Characterization of fiber fragments released from polyester textiles during UV weathering ☆

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ARTICLE INFO

ABSTRACT

Synthetic textiles are considered a prime source of microplastics fibers which are a prevalent shape of microplastic pollution. Whilst the release mechanisms and formation of such microplastic fibers have been so far mainly studied in connection with laundry washing, there are some studies emerging that describe also other release pathways for microplastic fibers such as abrasion during wearing. The aim of this study was to consider weathering as another process contributing to the formation of microplastic fibers and their presence in the environment. Four types of polyester fabrics were selected and exposed to artificial weathering by UV-light for two months. The fabrics were extracted every 15 days to quantify and characterize the formed microplastics. Microplastic fibers with the diameter matching the size of the fibers in the textiles were observed. However, additional microplastic fibers of different shapes were also formed. These included partially broken fibers, thin fibers with a diameter below the size of the fiber in the fabrics, fibers flattened into a ribbon, and non-fibrous microplastics. The released microplastics evinced physical alterations on their surface in the form of pits and cracks. The released microplastics exhibited a steep increase in number with progressing weathering; from hundreds of fibers per gram of textile from unaged fabrics, to hundred thousands fibers (150,000-450,000 MPF/g) after 2 months of weathering. Additional 10,000-52,000 unifbrous microplastics/g were released from the weathered fabrics. While plain fabrics showed higher releases than interlock and fleece, further research is needed to evaluate the importance of the textile architecture on the weathering process in comparison with the production history of the fabrics. Based on a comparison with washing studies with the same textiles, we can estimate that the potential of weathered fabrics to be a source of microplastic fibers can be 20-40 times larger than washing only.

1. Introduction

Microplastics (MP), plastic particles smaller than 5 mm, have been identified as a pollutant present in all environmental compartments (Browne et al., 2011; Koelmans et al., 2019; Zhou et al., 2020). Textiles have been identified as a major source of microplastic fibers (MPF) (Browne et al., 2011). The best known release mechanism of MPF from textiles is from washing. It has been shown that MPF in textiles are not formed during washing but produced during the production process and are only later released during washing (Cai et al., 2020a; Pinlova et al., 2022). Despite the fact that most studies have so far mainly focused on investigating MPF during washing, other release and formation pathways have been uncovered. A study with volunteers wearing synthetic garments suggested that the release to air during wearing is on the same scale as the release of MPF into water during washing (De Falco et al., 2020). The presence of microplastic fibers and smaller fibrils was reported in a textile abrasion study (Cai et al., 2021). These recent studies show that it is not only the well-known release of MPF during washing that is relevant for environmental exposure of particles released from textiles.

Synthetic textiles which end up in the open air as a result of littering and waste mismanagement or because they are used outdoors are exposed to weathering processes, leading to ageing and degradation of the polymer. Worldwide 87% of the textile waste ends up in landfills (Hermann et al., 2017). In Europe 15-20% of the textiles are collected and about half of them are then exported to developing countries (Sandin

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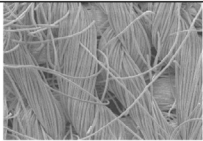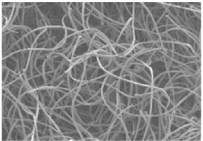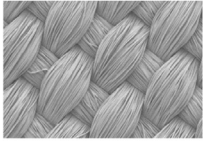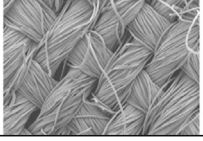
Table 1

Overview of the fabrics used in the study. Values of the fabric density and the fiber diameter were taken from a previous study (Cai et al., 2020a).

fabric	SEM	structure	yarn	surface processing	density [g/m^2]	fiber diameter [μm]
interlock		knit	spun	–	209	12.2 ± 0.8
fleece		knit	filament	✓	185	11.7 ± 1.3
plain filament		woven	filament	–	149	7.5 ± 0.6/7.9 ± 0.5
plain staple		woven	spun	–	100	12.7 ± 0.5/13.4 ± 0.9

and Peters, 2018). Such textiles are sent with the purpose of reusing and recycling but many of them are mismanaged and may end up in open dumps (Notten Philippa, 2020). An example of such mismanagement of textile waste can be found in Chile where a large textile landfill has emerged in the Atacama desert (Espinoza Pérez et al., 2022). The possible formation of fiber fragments during exposure of fabrics to environmental conditions is therefore a process that should be studied.

A prominent degradation pathway of polymers in the environment is photodegradation - a combination of photolysis and oxidative reactions (Gijsman et al., 1999; Booth et al., 2018). Other degradation pathways include hydrolysis, biodegradation, thermal degradation, mechanical or chemical stress induced degradation (Gewert et al., 2015). The single degradation pathways have been studied since the 1960s but their combination during weathering increases the complexity of the process due to a large number of factors involved (Chamas et al., 2020). Moreover, the focus of the degradation studies was primarily on assessing durability and the degradation pathways including the degradation products (Day and Wiles, 1971; Yakimets et al., 2004; Singh and Sharma, 2008). The increased brittleness of weathered plastics was not linked to a breakdown into microplastics in these studies. Fragmentation of plastics is a reverberation of the changes in the material properties due to weathering, often triggered by a mechanical force. Prior to fragmentation, cracks and other defects develop on the surface. These have been frequently documented on MP, including MPF, in field studies (Sathish et al., 2019) as well as in laboratory settings (Bai et al., 2022; Sørensen et al., 2021). Similarly to degradation, describing and modeling the fragmentation process is still in development (Andrady, 2017) and further understanding is needed, including the characterization of the fragments.

Despite the high abundance of MPF in the environment and the concerns regarding the impacts of weathering on MP properties (Liu et al., 2020; Wang et al., 2021b), the number of studies investigating weathering of synthetic textiles and the link to microplastic formation is so far limited. Nguyen et al. investigated nanoscale changes upon photodegradation of PET fibers (Nguyen-Tri and Prud'homme, 2019). Bai et al. studied the impact of artificial UV exposure for 35 days on PET geotextiles in seawater, reporting on chemical and structural changes, as

well as a number of small released particles (Bai et al., 2022). Sørensen studied the impact of UV degradation on synthetic (PET, PA) and wool yarns in seawater, primarily focusing on the analysis of leachates containing degradation products and additives (Sørensen et al., 2021). A similar experiment was performed comparing degradation in fresh water and seawater (Sait et al., 2021). With the COVID-19 pandemic, the focus has also been directed to disposable facemasks. Wang et al. studied the impact of UV and mechanical exposure on polypropylene face masks, reporting on morphological changes to the fibers in the mask already after 18 hours of weathering and fractured fiber fragments after 36 h of artificial weathering. (Wang et al., 2021a).

The studies presented above show that some information is available about the behavior of textiles during weathering but no detailed study has so far been performed to characterize in detail the released fiber fragments. The objective of the current study was therefore to push forward our understanding of weathering of polyester textiles exposed in air with a focus on MP quantification and to assess how relevant as a source of microplastics weathered textiles could be. Four different polyester fabric structures were exposed to UV radiation for two months. Every 15 days the fabrics were washed, combining application of mechanical stress on the textiles and extraction of the formed MPF. Investigating different fabrics allowed for assessing to which extent the results were fabric specific and what conclusions can be applied to PET fabrics in general. In addition, the results were not only compared within the frame of this study but the differences from MPF known from washing studies were described.

2. Method

2.1. Sample preparation

Four types of black polyester fabrics were selected for the study (Table 1): interlock, fleece, plain staple and plain filament. The black fabrics were chosen as the same fabrics were used in previous MPF-release studies by washing and abrasion which allowed for a direct comparison (Cai et al., 2020a,b, 2021). The subset of fabrics was selected to represent fabrics from different yarns (staple versus filament),

with different architecture (knit versus woven) and surface treatment (processed versus unprocessed).

The fabrics were cut with a laser-cutter (tt-1300, Times technology) into six 4 x 10 cm pieces from each fabric type. The size was chosen according to an ISO standard test for color fastening (ISO 105-C06, 2010) The weight of the samples depended on the fabric type with plain staple being the lightest (0.39 ± 0.01 g) and interlock the heaviest (0.88 ± 0.01 g).

The samples were prewashed three times to remove most of the MPF already contained in the textiles. A previous study showed that most MPF are released within the first few washes and after the third wash, the number of released MPF during repeated washing stabilized at a very low level Cai et al. (2020b). The prewash was done in a Gyrowash lab washing machine (James Heal, GyroWash, model 1615) at 40 °C for 45 min based on ISO standard 105-C06 and previous washing studies (Hernandez et al., 2017; Cai et al., 2020b). 150 mL of linear alkylbenzene sulfonic acid (LAS) solution (0.75 g/L, pH 9.2 ± 0.1) was used as a washing liquid. 10 steel balls (diameter = 6 mm) were placed in the vessels during the washing.

2.2. Ageing and extraction

Prewashed triplicates of the different fabrics were placed into the weathering chamber (Q-SUN XE-1, QLab). The samples were locked in place with ferrite magnets (7 x 7 mm) from top and neodymium, nickel plated magnets from the bottom of the sample shelf. The top magnets were carefully placed to minimize the covered area on the fabrics (Fig. S1). The weathering was performed with Daylight-Q filter to mimic the wavelength range and profile of natural light. The settings were chosen based on ISO 4892-2 for UV testing of plastic materials with xenon-arc exposure but altered to fit the goals of the study. The irradiance was kept at 60 W\m^2 (total UV), the temperature at the black panel was decreased to 55 °C, the lowest temperature at which the weathering chamber operates smoothly under the selected settings. The samples were left in the chamber for 15 days of UV exposure and then taken out to extract the MP(F) from the fabrics. The extraction was done by washing the samples, applying the same procedure as during the prewashing step. After the washing, the fabrics were left to drip and then placed in a centrifuge tube with a holder for the fabric (Fig. S2) and centrifuged at 550 xg for 5 min (Eppendorf 5810R, Eppendorf Germany). The accumulated liquid at the bottom of the centrifuge tube was added to the washing jars containing the washing liquid. The holder was made by cutting off the filter part from centrifugal filter (100 kDa Amicon Ultra-15 centrifugal filters, Millipore, Ireland). The spin-dried fabric was then placed back in the weathering chamber. The ageing and washing step was repeated in total four times after 15, 30, 45, and 60 days of weathering. The estimated time simulated by accelerated weathering was calculated and is given in the SI. The same procedure was followed for a matching control set of fabrics kept in a dark cabinet packed in an aluminium foil.

2.3. SEM analysis

The solutions with the washing liquid were filtered with a vacuum filtration system and the MPF were deposited on a cellulose acetate membrane (13 mm diameter, 0.45 μm pore size; Sartorius GmbH). The filtered volume varied based on the MPF concentration in the solution between 0.2 and 10 mL. The samples were sputtered with a Au/Pd layer (7 nm, LEICA EM ACE600). The whole area of the filter was captured with SEM (7 kV, Quanta FEI 650) with the Maps software (Thermo Fisher Scientific). The mapping was done at 200 magnification, 1536 x 1024 pixel resolution, with 3 μs dwell time, and 1 frame. A total of 77 images were stitched together to yield a final image of the whole filter with an approximate size of 9700 x 10100 pixels (Fig. S3). Additional SEM pictures were taken at higher magnification to obtain detailed information about the structure of randomly selected MPFs. Furthermore,

SEM pictures were taken of the fabrics prior and after weathering with SEM (8 nm Au/Pd layer, Hitachi S6200).

2.4. Image analysis

The merged images of the filters were analyzed in ImageJ. The MPF on the filters were labeled manually by tracing them with a Microsoft Surface pen. The top threshold for MP(F) length was set at 5 mm, the lower at 10 μm. The filter was analyzed multiple times to count separately the different types of MPF forms that were found in the samples. Due to the challenge of the high time demand for this step, reproducibility of the results was tested on one sample (Table S2). Based on those results, we decided to perform the count analysis only on duplicates and the complete analysis for triplicates was performed only for selected samples.

2.5. Microplastic categorization

The materials released from the fabrics were placed in five categories (Fig. 1). The first category represents MPF which have the standard appearance of a polyester fiber (round and smooth) and match the visual aspects of the fibers from which the studied textile are made. They were also previously described in textile washing studies (Cai et al., 2020b) and were labeled as "regular" MPF. The diameters of the "regular MPF", determined as an average of 8-12 randomly selected fibers (Table S3), fell within the range of diameters of the fibers in the fabrics (Table 1). The second category covers fibers which are about to brake into two or more pieces but the process has not been completed. Consequently, they are labeled as "split MPF". The next category of "thin MPF" encompasses MPF which have smaller diameter than the original fibers. The fourth category represents fibers which appear flat and therefore are named "ribbons". The last category includes particles which were not of a fibrous shape and were labeled "odd MP". For simplification, "thin" and "ribbon" MPF were in most instances grouped together as "other MPF".

2.6. Mass calculation

The information about the size (diameter and length) of all the tagged MP(F) was used to estimate their mass. The value of 1.38 g/cm^3 was used in the calculations for polyester density. The calculations varied for some of the MP(F) categories due to their specific shape characteristics. The mass of "thin" fibers was calculated as the worst case scenario weight, using the same diameter as for regular fibers. The mass of "ribbons" was also calculated the same way as they seem to be unfolded regular fibers. Considering that the mass of "ribbon" and "thin MPF" was determined using the same formula, it was possible to do the calculations on the supercategory of "other MPF". The mass of "odd MP" was calculated by considering them to be of a cylindrical shape with height half of the fiber diameter. The "split MPF" were omitted due to the fact that their length was not measured.

2.7. FT-IR

Attenuated Total Fourier Transform Infrared (AT-FTIR) spectra were recorded with a Varian 640-IR FTIR (Agilent Technologies, Santa Clara, CA, USA) to observe the chemical composition of the weathered fabrics and dark controls. The spectra were recorded for every fabric type from 4000-600 cm^{-1} as the average of 32 scans, with a spectral resolution of 4 cm^{-1}. The carbonyl index was calculated as the ratio between the absorbance of carbonyl group at 1712 cm^{-1} and an unalterable band (721 cm^{-1}) was selected.

2.8. Contamination evaluation

Basic measures were taken to reduce potential sample contamination. Lab surfaces and utensils were cleaned before work, nitrile gloves
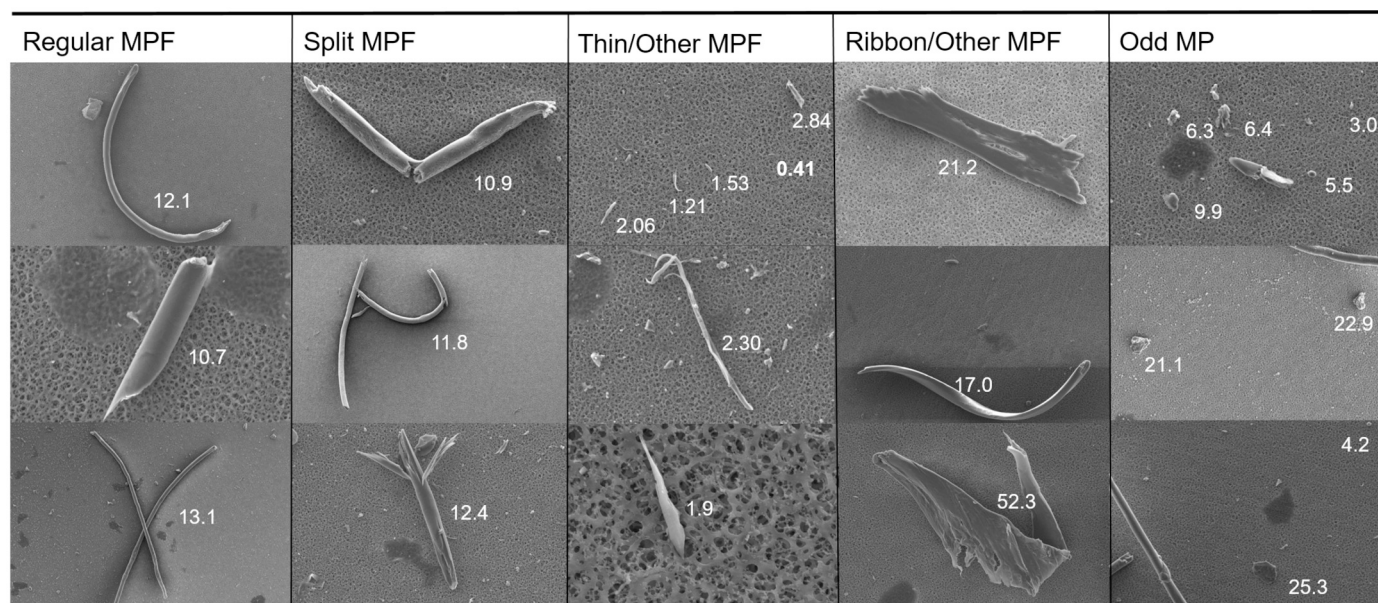
Fig. 1. Characterization of the different MP(F) types released from the studied samples. Selected examples with their diameters in μm are shown for each category. In case of "odd MP", the widest part of the particle was measured.

were worn, the lab coat was daily cleaned with a sticky lint roller, and high-release clothes were avoided to be worn underneath. The sample shelf was also wiped between the cycles. The weathering chamber was maintained based on the user guide. During the washing step, one jar was used to perform a blank method test, resulting in having one blank for two sets of triplicates. These blanks followed the same procedure of centrifugation and filtration as the tested samples.

Cross-contamination in the weathering chamber was tested after the main experiments were performed. In the first step, the weathering chamber was left running for 15 days. Inside were placed three laser-cut pieces of rinsed glass-fiber fabric (plain warp, 163 g/m², Vamliera Glass) and a piece of tape was placed on the venting grille at the back of the weathering chamber. After 15 days the glass fabrics were taken out without stopping the weathering program and a new set of glass fabrics was placed inside. Then the weathering was stopped which triggered the ventilator in the chamber to blow on maximum to cool it down. After 30 min, the fabrics were taken out and the tape from the back was removed. In the next step, one 4 x 10 cm piece of fleece fabric was placed in the chamber and fiber glass fabrics were placed around it at varying distances. A new tape was placed on the outside grille. The chamber was turned on for 15 days. Prior to turning off the chamber, the glass fabrics were taken out and replaced with a new set of fabrics and two-sided tapes, both attached to glass slides. After that the program was stopped and after 30 min of cooling down, everything was removed from the chamber. The glass fabrics and the tapes were then analyzed under SEM.

3. Results

### 3.1. Quality assurance and control

The average MPF count of four blanks was 86 (RSD = 63%) with a maximum value at 165 per 150 mL of the washing liquid used in the blank. Two of those four blanks were also analyzed for the other types of MP(F): there were 510 ± 45 "other MPF", 743 ± 38 "odd MP" and no "split MPF". In total the blanks contained on average 578 ± 37 MPF and 1320 MP per blank. In comparison, the values for the MPF found in the weathered samples ranged between 3,300 and 500,000 MPF/g textile and 3,500-570,000 MP/g. The contamination did not substantially affect the results of the weathering experiments as it represented only a small percentage of the measured values.

The washing and filtering procedure was done based on previous MPF washing studies, which included proofing of the reproducibility of the protocol (Hernandez et al., 2017; Cai et al., 2020b). Additional tests were run to confirm the reproducibility as very small volumes were filtered compared to the previous studies. For details see the SI. It was concluded that despite relatively high standard deviations of 40-60%, the overall trends and messages of the study were not impacted as the variation between the numbers between different cycles was greater than the standard deviation range.

Testing of potential cross-contamination within the weathering chamber was done to exclude the potential that fibers released from one piece of fabric contaminated the other fabrics inside the chamber and to quantify the background fiber contamination. Fiber glass fabric was selected for the testing due to its resistance to UV degradation compared to PET. "Regular MPF" were found only on double-sided tapes which were placed in the chamber after a fleece fabric was weathered and the ventilator was turned on to cool down the chamber. In total only two regular fibers were found on two tapes placed in a close proximity of the fleece fabric. It must be noted that the ventilator was never activated during the main phase of testing. Very few flat fibers and flakes were found on some of the tested surfaces during all phases of the contamination testing, including the tape on the grille, glass fabrics and the double-sided tape (Fig. S4). The flat fibers appear to be of natural origin (thin, twisted, or textured). As a conclusion, the cross-contamination within the chamber was not considered to be an issue.

### 3.2. Morphology of the weathered microplastic fibers

The weathering process was clearly visible on the fabrics as the black color faded, leaving the exposed side light grey/white, while the bottom side stayed black (Fig. S1). For fleece fabric, some discoloration was also visible on the bottom side (Fig. S5). The fabrics were observed under SEM and compared to control samples (Fig. 2A). The exposed side of the weathered fabrics showed clear signs of structural damage with many fibers being broken off. Due to the organized structure of plain filament fabric, it showcases well how the top layer of fibers was largely broken and missing, while a new layer of fibers beneath it was exposed. On the other hand, the unstructured fleece did not show pronounced changes. Upon further examination at higher magnification, specific structural damages were visible (Fig. 2B). The fibers were covered with indentations, pits and cracks, mainly perpendicular to the fiber and some of the
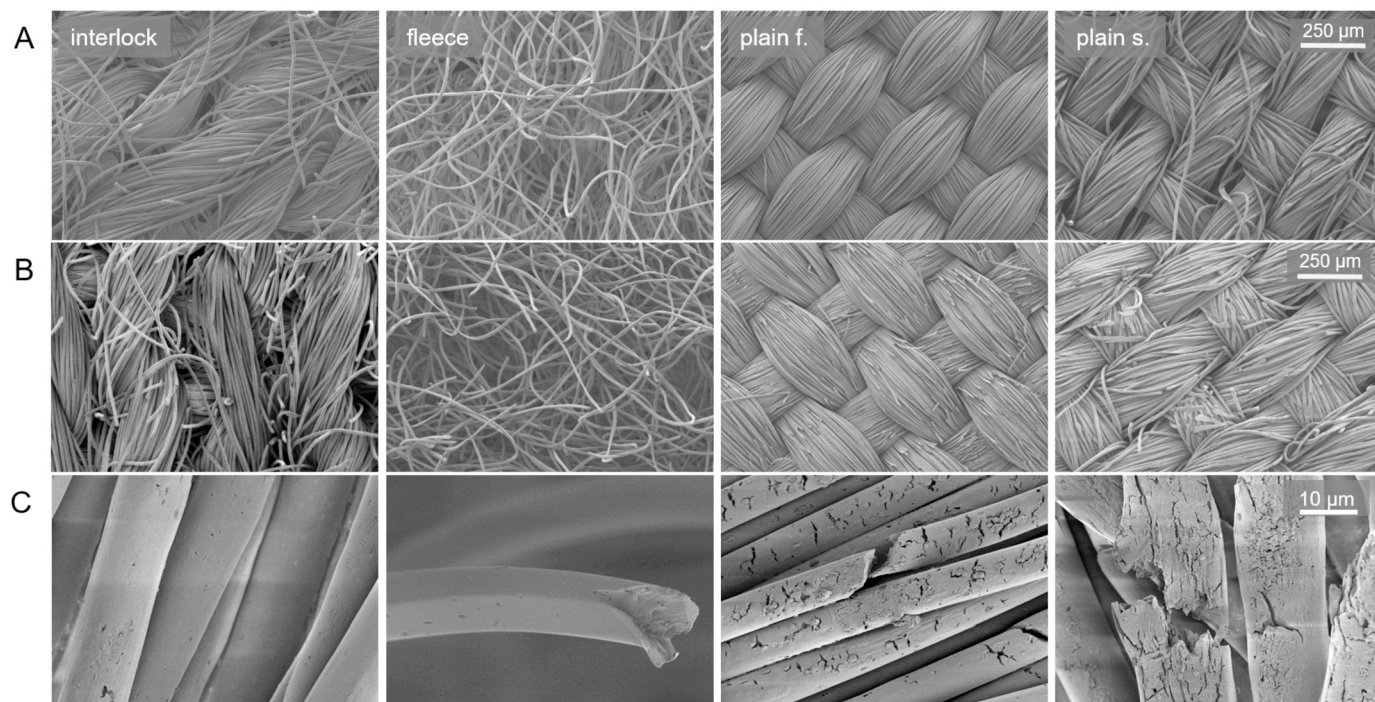
Fig. 2. Impact of UV exposure on the fiber structure. A: fabrics before weathering, B: fabrics after 60 days of artificial UV exposure, C: details of the weathered fabrics. Additional images of singular weathered fibers are displayed in Fig. S8.
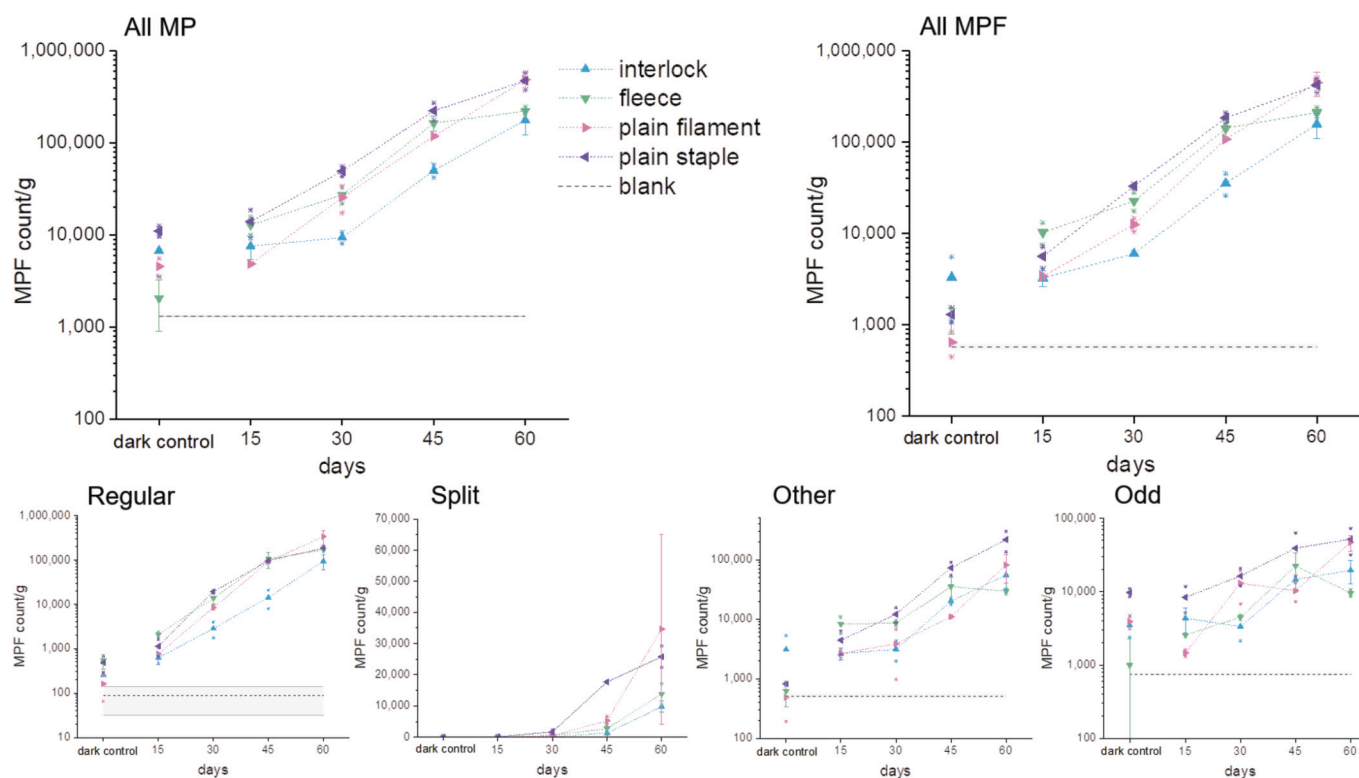
Fig. 3. Number of MP(F)/g textile sequentially extracted from the progressively weathered fabrics every 15 days. The results are shown for the different MP(F) categories, including the combined results for all MPF (regular, split, and other MPF) and all MP (all MPF and "odd MP"). Results from dark controls are used as the first data point. The MPF counts represent the MPF extracted at the specific time point and are not cumulative. All graphs are shown on logarithmic scale with the exception of "split MPF" which were not present in all samples. Error bars are shown for data points which were calculated as an average of analysis of triplicates. For samples, where only duplicates were analyzed, those two specific values are shown as asterisks. The blanks values are also represented in the graphs. The numerical values can be found in Tables S5-S9. (For interpretation of the colors in the figure(s), the reader is referred to the web version of this article.)
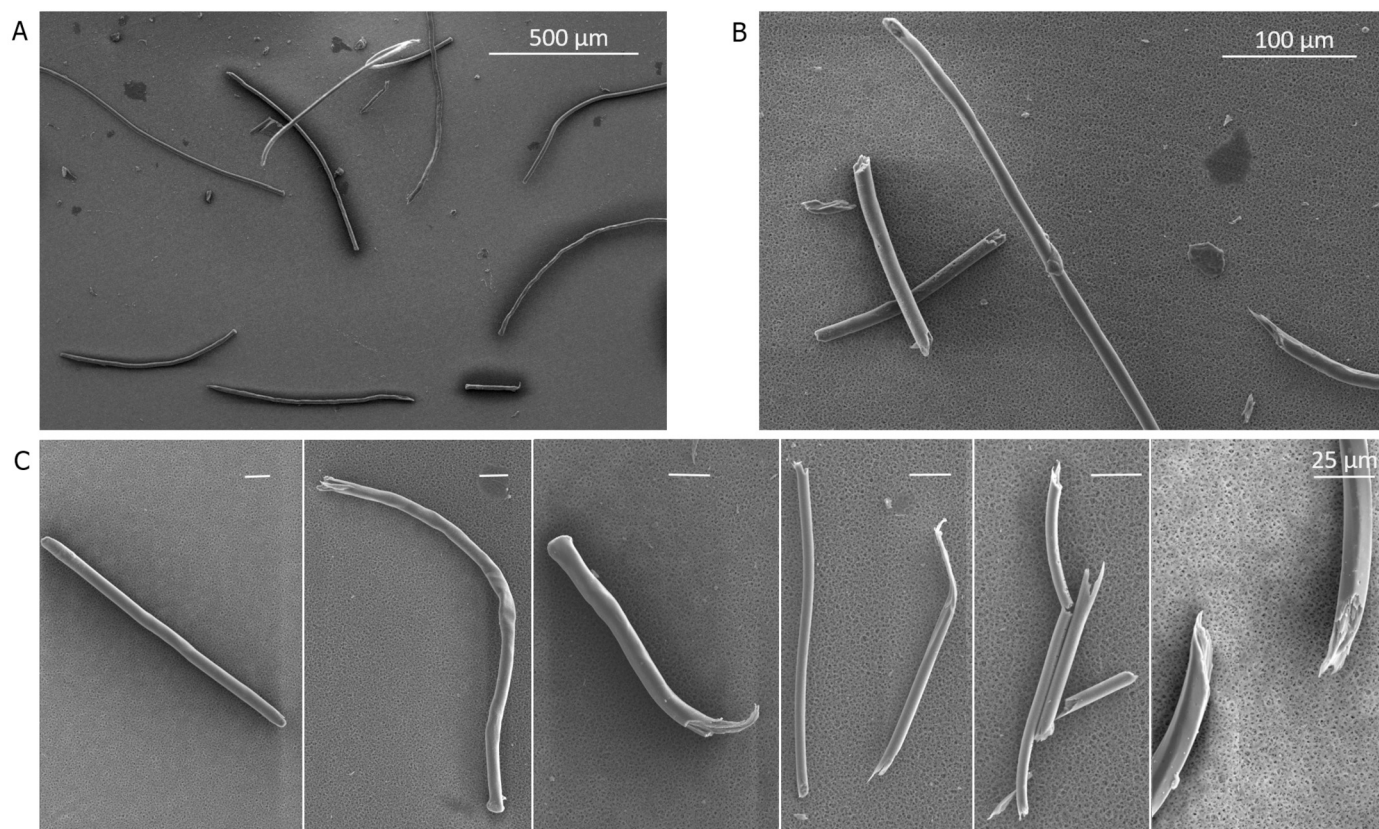
Fig. 4. Ends of regular fibers released; A: an example of fibers released after 15 days, most ends were flat and sharply cut, B: an example of fibers released after 60 days, most ends uneven, split, C: single fibers found throughout the different samples with some fibers having even ends but most uneven and splitting.

fiber ends featured splitting and flattening. Interlock and fleece fabrics appeared less damaged as the fibers were covered only with small pits, while the plain fabrics were covered in a dense network of cracks and indentations. No features of weathering were found on the unexposed side of the fabrics, nor on the dark controls (Fig. S6, S7).

3.3. Formation of fiber fragments following UV weathering

The fabrics kept at dark were extracted at every time point but the analysis showed that there was minimum variety between them (Table S4) and therefore the dark controls are represented by a single point in Fig. 3. For all fabrics, the dark controls contained MPF ranging between 160-540 per g of fabric (74-454 if the average blank value is subtracted). The MPF for weathered fabrics featured an exponential growth from the first time point at 15 days to the last measurement at 60 days, ranging from hundreds of MPF per g of fabric to hundred thousands of MPF (Fig. 3, Fig. S9, Table S5-S9). This applies to all four types of fabrics. If the values are fitted to an exponential function, the coefficient of determination ranges between 0.923 for plain staple fabrics and 0.997 for interlock fabric (Table S10).

Even though the four different fabrics followed the same general trend of exponential growth with extended exposure time, there were some differences. Interlock fabric showed consistently the smallest releases for all time points, whereas plain filament had small MPF releases for dark control and at the first measurement point but then exhibited an increase reaching a value of 330,000 MPF/g of fabric at 60 days. Releases from other fabric at the same time point ranged between 93,000 and 185,000 MPF/g.

The "split MPF" were not found in dark controls, nor blanks but they started appearing for some samples already after 15 days (Fig. 3). Specifically, they were first found in interlock and plain staple samples after 15 days of UV exposure, and fleece and plain filament after

30 days. With increasing UV exposure, the number of "split MPF" increased strongly, copying many trends seen with "regular MPF". Interlock showed the lowest release of "split MPF" after 60 days of exposure (9,800 MPF per g of fabric), while plain filament had highest release at 35,000 MPF per g and fleece and plain staple fabrics ranged in between them (14,000 and 26,000 MPF per g respectively). The number of "split MPF" represented 8-14% of the "regular MPF" (Fig. 5).

The category of "other MPF", which includes "thin" and "ribbon" MPF, were present in all samples, including the blanks and dark controls (Fig. 3). The two subcategories were only differentiated for fleece samples (Table S7). The "ribbons" represent only a small fraction of the category, while the "thin MPF" amount to 86-94% of the "other MPF". The "other MPF" also showed a growth in the numbers as the weathering progressed. Interestingly, interlock showed high values of "other MPF" in the dark control compared to the other fabrics which were just slightly higher than the blank values (3,100 "other MPF" per g for interlock, < 820 "other MPF" for other fabrics, 510 "other MPF" for blanks) but by the end of the weathering experiment, the highest releases were recorded for plain staple fabric (210,000 "other MPF"/g), while the other ranged in ten thousands of MPF per g, and the smallest release was seen from fleece (29,000 "other MPF"/g).

The number of "odd MP" found in the samples, both weathered (1,500-52,000 MP/g) and dark controls (990-9,800 MP/g), were higher than in the blanks (743 ± MP/blank), proving that also non-fibrous MP were released from the fabrics. In addition, the data show an increase in their count from the first measurement to the last, even though it is not as pronounced as with the other categories. The number of "odd MP" quadruples for interlock, fleece and plain staple fabric. For plain filament the values increased by a factor of 32, reaching values of 47,000 "odd MP"/g. Plain staple fabric showed highest values for each data point, including the dark control.
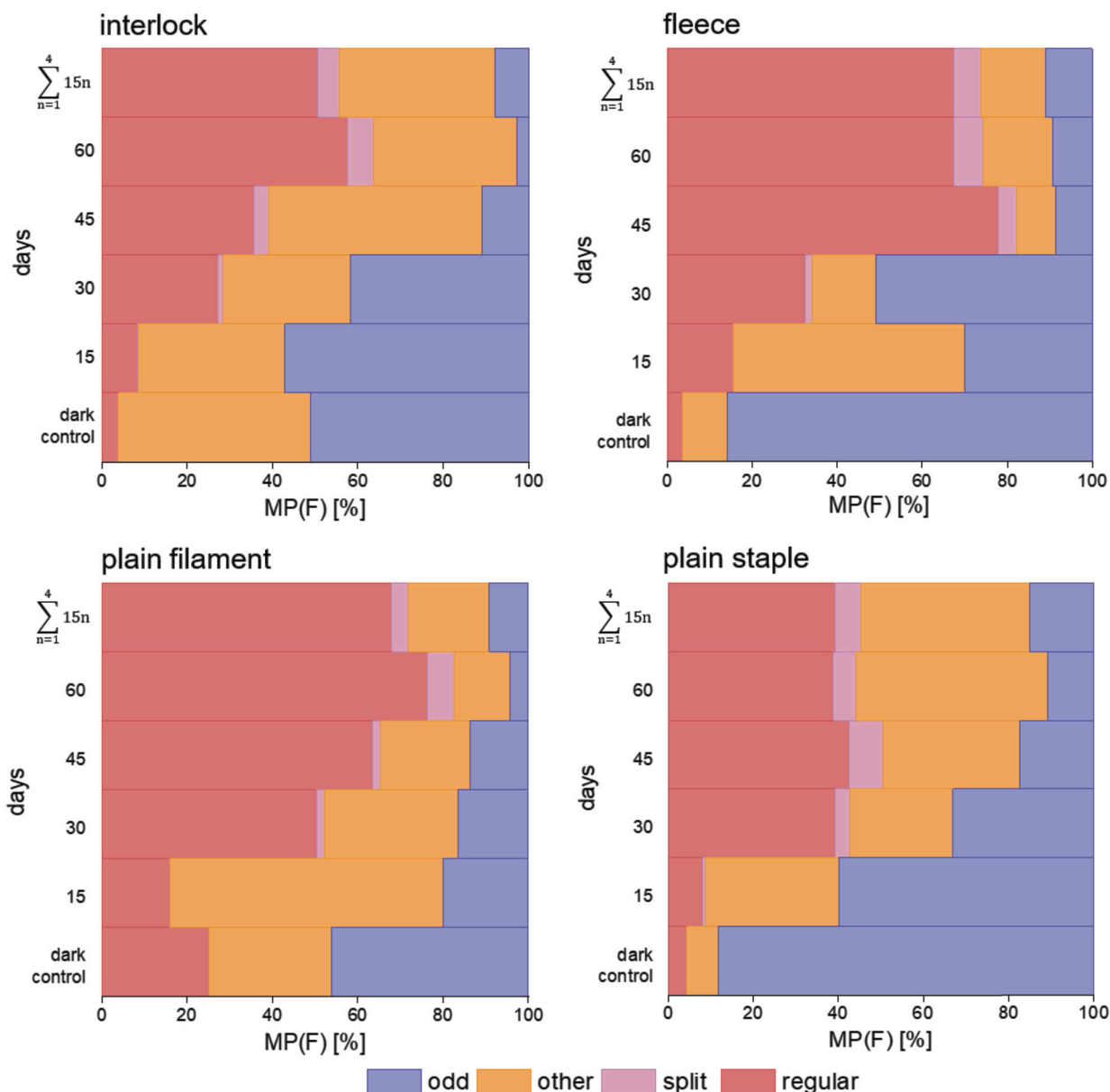
Fig. 5. Representation of how the MP(F) composition changes with progressing weathering for all four studied fabrics. Each row shows the MP(F) composition related to the extraction time point. The top row shows the cumulative composition of the MP(F) released during all four extraction points. While the dark controls showed primarily releases of "odd MP", the ratio taken up by "regular MPF" increased with progressing weathering. The ratios taken up by "other MPF" varied greatly based on the fabric type. (For interpretation of the colors in the figure(s), the reader is referred to the web version of this article.)

The changes in contribution of each MP(F) category to the total count with the progressing UV exposure are shown in (Fig. 5). The ratios between the different MP(F) categories varied both with time and the fabric type. In dark controls, most of the MP(F) found were "odd MP" contributing 50-85% to the total MP(F) found. As the weathering advanced, the number of "odd MP" was reduced to under 11%. On the contrary, "regular MPF" were represented little in the dark controls, and became the dominant component over time. "Split MPF" constituted only a small fraction of the MP(F) for all fabrics at all times. The ratios of "other MPF" to the other categories varied for the different fabrics. While in interlock, the contribution of "other MPF" did not vary with time, plain staple showed a small increase in the importance of "other MPF" over time, and plain filament and fleece showed the opposite trend.

Ends of randomly selected "regular MPF" from the filter papers were examined in detail (Fig. 4), Initially, mostly clean cut ends were observed but with progressing weathering, uneven and split ends be-

came predominant. Such ends had jagged, irregular surface (also seen in Fig. 2C) or showed signs of fibrillation, resembling a broken twig.

3.4. Length of microplastic fibers from weathered fabrics

The length distribution of all MP(F) categories for fleece fabric after 45 days of UV exposure is shown in (Fig. S10). "Thin" and "odd" MP(F) reached a median length of 21-22 μm. "Ribbon MPF" lengths were distributed over a range between 30 and 1,300 μm. The "regular MPF" showed two peaks in the distribution around 350 and 800 μm, with a median length at 429 μm. Based on this analysis and the small range of sizes of "other (thin) MPF" and "odd MPF", only "regular MPF" were further analyzed for other fabrics and time points (Fig. 6). The "regular MPF" length distribution varied with the fabric type. While interlock samples included MPF up to 4 mm in length, plain filament contained MPF only below 1 mm. With progressing weathering the length distribution curve started to bulge around the median length for all fabrics and
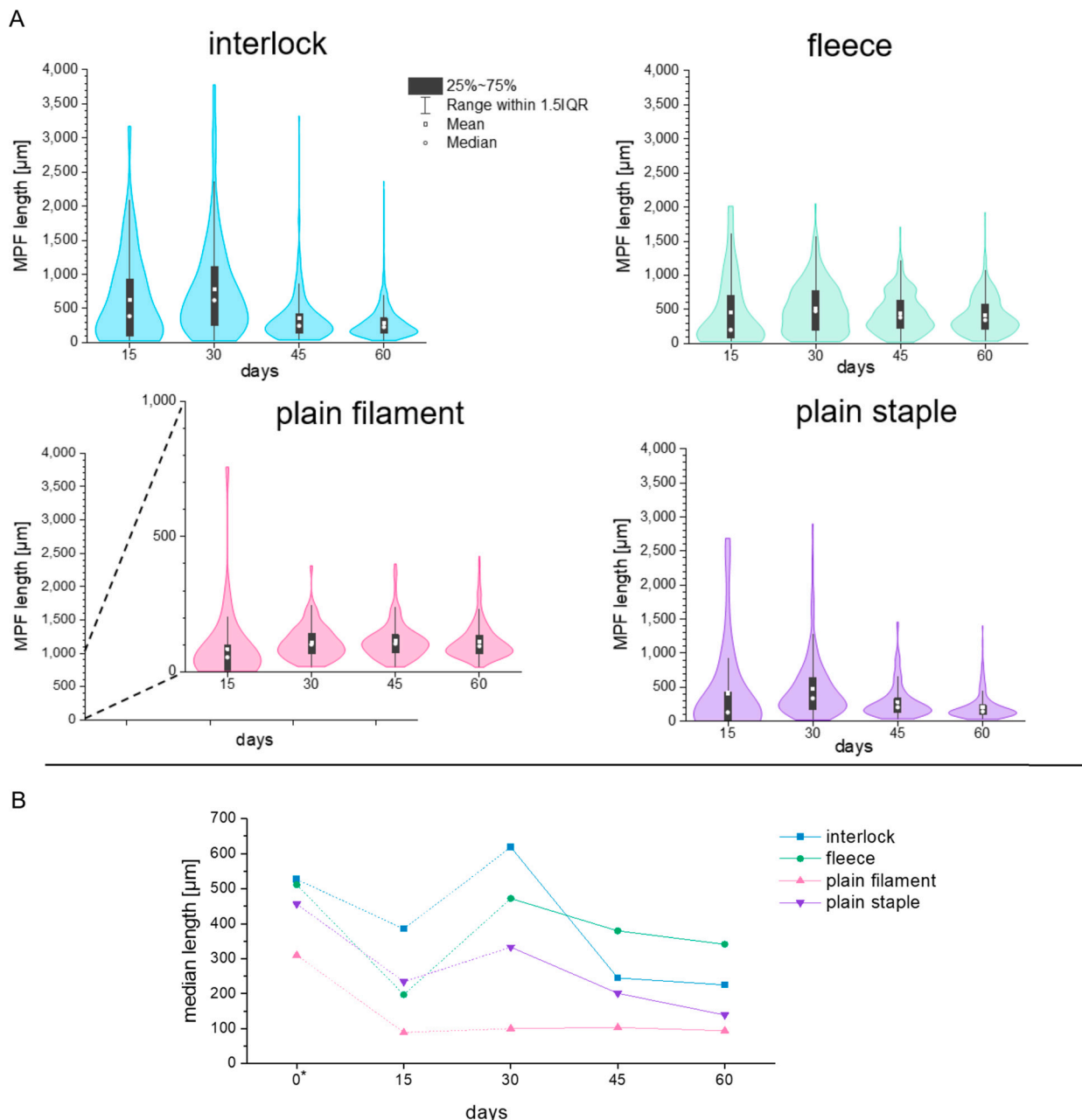
Fig. 6. A: "Regular MPF" length distribution based on fabric type. B: Median values extrapolated from the previous graph. *Time point 0 represents values for MPF median after 3 cycles of washing taken from a washing study of the same fabrics (Cai et al., 2020b). (For interpretation of the colors in the figure(s), the reader is referred to the web version of this article.)

the contribution of the longest and shortest fibers decreased. Median MPF lengths from a previous washing study are included for comparison (Cai et al., 2020b) (Fig. 6B, Table S11). With the exception of interlock, all fabrics showed lower median length values throughout the study compared to the unweathered reference fabrics. The median MPF lengths did not only vary with the extent of weathering but also with the fabric type. Plain filament, which showed almost no changes in median length throughout the UV exposure, had a median length of 97 µm. Interlock had initially the longest MPF, reaching a median value of 620 µm at 30 days but then the value decreased to 230 µm. Fleece showed smaller decrease in median length with the final value reaching 340 µm. Plain staple had a median MPF length ranging between 330 and 140 µm. With the exception of interlock after 30 days, all values from the weathered samples were lower than those reported in the aforementioned washing study.

3.5. Microplastic fiber mass

The contribution of the different MP(F) categories to the total mass of MP(F) released was assessed (Fig. 7, Table S12). The dominance of "regular MPF" was accentuated for all fabrics. This is a result the shorter nature of "other MPF" and generally small size of "odd MP" compared to the "regular MPF" (Fig. S10). For fleece fabric, the calculations were done also with separating the "thin" and "ribbon" MPF (Fig. S11). The results showed that the "regular MPF" are the main contributor to the weight of MP(F) released. This dominance was already seen with the MP(F) counts (Fig. S12) but the results expressed in mass highlight the fact even more as the greater length of "regular MPF" compared to the other shorter MPF impacts the results. While "regular MPF" represent 40-68% of the total MP count, in mass units the "regular MPF" represent 78-94% of the total MP released. Plain filament released 12 mg of
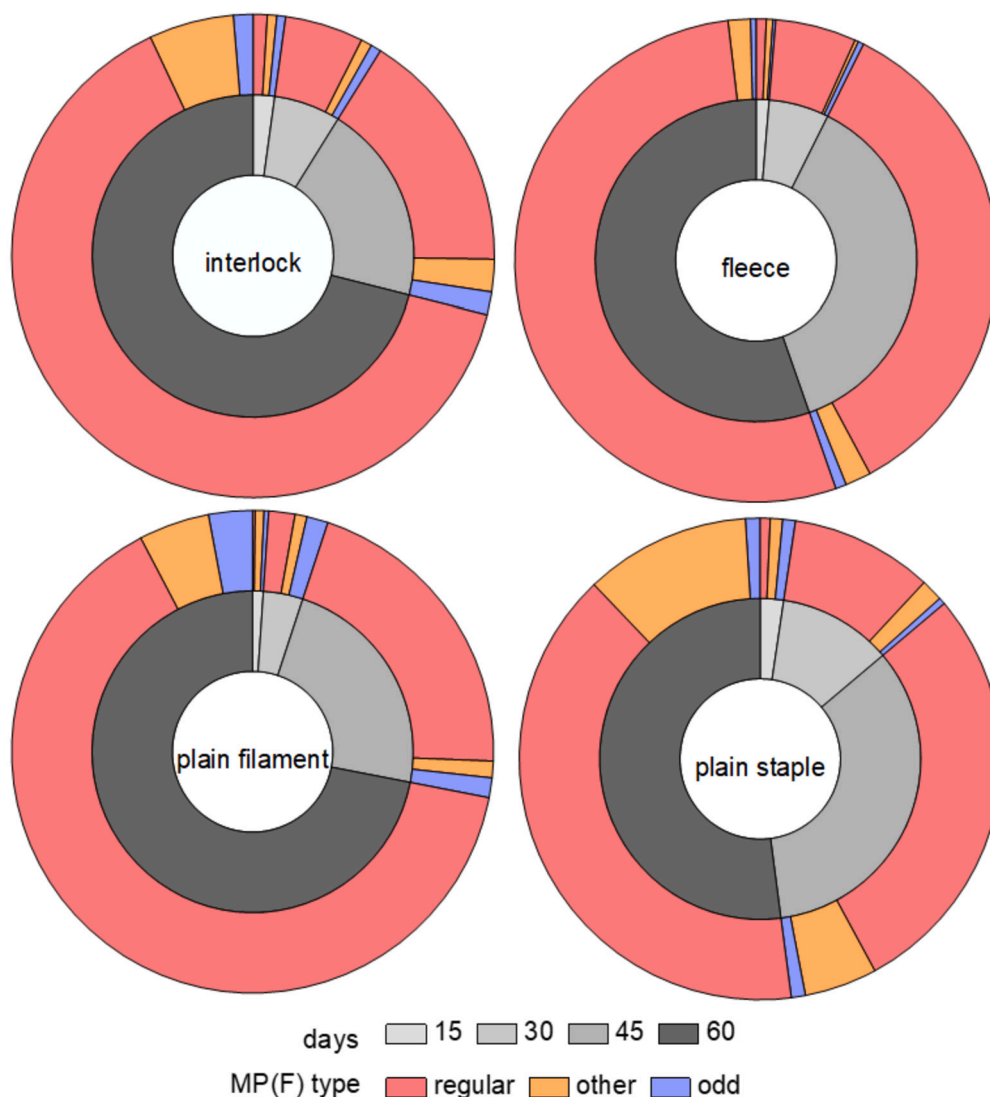
Fig. 7. Sunburst graphs for the different fabrics showing the contribution of the different MP(F) categories (outer circle) as well as from the different time points (inner circle) to the total mass of the MPF released. Moreover, the MP(F) category distribution is also visible for the different time points. (For interpretation of the colors in the figure(s), the reader is referred to the web version of this article.)

"regular MPF" per g of fabric, interlock 23 mg/g, plain staple 58 mg/g, and fleece 73 mg/g.

3.6. Chemical characterization

No significant changes in the absorbance bands in FT-IR spectra were observed after weathering (Fig. S13). The carbonyl index slightly varied between the different fabrics, ranging between 0.57 and 0.67 for the dark controls. When comparing the weathered fabrics to their control counterparts, they matched with ± 0.01 variation. PET already contains carboxyl groups and therefore observing formation of oxidized moieties during weathering is not as direct as in the case of polyolefins, where already small changes can be observed.

4. Discussion

4.1. Characteristics of microplastic fibers produced during weathering

So far MPF from textiles have been mainly studied in washing studies. MPF in such studies are commonly smooth round fibers, with diameters ranging between 10 and 30 μm, corresponding to the fibers used in the studied textiles (Cai et al., 2020b; Salvador Cesa et al., 2017; Choi

et al., 2021). A textile abrasion study revealed a finer type of MPF, the so-called fibrils (Cai et al., 2021). The fibrils were of much smaller diameter than the "regular MPF" (2-5 μm) and were also shorter (30-150 μm) than the "regular MPF". The MPF released from weathered fabrics showed different and novel features compared to those known from such standard washing or abrasion studies. One of the key differences was that not only regular fibers were found but a range of shapes including non-fibrous MP. The category of thin MPF may be compared with the fibrils due to their smaller diameter. However, the median length of the thin fibers was shorter than the one of fibrils with a median length ranging between 17 and 19 μm for fleece samples.

Aside from the size and shape of the MPF, the ends of the fibers showed more diversity after weathering than after washing. In a washing study investigating the same fabrics, the end of the released fibers were all cleanly cut and slightly broadened (Cai et al., 2020b). Such features correspond to tensile failure and are representative of a high-speed break (Morton and Hearle, 2008). These fibers were therefore formed during fiber or textile manufacturing and were only released during washing but not newly formed. In this study, clean ends were seen with dark controls and in the first set of samples weathered for 15 days. With increasing exposure, the fiber ends became predominantly rugged or split (Fig. 4). The rugged ends are characteristic of a gran-

ular failure which is related to the failure of single elements (fibrils) which further propagates across the material (Hearle et al., 1998). The split ends, resembling to splitting seen in a broken twig, are a sign of a separate fibrillar failure.

Shorter and smaller MP(F) compared to "regular MPF" are produced in the form of "thin MPF" and "odd MP". During the fiber cleaving, a fibril, or multiple adjacent fibrils, detach from the broken end of a fiber. An example of such an incomplete process can be seen in Fig. 1 (the bottom image in the "split MPF" category). The "odd MP" may also originate from the breaking point, or might be separated flakes from the cracked surface. Fibrillation and formation of thinner MPF was also reported in a textile abrasion study but there the textiles were exposed to a cyclic load and a different mode of fiber failure was proposed (Cai et al., 2021).

Another feature of the MPF released from the weathered fabrics was the roughening of the surfaces. The extent and form of damage varied from fabric to fabric. Interlock and fleece fabrics were primarily covered with pits, while plain fabrics were covered in cracks (Fig. 2). Transverse cracks were predominant but networks of cracks going in all direction were also seen. Emergence of indentations and cracks on weathered plastic samples have been previously reported, both in artificial weathering experiments, as well as in MP samples from field studies (Wang et al., 1998; Yakimets et al., 2004; Sørensen et al., 2021). Deng et al. (2022) described many different patterns of cracking on environmental plastic samples. Bai et al. (2022) reported on changes in morphology with proceeding weathering of a PET geotextile, first showing that the surface was heavily covered in flakes, followed by a build up of a series of cracks of the same orientation. In another study done on PET fibers, few small pits were visible at first time point at 14 days (but no cracks) and little changes were seen as the weathering progressed for another 42 days (Sørensen et al., 2021). Another study compared weathering of black and white PET with two data points at 5 and 10 months (Sait et al., 2021). While UV exposure of black fibers resulted in formation of cracks, white fibers were covered with large pits. It is important to note, that the weathering conditions varied between the different studies. Further research is needed to investigate whether the differences are related to the UV exposure conditions, the medium, the fabric type or the different grades of PET and the production history. For example a variation in the distribution of crystalline and amorphous regions may have an impact on the weathering as the photooxidative degradation is known to primarily occur in the amorphous regions (Andrady, 2017; Singh and Sharma, 2008). However, there is one more big unknown in the process which is the presence of additives such as dyes. Additives and impurities are considered as a starting point for the radical reactions caused by UV irradiation (Chamas et al., 2020). Therefore, the variation in morphology and the rate of weathering of the PET fibers could be also linked to the type and quantity of additives present. The presence of UV stabilisers would definitely have a great impact on the weathering process. However, it is not expected that they would be present in fabrics for casual textiles such as the ones used in this study.

Despite the clear structural changes observed under SEM, FT-IR analysis did not recognize any chemical change between the weathered and unweathered samples. This is likely due to the fact that the measurements were taken through the whole cross-section of a fabric and the signal from the unexposed bulk hinders seeing whether there are some changes in the weathered surface layer.

4.2. Impact of fabric type on microplastic fiber counts

All studied fabrics showed an increase of MPF releases of a similar magnitude from the first data point to the last one, but the ranking of the different fabrics varied. When the MP(F) counts were compared, plain filament showed approximately two to three times higher releases than interlock and fleece, not only when "regular MPF" were considered but also when all MPF and MP were added up. The same result was

seen when also non-fibrous MP were added to the calculations. When the results were translated into mass, the order of the fabrics changed. An important factor that plays a role is the diameter of the fibers. Plain filament is made of fibers ranging between 7.5 and 7.9 µm, while all the other fabrics were made of fibers with a diameter between 11.7 and 13.4 µm. The thinner diameter of the fibers in plain filament allows for exposure of a higher number of fibers per area as in the other fabrics. At the same time, the MPF released from the plain filament are lighter. The low mass of the MPF from plain filament was also influenced by their shorter length. The same reasoning regarding the mass of MPF can be applied for the fleece fabric. Even though the MPF counts were smaller than for plain fabric, the fiber diameter is greater.

The architecture of the fabric likely played a role in the specific release numbers. Plain fabrics have a neat structure, creating a full cover of aligned fibers in the woven thread on the surface of the fabric (Fig. S14). This is even more pronounced with the plain fabric made with filament yarn as the surface of the fabric is very smooth and no single fibers are protruding (Fig. 2A). The potential release from one piece of plain filament fabric was calculated based on some simple assumptions. The fabric was imagined as a set of fibers aligned with each other covering the whole surface. Taking into account the fiber width as well as mean length of 100 µm for the "regular MPF" released, one single layer has a potential of breaking into 130,000 MPF/cm^2. The potential release was calculated also for plain staple and interlock, where the void space in between yarns in the knitted fabric was taken into consideration. The potential releases were 55,000 MPF/cm^2 for plain staple and 21,000 MPF/cm^2 for interlock. In contrast with plain fabrics, the fleece surface is very disorganized as it is made of protruding fibers creating a loose entangled network. Such a structure does not result in a homogeneous UV exposure of the surface fibers. The more complex structure of knitted interlock also reduces the amount of potential fibers exposed to irradiation. It can be estimated that 5% of the potential fibers from the surface layer of the plain fabrics was released, while interlock released 11% of the potential regular fibers. Including split fibers in the calculations would result in changes < 1%.

4.3. Fragmentation of fibers

In textile washing studies, the washing step is usually considered as an extraction for removing the trapped MPF. The role of the washing process as a source of mechanical action was studied by altering the number of steel balls used and no significant difference in MPF release was noted (De Falco et al., 2018; Cai et al., 2020b). Together with the analysis of the fiber ends (Cai et al., 2020b), this clearly shows that washing is not producing any MPF but is primarily an extraction process. However, in our weathering study, the role of washing is not only to extract fibers but also to produce fiber fragments from the weathered fibers. Polymer ageing results in its friability as its chemical and physical properties change and cracks and pits emerge on the surface but it is not a direct cause of fiber breaking. It is expected that mechanical force is needed to complete the process of fragmentation. In this study, the washing step was considered to be a combination of source of mechanical action as well as simultaneous extraction of the produced fragments.

The mechanism of fragmentation of plastics as a result of weathering is still under discussion. There are theories applying cascading fragmentation to environmental plastic fragmentation (Turcotte, 1986; Kaandorp et al., 2021), suggesting initial breakdown into big pieces, which continue fragmenting down to the nanoscale. Another model describes plastic fragmentation through surface ablation, where only the top layer separates into small pieces (Andrady, 2017). In this study both models can be applied. If single fibers are considered, they split into shorter fibers with additional formation of fragments detached from the broken ends. However, when the whole textile sample is considered, the surface-ablation fragmentation model is more suitable as only

the surface fibers exposed to the UV irradiation are broken off and the size of the detached MP(F) is small compared to the size of the fabric.

The length of the "regular MPF" released from the fabrics either decreased or stayed constant during the weathering. In standard washing studies the opposite trend was generally observed, as longer MPF were released after repeated washings (Cai et al., 2020b). With progressing weathering the length distribution of the "regular MPF" narrowed. The architecture of the fabrics and the different length of the fibers cannot explain variations in length decrease and therefore more studies with different fabrics will be needed.

4.4. Contribution of weathered textiles to MP found in the environment

The discussion about MPF most frequently targets MPF released during washing. A direct comparison can be made between our weathering results and a washing study done exactly with the same fabrics (Cai et al., 2020b). This study reported the highest releases during the first wash, followed by a steady decrease in MPF numbers over ten washing cycles. To compare these results to our study, a simple model was created considering a piece of textile washed weekly for 17.5 months. This time is an approximate equivalent to 60 days of artificial weathering under the conditions of this study. The translation to a real-life scenario depends on the specific location. The suggested value of 17.5 months was calculated for an average European light exposure (detailed calculations can be found in SI). It was assumed that the MPF release during washing after the 10th cycle (which was not measured) remained constant. After 76 washing cycles, the total amount of MPF released would be between 2,700 MPF/g from plain filament to 21,000 MPF/g from fleece fabric. In comparison, the releases by weathering over the same time period ranged from 110,000 to 440,000 MPF per g. The potential of weathered fabrics to be a source of MPF upon exposure to mechanical stress is therefore 20-40 times larger than of washed garments. However, this is only a theoretical calculation as the amount of textiles exposed to UV weathering is likely to be small compared to the amount of textiles washed. For some textiles used outdoors and exposed to sunlight, the direct release to the environment by weathering may be very relevant. Weathered textiles should therefore be considered in the debate regarding the sources of MP in the environment. The same applies to textiles exposed in open landfills such as the one in Atacama desert. The weathering performed in this study corresponds to 8.4 months exposure in the Atacama desert (calculations in SI). The textiles in such scenarios do not experience washing like textiles in the study. However, they can meet other forms of mechanical stress which may lead to disintegration of the weathered fibers from the textile and formation of MP. It should also be noted that the "washing" used in this study has been shown in several previous studies to be mainly an extraction technique that is able to quantify the fiber fragments present in fabrics, so representing a way to quantify the potential of a fabric to release fiber fragments (Cai et al., 2020b).

We also showed that not only "regular MPF" are released from weathered fabrics, but also smaller fragments of fibers, either in the form of fibrils or odd shapes. When such MPs are identified in environmental samples, they are likely not linked to textiles. In addition, the size of such MP(F) is below the detection limits of many environmental studies, as primarily MPF of length ranging from hundreds and thousands of μm are reported (Gago et al., 2018; Silva and Nanny, 2020), while the small MP(F) found in this study average at 21-22 μm. However shorter MPF are not an unseen phenomenon, as MPF of length below 20 μm were reported to be the most abundant fiber type in urban air (Li et al., 2020). An important aspect that is related to the MP size is its availability to become airborne, especially as PET is the dominant polymer type found in the atmosphere (Xu et al., 2022). Textiles are often considered as one of the prime sources of the atmospheric MP (Dris et al., 2016; Prata, 2018) but the link to weathered textiles has not been previously formed. A study of MP in the air showed that 88% of the MP found was shorter than 100 μm (Xie et al., 2022). Even though primar-

ily particles below the limit of our measurements (<5 μm) are inhalable and can cause a biological response (Donaldson and Tran, 2008), small MP(F) should be looked at with caution. The smaller fragments released from textiles may behave differently than MPF in regards of their mobility, fate as well as (eco)toxicity (Waldschläger and Schüttrumpf, 2019; Piccardo et al., 2020; Bucci et al., 2020).

5. Conclusions

Our study represents the first detailed study of MP(F) extracted from four different polyester textiles exposed to UV ageing. We found a steep increase in the MP(F) released with progressing weathering. Extraction after 60 days of exposure to UV lead to the release of 160,000-450,000 MP(F) per g of fabric. Additional 10,000-52,000 non-fibrous MP per g of fabric were observed. These results allow to conclude that 1 gram of fabric has 20-40 times greater potential to release MP(F) during weathering compared to washing. The MPF released after weathering have jagged fiber ends or fiber splitting, similar to a broken twig. These features, which are different compared to the fiber ends seen in washing studies, may be useful to identify the source of fibers found in environmental monitoring studies. We have also shown that weathering produces a much larger variety of different types of fibers and other forms than washing. Especially the formation of "thin MPF" can be relevant from a human or environmental hazard perspective. Overall, the work shows that textiles can be a source of many different types of fibers and other forms of MP that extend in size, form and structure way beyond the MPF usually reported in standard washing studies.

CRediT authorship contribution statement

Barbora Pinlova: Formal analysis, Investigation, Methodology, Visualization, Writing – original draft. **Bernd Nowack:** Conceptualization, Funding acquisition, Investigation, Project administration, Supervision, Validation, Writing – review & editing.

Declaration of competing interest

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

Data availability

Data will be made available on request.

Appendix A. Supplementary material

Supplementary material related to this article can be found online at https://doi.org/10.1016/j.envpol.2023.121012.

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