

Systematic study of the presence of microplastic fibers during polyester yarn production

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ABSTRACT

Synthetic fibers, especially polyester, have over past decades managed to overtake and dominate the textile industry. Microplastic fibers are shed from synthetic textiles during their user phase, primarily during washing. However, there is little known about their origin, except that they are likely embedded in textiles already since their production. Therefore, we systematically examined the presence of microplastic fibers during the process of yarn production. We started with one bale and took samples from the bale opening step throughout carding, sliver handling and finally to spinning. We extracted microplastic fibers from all samples in order to quantify and characterize them. We also investigated the impact of process parameters, especially 4 different spinning methods (ring, compact, rotor, and air-jet spinning). We found microplastic fibers in all studied samples, ranging between 44 fibers/g to 8057 fibers/g. Rotor-spun yarns were identified as a material with a high content of microplastic fibers (2000–8000/g) while the other samples, including yarns spun with alternative methods, showed fiber numbers in tens and hundreds of fibers/g. Varying the operational settings of carding and spinning had none to minimal impacts on fiber number with the exception of rotor spinning, where we observed a 4 fold increase when the speed was increased by 25%. The released fibers have each a unique fiber length distribution with varying medians: 210 μm for rotor yarns, 330 μm for air-jet yarns and 530–580 μm for ring and compact yarns. The results from this study will allow textile companies to select processes or operating conditions that minimize the presence of microplastic fibers.

1. Introduction

The occurrence of microplastics in all environmental compartments all over the world is one of today's major environmental issues and a lot of research is carried out to identify the sources, transport and effects of microplastics. Textiles have been identified as a major source of microplastics as fibrous microplastics were found to be a frequent type of microplastics in marine (Browne et al., 2011; Barrows et al., 2017), freshwater (Koelmans et al., 2019; Wagner et al., 2018) and soil environments (Li et al., 2018; Zhou et al., 2020). Microplastics, including microplastic fibers (MPF), are defined by a particle size smaller than 5 mm (Barnes et al., 2010; Andrady, 2017).

It is likely that a large part of the MPF observed in environmental samples originate from laundry. Even though waste water treatment plants can have up to 99% removal efficiency (Talvitie et al., 2017), there are still large numbers of MPF discharged into natural waters. In some countries the sewage sludge is used as a fertilizer in agriculture and the MPF are as a result directly introduced into the soil environment (Corradini et al., 2019). In addition, globally only about 20% of

wastewater goes through any treatment prior to being discharged into natural waterbodies (UNESCO World Water Assessment Programme, 2018).

Based on the identification of textiles and laundry as an important source of microplastics, many washing studies have been performed in order to understand the number of MPF released and the mechanisms behind the release. Polyethylene terephthalate (PET) fibers have a dominant role in textile industry, taking up 52% of the total fiber market (Textile Exchange, 2021) and more than 80% of the synthetic fiber market (Bartl, 2020). Therefore, most studies primarily focus on PET fabrics. The results from the washing studies vary depending on the types of fabrics that were used and the experimental conditions. The number of released MPF per gram of textile ranges from few MPF to ten thousands of MPF (De Falco et al., 2019; Carney Almroth et al., 2018; Sillanpää and Sainio, 2017). One feature that the washing studies have in common is that the number of MPF decreases with repeated washing cycles (Cai et al., 2020b; Belzagui et al., 2019). Cai et al. hypothesized that the MPF in textiles are inherited from yarn and fabric

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Abbreviations

MPF	Microplastic fibers
PET	Polyethylene terephthalate
LAS	Linear Alkylbenzene Sulfonate
CS	Carding speed
S	Spinning speed
T	Traveler
ORS	Opening roll speed

production and are trapped within the fabric until they are washed out. This suggestion was also supported by the analysis of the ends of the extracted MPF. The MPF showed a sign of breaking upon high energy processes instead of fatigue failure fractures which would be expected to be a product of long exposure to forces during washing. In addition, the median length of the MPF increased with increasing number of washing cycles, which can be related to the fact that initially the shortest MPF are extracted and the more entangled MPF debris needs more time to be extracted from the textiles.

In addition to the washing studies, there is another strong proof of MPF formation during textile production. It is known that there is dust produced in the manufacturing process and therefore most machinery contains a mechanism for removal of such dust (Goyal and Nayak, 2020). Such dust and impurities are collected in filter bags and disposed of based on the regional waste treatment strategy (eg. incinerated). Dust from PET brushing has been characterized and short fibers were identified (Mellin et al., 2016).

Yarn production starts with the opening of a bale of short staple fibers. By then, these fibers have already gone through a series of production steps. PET pellets are melt-extruded and spun into filaments, which are quenched, drawn, crimped and cut into short fibers with lengths of several centimeters, and finally pressed into a bale (Hufenus et al., 2020). So produced staple fibers are called PET cotton-like fibers. The bale then travels to a yarn production plant where the bale is opened and fed into a drawing machine (Fourné, 1999). The output is a sliver made of parallelized fibers which is then drawn repeatedly to align the fibers into a finer sliver (Lord, 2003a). Polyester staple fibers represent 18.5% of the total fiber market which translates to production of 23 m tons of such fibers in 2020 (Industrievereinigung Chemiefaser e. V., 2022).

Nowadays, there are different spinning methods available on the market. The first developed industrial method is ring spinning, which is still widely used as the final yarn is strong and of a good quality. Slivers for ring spinning go through one more pre-spinning process, where their diameter is further reduced and a twist is imparted. The resulting pre-yarn is called roving. A key component of the ring spinning machine is the so-called traveler which is placed on a ring and is responsible for twisting the drafted roving and for winding the yarn onto the bobbin (Rengasamy, 2010). Compact spinning is an updated version of classic ring spinning, where some parts of the apparatus are slightly modified. The resulting yarns are expected to have better fiber orientation than the classic ring-spun yarns, including better parallelism, compactness and lower hairiness (Alagirusamy and Das, 2015).

Another method for yarn production is rotor spinning which works on the open-end spinning principle. In this method the sliver is opened and individual fibers travel into the groove of the rotor where they get in contact with the tail end of a yarn and new yarn is propagated as twist is introduced to it thanks to the rotational motion. Rotor spinning is suitable for production of coarse and medium-fine yarns as the fibers are less parallel, less twisted, and less compact. However, the method exceeds the ring spinning in productivity (Das and Alagirusamy, 2010b).

Another high delivery method is air-jet spinning. In this method the twist is introduced by an air vortex (Elhawary, 2015). Even though the resulting yarn looks similar to ring-spun yarn, the twisted fibers make only 6% of the yarn and are wrapped around a core consisting of parallel fibers (Das and Alagirusamy, 2010a).

Currently, only ring spinning and rotor spinning technologies are used to produce pure polyester yarns, with both systems covering a similar share of the market. Compact spinning and air-jet spinning are used almost exclusively for blends with regenerated cellulose or cotton (Rudolf Härdi 2022, personal communication). The primary objective of this study was to systematically analyze the yarn production process in order to assess whether a certain step leads to formation of MPF which are then trapped in the yarns and textiles, until they are washed or worn out. In addition, different settings of the machines are studied with the aim to enable an optimization of the process to minimize MPF content in the output yarns. Cai et al. (2020a,b) reported a higher number of MPF in a rotor yarn compared to other spinning methods but an exhaustive study of the yarn production process is missing so far. To allow a clear identification of the relevant processes, we started with one single sample of bale and followed the MPF content throughout the full chain of machines until the final yarns were obtained. Intermediate samples from yarn production, as well as the final yarns produced under variable settings, were washed to extract the MPF to be able to count and to characterize them.

2. Materials and methods

2.1. Samples

A bale of black, dope-dyed PET staple fibers (38 mm, 1.5 dtex) supplied by Swicofil (Emmen, Switzerland) was processed by the Rieter Spin Center (Winterthur, Switzerland). This bale was then further processed and mid-products were collected from several process stages along the production line. Different settings were tested and 4 spinning methods were used. In total 6 production pathways were assessed, creating 22 mid-products and 22 different yarns to test (Fig. 1). The mid-products studied were staple fibers from bale, chute feed, slivers sampled after carding and drawing steps, as well as roving where it was applicable. A detailed table with the specific parameters can be found in the SI. All yarns were spun to the same linear density of 30 Ne (English), which is equivalent to 19.7 tex in universal yarn numbering system. Twist introduced to ring, compact, and rotor yarns was set at 820 tpm. (Twist of air-jet-spun yarns cannot be measured due to the spinning principle.) The detailed structure of selected yarns can be found in Fig. 2. Speed and traveler are further abbreviated as S and T, for example a ring spun yarn spun at speed 1, with traveler 2 from sliver carded at speed (CS) 2 will be abbreviated as “ring S1T2, CS2”.

2.2. Sample preparation

From the bulk-produced samples, triplicates of smaller pieces were prepared which were then used in the experiments. Slivers were cut with a laser cutter (tt-1300, Times technology) to pieces of the weight of 1.0 ± 0.1 g. The length of the pieces was calculated depending on the linear density of the specific slivers which varied between 0.7 and 7 ktex ($1 \text{ ktex} = 1 \text{ g/m}$). Bale and chute feed samples of 1.0 ± 0.1 g were prepared without cutting.

The yarn sample weight was adjusted to 1.5 ± 0.1 g to have enough extracted fibers from all samples to be able to perform statistical evaluation. Yarns were unwound from bobbins (ring, compact yarns) or cones (rotor, air jet yarns) and the ends were heat sealed with a lighter.

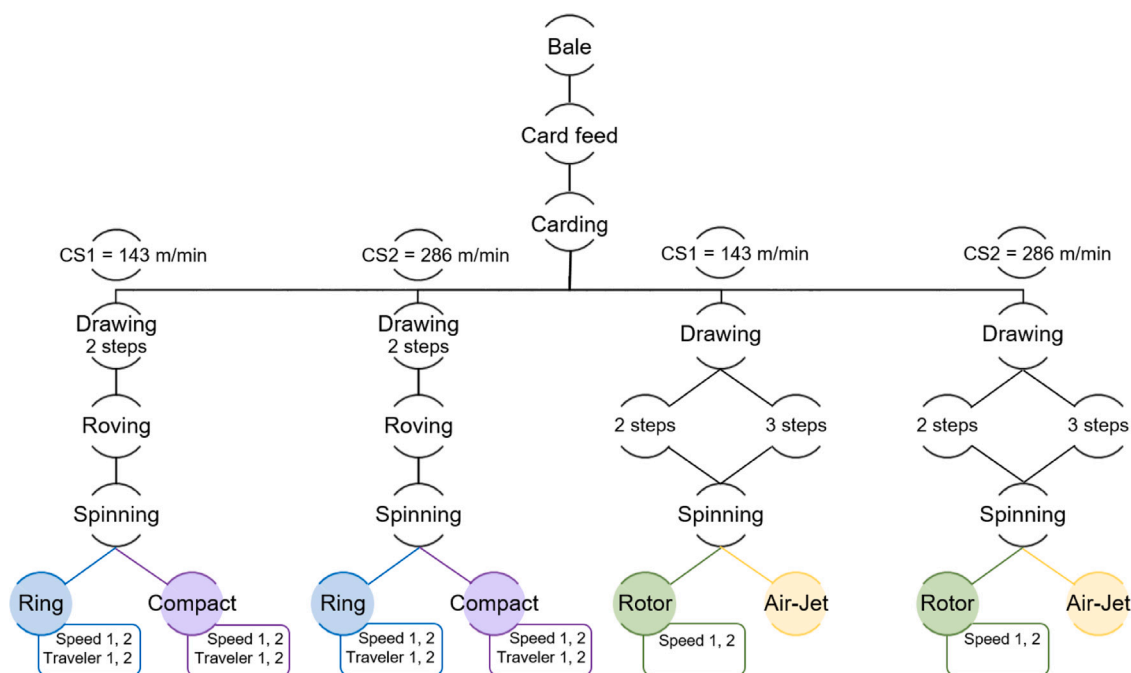


Fig. 1. Scheme of samples from yarn production; Speed 1 is always lower than speed 2 but differs for the different processes (S1 = 15000 rpm, S2 = 19000 rpm for ring and compact spinning; S1 = 7500 rpm, S2 = 9500 rpm for rotor spinning). In the case of rotor spinning, the speed represents the opening roll speed (ORS). For details about the conditions see table S1 in the supporting information. Yarns are color coded based on the type of spinning mechanism.

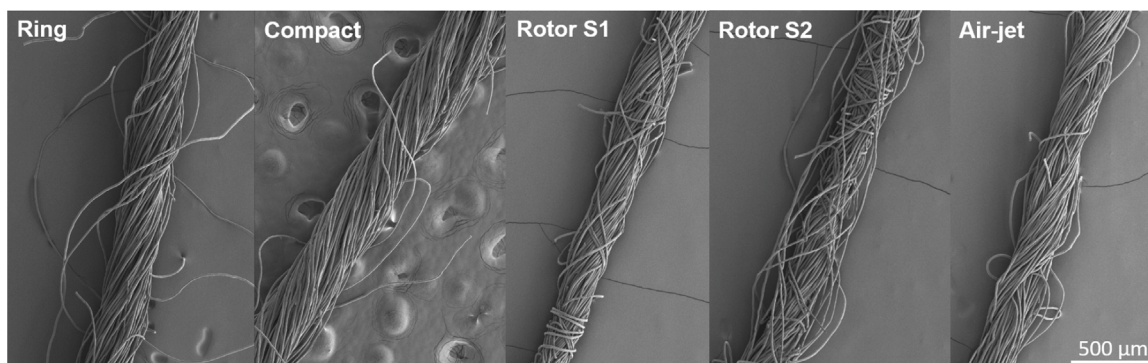


Fig. 2. SEM images of yarns made by different spinning methods. Shown yarns are specifically: ring yarn CS1, S1T1; compact yarn CS1, S1T1; rotor yarn CS1, S1; rotor yarn CS1, S2; air-jet yarn CS1.

2.3. Washing - extraction experiments

Washing experiments were done with the aim to extract MPF embedded in the material. The experiments were performed in a Gyrowash lab washing machine (James Heal, GyroWash model 1615). The experimental setup was based on the standardized procedure for testing color fastness in textiles in laundering processes (ISO 105-C06, 2010). In the first step, the 500 mL steel vessels and 6 mm steel balls were thoroughly cleaned and rinsed with DI water, followed up by two 5 min cycles in the Gyrowash at 40 °C with 50 mL of linear alkylbenzene sulfonate (LAS) solution (ThermoFisher) and 100 mL of DI water. The LAS solution was used to substitute for a commercial laundry detergent. The 0.75 g/L LAS solution pH was adjusted to 9.2 ± 0.1 with 1 M NaOH solution.

The prepared sample was put into the steel vessel with 10 steel balls and LAS solution (150 mL). The experiment was conducted in triplicates for all samples. For every two sets of triplicates, one blank experiment was performed by running the experiment without inserting any material into the vessel but with LAS solution and the metal balls. To minimize the likelihood of contamination of the samples, working

surfaces were wiped with water and ethanol in the morning, clean lab coats and gloves were used, and wearing black clothes under the lab coat was avoided.

The prepared vessels with samples were placed in the Gyrowash and washed for 45 min at 40 °C. Once done, the vessels were taken out and left to stand for 5 min to avoid foam leaving the vessel upon opening. The soaked sample was lifted from the vessel with tweezers and a custom-made sieve was placed in the vessel. The sample was then placed on the sieve and a circular weight (989 g, $r = 3.6$ cm) was placed on top for 15 s to let the soaked-up liquid drip off into the vessel. The picture of the apparatus can be found in the SI (Fig. S1). To compare the force of the weight with the force applied on clothes in a washing machine: the weight exerted 10 N on the sample, while a regular T-shirt washed in a washing machine with spin speed set to 1000 rpm experiences about 307 N. (Calculations can be found in SI, page 3.)

2.4. Filtration

After the removal of the sample from the vessel, vacuum filtration was performed on the washing liquid remaining in the vessel. The fibers

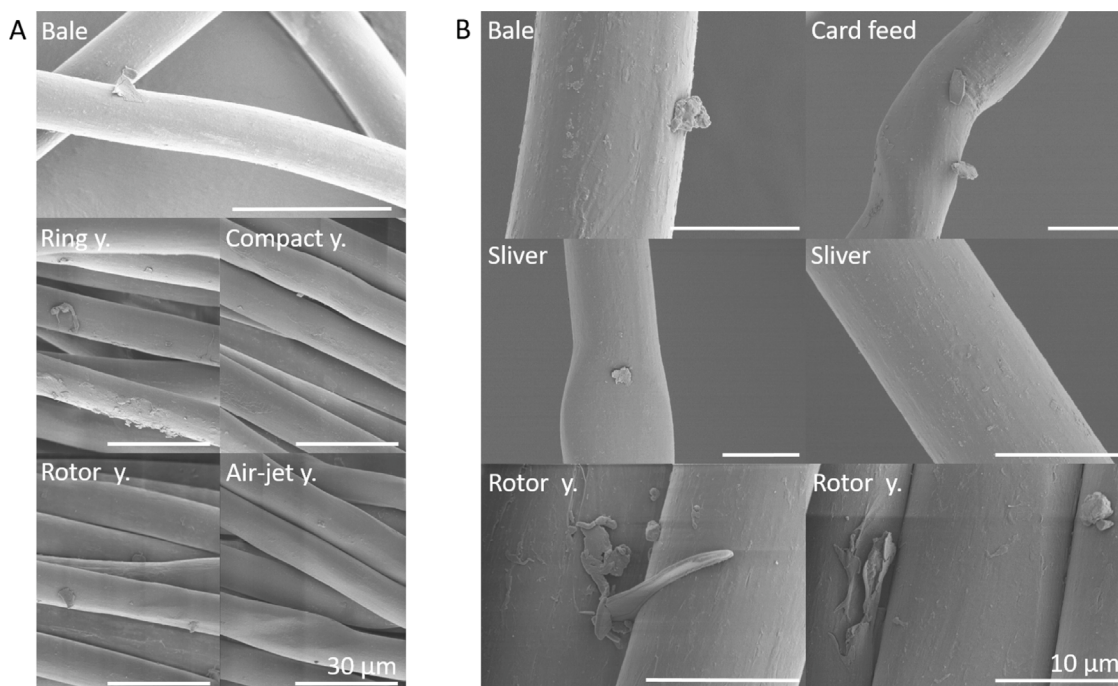


Fig. 3. Structure of the studied material: Figure A show surface unevenness of bale and selected yarns at lower magnification; Figure B shows details of some of the irregularities found on different samples at higher magnification.

were collected on a cellulose nitrate membrane (GE Whatman diameter 47 mm, pore size 0.45 μm). The volume of the liquid filtered was adjusted for each sample based on the expected quantity of the fibers on the filter to allow optimal image analysis. It ranged between 5 and 110 mL. The full volume (150 mL) was filtered for blanks. The filters were then left to dry in a single-use Petri dish.

2.5. Filter imaging and analysis

An image of the filter together with a microscope calibration slide was captured with the use of a single-lens reflex camera (Nikon D850) with a macro lens (Nikon 105 mm/2.8) (Fig. S2). The contrast of the photo was enhanced in Adobe Photoshop CC 2018. The photo was analyzed in the software FiberApp, which allows semi-automated caption of the fibers on the filter (Usov and Mezzenga, 2015). A closer look on the filter papers with the microplastic fibers can be found in SI (Fig. S3). Data about the number of fibers and their length was collected. As the analysis was performed on color-inverted images, a comparison was always made with the original photo to make sure only black fibers were labeled. In addition, only particle shapes which were clearly fibers were tagged. The detection limit is 4–5 pixels, which corresponds to 40 μm . However, in practice anything below 60 μm was difficult to assess if fibrous in shape. Therefore, in the later analysis any tagged particles and fibers shorter than 60 μm were removed from the assessed data lists. Fibers longer than 5 mm were also not considered.

Scanning electron microscopy (SEM) (Hitachi S6200, 2.0 kV) was performed on selected coated samples (7 nm Au/Pd) to obtain information about the structure of yarns and MPF and their ends.

2.6. Statistics

One-way ANOVA test was performed on the length distribution of different groups of the samples to assign whether they are statistically significant differences between them. Tukey post-hoc test was further run for those groups which had p-value smaller than 0.05. Same statistical tests were performed on the MPF counts for relevant sample groups.

3. Results

3.1. Quality assurance/quality control

The washing procedure used in this study had been used in previous studies and tested for its quality assurance and reproducibility (Hernandez et al., 2017; Cai et al., 2020a,b). As it is difficult to secure the lab from any contamination despite of frequent cleaning, and the samples could be contaminated already from the production site, we worked with black-dyed samples and only black fibers were investigated. The use of black yarns helped to differentiate from contamination coming from the lab room but also from the production site, as the test center usually works with white fibers. In addition, to prevent ambiguity, the smallest particles on the filter were omitted from the results as it was difficult to confirm their color as well as fibrous shape. To quantify contamination with black fibers, one vessel per wash was used as a blank and was further analyzed. The blanks performed for both yarns and slivers showed on average 9 MPF per wash with the maximum of 32 MPF found. Those numbers represent 4%, respectively 12% of the median number of MPF found on the tested filters. Some blanks contained only MPF shorter than 100 μm , some blanks were dominated by longer MPF. The median length was between 73 and 670 μm , with average median length of 255 μm .

All experiments were carried out in triplicates. Furthermore, an additional standardized step was introduced to the method to press out liquid from a sample after washing in a reproducible way. Initial experiments without this step showed high variety in results between the triplicates due to the fact that bale and chute feed samples retained large amounts of washing liquid (Fig. S3). An experiment was done with chute feed samples to test the effect of pressing on the number of MPF. It was observed that pressing out the liquid resulted in doubling the number of MPF in the analyzed washing liquid. In addition, the standardized method showed a decreased variability between replicates compared to performing the process by squeezing the sample by hand (Fig. S4). Using the standardized pressing method, the average relative standard deviation of the MPF count between the replicates in this study was 20% for yarn samples and 14% for samples from the

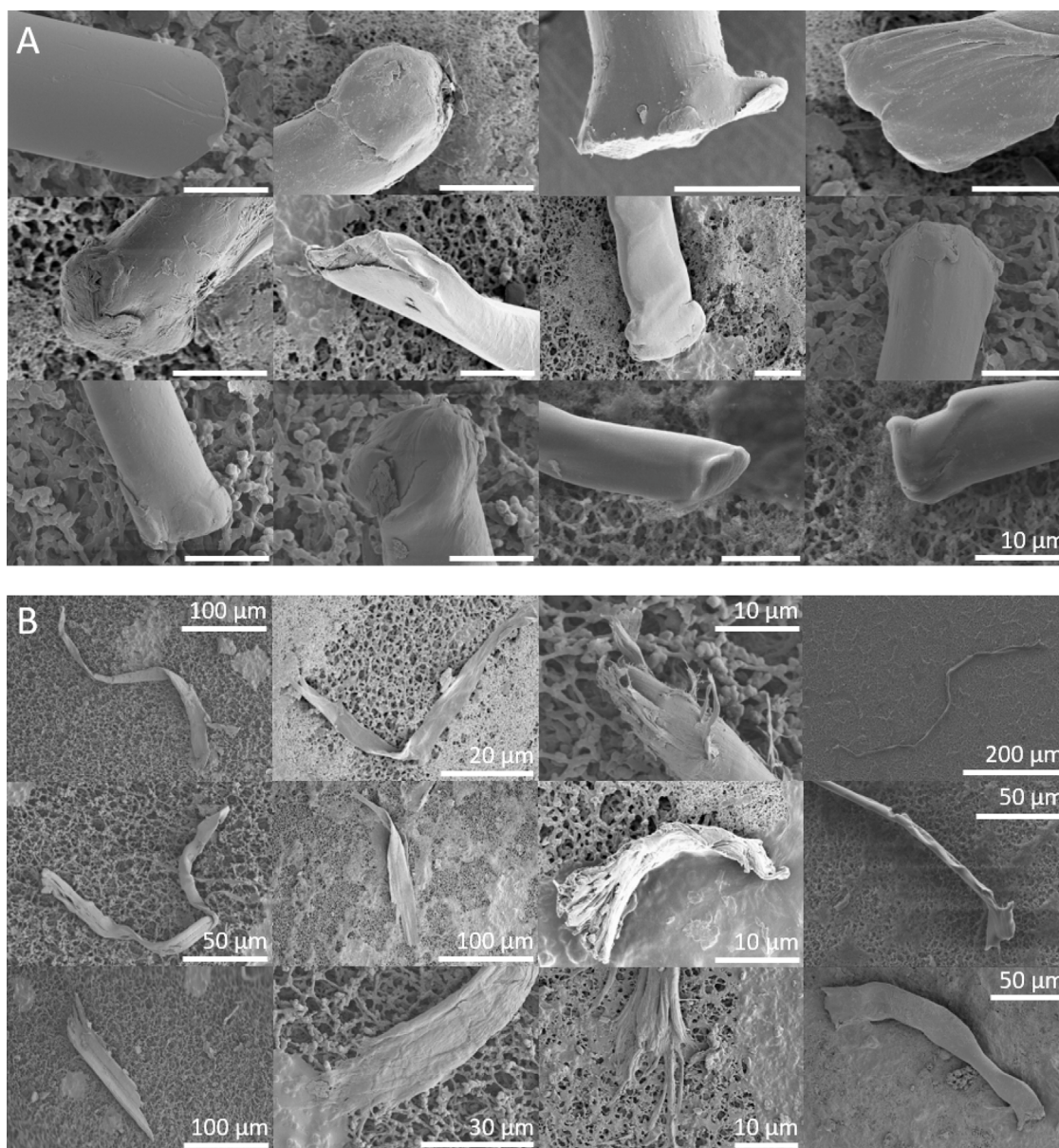


Fig. 4. SEM pictures of details of the extracted fibers. Figure A shows the typical round fiber ends cut by a high energy force. Figure B displays a variety of other types of fragments and defects of fibers detected. Those fragments include flattened fibers, signs of fibrillation, and structural inhomogeneity.

processing part of production, ranging between 2% and 45%. Such standard deviations between replicates are common for similar washing and extraction studies (18% Cai et al., 2020b, 20% De Falco et al., 2018, 29% Cai et al., 2020a, 36% Hernandez et al., 2017).

3.2. Imaging of yarns and microplastic fibers

SEM pictures of the different types of yarns were obtained to get an insight into the structural differences between them (Fig. 2). The direct structural differences from different spinning methods described in the introduction cannot be observed such as the core made of parallel fibers in air-jet yarns. However, differences in hairiness can be observed. Hairiness describes the phenomenon of protruding fiber segments from the body of a yarn (Tyagi, 2010). Based on the visual evaluation, the ring yarn shows a higher level of hairiness compared to the other yarns. This finding was expected based on the literature about the production methods (El Mogahzy, 2009). The sampled air-jet yarn has protruding fiber loops rather than sticking out fibers ends which is again a result of the production method.

When two rotor yarns spun at two different speeds were compared, the yarn spun at higher speed appeared to be more loosely wrapped.

The yarns, as well as bale and selected slivers, were also surveyed at higher magnification (Fig. 3). The slivers appeared to be the smoothest with the least amount of imperfections or attached particles. The final fibers are not completely smooth and there are small irregularly shaped particles found on them. Most frequently these particles are flat. These odd structures were more pronounced on the surface of a ring and compact yarn compared to the other yarns, bale and slivers.

Selected filter papers were also placed under SEM to study the character of collected MPF and their ends. Most ends of the MPF suggest a history of tensile stress and high energy cuts which result in sharp edges, and mushroom like heads on the MPF (Fig. 4A) (Morton and Hearle, 2008; Hearle et al., 1998). However, other types of fragments were also observed (Fig. 4B). These fragments were also of fibrous shape but were either flat, showed signs of fibrillation, or showed irregular shapes. This variety of fiber shapes was not reported so far in previous similar washing studies with textiles (Cai et al., 2020b).

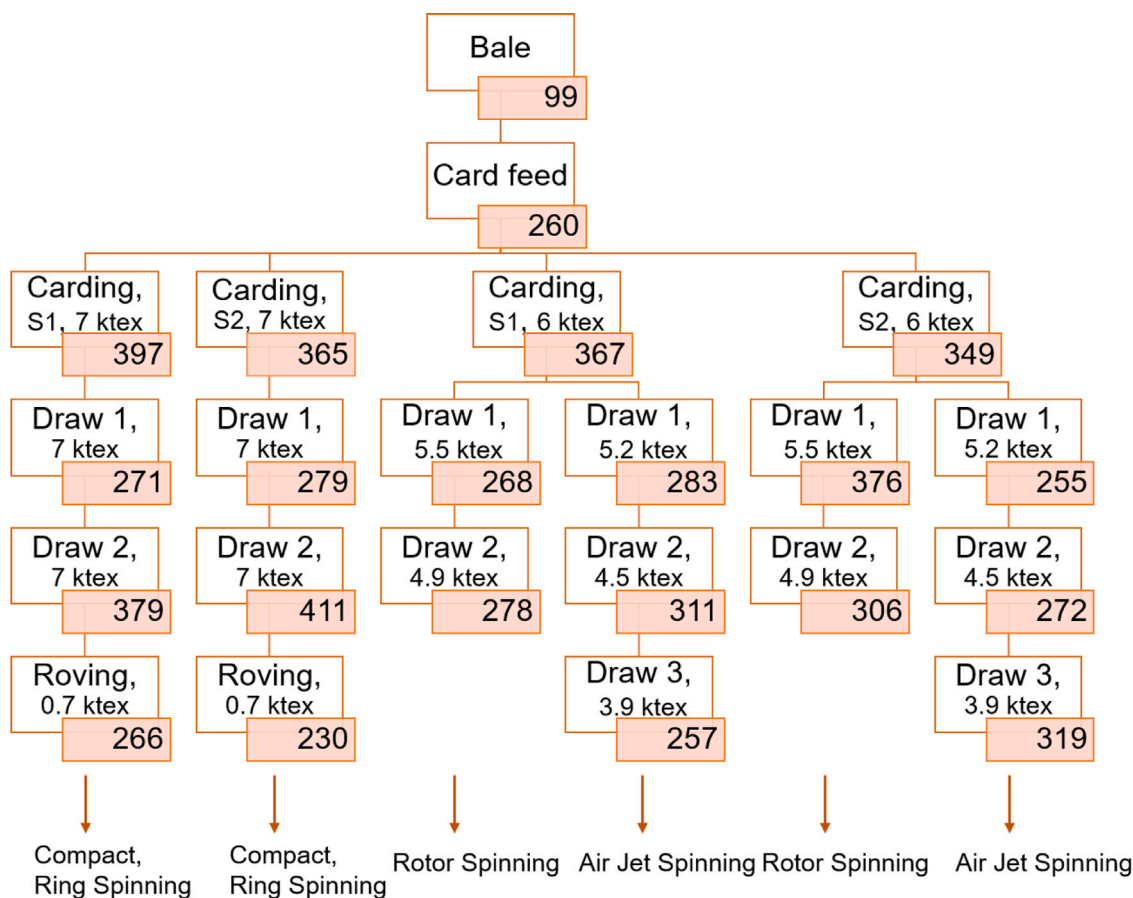


Fig. 5. Number of MPF extracted from slivers. Values are given as MPF/g of material. Numbers are the average of triplicate measurements. The complete dataset including standard deviations can be found in Table S2. Charts with median MPF length and total mass of MPF can be found in the SI as Fig. S5 and S6.

3.3. Microplastic fibers contained in slivers

MPF were found throughout the whole production line including the starting material (bale) where an average of 99 MPF/g was determined. The number of MPF in the slivers ranged between 230 and 397 per g of material (Fig. 5, Table S2). The median length of the MPF increased as the bale was going through the different production steps, from 140 μm to 349 μm on average for the final slivers (Fig. S5, Table S3). This trend can be well seen when looking at Fig. 6, displaying length distributions for each sample from bale to the sliver prepared for ring and compact spinning.

The mass of the MPF extracted was also calculated based on the number of MPF and their length. The values ranged between 20 mg/kg and 114 mg/kg (Fig. S6, Table S4). The results for specific samples correlate with the differences in the number of MPF and their length, meaning that bale contributed the smallest mass of MPF. The complete set of results can be found in the SI.

Carding speed and linear density of the produced sliver seemed to have no impact on the MPF extraction as there was no statistically significant difference (p-value = 0.937) between their length distributions, nor their quantity (Fig. S7). When the MPF length distributions of the output (ready for spinning) slivers were compared, it was recognized that there was a statistically significant difference between them (p-value < 0.001). Further post hoc analysis identified that the length distributions of slivers prepared for ring and compact spinning were statistically different from those prepared for rotor and air-jet spinning.

The comparison between first slivers from carding and output slivers showed a minor decrease in MPF count, on average by 89 fibers (SD = 45). For 2 of the 6 tested sliver couples, the difference was within the range of the average standard deviation between replicates (RSD = 14%).

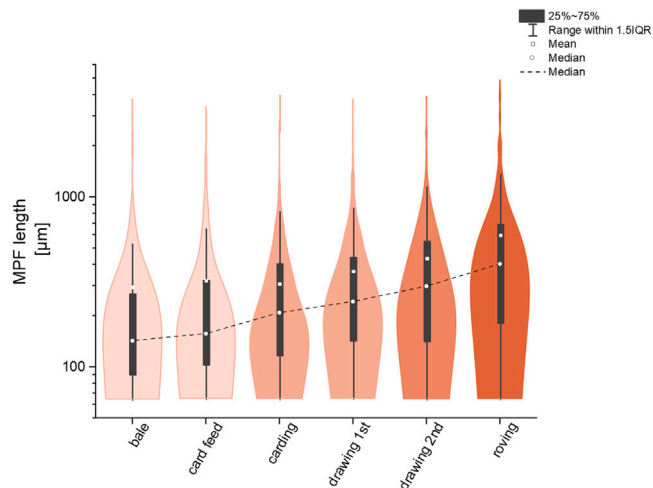


Fig. 6. Full distribution of MPF length of samples from bale to the roving fed into ring and compact spinning and the steps in between, including sliver from carding at speed 1. The width of the plots is normalized. The violin plot show steady growth in median and mean MPF length, as well as the change in distribution and reduction of the shortest fibers in the later steps. Violin plots for the other branches can be found in SI (Fig. S8). They all show the same trend of increasing median length.

3.4. Microplastic fibers contained in yarns

The results for the MPF extracted from yarns showed a large dependence on the spinning method (Fig. 7, Table S5). The lowest extraction

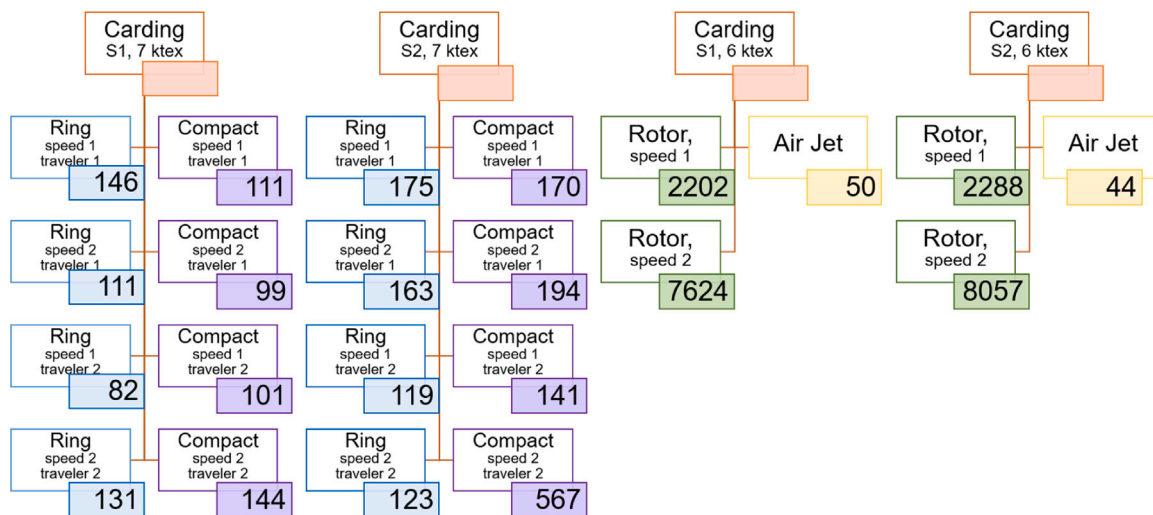


Fig. 7. MPF extracted from yarns made by different spinning methods. The values are given as MPF/g of material.

showed air-jet-spun yarns with 44–50 MPF per g of yarn. Ring- and compact-spun yarns resulted in similar numbers ranging between 82–194 MPF/g with one exception: a yarn produced from a sliver carded at higher speed and spun at higher speed with traveler 2 (CS2 S2T2) released 567 MPF/g. The extraction was repeated one more time with a new sample but the result of the 4th repeat agreed with the previous finding. The highest number of MPF was recorded with rotor-spun yarns, where 2245 MPF/g at slower spinning speed and 7840 MPF/g at higher speed were extracted on average.

Not surprisingly, it was found that there is a statistical difference between the MPF count from yarns from different methods (p -value = 0.00004). In addition, we looked at differences in MPF count between ring and compact yarns and the different settings applied during the production. No statistically significant variation was found neither for ring versus compact yarns in general (p -value = 0.311), nor for the alternative settings: carding speed (p -value = 0.109), spinning speed (p -value = 0.295), and the traveler (p -value = 0.610).

Differences between the MPF released from yarns produced with different spinning methods were not only found for the number of MPF but also their length distribution, including median fiber length (Fig. 8, S9, Table S6). For air-jet-spun yarns the median length of MPF ranged between 300–356 μm , 425–705 μm for ring and compact yarns, and 194–217 μm for rotor-spun yarns. The difference between the different types of yarns was confirmed with a statistical analysis (p -value < 0.001). Post-hoc Tukey test revealed that there was no statistical difference between the groups of compact and ring yarns (p -value = 0.5).

Unlike the number of MPF, on the first look it seems that the change in speed of rotor spinning did not affect their length as the median length for all rotor yarns are in similar range and the visual observation of a violin plot of the MPF length distributions also does not suggest any differences (Fig. S9, S11). However ANOVA analysis showed that there is a statistically significant difference between them (p -value = 0.004).

In the next step, we focused on the potential differences between different settings for ring and compact yarns. Carding speed showed no statistical difference (p -value = 0.082), nor any significant impact of changing the traveler was noticed (p -value = 0.4). Altering spinning speed showed statistically significant difference in MPF length distributions (p -value = 0.011). Further analysis showed that this difference is not relevant for ring yarns (p -value = 0.303) but only compact yarns (p -value = 0.012). The shapes of violin plot of the distributions of compact yarns evince the same, despite median length and MPF counts being of similar values (Fig.S12). The yarns spun at higher speed have a bell shape, showing an even distribution of the MPF lengths at shorter lengths and narrowing at higher values, while yarns made at

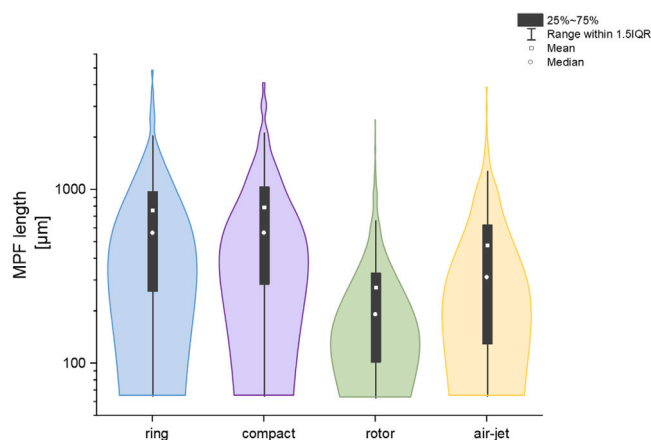


Fig. 8. Distribution of MPF length for selected yarns made by different spinning methods. The width of the plots is normalized.

lower speed show distributions with less very short MPF present in the measured samples and the MPF are most likely to be median length. The trend seems to be more visible with yarns made of slivers carded at higher speed. Violin plots of ring yarns do not suggest that the yarns would be affected by altering the spinning speed (Fig. S13).

The results were also expressed in terms of mass of the extracted MPF (Table 1). Detailed results are reported in the SI (Fig. S10, Table S7). The differences in mass of MPF from different spinning methods matched the trends in their count. Rotor-spun yarns, spun at higher speed, released on average MPF of weight 1709 mg/kg. In comparison, the weight of MPF extracted from air-jet-spun yarns was 100 times smaller, 16 mg/kg of yarn.

4. Discussion

Previous washing studies with different types of textiles or yarns had sourced their materials from industry and thus had limited control over the process steps the materials were exposed to, e.g. the type of spinning process that was used. These studies suggested that the type of spinning method may be relevant to determine the fiber number contained in the yarn. (Cai et al., 2020b; Belzagui et al., 2019). The current work thus took a very systematic approach in starting with one sample of bale and following the slivers and yarns throughout the production process. Therefore, the differences in the numbers of

Table 1

Summary of the results for yarns produced by different spinning methods. The values represent the mean value of all the yarns from the same method with the exception of compact yarns, where one yarn was excluded based on its different behavior. Rotor-spun yarns are differentiated by the spinning speed as the yarns follow different trends, while the other yarns (ring, compact) did not show significant difference between the yarns spun at different speeds.

Spinning method	Count [MPF/g yarn]	Median length [μm]	Mass [mg/kg yarn]
ring	131 \pm 30	528 \pm 66	66 \pm 21
compact	137 \pm 36	575 \pm 115	73 \pm 19
rotor, speed 1	2244 \pm 60	206 \pm 16	441 \pm 35
rotor, speed 2	7840 \pm 306	216 \pm 1	1709 \pm 28
air-jet	47 \pm 4	328 \pm 39	16 \pm 2

extracted MPF from different production steps can answer the question which steps in the yarn production are actually responsible for MPF formation and then further carried along down the production line to final textile products. From the first look at the results, the key finding of this study is how the MPF counts from rotor-spun yarns exceed the values of all the other samples. Even though many other trends can be found within the data, the scale of those variations is relatively small compared to the influence of rotor spinning on MPF formation. Nevertheless, we also analyzed those other trends found in data.

4.1. The origin of microplastic fibers in bale and slivers

We found that MPF are present in yarn production from the starting material to the final yarns. Yarn production usually starts with a bale of compacted, disorganized staple fibers. However, before the bale reaches the mill, these staple fibers have already gone through multiple production steps. Typically, the preceding steps are melt-spinning, crimping, and cutting (Lord, 2003b). In melt-spinning, PET pellets are melt-extruded from a spinneret into quenching air and drawn (stretched up to several times its original length) to produce filaments with high tensile strength (Hufenus et al., 2020). The continuous filaments are given a texture by crimping and then they are cut into staple fibers and packed into bales. This cutting step is reported by the industry to be sharp and clean (Oerlikon Neumag, 2018). Despite such claims, it can be seen as a potential source of MPF in bale and would explain the presence of MPF in the bale. Opening the bale in a blowroom and preparing the staple fibers for carding does not involve any mechanically harsh treatments, yet the number of MPF found in the chute feed doubled compared to bale. We suggest that this reflects the fact that the fibers in a bale are densely packed, including the embedded MPF, and therefore they are less easy to be extracted. In comparison, the MPF in chute feed are more readily accessible for extraction.

The results of the MPF count for slivers from carding, drawing and roving do not indicate that one of the processes would be key to MPF formation. There are only small differences between the samples but the relative standard deviation of 17% is small in consideration of the standard deviation between replicates and the overall scale of the study (tens of MPF versus thousands of MPF per g). However, the length distribution and changes in the median length prove that the machinery has an impact on the final MPF output. The steady increase in MPF median length with the number of process steps the sliver goes through, indicates that the shorter MPF are partially removed by the dust-removal mechanisms built into the machines. Based on this finding, it would be expected that the MPF count should decrease with each process step. However, the results state the opposite which leads to the hypothesis that while the shorter MPF are removed, each process also produces MPF by creating physical stress on the processed fibers. Therefore, the net change in the count of extracted MPF is close to zero.

Another strong indication about the type of process that produces the MPF is given by the nature of the MPF ends. Fatigue failure, which would be linked to the extraction cycle, would likely manifest

itself by splitting of the ends, cracks both perpendicular and along the fiber (Morton and Hearle, 2008). However, we have seen only rare examples of fibrillated parts of the fibers. Most ends of the MPF suggest a history of excessive tensile stress and high energy cuts which result in sharp edges and mushroom-like heads on the MPF. Morton and Hearle (2008) The MPF ends from washing were also analyzed in previous textile washing studies and the conclusion was reached that washing did not result in formation of new MPF based on the analysis of fiber ends (Cai et al., 2020b).

4.2. Impact of spinning on the presence of microplastic fibers

The most striking result of our investigation was the difference of MPF produced by different spinning methods, particularly rotor spinning in comparison with the other spinning systems. The difference is further highlighted by the fact, that while we see a major increase in MPF count for rotor-spun yarns compared to the slivers, all the other spinning methods result in reduction of the MPF number (with one exception). The results showed that the slivers which were used as an input material for spinning, released MPF on the same scale (276 \pm 33 MPF/g of material), regardless of the difference in treatments (carding speed, different number of drafting steps, an extra roving procedure in case of slivers prepared for ring and compact spinning). Therefore, the differences in the spun yarns are the result of the different spinning methods.

Ring and compact spinning are used to produce yarns of higher quality, whereas rotor spinning focuses on production quantity. It can be anticipated that during the development of rotor spinning less focus was placed on the removal of impurities, including MPF. The removal of fine fibrous dust is not only adding further complexity to the machine but including short fibers in the final yarn increases its weight and potentially also profit. The opening roller speed has a major impact on the MPF formation as we saw an almost 4 fold increase in the numbers when the speed was increased by approx. 25%. At the same time, the fiber length distribution, including the median MPF length, was affected only minimally. Fiber rupture during rotor spinning has been previously described, including the impact of raising the speed of the opening roller (Salhotra and Chattopadhyay, 1982). It is possible that the increased speed of the opening roller causes more ruptures of the fibers and produces fiber fragments on the microplastics size scale. Concurrently, the visual comparison of the rotor yarns under SEM indicated that the yarns produced at higher speed seemed to be more disorganized and looser (Fig. 2) which could allow an easier release of the embedded MPF compared to the tighter structure of the yarn spun at lower ORS. It is also possible that a combination of these two factors is behind the major difference between the MPF numbers in yarns produced with different ORS.

Considering that the opening roller system is specific for rotor spinning, it can explain why the other yarn types do not report MPF counts as high as rotor-spun yarns. Nevertheless, the high release in rotor yarns may be also facilitated by the shorter length of MPF compared to the other yarns. Short fibers are less likely to be entangled in the yarn structure and therefore can be more readily extracted.

Air-jet spinning showed the lowest releases of MPF despite also being a high throughput method. The results again can be a consequence of the process mechanism, or the different structure of the yarn, or their combination. Air-jet spinning is unique by introducing a false twist, meaning that most fibers in the core are parallel and only a small percentage is twisted around the core fibers. This alignment could hinder the MPF in the core section from being extracted. In addition, air-jet yarns are less hairy than other yarns and unlike the other yarns, the fibers sticking out rather create loops with the ends hidden in the yarns than having loose ends. In case the hairiness was related to MPF extraction, the air-jet yarns lack of fuzziness and the sticking loops suggest a higher degree of entanglement, which would explain the low extraction numbers. Positive correlation between yarn hairiness and release of

MPF was observed by Özkan for hairiness higher than 4 mm (Özkan and Gündođdu, 2020). Another study also correlated hairiness together with easiness to form fuzz and MPF extraction (Zambrano et al., 2019). However, the hairiness was compared between different types of fabrics (cotton, rayon, and PET) and the study discussed potential mechanisms of MPF production during washing, also taking the wear phase into account, and did not test the hypothesis that most fibers were already embedded in the textiles. Another indicator suggesting that the loose fibers may have an impact on MPF extraction is the fact that in textile washing studies the major source of MPF are edges of fabrics where loose fibers are also considered to play a key role (Cai et al., 2020b).

Ring and compact spinning are similar methods as compact spinning is altered ring spinning with the aim of producing more organized yarns. As the yarns were exposed to similar spinning mechanism, it was expected that the results for corresponding yarns would be on the same scale and in most instances they were. However, some differences were observed. Compact spinning is affected by spinning speed unlike ring spun yarns (Fig. S12, S13). With increasing spinning speed, more shorter MPF are produced, though the median MPF length barely changed. On top, one of the compact fibers made at higher speed did not match the numbers of the other compact and ring yarns, and released 4 times more MPF whose median length was 5 times shorter. This implies that the higher speeds of carding and spinning, and traveler 2 apply more stress on the fibers, however the changes are very small and therefore only when joined, they reach a threshold resulting in a change in pattern of MPF formation. There is no comparison available for the yarns but regarding studies on textiles, it was shown that the more stress is applied on textiles, be it mechanical or chemical, the higher is the number of MPF released (Ramasamy and Subramanian, 2021).

Only one indirect comparison with another study can be made as most washing studies look at textiles and not slivers and yarns. Cai et al. (2020a) included different yarns and a sliver in their study but a different extraction method was used and therefore only the ratios and fiber length distribution can be compared. In addition, the samples were from different producers and the production history was not known. In their study the MPF extracted from sliver had a median length of 405 μm which would correspond to some of the more processed slivers in our study. They also found that rotor yarns shed shorter MPF than other methods, 226 μm compared to other yarns, which ranged between 300–500 μm . Our results match those value ranges (Table 1). The MPF quantity assessment showed that the ratios between different yarns do not coincide but the ranking of yarns from least shedding to most shedding is the same. In addition, in both studies the difference between rotor yarns and other yarns is well pronounced. Specifically, we observed 47 times more MPF released from rotor yarns compared to air-jet yarns, while Cai et al. noted 35 times more MPF for the same pair (Cai et al., 2020a).

The length measurements of MPF reported in textile washing studies are difficult to compare with our results because different studies counted over various ranges of MPF length and because of inconsistencies of reporting median or average MPF length. Nevertheless, the reported values start at 400 μm and reach more than 1 mm length (Vassilenko et al., 2021; Özkan and Gündođdu, 2020; De Falco et al., 2020; Cai et al., 2020a,b). We can however compare the results with previous work done in our lab under very similar conditions by looking specifically at laser-cut textiles from staple fibers: the median average length of the MPF extracted from textiles was 428 μm and the MPF count ranged between 376 and 1222 MPF/g of textile (Cai et al., 2020b). The yarn type was not reported, which is why we can only speculate about the differences in the numbers. Based on the MPF length comparison, we can suggest that most textiles in the study by Cai et al. were from ring-spun yarns and that knitting and weaving produced further MPF in the textiles. It is also possible that the 3D structure and bending of the yarns in the textile allows for easier extraction. However, it is also feasible that the short MPF in rotor

yarns are removed during further processing and their count drops and primarily the long MPF stay embedded in the textiles. Further research is necessary to fully understand the importance of producing textiles from different types of yarns regarding the MPF formation and release.

The difference in length is an important factor when the fiber count is translated to mass of the MPF. However, the results show that despite the short nature of MPF from ring spinning compared to the other spinning systems, the comparison of mass of MPF in yarns still makes rotor-spun yarns stand out above the others (Table 1). In addition, the shorter fibers from rotor-spun yarns might be harder to retain by washing machine filters and waste water treatment plants than the longer fibers from other yarns.

4.3. Impact of yarn structure on the presence of microplastic fibers

Based on our results, we are currently not able to definitely conclude which differences in the extracted MPF are a result of the level of stress produced on the fibers by the different machineries and which differences could be caused by the different yarn and sample structures. For example, the variation between the compact yarns seems to be a result of the increase in stress by increasing the spinning speed. On the other hand, the general decrease of the MPF count from slivers to yarns (with the exception of rotor yarns) could be explained by the fact that the fibers are twisted into a tighter structure and are less available for extraction. The same reasoning could be applied to the minor decrease between initial (carding) slivers and the output slivers as the slivers are more organized as they progress along the production line. The difficulty in assessing the extent of the impact of the structure of the yarn (or sliver) hinders making clear conclusions on the formation of MPF in yarn production.

SEM pictures of the tips of the MPF show high-energy cuts, similar to those reported in the preceding textile study (Cai et al., 2020b). However, we see the same tips from bale samples to yarn samples, so it is not possible to determine which MPF were produced during cutting of filaments into staple fibers and which were produced during yarn production. Intriguingly, a variety of other fragments and fibers was also found on the sample filters which was not previously reported (Fig. 4B). The published washing studies which reported details about the shape of the released fibers only identified fibers with the diameter of the original fiber. In particular Cai et al. performed a very careful analysis of the fiber ends and diameters and concluded that no fiber breakage or splitting occurred during washing (Cai et al., 2020b). However, fibrillation of PET fibers was observed during abrasion and fibrils with a diameter of 2–5 μm and a length of 30–150 μm were formed and could be released during washing (Cai et al., 2021). The fibrils observed by Cai et al. resemble some of the fiber fragments shown in Fig. 4B. This indicates that to some degree fibrillation of fibers can occur during manufacturing although Cai et al. only observed very few fibrils in unabraded textile samples. Fibrillation as well as other odd shapes of some fibers may be another consequence of the stresses from production aside from complete fiber breakage. Cai et al. have stated that the absence of fibril observation in previous studies may be due to the analytical techniques used in previous studies which were not able to detect the much smaller fibrils. We confirm that the anomalies were found under SEM only at high magnification. Recently it was also shown that PET fragments smaller than 1 μm , so-called nanoplastics (Mitrano et al., 2021), were released from textiles during washing (Yang et al., 2021). This work also analyzed for fibrils and also found them just in abraded textiles and not the pristine ones (albeit after prewashing before abrasion to remove any debris). Nanoplastics may also be related to the structural impurities on the fiber surfaces as seen in Fig. 3.

Another question is how the results of MPF release from unprocessed yarns translate when they are woven or knitted into fabrics. We have already discussed the differences between the releases from textile studies and our study. However, a systematic continuation of this study is needed in which fabrics made with different types of yarn are made to fill in the knowledge gap.

5. Conclusions

In this study we have followed up on the research of textile washing and MPF extraction with the aim to further expose the origin of MPF in textiles. Working with the hypothesis that MPF are primarily generated during production, we directed our attention to yarn production, a second step in the textile manufacturing process. MPF were found in every sample studied, from initial bale to all types of yarns, confirming the hypothesis. The fact that MPF were also in the bale suggests that some MPF are already introduced into the chain during staple fiber production. Nevertheless, that does not mean that no more MPF are formed during yarn production. Changes in the MPF counts as well as their fiber lengths suggest that more MPF are generated during yarn production, while some, especially the shorter MPF, are efficiently removed.

The most striking finding of the study was, how different the results of rotor-spun yarns were compared to other yarns, as well as to slivers. While with most samples the MPF numbers observed were on the scale of tens or hundreds of MPF per gram, rotor yarns released thousands of MPF per gram. It is of a question to what extent these results are determined by the different spinning mechanisms or by the structural differences of the yarns as the results of this study provide an insight into the MPF counts and their characteristics but cannot directly unravel their formation mechanism.

Nevertheless, we would like to advocate for favoring ring spinning over rotor spinning, or to start using air-jet spinning also with pure polyester yarns, if a low MPF release is targeted. In case rotor spinning cannot be avoided, the opening roller speed should be set to low speed to minimize the MPF counts in the yarns. The results may help the industry to take pro-active steps to reduce the impact of the textile industry on the environment.

CRedit authorship contribution statement

Barbora Pinlova: Conceptualization, Formal analysis, Investigation, Visualization, Writing – original draft. **Rudolf Hufenus:** Conceptualization, Writing – review & editing, Funding acquisition, Supervision. **Bernd Nowack:** Conceptualization, Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

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

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Appendix A. Supplementary data

Supplementary material related to this article can be found online at <https://doi.org/10.1016/j.jclepro.2022.132247>.

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