to spin slivers into yarn. The yarn made from short staple fibers are called “spun yarns”. Correspondingly, there are yarns made from endless filaments which are called “filament yarns”. These yarns can then be further woven or knit into textiles. The woven textile is made by interlacing two threads perpendicularly and the knit textile has only one thread

following a course to produce symmetric loops on both sides of the mean path. In the finishing step, various techniques can be applied to enhance the performance, look and feel of the final product. Some surface treatments can be applied at this stage to produce textiles with special textures, such as fleece. Finally, the finished textiles are cut and tailored into garments and delivered to customers.

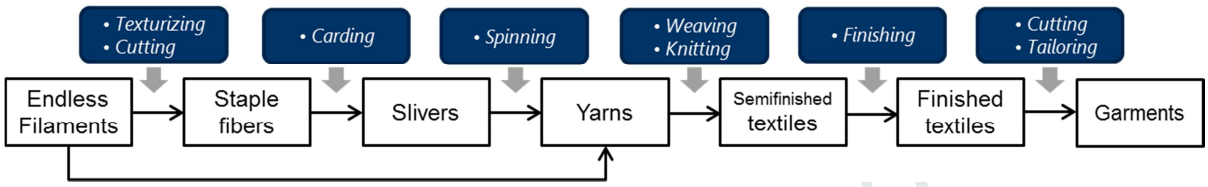

Figure 1. A simplified flow chart of the important stages for the manufacturing of polyester textiles. Blue blocks represent processes and white blocks represent the corresponding products.

Understanding the source of MPFs is relevant since different origins would advocate different mitigation strategies. If the majority of MPFs are formed during the washing process, then changes in washing methods, such as improvements of washing detergents, or entrapments of released MPFs during washing may be needed in the future. However, if MPFs are already generated in the textile throughout the manufacturing process, then efforts should be made to localize and improve the culprit process(es) in the production line or submit textiles and garments to additional washing/cleaning steps before they are shipped to the consumer.

Many studies have investigated MPF release from finished textile products during washing, but none of them have addressed the presence of MPF in intermediate fiber products. Since mechanical stress affects yarns and textiles at different textile processing steps, a systematic study with the products along the production line can help in identifying the crucial steps in MPF formation. Therefore, the aim of our study was to investigate the presence of MPFs in various intermediate fiber products and for a number of different finished polyester textiles. We first developed a method to quantify the extractable MPFs which were already present in textiles and applied it to 18 samples at different stages along the production line. The extracted MPFs were characterized (length and diameter) and counted to identify which stages along the production line were most relevant for MPF formation. The results from this work enable comparisons of the presence of MPFs from different textile products leading to the understanding of the origin of MPFs, and may provide the basis for engineering options to produce textiles with reduced MPF release.

2. Materials and methods

2.1. Sliver, yarns and textiles

A representative set of 18 products along the polyester textile production line was obtained directly from manufactures and suppliers located in China and Switzerland (Table 1). One sliver, one filament yarn and four spun yarns were selected to investigate the influence of sliver production and different yarn spinning methods. To further determine the existence of MPFs in textiles, we selected 12 textiles with different textile structures, types of yarn and post processing finishing steps, which was also used in a previous study (Cai et al., 2020). In the manuscript, a suffix is given to distinguish the textiles made of spun yarns "S" or filament yarns "F", respectively. Additionally, the suffix "B" was given to the plain textile with a brushed surface. Most of the samples were dark-colored, except for one sliver and three spun yarns which were white. This allowed us to more easily and accurately quantify the MPF extracted from the sample using our detection methods (see below). The density of the textiles ranged from 75 g/m^2 to 294 g/m^2, which was determined by weighing three pieces of 36 cm^2 swatches. The chemical composition (polyester) of the textiles was further confirmed by FTIR analysis (Varian 640-IR).

All samples were characterized by scanning electron microscopy (SEM) (Hitachi S6200) to obtain structures of the textiles and yarns as well as fiber diameters (Figure 2, Table S1). To enhance SEM contrast, the samples were sputtered with a layer of Au/Pd (nominally 7 nm thick) in a high vacuum sputter coater (LEICA EM ACE600). Fiber diameters were characterized by randomly measuring 10 fibers from the SEM images.

Table 1: Textiles, yarns and sliver used in the study. The values for textiles were taken from a previous study (Cai et al., 2020).

Product	Surface	Structure	Type	Yarn	Color	Density [g/m^2]	Fiber diameter [µm]
Sliver	-	-	-	-	White	-	12.0±1.3
Yarn	-	-	Rotor*	Spun	White	-	12.5±0.7
	-	-	Air-jet*	Spun	White	-	11.9±0.7
	-	-	Ring*	Spun	White	-	12.3±0.6
	-	-	Ring*	Spun	Black	-	12.8±1.5
	-	-	-	Filament	Black	-	10.8±0.4
Textile	Unprocessed	Knit	Interlock	Spun	Black	209±1	12.2±0.8
			Jersey	Spun	Black	226±1	12.8±0.8
			Rib	Spun	Black	294±2	12.7±1.1
			Rib	Filament	Black	199±1	15.9±2.2
			Terry	Spun	Black	208±2	13.0±1.3

	Woven	Plain	Spun	Black	100±0	12.7±0.5/13.4±0.9**
		Plain	Filament	Black	149±1	7.5±0.6/7.9±0.5**
		Twill	Filament	Black	154±1	12.4±1.8/19.9±1.7**
		Satin	Filament	Black	75±0	13.0±0.7/16.4±1.7**
Processed	Knit	Fleece	Filament	Black	185±1	11.7±1.3
	Woven	Plain brushed	Filament	Black	131±0	9.0±1.2/10.1±1.5**
	Woven	-	Filament	Grey	191±3	19.9x8.9/7.7x2.2*** (microfiber)

* Spinning methods of spun yarns

**The diameters of the weft and the warp yarns of the woven textiles

***The width and length of the weft yarn (19.9±1.1 x 8.9±1.2 µm) and the warp yarn (7.7±0.9 x 2.2±0.5 µm) with a rectangular cross section for the microfiber sample

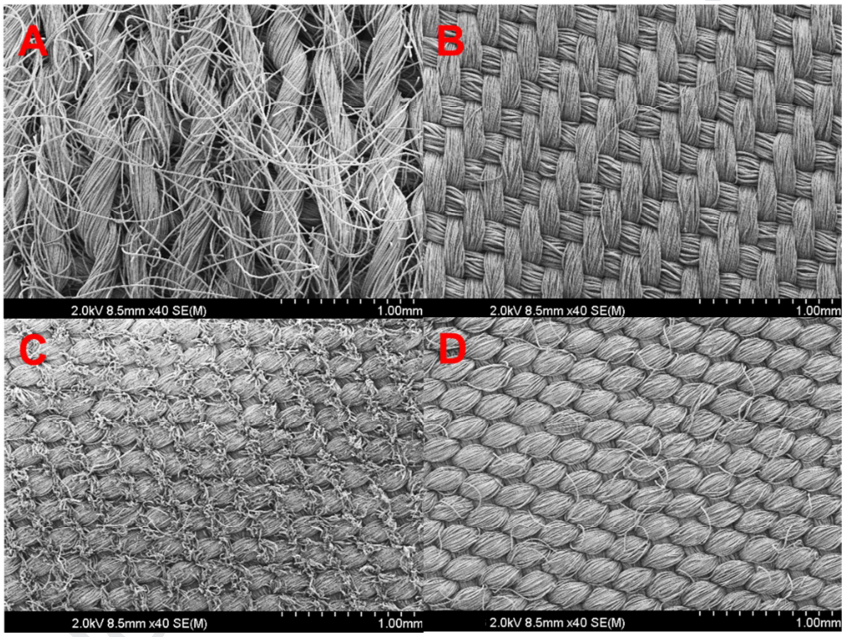

Figure 2. SEM images for three textile samples: A) Interlock S; B) Twill F; C) Plain B: front side (brushed); D) Plain B: back side (unbrushed)). For the textiles made of spun yarns (Figure 2A), there are many fibers protruding from the textile surface. In contrast, the filament textiles (Figure 2B) have a surface with few protruding fibers. Figure 2C and 2D display the front and the back side of Plain B, where one can observe how the abrasion (brushing") process affects the surface of the textile. The details for the other products can be found in Table S1.

2.2. Sample preparation

Textile scissors or a laser cutter (tt-1300, Times technology) were used to cut the textile into swatches with a dimension of 6 cm x 6 cm. The edges of the laser- and scissor-cut samples were characterized by SEM (Hitachi S6200) (Figure S1). Depending on the textile sample, the weight of the sample ranged between 0.16 g to 1.05 g. The sliver and yarns were cut by a laser cutter into pieces of approximately 1.50 g.

The white-colored fibers were dyed using a blue-colored pigment (BEMACRON E-RD, CHT, Switzerland) in order to provide increased contrast when imaging the fibers in subsequent analysis steps. In brief, the dyeing process compromised of two steps, where the first step was to dye the polyester and the second step was to fix the color and remove the extra dye on the surface. See the supplementary information (SI) for additional details on the dyeing procedure. The solution from the two dyeing steps was also analyzed to determine if fibers were released through this process as well. The dyed samples were then used in extraction experiments.

2.3. Extraction experiments and filtration processes

Extraction of MPFs was carried out in a 0.75 g/L linear alkylbenzene sulfonic acid (LAS) solution (Alfa Aesar) that was used in previous washing experiments to mimic domestic laundry detergent (Hernandez et al., 2017). LAS is the main surfactant ingredient in many commercial laundry detergents. A 1 mol/L solution of sodium hydroxide (Sigma-Aldrich) was used to adjust the pH of the LAS solution to approximately 9.0-9.5.

For the extractions, a sample was placed into a 600 ml beaker filled with 200 ml LAS solution. No prewashing step was performed for any of the samples. The solution, together with the sample, was sonicated for 10 min with an ultrasonic probe with an output power of 70 W and a frequency of 20 kHz (Sonopuls HD 2070, with probe VS 70T). The tip of the probe was submerged in the solution 1 cm under the air-water interface. Preliminary experiments were conducted suggesting that the majority of the extractable MPFs could be extracted from textiles within 90 minutes. Therefore, for each sample, the experiment was stopped at either 90 min (9 sequential extraction steps) or when there were less than 20 MPFs per cycle extracted, whichever was reached first. The temperature of the solution was between 23 to 27 °C during extraction. For all experiments, three independent replicates were performed for each product. Between each extraction step, the beaker was rinsed with DI H_2O three times to avoid any contamination between extractions.

After each extraction, the sample was taken out of the beaker with tweezers and allowed to drip for 15 s to remove excess liquid. The remaining solution was continuously stirred and a 10 mL pipet was used to transfer the solution to a vacuum filtration system. A vacuum pump with a filtration unit was used to filter the liquid through a cellulose nitrate membrane (GE Whatman, diameter 4.7 cm, pore size 0.45 µm). We attempted to avoid too many fibers on the filters, which would result in extensive overlapping of fibers and lead to difficulties in analyzing the number and the length of fibers in the extraction solution. Therefore, the volume of water filtered was between 10 ml to 200 mL, depending on the expected concentration of MPFs in the solution. The filters were put into separate petri dishes (VWR, diameter 90 mm, height 16 mm), covered, and left to dry overnight at room temperature. Moreover, to determine the

194 reliability of the sample collection and filtration method, triplicate aliquots from the same extraction
195 solution for two randomly selected textiles (Interlock S, Microfiber) were filtered through separate filters
196 and dried overnight as described above.

197 Blanks were measured three times for each experimental day, before the daily analysis, mid-way through
198 the analysis, and after the last extraction. This involved sonicating 200 ml of the LAS solution for 10 min
199 without the textile sample.

200 **2.4. Filter imaging and analysis**

201 A single-lens reflex camera (Nikon D850) with a macro lens (Nikon 105 mm/2.8) was used to image all
202 filters. A ruler was added to each filter as a scale and images (8256 x 5504 pixels) were edited in the
203 software Adobe Ligthroom CC (version: 2015.14) to enhance contrast. Most of the filters were analyzed
204 for fiber number and length with the software FiberApp (version: 1.51) (Usov and Mezzenga, 2015). By
205 manually selecting the starting point and the end point of each fiber, the software automatically calculated
206 the fiber length and recorded the fiber number. One exception was the sliver samples, which were
207 analyzed using ImageJ. This is because the sliver sample shed many long fibers which were difficult to be
208 tracked with FiberApp. Both methods had a lower length detection limit approximately 3-4 pixels,
209 corresponding to about 40 µm fiber length. Only dark-colored fibers were counted. The number of MPFs
210 on each filter was between 4 to 1'320, with an average of 186 MPFs per filter. In total, 615 filters were
211 analyzed and the length of approximately 120'000 MPFs were collected individually. Additionally, the
212 mass of extracted fibers was also calculated for knit textiles, but not for woven textiles. This is because
213 woven textiles have two threads, which made it difficult to determine the diameter of fibers and
214 subsequently which of the two fibers were shed from the textile. The mass calculation was done by using
215 the measured length and the known diameter of the fiber (see Table 1) and multiplying by the polyester
216 density of 1.38 g/cm^3 (Kallay et al., 1990).

217 In addition, for seven selected scissor-cut samples, the diameters of the extracted MPFs on the filters were
218 determined by SEM, which were then compared with the fiber diameter in the unused textile. A high
219 vacuum sputter coater (LEICA EM ACE600) was applied to sputter the filter with a layer of 7 nm Au/Pd.
220 The fibers were observed by SEM (Hitachi S6200) at a voltage of 2.0 kV and a magnification of 400. Ten
221 SEM images were randomly captured from each filter and one fiber was randomly chosen from each
222 image to obtain the fiber diameter.

223 **2.5. Influence of edge processing methods on MPF extraction**

224 Additional experiments were performed to investigate if the number of fibers extracted from the textiles
225 scaled linearly with the total length of the edge of the textile samples. Interlock S samples with a constant

area of 36 cm^2 were cut into smaller pieces with different perimeters of 24, 36, 48, 60 and 72 cm either by a scissor or by a laser cutter. All pieces from one 36 cm^2 textile were then sonicated together for a 10-minute extraction step. For each length of the perimeter, three replicates were performed. The filtration process and image analysis was done according to the previous experiments.

2.6. Statistics

A linear mixed model (package "lmerTest") in R (version 3.4.3) was used to determine the influence by factors on the number of extracted MPFs from textiles. The four factors included the surface treatment (unprocessed, processed), the textile structure (knit, woven), the yarn type (spun, knit) and the cutting method (scissors, laser). Each factor was taken as a fixed effect. The textile types (Interlock, Jersey and etc.) were considered as random effects. Since there were several extraction steps throughout the experiment, the number used in the model was the cumulative number of extracted MPFs. A similar model was established to determine the influence on the length, in which the median length of extracted MPF only during the 1st extraction step was considered. Additionally, a non-parametric Kruskal-Wallis (K-W) one way ANOVA test in IBM SPSS statistics (version 25) was used to compare the length distribution in the following groups: 1) the length distribution of MPFs extracted from the sliver and yarns; 2) the length distribution of MPFs extracted from the scissor-cut and laser-cut textile samples; 3) the length distribution of MPFs extracted from textile samples in sequential extraction steps. A p-value below 0.05 was considered to constitute a significant difference for all the statistical tests.

3. Results

3.1. Assessment of experimental procedures

We conducted several measurements to determine the reliability of the analytical workflow and the potential for contamination throughout the experiments. First, to quantify the contamination from dark-colored fibers, three blanks were investigated three times; before the daily analysis, mid-way through the analysis and after the last extraction on each experimental day. The average number of fibers was found to be 2 ± 2 MPFs per filter (n=60), as monitored over a period of four months. Light-colored fibers do not interfere with the measurements, since we only counted dark-colored fibers through the entire experiment. An example of extracted MPFs collected on the filters for four types of textiles is shown in Figure 3.

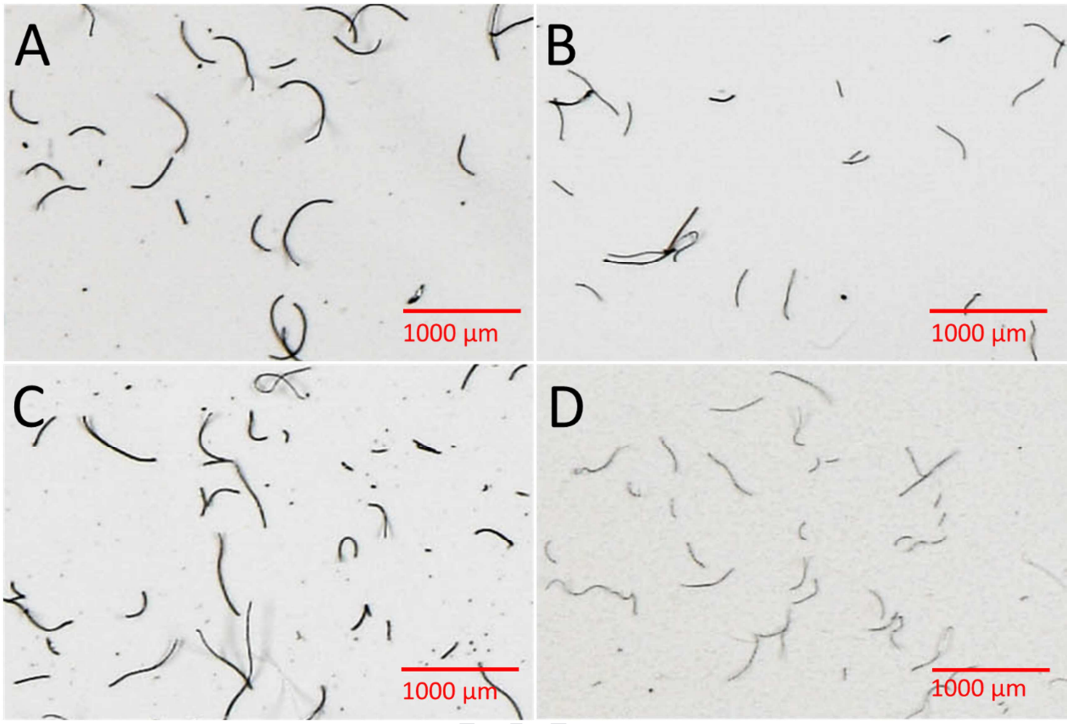

Figure 3. MPFs extracted from four types of textiles: A) Interlock S; B) Plain F; C) Fleece and D) Microfiber.

By analyzing three aliquots from the same washing solution for two randomly selected samples (Interlock S and Microfiber), we found a similar fiber number (relative standard deviation < 4%) and MPF length distribution (Figure S2). These results confirm that the sub-sampling method used was representative for quantification of MPFs in our system and that the chosen workflow yielded reproducible results. Moreover, all experiments were carried out in triplicate. The average relative standard deviation of MPF number for different samples was 29%, ranging from 1% to 83%. In previously published studies, the average relative standard variation of fiber number ranged between 20% (De Falco et al., 2018) to 36% (Hernandez et al., 2017).

3.2. Extraction dynamics of MPFs

To track the origin MPFs along the manufacturing process, we investigated one filament yarn, one sliver, and four spun yarns. All the dyed products (Sliver, Rotor, Air-jet, Ring-white) demonstrated an elevated initial MPF release during the dyeing steps, followed by a decrease in subsequent extractions. The decreasing trend of MPF release during extractions was also observed for the undyed black-colored ring

yarn. For the sliver and the rotor yarn, the number of extracted MPFs remained high after several extractions at approximately 50 and 220 MPF/g respectively, compared to the air-jet yarn, the white-colored and the black-colored ring yarns with less than 20 MPF/g (Figure 4). The number of extracted MPFs from the filament yarn remained at low level (smaller than 20 MPF/g) during the extraction steps.

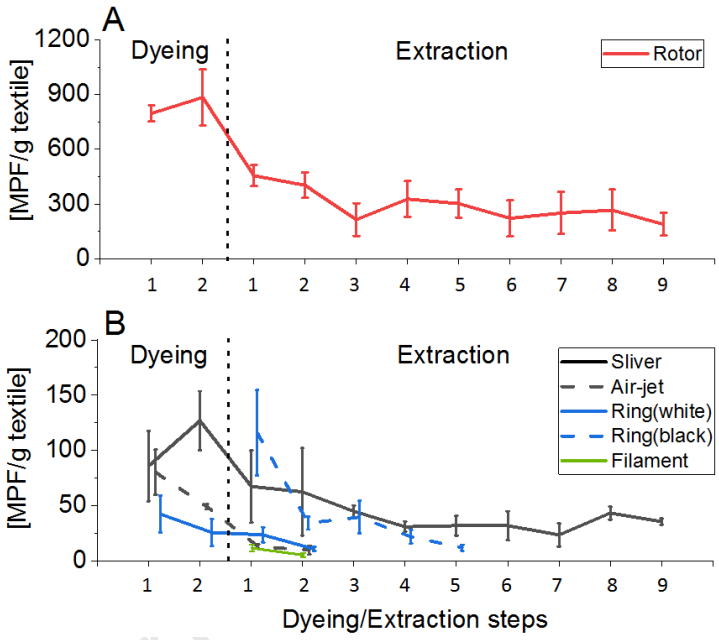

Figure 4. Number of extracted MPFs from slivers and yarns. All results are presented in number of MPFs per gram of textile. Standard deviations were calculated from triplicate experiments.

The number of MPFs extracted from 12 textiles after each extraction step was also determined (Figure 5 and Figure S3). In general, we observed a strong decrease in the number of extracted MPFs between the 1st and 2nd extraction steps, followed by a relatively slow decrease in subsequent extractions. One exception was the scissor-cut Plain B, which demonstrated a slight increase in the 8th and 9th extraction steps. Therefore, we continued the extraction for this sample with three more extractions. The results showed that the number of extracted MPFs from scissor-cut samples dropped to a mean level of 62 MPF/g, compared with the ones extracted from the 9th step (689 MPF/g). Since there was no elevated number of extracted MPFs for the laser-cut samples and the standard deviation of this 9th sample was much higher than for the extractions before and after, this suggest that the peak at extraction step 9th was an outlier.

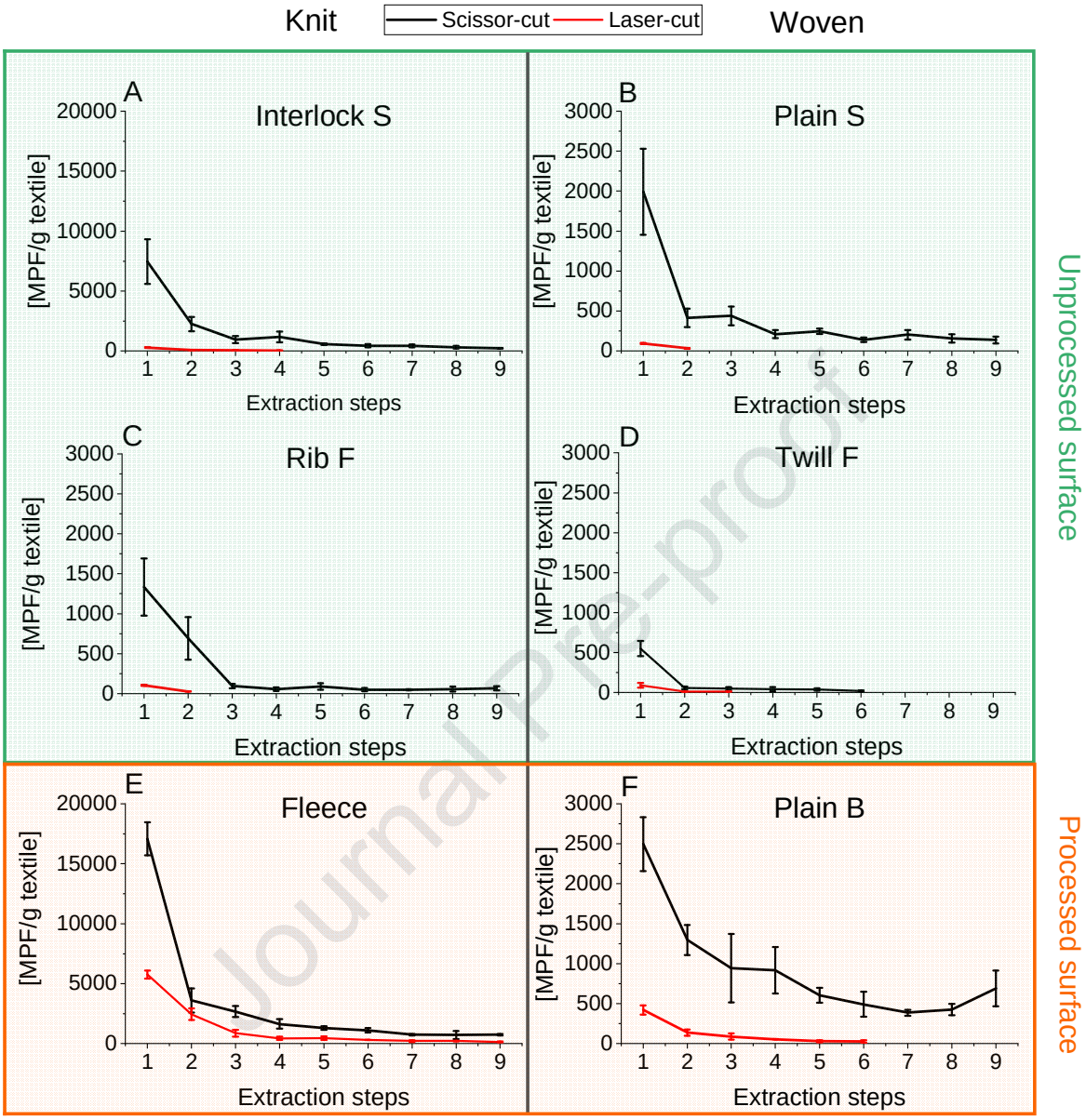

Figure 5. The number of extracted MPFs from textiles as a function of the number of extractions steps. Six textiles with different textile structures, yarn types and surface treatments were selected to present here and the remaining six samples are shown in Figure S3. All results are presented in number of MPFs per gram of textile. The black lines represent the scissor-cut samples, the red lines show results for laser-cut samples. Error bars represent the standard deviation for triplicate experiments.

3.3. Cumulative number of MPFs extracted from sliver, yarns and textiles

To estimate the total amount of MPFs present in the products, we summed the extracted fibers from all extraction steps (Figure 6). A logarithmic scale was used to cope with the large differences in the number of extracted MPFs from different samples. For the sliver and yarns, the lowest number of extracted MPFs was found for the filament yarn with a cumulative extraction of 15 MPF/g. That was about 40 times lower than the number of MPFs extracted from the sliver (590 MPF/g), which suggested that MPFs may be formed during sliver production. Moreover, the number of extracted MPFs from spun yarns was influenced by the spinning method. The majority of the yarns (Air-jet, Ring-black, Ring-white) exhibited a lower number of extracted MPFs than the sliver. On the other hand, the amount of MPFs extracted from the rotor yarn (4'310 MPF/g) was approximately seven times higher than the MPFs extracted from the sliver, suggesting that the rotor-spinning process may be responsible for some MPF formation.

For the textiles, the cutting method was one of the most critical factors which significantly influenced the number of extracted MPFs (p-value < 0.001). The ratio of the cumulative number of MPFs extracted from scissor-cut (black symbols) to laser-cut (red symbols) samples was determined (Figure 6), where this is a constant ratio between the two variants. In practice, one can think of this difference between black and red symbols as an indication for the "extra" MPFs extracted due to the cutting method alone. It is interesting to note that the textiles with unprocessed surfaces exhibited a relatively high scissor-to-laser ratio with an average of 19. Meanwhile, the surface-processed textiles demonstrated a much lower scissor-to-laser ratio, averaging approximately 6. This suggested that there was a relatively higher share of MPFs extracted from the entire surface area when the surface underwent additional surface treatment.

Besides the cutting method, the number of extracted MPFs from textiles was influenced by the surface treatment. The linear mixed-effect model showed that the number of extracted MPFs from the textiles with processed surfaces was significantly higher than those from the textiles without surface treatment (p-value < 0.001). The highest number of extracted MPFs was found for the Microfiber textile, with 45'400 MPF/g and 11'300 MPF/g for the scissor-cut and laser-cut samples respectively. That is approximately 60 times higher than the number of MPF extracted from Twill F, which exhibited the lowest number of extracted MPFs (scissor-cut: 760 MPF/g; 120 MPF/g laser-cut). On the other hand, there were no significant influences observed by the yarn type (spun, filament) or the textile structure (woven, knit).

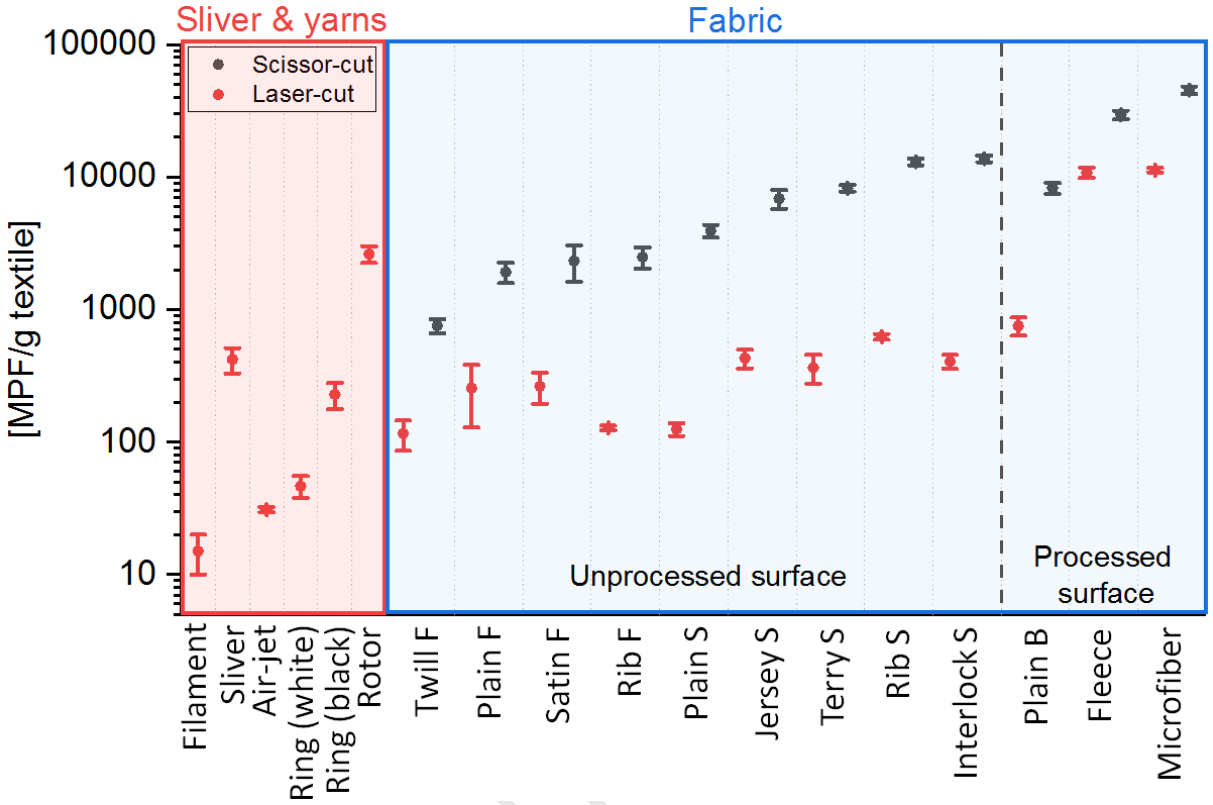

Figure 6. Cumulative number of MPFs extracted from the sliver, the yarns and the textiles in number of MPFs per gram of material. The mean values for the scissor-cut and laser-cut methods are represented by the black dots and red dots, respectively. Error bars indicate the standard deviation from triplicate experiments.

Furthermore, we found that the number of extracted MPFs during the 1st extraction step usually accounted for a high percentage of the total cumulative extraction (Figure S4). The results show that for 9 out of 13 scissor-cut samples and 12 out of 13 laser-cut samples, more than 50% of their cumulative MPFs were extracted during the first extraction. In comparison, the number of the MPFs extracted in the last step only accounted for a few percent of the cumulative extraction; only 3% for the scissor-cut samples and 8% for the laser-cut samples, on average. For nine out of 13 laser-cut samples, the experiments were stopped in less than 90 minutes, meaning that there were fewer than 20 MPFs per cycle extracted.

3.4. Length distribution of extracted MPFs

Although the number of extracted MPFs varied significantly between samples, the length distribution of released MPFs was relatively similar. The majority of the extracted MPFs were found to be between 100 and 800 µm in the 1st extraction (Figure 7). Significantly longer MPFs were extracted from the sliver (median: 405 µm) than those from the filament (median: 285 µm, p-value of 0.009). Another notable

feature for the sliver was that there were some much longer fibers, as indicated by the outliers in Figure 7, some of which were over 10'000 µm. The MPFs extracted from rotor yarns were significantly shorter (median: 226 µm, p-value < 0.001) than those from other spun yarns (median: 393 µm).

The shortest MPFs extracted from textiles were found in laser-cut Plain B, with a median length of approximately 184 µm, while the longest was found in scissor-cut Rib F (median: 595 µm). The length profile of extracted MPFs from textiles was affected by several factors. In particular, the surface treatment was identified as one of the critical points influencing the length of MPFs, with processed surfaces having shorter lengths than those from textiles with unprocessed surfaces (p-value < 0.004). Moreover, we found that the knit textiles tended to have significantly longer MPFs than the woven textiles (p-value of 0.04). No statistical differences in the length of MPFs was observed between the textiles made from spun yarns and the textiles made from filament yarns (p-value of 0.882).

Furthermore, we found that the length of fibers which were extracted was significantly affected by the cutting method. The MPFs extracted from seven scissor-cut textiles (Interlock S, Jersey S, Rib S Terry S, Rib F, Fleece and Microfiber) had longer lengths than those from the laser-cut ones. In contrast, five textiles, including Plain S, Plain F, Plain F, Twill F and Plain B, had longer MPFs extracted from laser-cut samples. It is notable that all of those that shed longer MPFs from laser-cut samples have a woven structure.

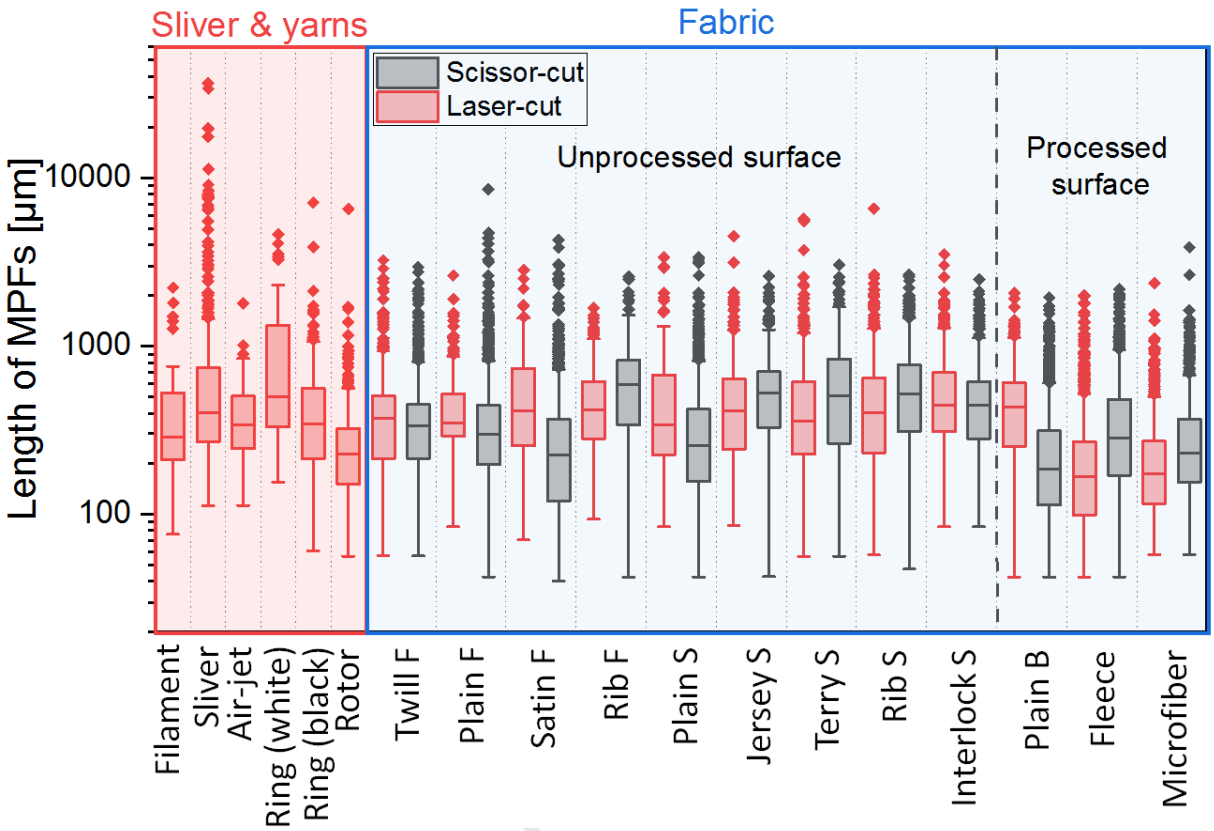

Figure 7. Length distribution of MPFs extracted during the 1st extraction step. The MPFs extracted from 1st extraction steps were present here, except for the dyed samples (Sliver, Air-jet, Ring-white, Rotor) where the MPFs from the 1st dyeing solution were present here. 25th and 75th percentiles were plotted in the boxes with a line indicating the median. Whiskers represent 95% of length distribution and outliers were labelled dots representing 0.7% of total distribution. The values presented here were a summation of three experimental replicates. The number of MPFs plotted per sample was between 59 for Air-jet to 3'498 for Plain B, with an average of 993.

Additionally, we analyzed the changes in length across all extraction steps (Figure 8, Figure S5). In general, after a few extraction steps, the length of MPFs extracted from the same textile was more or less constant. A pairwise K-W test was used to compare the length of MPFs from the neighboring steps. For example, the extraction from 1st and 2nd, 2nd and 3rd were compared in pairs, and so on. For the scissor-cut samples, the length of the extracted MPFs often increased significantly between steps in the first few extractions. However, after the 3rd or 4th extraction, there were no longer statistical differences between the steps. On the other hand, no significant difference in length between steps was observed for most of the laser-cut samples.

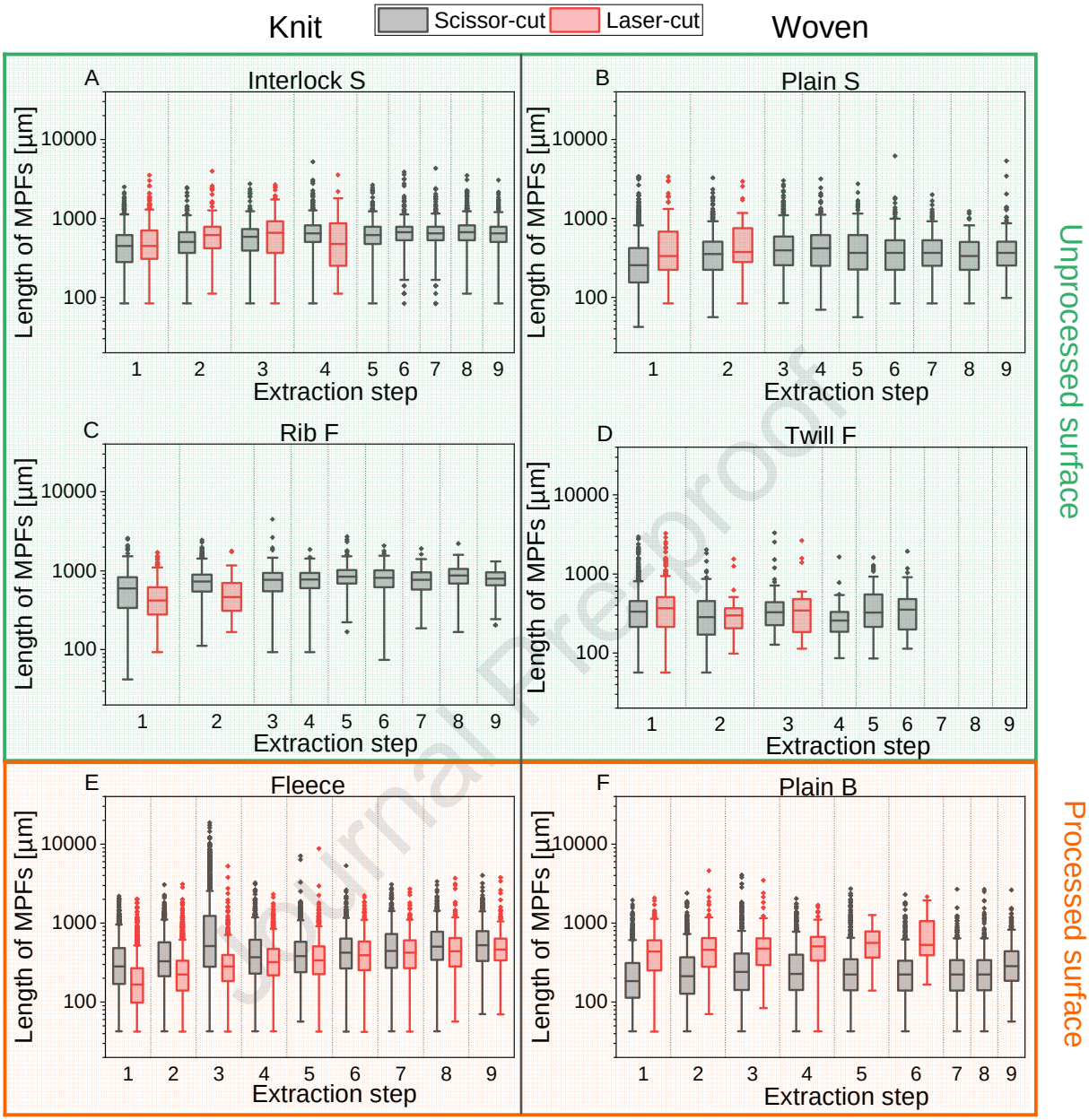

Figure 8. Length distribution of MPFs extracted during the sequential extraction steps. 25th and 75th percentiles were plotted in the boxes with a line indicating the median. Whiskers represent 95% of length distribution and outliers were labelled by dots representing 0.7% of total distribution. The value present here was a summation of three experimental replicates. The number of MPFs plotted per distribution was between 20 to 3'498 with an average of 663.

The diameter of extracted MPFs on the filters was compared with the fiber diameters within the original textile swatches to gain further insight into the origins of the extracted MPFs (Figure S6). The results

show that most of the liberated MPFs shared a similar diameter as the fibers in the parent textile. This is also true for the Plain F, which possesses the smallest fiber diameter in our sample set.

3.5. Investigating the edge effect

Additional experiments were performed to further investigate if the number of fibers extracted from the textiles scaled linearly with the total length of the edges. The Interlock S textile of fixed surface area (36 cm^2) was cut into a number of smaller pieces, resulting in additional edges. For the laser-cut samples, an average extraction of 188 MPF/g was observed regardless of the length of the edges (Figure 9), which corresponds to 5.2 MPF/g textile*cm^2. In contrast, for the scissor-cut samples, there is a linear correlation (Equation 1) between the number of extracted MPFs and the total edge length (correlation coefficient of 0.984 with a p-value < 0.001). The intercept of the regression line was set at 5.2 MPF/g*cm^2, representing the number of extracted MPFs from the textile surface of a 36 cm^2 swatch alone (i.e. the number of fibers detected upon extracting fibers from the laser cut textile swatches). The slope of the equation suggests that 346 MPFs can be extracted from one centimeter of cut textile edge.

$$Extraction\left[\frac{MPF}{g}\right] = \underbrace{5.2\left[\frac{MPF}{g * cm^2}\right] * Surface[cm^2]}_{188\ MPF/g} + 346\left[\frac{MPF}{g * cm}\right] * Perimeter\ [cm]$$

Equation 1

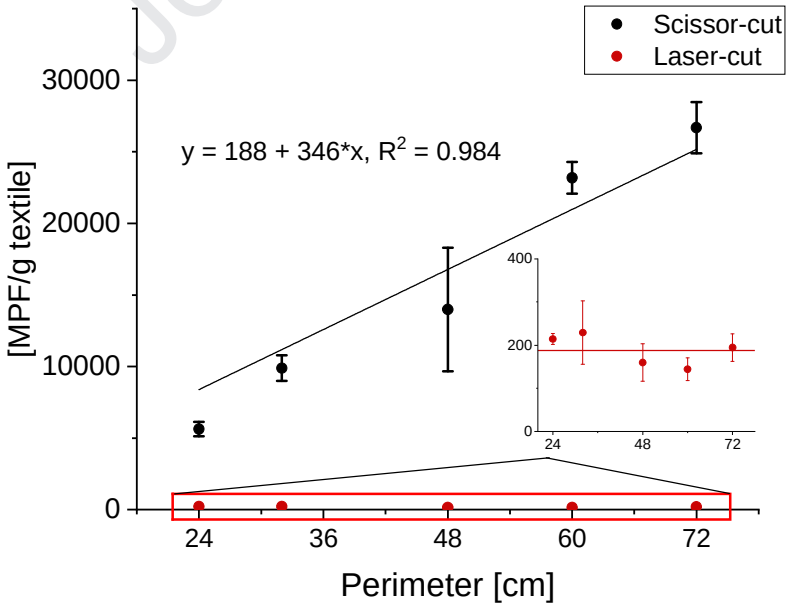

Figure 9. Correlation between the number of extracted MPFs and the samples' perimeter. Black dots represent the extraction from the scissor-cut samples and the red dots represent the extraction from the laser-cut samples. The black line indicates the linear regression between the number of MPFs extracted and the length of the perimeter for scissor-cut samples. The red line indicates the average extraction from the laser-cut samples. Error bars represent the standard deviation from triplicate experiments.

4. Discussion

4.1. Ultrasound as a method to extract embedded MPFs

In this manuscript, the method we investigated to extract fibers from textiles was an ultrasound extraction. A short-duration (2 to 5 minutes) ultrasonication has been found to cause little damage to textiles in several previous experiments which assessed the removal of soil contaminants from textiles (Gotoh and Harayama, 2013; Gotoh et al., 2015a; Gotoh and Hirami, 2012; Gotoh et al., 2015b; Hurren et al., 2008; Ma et al., 2014). That is not only true for polyester textiles (Gotoh and Hirami, 2012) but also for silk textiles (Gotoh et al., 2015a). By forming and collapsing bubbles or cavities, ultrasound is considered as a gentler way to remove surface contamination from textiles than methods using mechanical agitation, which causes textile-textile friction.

Although the duration of the ultrasonication and the input energy is different between the experiments in the literature and our present study, the results from our study suggest that ultrasound itself does not produce a significant amount of MPFs. On one hand, we observed that the number of extracted MPFs from the textiles dropped to 3-8% of the cumulative amount after several extractions, suggesting that the sonication is not responsible for the major amount of extracted MPFs. On other hand, we also have not observed any sign of damage or pills on the fabric and the fiber structure itself remained intact after several extraction steps, as verified by SEM images. However, since there was still a relatively small amount of MPFs extracted from the samples at 90 minutes, we cannot exclude the possibility that the ultrasound treatment generated a small amount of MPFs. Additionally, some MPFs in the textiles may only be extracted after a longer period of time, and thus would not be accounted for in our study.

4.2. The origin of MPFs in textile products

To track the origin of MPFs, we need to recall the manufacturing process of polyester textiles. Our hypothesis was that fibers and fabric are subjected to strong forces during certain steps in manufacturing and are responsible for the formation of MPF. Endless melt-spun fibers are converted into filament yarns or cut into staple fibers of lengths between 25 to 150 mm, which are further carded into slivers (Gries et

al., 2015; Krifa and Ethridge, 2006). As we can see in Figure 7, the extracted MPFs typically had a length below 1'000 µm, which is 25 times shorter than the length of the shortest staple fiber (25 mm). Therefore, neither the endless filaments nor the sliver are likely to be the origin of MPFs found in our experiments. The number of MPFs extracted from the filament yarn was below 15 MPF/g. That is in agreement with our hypothesis that there should not be any MPFs originating from "endless filaments", except for cross contamination from the production site, which can be removed in the first few extractions. On other hand, we found a broad length distribution of MPFs in the sliver ranging from 80 to 50'000 µm. There are many possibilities of how these fibers may have formed during the sliver production. First, filaments are often texturized before being cut and as a result they might not always be perfectly straight when they are caught by the blade. Second, the machines usually work on a rotating-cutting principle leading to unintentional cuts of protruding fiber ends. Moreover, staple fibers are processed into a carding machine where very sharp edges are applied to merge and parallelize fibers. Thus, it is also possible that these edges may cut fibers in a non-controlled way. The liberated MPFs shared a similar diameter as the fibers in the parent textile, suggesting that the majority of the shed MPFs we analyzed originated from the textile and that abrasion by cross-sectional fibrillation (resulting in smaller fiber diameters) is a negligible mechanism of MPF production.

Although fibers underwent additional mechanical stress during spinning the sliver into yarn, most of the spun yarns exhibited a lower number of extracted MPFs than the sliver. One reason may be because yarn has a tighter structure and twisting the yarn creates tensile force, which prevents the extraction of MPFs present in the yarn core. Moreover, the air-jet and ring spinning processes often involve a stage where the fibers are drawn and parallelized and where some of the ultrashort fibers may already be removed. However, there is one exception, the rotor yarn, which shed substantially more MPFs than the sliver as well as the other yarns, suggesting that the rotor spinning process might be the primary step responsible for the formation of MPFs. In rotor spinning, opening rollers with very sharp edges are used to open up the fiber bundle before twisting it together into a yarn. In addition, it is also known that the accumulation of fiber dust (in other words: MPF) in the rotor groove can result in yarn defects and end breakages (Lawrence, 2010), which may also contribute to the formation of ultrashort fibers. However, we must consider that in the current experimental regime there are only a limited number of fiber samples and they are sourced from different companies. Therefore, they do not represent samples from one production line. Further research needs to be performed to better quantify how much variability exists in the MPF content of fiber samples from a larg(er) variety of machinery.

The next step along the production line is to weave or knit yarn into textiles. Although we found a similar number of MPFs extracted between the spun yarns and the laser-cut textiles made of spun yarns, we cannot draw a conclusion that the weaving or knitting of yarn into textiles does not form MPFs. That is

because textiles may be intensively washed during the finishing process, which could remove some short fibers from the yarn. Therefore, future studies are needed to investigate the influence of knitting or weaving from yarn to textile with more controlled samples.

Some treatments can be applied to the textile surface to achieve a special texture. For example, a screw-like shearing blade is commonly used to cut the surface fibers of fleece to create a fuzzy feel. Additionally, several abrasion processes (e.g. sand blasting or rubbing with sandpaper) can also be used to improve the look and feel of the textile. On one hand, a large quantity of MPFs may be formed during this stage and remain in the textile. On the other hand, with a looser surface structure, the MPFs present in the textiles may be more readily extracted. Therefore, it is not surprising to find a statistical difference between the amount of MPFs extracted from the textiles with processed and unprocessed surfaces.

One of the most important findings in our study is that the amount of MPF extracted from textiles was significantly influenced by the cutting method. From the SEM images, we know that the textile edges of laser-cut samples exhibited a seal of molten polymer and the scissor-cut samples consisted of a large number of open ends near the edge. Therefore, the major source of MPFs from laser-cut samples is the surface and for the scissor cut ones the source is both the surface and the edges. We observed a much higher number of extracted MPFs from all 12 scissor-cut samples compared to the laser-cut samples, suggesting that the cutting method plays an important role in the number of MPF which can be extracted from textiles. There is the possibility that the textile samples were contaminated by other fibers during production or shipping. However, this contamination level should not be above the amount of extraction from the laser-cut ones, which means the difference of extracted MPFs between the scissor- and laser-cut samples was indeed due to the cutting method. Since cutting textiles is normally undertaken when tailoring garments, as a consequence, tailoring could be jointly responsible for the formation of MPFs present in garments.

There are two ways that scissor cutting can contribute to the MPFs extracted from textiles. One is that cutting creates openings at the ends of the yarns by fraying, which enables the extraction of MPFs present inside the yarn trapped during spinning. The other possibility is that the fibers are formed during the cutting process itself. All knitted textiles share a basic loop structure. Therefore, cutting through the loops may produce loose fiber fragments sitting on the edge that can easily be removed by extraction. For woven textiles, the cutting line can pass at random fiber positions leading to the formation of MPFs. We observed that the length of extracted MPFs from scissor-cut knit textiles was longer than those from woven textiles. However, there is no difference between the two kinds of structures in terms of the number of extracted MPFs.

Based on the results, we propose to use a combination of extractions from laser- and scissor-cut samples to develop an "extraction equation" for each textile (Equation 2):

$$Extraction_{textile} = \underbrace{\frac{extraction_{laser}}{area_{sample} * mass_{sample}} * area_{textile}}_{Extraction_{surface}} + \underbrace{\frac{extraction_{scissor} - extraction_{laser}}{perimeter_{sample} * mass_{sample}} * perimeter_{textile}}_{Extraction_{edge}}$$

Equation 2

where "$extraction_{laser}$" is based on the laser-cut samples and "$extraction_{scissor}$" on the scissor-cut samples. With this equation, we can predict the number of extracted MPFs from a larger piece of textile by considering both the surface area as well as the length of the edges (Table 2). The ratio of edge/surface release is between 3 for Fleece and 53 for Plain S. This procedure could be used as a standardized test that would be suitable to estimate the number of extractable MPFs present in textiles based on extraction of small textile swatches. For example, upscaling from the scissor-cut sample of 36 cm^2 to a fleece textile of 1 m^2 (edge 4 m) would result in an extraction of approximately 2'234'000 MPFs. The influence of the area/perimeter ratio decreases with increasing surface area. For a 1 m^2 fleece textile, only 3% of the MPFs originated from the edge, but the percentage increases 50 times for a 0.01 m^2 textile. For Plain S, with the highest ratio of edge/surface, the corresponding release from a 1 m^2 piece would result in an extraction of 180'000 MPF, with 56% of MPFs originating from the edge.

Table 2. Estimated MPFs extracted per cm^2 surface area and per cm perimeter. The calculations were performed according to Equation 2 and based on the cumulative number of extracted MPFs.

Textile	Extraction from Surface [MPF/(g*cm^2)]	Extraction from edge [MPF/(g*cm)]	Ratio edge/surface [cm]
Twill F	3	27	9
Plain F	7	69	10
Satin F	7	86	12
Rib F	4	98	25
Plain S	3	159	53
Jersey S	12	268	22
Terry S	10	328	33
Rib S	17	518	30
Interlock S	11	556	51
Plain B	21	312	15
Fleece	300	781	3
Microfiber	313	1422	5

In addition, it is interesting to note that although the length of extracted MPFs from different textiles exhibited a degree of variation, the majority of extracted MPFs shared a similar length range between 100

µm to 800 µm. The similarity in the length of extracted MPFs may be explained by the origins of MPFs. We have identified that there are two plausible origins of MPFs in textiles. The first is a "liberation" of existing MPFs which are produced during the manufacturing process. Although there are many textile varieties for different purposes on the market, the manufacturing processes of slivers, yarns and textiles are rather similar. Therefore, it is not surprising that the length of MPFs produced during the manufacturing exhibit a high degree of similarity. The second origin of MPFs is suspected to be "production" through the textile cutting process. Apart from the fact that there are many different textile structures such as interlock, jersey, plain, and twill, there were often variations derived from some basic structures. For example, all knitted textiles are knitted by the yarn following a meandering path to form symmetric loops and all the woven textiles are made by two or more threads interlaced at a right angle. This might be another reason that the length of MPFs is limited to a relatively narrow range.

4.3. Comparison with previous studies on the MPF release from textiles

Several studies have investigated the MPF release from synthetic textiles during washing, using either real domestic washing machines (Browne et al., 2011; Dris et al., 2016; Hartline et al., 2016; McIlwraith et al., 2019; Napper and Thompson, 2016; Sillanpaa and Sainio, 2017) or lab washing machines to simulate the domestic washing process (Almroth et al., 2018; De Falco et al., 2018; Hernandez et al., 2017; Jonsson et al., 2018). We cannot directly compare our numbers of extracted fibers to these studies or estimated releases from different textiles during washing as the release mechanism may not be the same. Further studies have to show how the extraction used in our work relates to the number of MPF released during washing. Moreover, the MPFs can be also formed in the use phase of textiles. Therefore, the number of the MPFs extracted in our study do not correspond to the total release including the use phase. The MPFs extracted from textiles products may correlate with the MPF release during the first few washing cycles, which needs to be confirmed in future studies.

However, several conclusions regarding the release mechanisms can still be made. Because the washing studies investigated different textiles with various experimental setups and analytical methods, the amount of MPFs released per wash reported varied, ranging from 0.012 mg/g (Pirc et al., 2016) to 3.3 mg/g (Sillanpaa and Sainio, 2017), from 23 MPF/g (Pirc et al., 2016) to 1'273 MPF/g (De Falco et al., 2018). Our work has shown that the type of textiles and the treatment of edges can also strongly influence the magnitude of release. The material used in different studies varied from whole garments (Browne et al., 2011; Hartline et al., 2016) to pieces of textiles (De Falco et al., 2018), to double folded and sewn edges (Hernandez et al., 2017) to scissor-cut edges (Jonsson et al., 2018). These variables make it difficult to directly compare the results amongst or between the studies with the data we have collected here. For

instance, we found that the MPF extracted from the scissor-cut Jersey S during the first extraction was 0.4 mg/g, which is much higher than the 0.1 mg/g release for the jersey textile per wash reported by Hernandez et al. (2017), using doubled folded and sewn edges. Because we demonstrated the importance of the cutting method and the ratio edge/surface is, different studies cannot be compared without a fully standardized procedure.

While there are some studies quantifying the number or the mass of MPFs released during textile washing, there is limited information regarding the length of MPFs released. A few recently published studies provided more detailed length distributions of released MPFs by characterizing a larger number of MPFs (De Falco et al., 2018; Hernandez et al., 2017; McIlwraith et al., 2019). Sometimes the length profile was also provided in the form of a "size range" with intervals usually above 200 µm (Almroth et al., 2018; Hartline et al., 2016). Some previous studies only suggested an average length of MPFs released by sampling a limited number of MPFs on filters (Napper and Thompson, 2016; Pirc et al., 2016). The length of the released MPFs ranged from 100 µm (Hernandez et al., 2017) to 25 mm (Pirc et al., 2016), depending on the application of different filtering and analysis methods. The MPFs from our study fell into a range between 100 to 800 µm which is in accordance with the findings by Hernandez et al. (2017) and De Falco et al. (2018), using a similar filtration technique and analysis methods.

4.4. Implications for the textile industry

Our results confirm that a predominant fraction MPFs may already be present in textiles when manufactured. The rotor yarn production method leads to the most important number of extracted MPFs compared to the other yarns. This is a strong indication that the rotor spinning process may be a critical stage responsible for the MPF formation during the yarn spinning. A more representative study on rotor-processed yarns is now advised. Furthermore, a comprehensive investigation is needed to compare different spinning methods, which requires the collaboration between industry and academia. A precise location of the origin of MPFs in the production process will guide any future efforts to minimize MPF release. Additionally, we observed that textiles with processed surface treatments (fleece, brushed surface) exhibited a significantly higher release than the other types of textiles.

Furthermore, for all textile variants, the laser-cut samples demonstrated on average 17 times fewer extracted MPFs than the scissor-cut ones, suggesting that adopting cleaner cutting methods (e.g. during tailoring) is another option for the industry to help reduce the MPF release since the majority of the MPF originates from the edges of the textile and not from the textile surface. Scrutinizing the cutting and seaming processes thus offers another means to minimize MPF release from garments. Finally, our results

revealed a sharp decrease in the number of extracted MPF after the 1st extraction, which typically accounted for more than 50% of the total number of fibers extracted. Therefore, prewashing cut textiles or garments once at the factory and collecting the released MPFs before delivery to the customers may be an efficient way to remove a large part of the present MPFs in the products, which was also suggested by other researchers (Almroth et al., 2018).

5. Conclusions

Although there have been a number of textile washing/MPF release studies performed in the recent years, the origins of MPFs released during the wash cycle has remained unclear. In this study, we have developed an ultrasonic extraction method to extract and characterize MPFs already present in the textiles, which was used to discern MPFs exclusively generated in the production process. A representative set of 18 polyester products along the textile production line was investigated. We found that the rotor spinning and surface treatment are among the most critical steps responsible for the formation of MPFs during the yarn production and textile production, respectively. Moreover, the cutting methods to create textile swatches (and, to begin tailoring garments) had a significant influence on the number of extracted MPFs. We used this insight to differentiate between surface- and edge- contributions of total MPF in the extraction process.

Our results confirm the presence of MPFs in textiles throughout the manufacturing process. Since the first extraction consistently released the majority of the total MPFs, we propose that prewashing textiles may remove a significant portion of the production-initiated MPFs from a textile product at a point source (i.e. the factory). However, it should be noted that MPF which are intrinsically in the textiles from the manufacturing process do not necessarily influence the formation of additional MPF during washing, wearing or the later use phase(s). Therefore, the number of the MPFs extracted in our study does not correspond to the total release of MPFs from textiles during the whole life cycle. Instead, our results confirm that it is analytically possible to discern between production-inherited MPF and those which are produced as part of the use phase of the life cycle, which opens up new avenues to systematically study release scenarios that include abrasive washing and usage.

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Declaration of interests

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

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: