



Evaluation of the Microfibre Release from Textile Fabrics: Influence of Material Type, Washing, and Abrasion

Yanice Walraevens

P592783988

Supervisors: Prof. Maria Ángeles Bonet Aracil, Prof. Kazushi Yamada

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Abstract and keywords

The release of microfibrils (MFs) from textiles during domestic washing is a growing environmental concern. This study investigates the MF release of plain woven textile fabrics, focusing on the influence of material composition, washing, and abrasion. The fabrics were either entirely made of conventional polyester or composed of conventional polyester warp yarns combined with varying weft yarns, including biodegradable, recycled, staple fibre, entangled fibre polyester, or cotton.

Washing tests were conducted using both a laboratory-scale (Linitest) and a domestic washing machine. Fabric wear was simulated by applying abrasion using a Martindale device. The surface damage was examined with Scanning Electron Microscopy (SEM), and the MF release was quantified through optical microscopy.

The results showed that the MF release decreased over successive washes but increased significantly after abrasion. The cotton-polyester fabrics released the highest number of MFs among the non-abraded fabrics, possibly due to exposed fibre ends at the laser-cut edges. The recycled polyester fabrics performed as well, or in some cases better than, virgin polyester in terms of MF release. Additionally, nanoparticles were detected in the wash effluents of fabrics washed in the Linitest, with larger and more polydisperse particles observed in cotton-polyester samples.

These findings may support more informed material selection and manufacturing decisions to help reduce MF emissions from textiles.

Keywords: Microfibre release, Plain woven fabrics, Polyester, Cotton, Washing, Abrasion, Optical microscopy, SEM, DLS

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Abstract – The release of microfibrils (MFs) from textiles during domestic washing is a growing environmental concern. This study investigates the MF release of plain woven textile fabrics, focusing on the influence of material composition, washing, and abrasion. The fabrics were either entirely made of conventional polyester or composed of conventional polyester warp yarns combined with varying weft yarns, including biodegradable, recycled, staple fibre, entangled fibre polyester, or cotton. Washing tests were conducted using both a laboratory-scale (Linitest) and a domestic washing machine. Fabric wear was simulated by applying abrasion using a Martindale device. The surface damage was examined with Scanning Electron Microscopy (SEM), and the MF release was quantified through optical microscopy. The results showed that the MF release decreased over successive washes but increased significantly after abrasion. The cotton-polyester fabrics released the highest number of MFs among the non-abraded fabrics, possibly due to exposed fibre ends at the laser-cut edges. The recycled polyester fabrics performed as well, or in some cases better than, virgin polyester in terms of MF release. Additionally, nanoparticles were detected in the wash effluents of fabrics washed in the Linitest, with larger and more polydisperse particles observed in cotton-polyester samples. These findings may support more informed material selection and manufacturing decisions to help reduce MF emissions from textiles.

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I. INTRODUCTION

Microplastics are plastic particles larger than 1 μm and smaller than 5 mm that originate from various sources and are now ubiquitous in both marine and terrestrial ecosystems [1], [2], [3]. These microplastics can be classified as “primary” or “secondary” depending on their source [4]. Primary microplastics are directly released into the environment, while secondary microplastics originate from the degradation of larger plastic items discarded into the environment [5]. One of the major sources of primary microplastics is textile laundering [6], [7]. According to Boucher et al. [5], 35% of all primary microplastics in the oceans originate from textile laundering, in the form of microplastic fibres (MFs) [5].

The contribution of textile laundering to microplastic pollution is especially concerning due to the continually increasing production and consumption of textiles, because of the growing population and the fast fashion industry [3]. Additionally, there is a growing dominance of synthetic fibres,

such as polyester, due to their durability, affordability, and performance characteristics [3]. However, these same properties make synthetic MFs persistent in the environment once released, eventually leading to the accumulation inside ecosystems [8]. In addition to their physical presence, MFs can act as carriers of toxic pollutants, such as heavy metals and hazardous organic compounds [9], [10]. These particles can penetrate biological barriers, leading to the accumulation in vital organs [10], [11]. Although there is still a need to increase the understanding of the potential impacts of these microplastics on human health, their ability to accumulate in the supply chain and enter vital organs makes them a threat to human health [8], [9].

Although general trends such as increased MF release due to abrasion [12] and a gradual decrease over sequential washes [13], [14] are well established, there is still considerable variation in reported results across studies. These differences can largely be attributed to the wide range of textile types, washing conditions, and analytical methods used [13], [14], making direct comparison among studies difficult. This study investigated the MF release from plain-woven laser-cut polyester-based fabrics. The fabrics were either made entirely of conventional polyester yarns or conventional polyester warp yarns combined with recycled, biodegradable, staple fibre, entangled polyester, or cotton weft yarns. Washing tests were performed using both a laboratory-scale washing machine and a domestic washing machine, with and without intermediate abrasion of the fabrics. Additionally, the fabric surfaces were analysed using Scanning Electron Microscopy (SEM), the MF release was quantified using optical microscopy, and the nanoparticle release into the wash effluent was analysed using Dynamic Light Scattering (DLS) to investigate both visible and finer particle fragmentation.

II. MATERIALS & METHODS

A. Materials

The provided yarns consisted of white conventional polyester, biodegradable polyester, recycled polyester, staple fibre polyester, entangled polyester, and cotton fibres. The densities of these yarns were determined using the MESTEX according to the ISO 2060:1994 [15] standard method, and are represented in Table 1.

Table 1: Yarn materials and corresponding densities.

Yarn	Yarn density (Tex)
Conventional polyester	18.1
Biodegradable polyester	17.4
Recycled polyester	17.1
Entangled fibre polyester	33.1
Staple fibre polyester	40.2
Cotton fibre	19.4

Black cellulose nitrate membrane filters (pore size: 0.2 μm ; diameter: 47 mm; Scharlau) were used to filter the wash effluents.

Nitrile gloves and a cotton lab coat were worn to reduce MF contamination originating from the operator.

B. Methods

1) Fabric preparation

The yarns were used to weave plain woven fabrics, shown in Table 2, with the Smit Textile GS900.

Table 2: Fabric characteristics with the label, yarn types, warp and weft density, and fabric density.

Label	Warp yarn type	Warp density (yarns/cm)	Weft yarn type	Weft density (yarns/cm)	Fabric density (g/m ²)
N	Conventional polyester	60	Conventional polyester	21	150.4
S		60	Staple fibre polyester	20	208.6
T		61	Entangled polyester	21	189.7
R		59	Recycled polyester	21	151.6
B		60	Biodegradable polyester	21	151.6
C		61	Cotton	20	158.1

The exact content of cotton in the cotton-polyester blend fabric was determined by measuring the dry weight of a piece of fabric, dissolving the cotton, and measuring the dry weight again afterwards. The cotton-polyester fabric contained 27 wt% of cotton.

The fabrics were cut using a FLUX Ador laser cutter at an intensity of 15% and a velocity of 25 mm/s. The samples for the laboratory-scale washing machine were cut into circles of 38 and 140 mm according to ISO standard 12947-2:2016 [16]. In contrast, the dimensions of the samples prepared for the domestic washing machine were 30 cm x 60 cm.

Finally, the samples destined for the laboratory-scale testing were rinsed for 5 min in 150 mL at 30°C, while the samples destined for the domestic washing machine testing were rinsed on the “RAPIDO” program (20 min, 20°C, 400 rpm). This rinsing procedure was performed to remove all contamination originating from the weaving process.

2) Yarn material characterisation

The chemical composition and thermal properties of the yarn materials were analysed using Fourier Transform Infrared spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC). The University of Alicante performed both assessments. The FTIR analysis was performed in Attenuated Reflectance (ATR) mode, using the Jasco FTIR 4700 IRT 5200 at a resolution of 4 cm^{-1} . The DSC250 was used for the DSC analysis. The test was conducted in a nitrogen environment, with a gas flow rate of 50 ml/min. The starting temperature was

set at 30°C, while the ramp was set at 10°C/min until 300°C was reached. Afterwards, the temperature was decreased by 10°C/min to 30°C before starting the third heating cycle, identical to the first heating cycle.

3) Surface analysis

The surface analysis was performed using Scanning Electron Microscopy (SEM), with the Field Emission Microscope FESEM (ULTRA 55, ZEISS) at an acceleration voltage of 2 kV. However, before the SEM analysis, the samples' surfaces were coated with a layer of gold and palladium for 30 seconds to make them conductive. The coating was performed with a BAL-TEC SCD 005 sputter coater.

4) Washing and abrasion

The abrasion tests were performed with the M235 Martindale, applying 5000 rubbing cycles with a load of 12 kPa at a rotation speed of 47.5 rpm.

The laboratory-scale washing experiments were performed using a Linitest device. Each washing vial contained the prepared circular sample fabrics of 38 and 140 mm in diameter, 150 mL of distilled water, and 10 steel balls. The temperature was set at 40°C for 45 min. A triplicate of washing tests was performed for every sample type with and without preceding abrasion.

The following abbreviation structure was used to clarify the fabric type, the number of washing cycles, and the number of abrasion cycles: x_yR_zL, where “x” represents the sample type, “y” the number of abrasion cycles, and “z” the number of washing cycles. For example, N_1R_1L is made from conventional polyester yarns, underwent one abrasion cycle, and one washing cycle.

The domestic laundry tests were performed in a Samsung front-load domestic washing machine on the “LANA” (wool) program, including 54 min, at 40°C and 800 rpm. However, a rinsing cycle (54 min, 40°C, 800 rpm, 0.5 L of vinegar) was introduced to remove the remaining contamination inside the washing machine and tubes before starting the actual testing. Additionally, a rinsing cycle (“LANA” program) was performed after every test to clean the machine.

5) Filtration

Vacuum-assisted filtration, using black cellulose nitrate membrane filters with a pore size of 0.2 μm and a diameter of 47 mm, was performed to filter the wash effluents. The full content of the Linitest containers was filtered, and the containers were rinsed 3 times with 2 mL of distilled water. The domestic washing machine sampling was performed by capturing all the effluent water in three separate containers of 25 L, which were thoroughly shaken before taking a 1 L sample from each container. This 1 L sample was then divided into two samples of 0.5 L to avoid overpopulation of MFs on the filter surface, which could otherwise interfere with accurate MF counting.

6) Microfibre quantification

Two approaches were pursued to quantify the MF release: gravimetric analysis and manual counting by using optical microscopy. The gravimetric analysis was performed by taking the dry weight of the filters before and after the filtration. Each time, the filters were dried for at least 4 hours at 60°C and 50%

ventilation before being placed in a desiccator for 30 min to cool down. The optical microscopy was performed with the Leica SAP0, linked with a Flexacam C3 camera. The grid structure of the filter was used to divide the filter surface into multiple counting areas, as illustrated in Figure 1, each area included two squares of the grid. A magnetic closing piece was mounted on the filter during filtration, meaning that only the part of the filter inside this piece was exposed to the wash effluent. The position of this closing piece could always be relocated with optical microscopy due to the presence of a circular band without any MFs or other particles present on the filter, making it possible to locate the counting areas at the edges. The total number of MFs was determined by manually counting the number of MFs in the two squares of each zone, calculating the average number of MFs in a square, and multiplying this by the total number of squares on the filter grid exposed to the wash effluent (122).

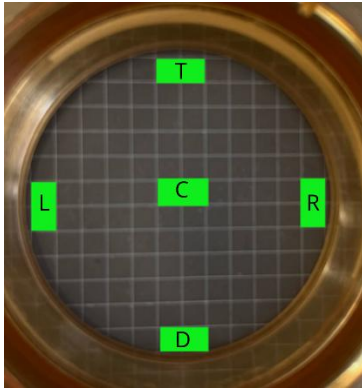


Figure 1: Filter surface with the green rectangular zones indicating the MF counting areas: centre (C), left (L), top (T), right (R), and down (D).

The images taken during the optical microscopy were analysed in ImageJ to count the MFs. Firstly, the contrast of the images was enhanced to increase the visibility of the MFs. Afterwards, the multipoint function was used to count the MFs manually. Only white yarns were used to weave the fabrics, meaning all MFs with a different colour were considered as contamination and not counted. Additionally, fibres longer than 5 mm were also not counted because these are not considered MFs.

7) Nanoparticle analysis

Dynamic Light Scattering (DLS) was used to analyse the size distribution of the nanoparticles present in the aqueous filtrate after filtration. The DLS measurements were performed using the ZETASIZER Nano Series with polystyrene cuvettes, at a fixed scattering angle of 173° and a temperature of 25°C .

III. RESULTS & DISCUSSION

A. Material characteristics and surface analysis

1) FTIR and DSC analysis

The FTIR and DSC analyses showed that the chemical composition and thermal behaviour of the studied polyesters were very similar, apart from the recycled polyester, which showed signs of degradation by having broader peaks with a higher absorbance intensity at the C-O stretching vibrations and a lower melting temperature compared to the other polyesters.

2) SEM analysis

The SEM images of the different fabric types after weaving and rinsing, illustrated in Figure 2, show that there are a significant number of partially detached fibres present on the surface of the staple fibre polyester fabric. These fibres were removed after the first washing cycle, as visible in Figure 3. This suggests that the washing process could remove the partially detached fibres still present from the weaving process, while the rinsing procedure was insufficient.

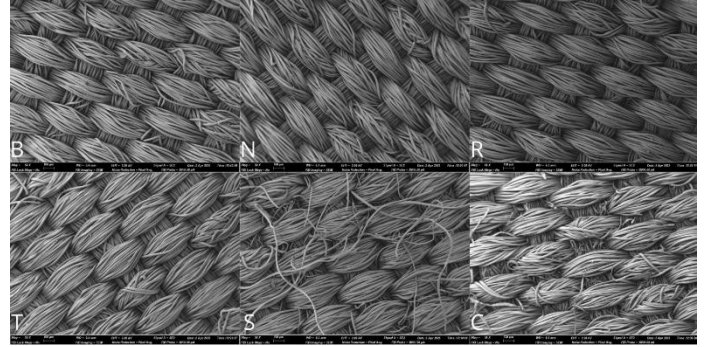


Figure 2: SEM images at x50 of the woven sample fabrics: biodegradable (B), conventional (N), recycled (R), entangled (T), staple fibre (S) polyester, and cotton-polyester (C).

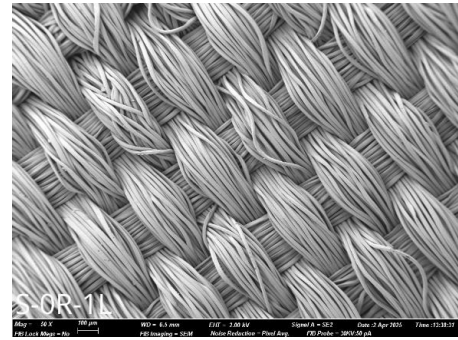


Figure 3: SEM image at x50 of the staple fibre polyester test fabric (S-0R-1L) after one washing cycle.

Scanning Electron Microscopy (SEM) revealed increasing surface damage on the fabrics after one and three sequential abrasion and washing cycles, as illustrated in Figure 4. The most common damage features were partially detached fibres and fractured remnants at the yarn interlacing points. Notably, the recycled polyester fabric retained a more intact structure after the first cycle compared to the biodegradable polyester fabric. Even after three cycles, the recycled polyester showed fewer fractured fibre remnants, suggesting better resistance to surface degradation under mechanical stress.

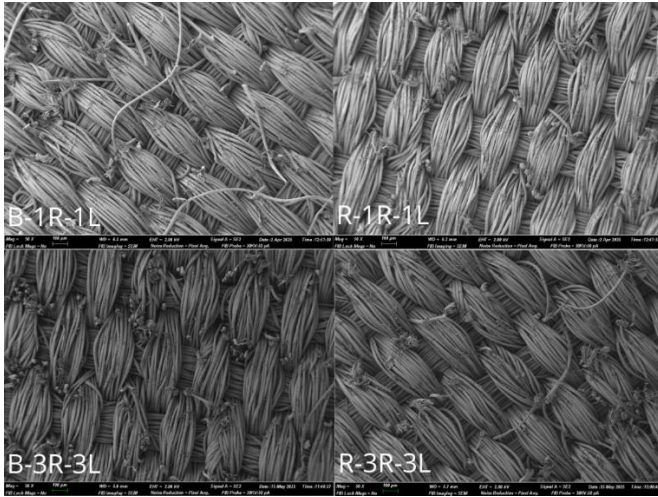


Figure 4: SEM images at x50: Biodegradable polyester (B-1R-1L) and recycled polyester (R-1R-1L) test fabrics after one abrasion and washing cycle; Biodegradable polyester (B-3R-3L) and recycled polyester (R-3R-3L) test fabrics after three abrasion and washing cycles.

SEM images of the laser-cut fabric edges, illustrated in Figure 5, revealed differences in thermal response across the fabric types. The staple fibre polyester fabric (S) showed a compact, smoothly fused edge, indicating effective thermal sealing. The recycled polyester fabric (R), which has a slightly lower melting temperature, had a broader and less uniform molten layer at the laser-cut edge, possibly due to an increased polymer flow during melting. However, surface cracks were also visible, suggesting increased brittleness. In contrast, the cotton-polyester fabric (C) showed partially exposed cotton fibre bundles, as the molten polyester warp only partially covered the cotton weft yarns. This limited sealing may increase the risk of fibre shedding at the edges under mechanical stress, despite some fibres being held in place by the surrounding melt.

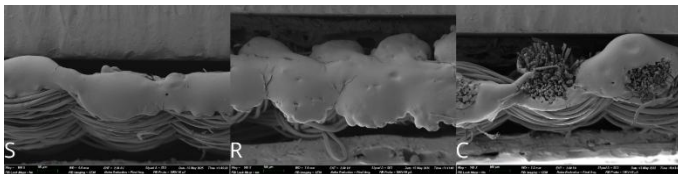


Figure 5: SEM images at x100 of the laser cut fabric edges of the staple fibre polyester (S), recycled polyester (R), and cotton-polyester (C) fabrics.

B. Microfibre quantification

1) Gravimetric analysis

Gravimetric analysis was used to assess the MF release by measuring the mass gain of the cellulose nitrate filters before and after filtration. However, this method proved unreliable due to the very low quantities of MFs shed, which were often below the detection threshold of the analytical balance. Additional variability was likely introduced by filter degradation, moisture absorption, and environmental factors such as air drafts and static charge. As a result, gravimetric analysis did not yield accurate or repeatable data and was excluded from the final MF quantification approach.

2) Optical microscopy

MF quantification with optical microscopy, illustrated in Figure 6, revealed a decreasing trend in MF release from unabraded fabrics over three washing cycles in the laboratory-

scale Linitest device, with the third cycle showing significantly lower release than the first for all fabric types. For most fabrics, the release between the first and second wash remained similar, except for the staple fibre polyester fabric, which showed a decrease after the first wash. SEM analysis suggested that this decline could be due to the removal of partially detached fibres present on the surface before washing.

Among all samples, the cotton-polyester fabric released the highest number of MFs. This aligns with existing studies reporting that natural fibres, particularly cotton, tend to shed more due to their irregular morphology, shorter lengths, and swelling behaviour during washing [3], [17]. Additionally, SEM images of the fabric's laser-cut edges showed that cotton bundles were only partially sealed by the molten polyester, increasing the risk of fibre shedding under mechanical stress. While staple fibre polyester also contains shorter fibres, its MF release remained comparable to other polyester variants, suggesting that fibre length alone does not explain the higher release observed in the cotton blend.

Additionally, the MF release measured for the conventional polyester fabric (341 ± 77 MFs/g) aligns closely with the value reported by Cai et al. [18] for similar plain-woven laser-cut polyester filament yarn fabrics (370 MFs/g), supporting the validity of the experimental procedure.

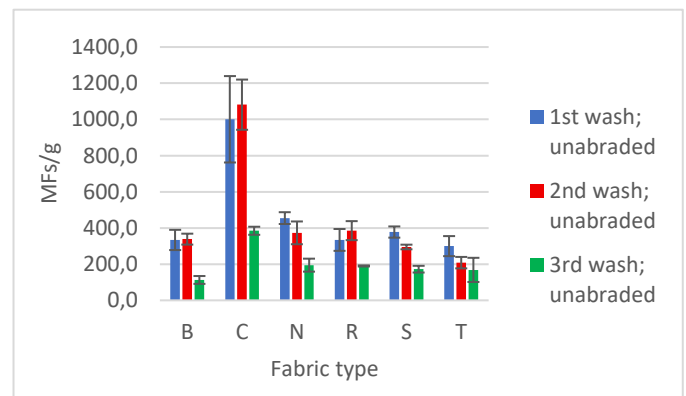


Figure 6: Released number of MFs/g of fabric during the first (blue), second (red), and third (green) washing cycles in the Linitest washing machine. The fabric samples include: biodegradable polyester (B), cotton-polyester (C), conventional polyester (N), recycled polyester (R), staple fibre polyester (S), and entangled polyester (T).

Figure 7 shows the MF release per gram of fabric across three combined abrasion and washing cycles. High variability was observed in the first cycle for several fabrics, particularly the biodegradable, conventional, and entangled fibre polyester, as indicated by the large error margins. This suggests inconsistent surface damage during the initial abrasion step. As a result, no clear trend could be established across sequential cycles for the biodegradable and conventional polyester fabrics.

The cotton-polyester blend showed a peak in MF release during the second cycle, followed by a decline in the third. In contrast, recycled polyester fabrics released fewer fibres in the first cycle, which is consistent with the SEM observations that showed less surface damage compared to the biodegradable polyester fabrics. MF release trends for some fabrics, such as staple fibre and entangled fibre polyester, could not be fully assessed due to high variability and sample limitations.

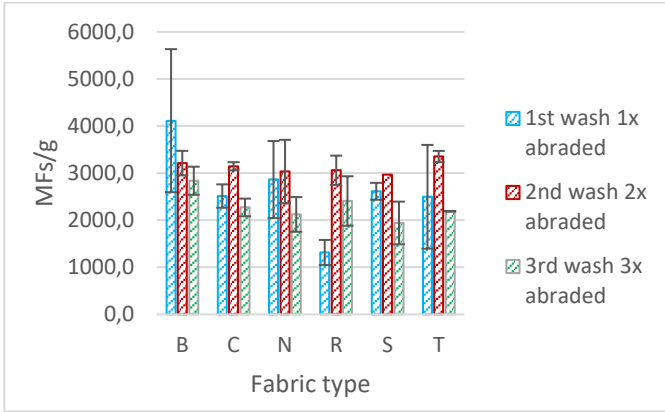


Figure 7: Released number of MFs/g of fabric during the first (blue), second (red), and third (green) washing and abrasion cycles. The fabric samples include: biodegradable polyester (B), cotton-polyester (C), conventional polyester (N), recycled polyester (R), staple fibre polyester (S), and entangled polyester (T).

Figure 8 highlights the difference between fabric treatments by showing the average release of MFs for unabraded and abraded fabrics. It is important to note that while unabraded samples were subjected to three consecutive washing cycles, the abraded samples underwent three abrasion and washing cycles, in which abrasion was applied before each wash. Therefore, the higher MF release from abraded samples reflects the cumulative impact of repeated surface wear combined with washing.

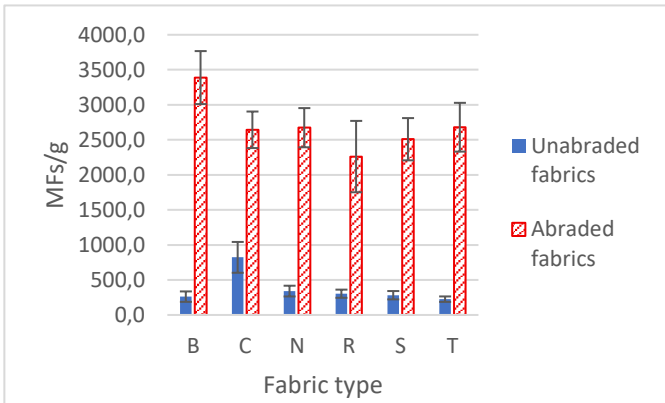


Figure 8: Average MF release over the three studied cycles of the unabraded (blue) and abraded (red) samples. These samples include: biodegradable polyester (B), cotton-polyester (C), conventional polyester (N), recycled polyester (R), staple fibre polyester (S), and entangled polyester (T).

Domestic washing tests did not yield conclusive results. No significant difference in MF content was found between the blank wash and the washes containing fabric, indicating residual contamination in the machine and insufficient rinsing between tests. Additionally, the small fabric load relative to the water volume (approximately 130 g of fabric in 50 L) likely diluted the MF concentrations below detectable levels. These limitations made it impossible to assess differences in MF release based on fabric type or washing cycle by using the domestic washing machine setup.

3) Environmental impact of the MF release

Based on the synthetic fibre consumption per capita estimated by Boucher et al. [5], the MF release measured for unabraded conventional polyester fabrics in this study suggests that approximately 5 million fibres per person could be emitted

during the first two household washes. In a scenario where fabrics are prewashed during manufacturing, this estimate decreases to around 3.5 million fibres, assuming effective filtration of the pre-wash effluent.

4) Nanoparticle distribution analysis

The Dynamic Light Scattering (DLS) analysis of the wash water filtrates, illustrated in Figure 9, showed that samples from the Linitest tests with polyester fabrics and the blank (distilled water) had an average particle size of approximately 150 nm and moderate polydispersity (PDI 0.2–0.5). In contrast, filtrates from cotton-polyester fabrics washed in the Linitest had a higher average particle size of approximately 600 nm and a broader size distribution. Samples from the domestic washing machine showed the highest particle sizes and PDI values, suggesting a high level of heterogeneity and likely contamination. This aligns with optical microscopy observations, which also revealed non-fibrous particulate contamination.

Due to the likely contamination in the domestic wash samples, the DLS results for these samples are difficult to interpret reliably. In contrast, the Linitest samples showed more consistent and interpretable particle size and dispersity patterns, making them more informative for assessing potential nanoparticle release from the fabrics in this study.

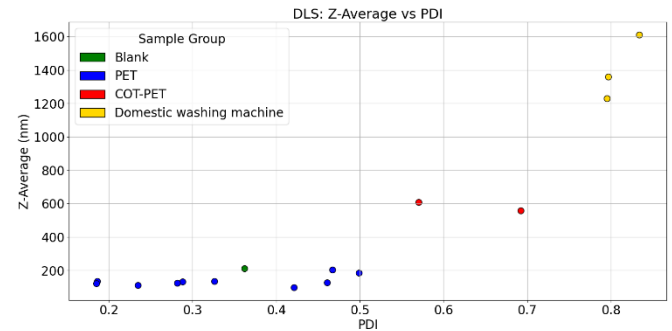


Figure 9: Dynamic Light Scattering (DLS) results showing the relationship between the average particle size (Z-average) and the polydispersity index (PDI) of the wash effluent filtrates. The blank sample (only distilled water) is indicated in green, the wash effluent filtrates of the polyester fabrics washed in the Linitest in blue, the cotton-polyester blend fabrics washed in the Linitest in red, and the filtrates of the wash water effluent from the domestic washing machine in yellow.

IV. CONCLUSIONS

This study demonstrated that material composition and surface abrasion significantly influence the release of microfibrils (MFs) and nanoparticles from plain-woven textile fabrics. Among the unabraded samples, the cotton-polyester blend released the highest number of MFs, possibly due to cotton's swelling behaviour during washing and the incomplete edge sealing observed on the SEM images. Recycled polyester fabrics performed similarly to, or in some cases better than, conventional polyester, suggesting that recycling polyester does not inherently increase the MF release. Based on scaled estimates, unabraded polyester fabrics may release approximately 5 million MFs per person over the first two household washes, with prewashing at the manufacturing stage potentially reducing this to around 3.5 million, assuming effective filtration of the prewash effluent.

These findings highlight the potential of production-stage interventions and material selection to reduce MF emissions. Recycled polyester, in particular, shows promise as a more sustainable alternative that maintains low MF shedding performance. Future research should investigate fibre shedding from complete garments under real-world conditions, including environmental factors such as UV exposure, microbial activity, and fluctuating moisture levels. Additionally, the broader molten edge observed in recycled polyester may offer design opportunities to encapsulate exposed cotton fibre bundles in blended fabrics, potentially reducing fibre shedding at cut edges.

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Chapter 1: State of the art

1.1 INTRODUCTION TO MICROPLASTICS

Thompson et al. [1] introduced the term “microplastics” for the first time in an article published in Science [2]. However, the term hasn’t been clearly defined yet. Nowadays, according to ISO/TR 21960:2020 [3] and ISO 4484-2:2023 [4] standards, plastic particles larger than 1µm and smaller than 5 mm are categorised as *microplastics*, while particles smaller than 1µm are categorised as *nanoplastics* [2]. Microplastics larger than 1mm can also be categorised as *macroplastics* [2].

1.1.1 Microplastics

It is impossible to imagine a life without plastics nowadays. This is due to their performance, low density, and versatile applications in the textile, construction, automotive, medical and many other industries [5]. Since 1950, plastic production increased from 1.7 million tonnes to 400 million tonnes in 2015, leading to an estimated 6300 million tonnes of plastic waste generated globally, with 79% accumulating in landfills or natural environments [5].

Microplastics can be classified as “primary” or “secondary” depending on their source [6]. Primary microplastics are directly released into the environment. They are either intentionally produced for commercial use, such as cosmetics, or originate from the abrasion of large plastic objects during manufacturing, use or maintenance [7]. Microplastic fibres (MFs) shed during the manufacturing, use and washing of textiles are primary microplastics [7].

Secondary microplastics originate from the degradation of larger plastic items such as discarded plastic bags or lost fishing nets. This degradation occurs due to exposure to photodegradation and other weathering processes in the marine environment [7].

1.1.2 Microplastic fibres, a dominant component of microplastic pollution

Global fibre production has increased from 57 million metric tons (MMT) in 2000 to 111 MMT in 2020. It is estimated to be 145 MMT in 2030, with synthetic fibres being the most popular, accounting for 65% of worldwide output in 2020 [8]. This increase in the production and consumption of textiles is due to the increasing population and the fast fashion industry [8].

When the distribution of MFs along shorelines worldwide was studied by Browne et al. [9], they found a higher number of MFs in densely populated areas. Their data suggests that MFs reach the aquatic environment through sewage water contaminated with fibres from washing clothes in domestic laundry. These MFs were mainly of synthetic origins, such as polyester (56%), acrylic (23%), polypropylene (7%), polyethylene (6%) and polyamide (3%) [10]. Textile laundering is also considered a significant source of MFs entering the environment by Napper et al. [11] and Tiffin et al. [12]. Additionally, Boucher et al. [7] estimated that 35% of primary microplastics in the ocean originate from textile laundering, illustrated in Figure 1.

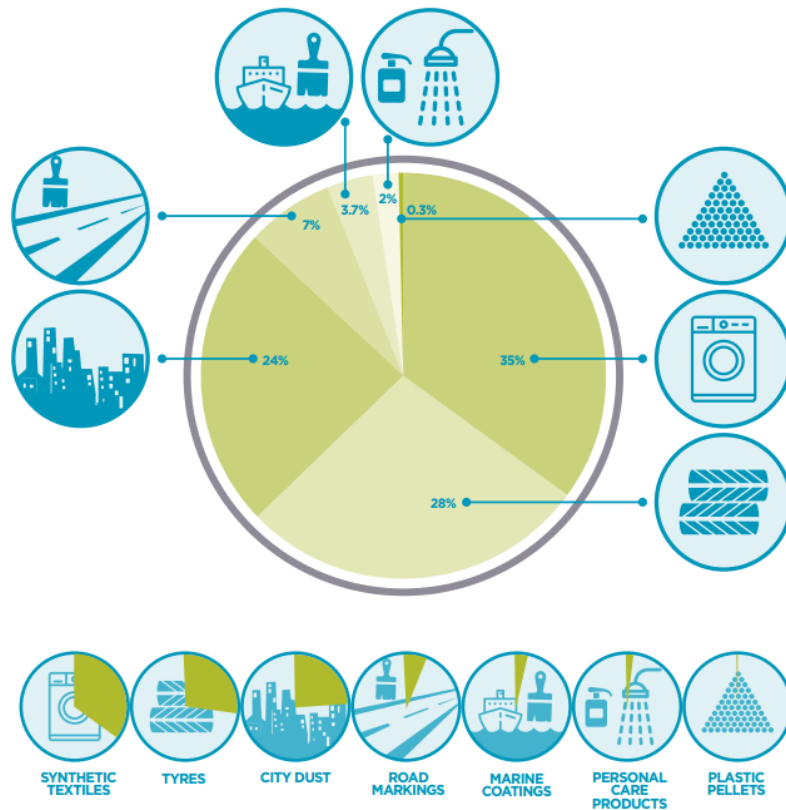


Figure 1: Schematic illustration of the global release of primary microplastics to the oceans [7].

Although primary sources, such as domestic laundry, remain the most significant known contributors to MF pollution in aquatic environments, it should be noted that secondary MFs originating from textile waste in landfills and fishing nets also contribute to global pollution [13]. The sources of the MFs and the pathways they follow to reach marine, freshwater and terrestrial ecosystems are shown in Figure 2 [14].

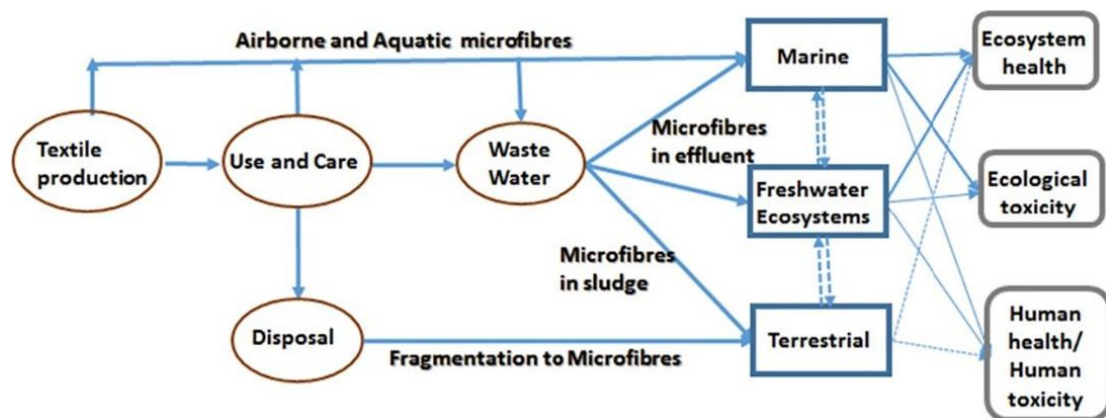


Figure 2: Textile microfibre supply chain [14].

1.1.3 Influence on Ocean and Terrestrial Life

Microplastics are highly ubiquitous in aquatic and terrestrial environments. They have been found in seawater, lake water, sediments, terrestrial soils and living organisms [8], [14], [15]. These small particles are ingested by marine, freshwater and terrestrial organisms, which is not only a threat to these organisms but also to humans [8], [14], [16].

Microplastics can disrupt the feeding and digestion of organisms, which substantially threatens their metabolic activities [8], [17]. These microplastics are transferred up through the food chain, resulting in an accumulation at the top [18]. Additionally, the small particles can penetrate biological barriers, leading to an accumulation in vital organs [14], [19]. This is shown by Mattsson et al. [18] and Barboza et al. [20], who investigated the presence of plastic particles in fish brains. They concluded that these particles can cross the blood-to-brain barrier and accumulate in the brain, resulting in brain damage and behavioural disorders.

Microplastics have a large surface area, which increases their ability to adsorb pollutants such as metals and hazardous organic pollutants such as polychlorinated biphenyls (PCBs), dichlorodiphenyltrichloroethane (DDT) and polycyclic aromatic hydrocarbons (PAHs) [16], [19]. This means that microplastics are not only accumulating in ecosystems, but also the contaminants attached to these microplastics [16]. Figure 3, originating from a study performed by Rai et al. [16], shows how the fragmentation of microplastics from larger debris can result in microplastics that adsorb pollutants, which affect the ecosystem. They focused on the effects on soil and concluded that the soil health and fertility were impacted because there was a change in soil pH, porosity, microbial enzymatic activity and water-holding capacity. The enzymatic activity in the soil decreased because the bacterial and fungal diversity was reduced by the presence of these microplastics, which can impact their physiology and metabolism [21]. This disrupts the nutrient cycling of plants, leading to a reduced uptake of substances by plant roots [22]. When the plant growth reduces, the crop yields also reduce, which impacts food security and can result in hunger, indirectly affecting human health [22].

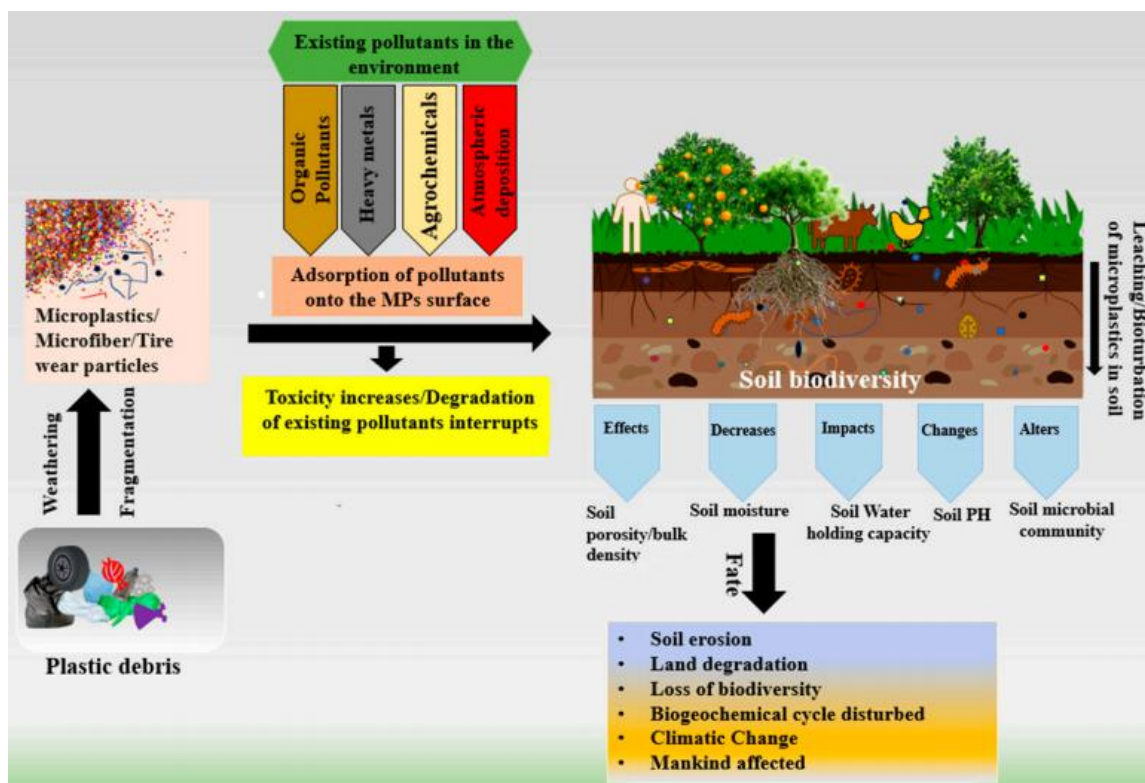


Figure 3: Adsorption of pollutants onto microplastics and their impact on the ecosystem [16].

1.1.4 Danger to human health

Humans are exposed to MFs through air, water and food. These MFs can be inhaled, ingested or pass through the skin by dermal contact, which is shown in Figure 4 [8]. There is a consensus among researchers that microplastics have been detected in the human body. However, there is still a need

to increase the understanding of the potential impacts of these plastics on human health [14], [23]. Although further research about the impact of MFs on humans is necessary, it is known that these MFs can carry dangerous pollutants, accumulate through the supply chain and can enter vital organs, making them a threat to human health [14], [16], [18], [24].

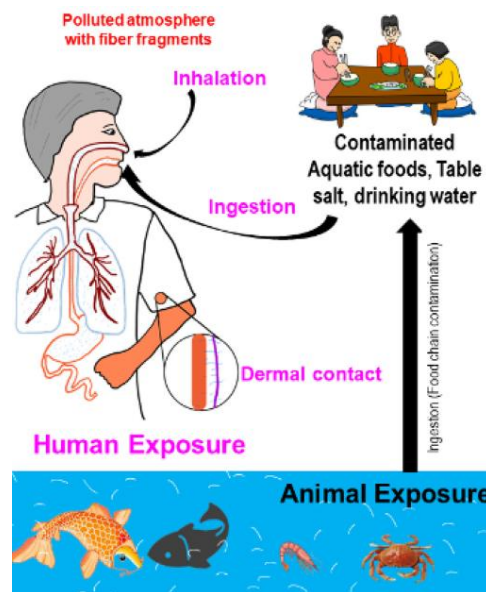


Figure 4: Human exposure to MFs [8].

1.2 FABRIC STRUCTURES

1.2.1 Natural and synthetic fibres

Textile fibres can be categorised into two main categories, natural and synthetic/manmade fibres, depending on their origin. Firstly, the natural fibres originate from animals, vegetables/plants, or minerals. These fibres are hydrophilic due to the presence of hydroxyl groups and other polar groups, increasing the tendency to absorb water [25]. Secondly, manmade fibres can be derived from a naturally occurring source with the help of a chemical process. They are commonly considered semi-synthetic fibres. However, manmade fibres can also be derived from chemical resources, such as petroleum products, and then they are categorised as synthetic fibres [26]. Cotton is an example of a natural fibre, while polyester is an example of a manmade synthetic fibre. The categorisation of fibres with examples is shown in Figure 5.

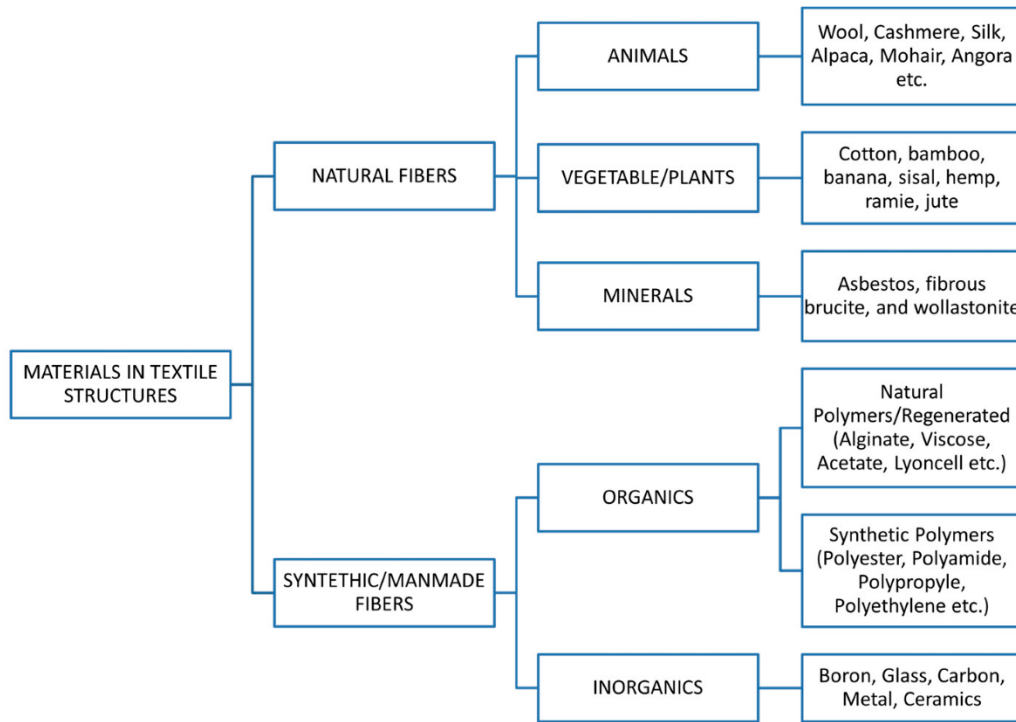


Figure 5: Commonly used textile fibre materials [25].

Material influence on microfibre shedding

Research regarding MF release often lacks clear constants and variables, making it difficult to compare the reported quantities of MFs released in different studies. One factor contributing to this issue is the variety of fibre types used, as fibres of natural origin have been reported to shed more MFs than synthetic fibres [2], [27], [28]. Natural fibres like cotton tend to have a more irregular shape and shorter lengths than synthetic fibres like polyester, influencing their MF shedding rate [2], [29]. Haap et al. [30], for example, studied the MF release of polyester-cotton blends and reported that $86 \pm 3\%$ of the released MFs originated from the cotton, while $14 \pm 3\%$ originated from the polyester. This is shown in Figure 6, where the number of shed MFs for the fibre types is expressed in MFs per gram of fabric.

In contrast, Napper et al. [11] reported that synthetic fibres shed more MFs than natural ones. However, this variation in results could be due to the use of commercially available sweatshirts, which may have varying constructions, dyes, and finishes [2].

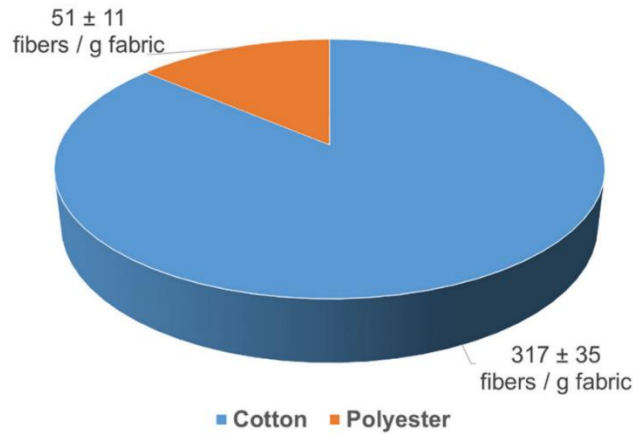


Figure 6: Number of polyester and cotton MFs shed per gram of 50%/50% polyester/cotton fabric (analysed with dynamic image analysis) [30].

Biodegradable fibres

Biodegradable fibres are made of polymer materials that can be broken down into carbon dioxide, methane, water, inorganic compounds or biomass [31], [32]. Soil is the primary medium for this decomposition, with the enzymatic activity of microorganisms as the dominant mechanism for this biodegradation [31]. However, Zambrano et al. [33] studied the aerobic biodegradation of MFs released during laundry in aquatic environments, including lake water, seawater and activated sludge by comparing the oxygen uptake with the theoretical oxygen demand (ThOD). Their methodology is illustrated in Figure 7. They evaluated the biodegradation in lake water and activated sludge by measuring the biochemical oxygen demand (BOD) according to ISO 14851 [34], while the carbon dioxide production over time was monitored according to ASTM D6691 [35] to determine the biodegradation in seawater.

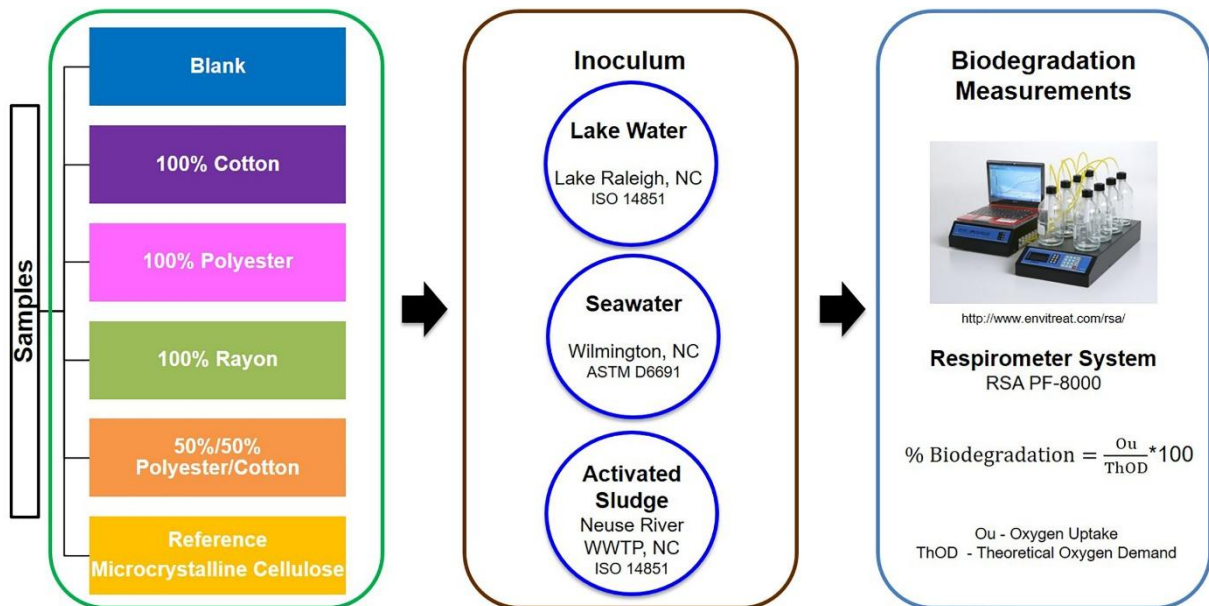


Figure 7: Experimental flowchart of the samples, inoculum, test methods and biodegradation measurements used in a study by Zambrano et al. [33].

As shown in Figure 8, they concluded that 70% of the cotton and rayon yarns were biodegradable in activated sludge and lake water, while this was only 50% in seawater. The biodegradation potential for all their performed tests was: microcrystalline cellulose > cotton > rayon > polyester/cotton >> polyester. They concluded that polyester was not noticeably biodegradable [33].

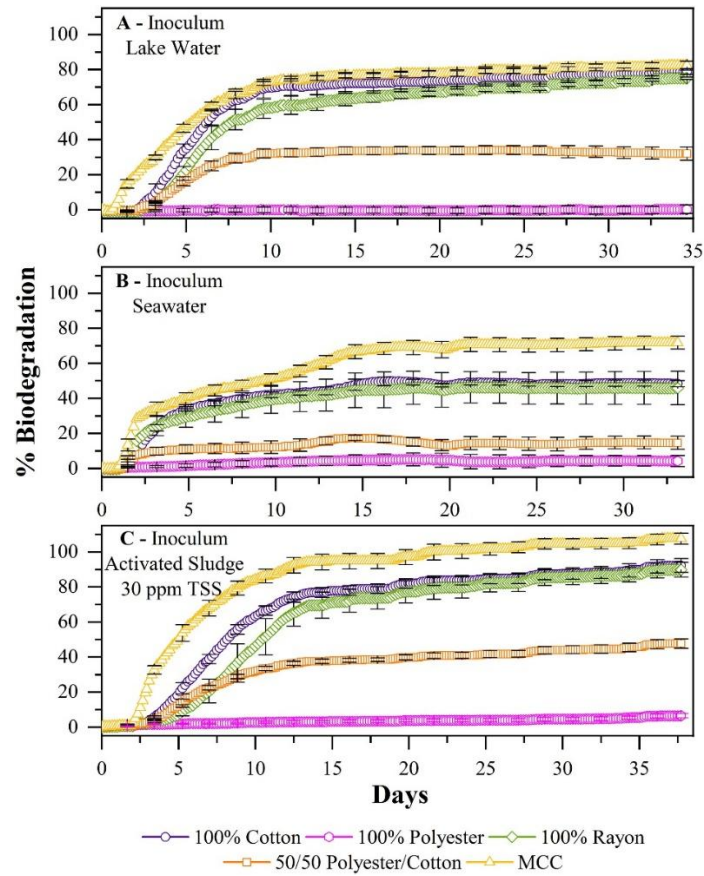


Figure 8: Biodegradation curves of the textile yarns in the following inocula: (A) Lake water, method ISO 14851; (B) Seawater, method ASTM D6691; (C) Activated sludge, method ISO 14851 [33].

Additionally, Zambrano et al. [36] investigated the influence of textile treatments such as dyes, softeners, durable press finishes, and water repellent coatings on the biodegradability of cotton. Their results, illustrated in Figure 9, showed that while these treatments alter the biodegradation rate in aquatic environments, they do not significantly impact the final extent of biodegradation.

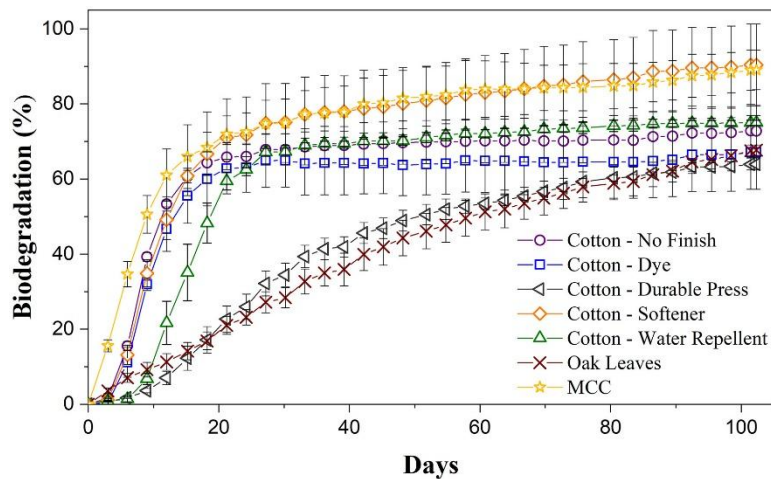


Figure 9: The effect of finishes on the biodegradation of cotton MFs in 30 ppm TSS of activated sludge according to the standard ISO 14851:2019 method [36].

Virgin and recycled polyester yarns

Akyildiz et al. [37] and özkan et al. [38] stated that recycled polyester yarns emit more MFs during washing than virgin polyester yarns. They attributed this to the lower fibre strength due to the shorter polymer chains of mechanically recycled polyester. However, this conclusion is not consistent with other studies [39], [40], [41], [42]. Akyildiz et al. [37] and özkan et al. [38] both did not mention the recycled content of the polyester yarns used, making it hard to compare their results with other research. Frost et al. [41], for example, showed that the percentage of recycled content matters. They concluded that the 70% recycled polyester yarns shed significantly fewer MFs than the 40% recycled polyester ones. At the same time, they also showed that the MFs of the 70% recycled were longer in mean length than the virgin and 40% recycled polyester. To conclude, the majority of the studies in the literature agree that recycled polyester yarns release more MFs during washing, but that the difference is statistically insignificant [2], [39], [40], [41], [42].

1.2.2 Yarn type

A textile yarn is a long, fine fibre strand of parallel continuous filaments or twisted staple fibres. These yarns can then be used to weave or knit fabrics, or be intertwined into braids, ropes or cords [43]. The type of yarn influences the shedding of microplastics. The parameters of the yarn that affect the shedding of microplastics are the twist, hairiness, length of the fibres, structure and nature of stresses built into the yarns during spinning, and the material of the fibres [44], [45], [46], [47], [48], [49].

Firstly, a high twist in the yarn reduces the microfibre shedding because a higher twist increases the fibre cohesion [47], [48], [49]. Secondly, theoretically, low hairiness should be preferred concerning microfibre shedding because fewer fibres can detach from the surface. However, De Falco et al. [46], [49] did not find a relationship between the release of microplastics during washing and the hairiness of the yarns. Thirdly, shorter fibres tend to fall off more easily during washing than long fibres, meaning that staple fibre yarns release more fibres than continuous filament yarns [19], [44], [45], [46], [49].

1.2.3 Fabric types

Textile fabrics can be defined as the assembly of fibres or yarns, which can be natural or manmade. Typical techniques to assemble these yarns or fibres are weaving, knitting, and braiding [25]. To make a woven or knitted fabric, the fibres need to be converted into a yarn before they can be used to create a fabric. However, fabrics can also be produced directly from the fibres, which are called non-wovens [50]. These different textile structures are shown in Figure 10.

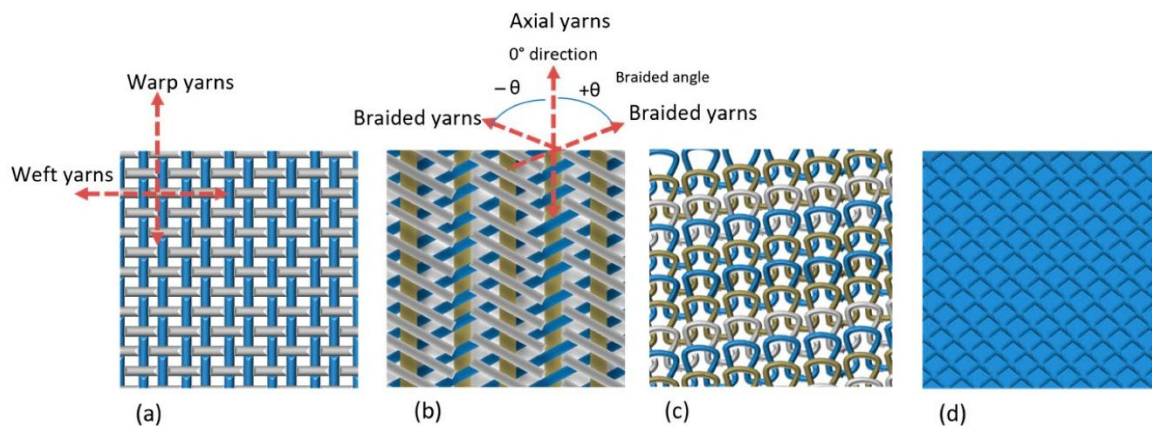


Figure 10: Schematic representation of (a) woven, (b) braided, (c) knitted, and (d) non-woven structures [25].

Weaving is the most commonly used fabric manufacturing technique. Woven fabrics are produced by interlacing two sets of yarns perpendicular to each other. The directions of these yarns are called weft and warp [50]. Woven fabrics are anisotropic and complex combinations of yarn spacings, fibre bundles, fibre orientations, yarn sizes, stacking sequences, fibre architectures and fibre volume fractions that determine their mechanical properties [25]. The yarn-yarn contact at the interlacing points, shown in Figure 11, affects the behaviour of the yarns under tensile load significantly, with more interlacing points leading to a higher fabric strength under tensile load. Additionally, this woven structure also increases the resistance to damage [25].

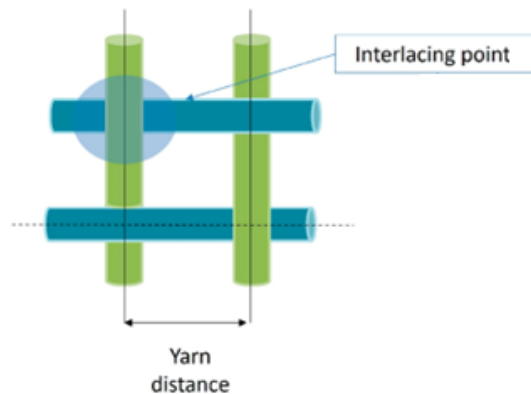


Figure 11: Schematic representation of the interlacing points in a plain woven structure [25].

1.2.4 Structure's influence on microfibre shedding

The fabric structure influences the shedding of microfibres during washing [19], [45], [47], [48], [51]. Petersson et al. [45] even concluded that the fabric structure is the most crucial factor influencing fibre release. Cui et al. [19] also noted a difference in microfibre shedding rate during laundry between different weaving structures. However, their statistical analysis showed that this difference was not large enough to be significant.

Most researchers agree that loose fabric structures shed more microfibres than tighter structures [19], [47], [48], [51]. This is confirmed by knitted fabrics shedding more MFs than woven fabrics, and plain woven structures shedding fewer MFs than satin or twill structures [19], [48], [51]. The interweaving points of the woven structure make the structure tighter, which increases the tendency of short fibres to remain at the surface during washing [19]. Plain woven fabrics have a higher interlacing coefficient and weft density than fabrics with a twill structure, which results in a tighter structure and a decrease in MF shedding [51].

However, the impact of the fabric structure on the MF release can be influenced by changing other parameters. De Falco et al. [46] concluded that their woven fabric released more microfibres during washing than their knitted fabric. This is not in line with other studies, where tight structures reduce the microfibre release [19], [47], [48], [51]. It is possible that in the case of De Falco et al. [46], their choice of yarn types influenced their results. They chose short staple fibres of 35 and 50 mm in length for their woven fabrics, while their knitted fabrics were made out of continuous fibres. The short fibres tend to slip away more easily from the yarn during washing.

1.2.5 Edge sealing

Fibre release at the edges of the fabrics due to the cutting can be avoided by folding the edges over twice and sewing them with a white thread. A filament thread with a different colour from the sample fabrics is chosen to minimise the microfibre release from the thread and not interfere during the counting of the microfibres [12], [44], [46].

However, Cai et al. [52] showed that laser-cut samples reduce the number of released MFs 3-21 times in comparison to scissor-cut samples. They found that, on average, 84% of the MFs released originate from the edges, which highlights the importance of the edge-to-surface ratio when comparing different studies on MF release. Additionally, Rathinamoorthy et al. [53] also stated that laser and ultrasonic cutting methods reduce the MF release by a factor of 20 in comparison to conventional scissor-cutting methods. Finally, Yang et al. [54] and Nowack et al. [55] used laser cutting to prepare their polyester fabrics for abrasion with the Martindale according to the ISO 12947-2:2016 [56] standard, which requires a small circular-shaped fabric with a diameter of 38 mm and a big one with a diameter of 140 mm.

1.3 WASHING AND ABRASION

1.3.1 Washing machine type

Two categories of washing machines can be used to investigate the release of MFs from textiles: domestic washing machines and laboratory-scale washing machines. The effect of the domestic washing machine type on the release of microfibres during washing was investigated by Hartline et al. [57]. They compared front- and top-load conventional laundering machines. They concluded that more fibres were released in the top-load washing machine because it applies a higher amount of mechanical action. However, the use of domestic washing machines to investigate the MF release of textiles has multiple drawbacks. Firstly, they have poor consistency and reproducibility regarding water intake/outflow, spin speeds and cycle duration [12]. Secondly, multiple wash samples are necessary to facilitate statistical analysis. However, to simulate a standard domestic wash load, each experiment should use 3 to 5 kg of test fabric, which means that hundreds of kilograms of fabric would be necessary to facilitate statistical analysis [12]. Finally, the large volume of effluent water leads to practical challenges in filtering the effluent, meaning that smaller aliquots of the effluent should be taken and analysed. However, this method assumes that there is a homogeneous distribution of fibres in the effluent and that the samples inside the aliquots are representative of the whole washing effluent [12].

The experimental variables among studies can be minimised by utilising lab-scale washing machines such as the Gyrowash or the Linitest [12]. These machines consist of a rotating shaft with stainless steel containers in a heated bath. The test samples are added to these containers along with stainless steel balls to replicate the agitation and abrasion that occurs inside a domestic washing machine. The main advantage of this machine is the reproducibility of the test conditions, and that the whole content of the container can be filtered [12]. These washing devices were used in multiple studies [30], [42], [44], [46], [47], [58], [59]. However, due to the small size of the containers, these devices only offer the possibility to test fabrics and not full-scale garments [12].

1.3.2 Subsequent washes

There is an agreement among researchers that the number of MFs released during washing decreases in a series of successive washes [27], [58], [60], [61], [62], [63], [64], [65]. This decreasing trend is visible in Figure 12, originating from a study performed by Cesa et al. [61]. Additionally, according to Pirc et al. [60], the release stabilises and remains constant at approximately 0.0012 wt%. However, Dubaish and Liebezeit [66] reported a weight percentage release ratio of 0.039 wt%. This weight percentage varies among researchers because it depends on various factors, such as fabric type, washing parameters, and filter type [57], [60], [61], [66].

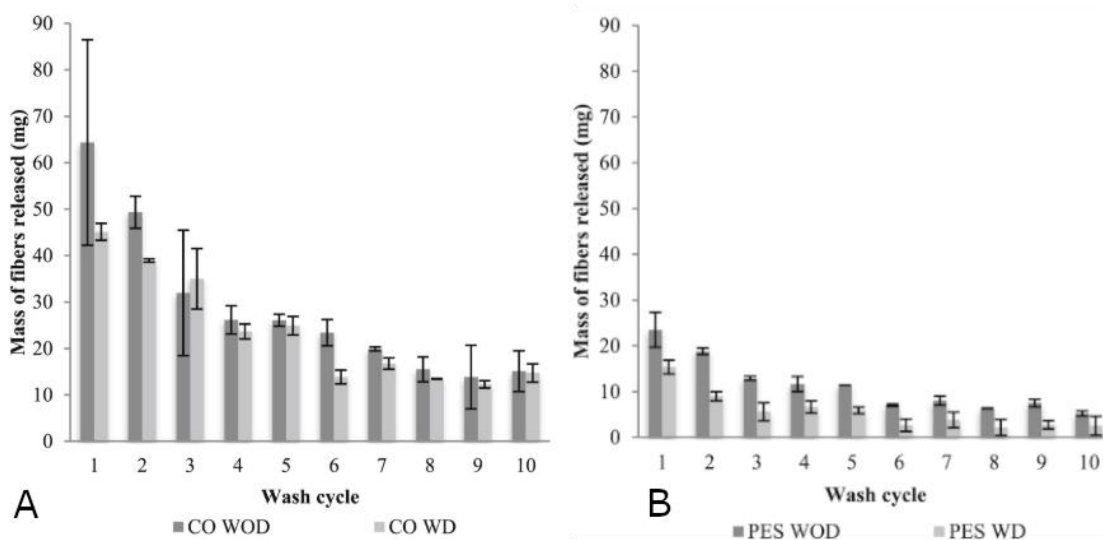


Figure 12: Mass of the MFs released in 10 sequential washes with (WD) and without (WOD) detergent for: (A) cotton and (B) polyester [61].

1.3.3 Washing parameters

Multiple washing parameters can be varied during washing, including temperature, water consumption, spin speed, pH, mechanical action, time, and water hardness [8], [19], [46].

Temperature

The articles in which the MF release during washing was studied, and the corresponding temperature that was used, are stated in Table 1. Most researchers conclude that higher washing temperatures lead to a higher MF release rate [8], [19], [44], [48], [59], [65], [67]. However, there is still uncertainty regarding the effect of temperature on MF release, as multiple studies, such as those performed by De Falco et al. [46], Cui et al. [19], and Hernandez et al. [44] who concluded that a higher washing temperature leads to a higher release of MFs, showed that there was no significant statistical difference in microfibre release between the tested washing temperatures.

Zambrano et al. [59] also concluded that the influence of temperature on the release of microfibres was not statistically significant for polyester, rayon, and a polyester/cotton blend. However, they state that this isn't the case for cotton, where there was a statistically significant difference between the different washing temperatures. They associate this difference with the swelling of the cellulose-based cotton fibres in water at increased temperature [59]. This is in accordance with Periyasamy et al. [8], who state that the swelling of the cellulosic fibres increases the free space between the fibres, inducing the mobilisation of shattered fibres. These shattered fibres are then shed from the fabric's surface due to exposure to the sheer force and mechanical action in the washing machine. However, synthetic fibres are hydrophobic, which reduces their tendency to swell in water [8], [59].

Table 1: Literature articles about the investigation of microplastic release, with the fabric type and temperature they used for washing [12].

Article	Fabric type	Washing temperature (°C)
Belzagui et al. [68]	Polyester, polyester-elastene, acrylic-polyamide	Ambient
Hernandez et al. [44]	Polyester, polyester-spandex	25, 40, 60, 80
Zambrano et al. [59]	Cotton, rayon, polyester, polyester-cotton	25, 44
Cotton et al. [69]	Cotton, polyester, polyester-cotton-rayon	25, 40
Pirc et al. [60]	Polyester	30
Napper et al. [11]	Polyester, acrylic, poly-cotton	30, 40
Hartline et al. [57]	Polyester, polyester-nylon	
Yang et al. [28]	Polyester, polyamide, acetate	30, 40, 60
Browne et al. [10]	Polyester	40
Sillanpää et al. [27]	Polyester, cotton	
Jönsson et al. [42]	Polyester	
De Falco et al. [48]	Polyester, polyester-cotton	
Haap et al. [30]	Polyester-cotton	
De Falco et al. [46]	Polyester, polypropylene	40, 60
Almroth et al. [47]	Polyester, nylon, acrylic	60

Washing load

Kelly et al. [58] and Rathinamoorthy et al. [70] state that the water-volume-to-fabric ratio is the most influential parameter for microfibre release during washing, with a high water-volume-to-fabric ratio leading to a higher microfibre release. Additionally, Volgare et al. [71] noticed that low washing loads can hide the effect of using different detergents and laundry additives on the MF release. As a consequence, they concluded that the washing load should be considered to estimate the MF release and to account for the high variability among the data reported in the literature.

Mechanical agitation

High spin speeds increase the agitation inside the washing machine, which results in more friction and thus increases the microfibre release [12], [58]. Additionally, steel balls can be added to simulate the increase in agitation, which increases the friction inside the washing machine [12], [19], [44], [46]. Finally, when the time of a washing program is extended, the microfibre release increases because the fabrics are exposed to friction over a longer time [19].

Detergents and softeners

The use of detergents has been found to increase the microfibre release during washing significantly [44], [46], [47], [59], [65]. Hernandez et al. [44] state that the detergent composition (liquid or powder) or overdosing a detergent does not significantly affect the microplastic release. In contrast, De Falco et al. [46] stated that powder detergent results in a higher microfibre release than liquid detergents. De Falco et al. [46] hypothesise that the powder detergents contain inorganic

compounds such as zeolites, which could cause friction to the fabrics. However, they noted a high error in their results of the powder detergent tests due to the formation of a thick layer on the filter, which increased the difficulty of counting the microfibrils. The formation of this layer is a recurring problem in other studies as well, such as Browne et al. [10], who chose not to use detergent or conditioner to avoid filter blocking. Nevertheless, Pirc et al. [60] concluded that the effect of detergent and fabric softener on the MF release is relatively small, while Kelly et al. [58] stated that detergent has no effect at all. Conversely, Cesa et al. [61] concluded that the use of detergent decreased the MF release during washing. These conflicting findings highlight that, while several studies suggest a significant influence of detergent use on MF release, there is currently no clear consensus in the literature regarding this relationship.

1.3.4 Fabric wear

Multiple researchers showed that fabric wear increases MF emissions [45], [54], [55], [57]. Nowack et al. [55] and Yang et al. [54] investigated the effect of abrasion of polyester textiles on the formation of MFs and fibrils. Nowack et al. [55] performed abrasion tests on interlock and fleece polyester fabrics with a Martindale tester. The extracted fibres were analysed with SEM. They identified two distinct fibre morphologies, shown in Figure 13. The MFs had a length between 200 and 800 μm , while the length of the fibrils ranged from 30 to 150 μm . The diameter of the MFs was the same as the diameter of the original fibres, while the diameter of the fibrils was much smaller. They concluded that the number of MFs increased by a factor of 5 to 30 for abraded samples in comparison to non-abraded samples, while the number of released fibrils increased by more than a factor of 200 in the case of the abraded samples.

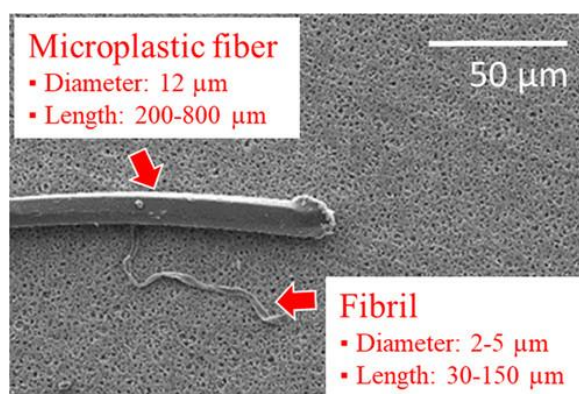


Figure 13: SEM image of microplastic fibre (MF) and fibril [55].

Yang et al. [54] performed a similar experiment. However, they also investigated woven polyester fabrics. They concluded that a higher number of rubs results in an increase in the total number of MFs and fibrils released. The visible pilling on the textile surface, shown in Figure 14(A, B), is an indicator for the formation and release of MFs and fibrils. Their data showed that the plain structure resulted in the highest release in the woven category. Additionally, they saw that the warp yarns remained undamaged, while loose fibre ends appeared at the intersection of the warp and weft yarns, which is visible in Figure 14(B). The fibre ends before and after abrasion were investigated by Nowack et al. [55] and are shown in Figure 14(C, D), where it is visible that the ends of the fibres in Figure 14(D) are fractured.

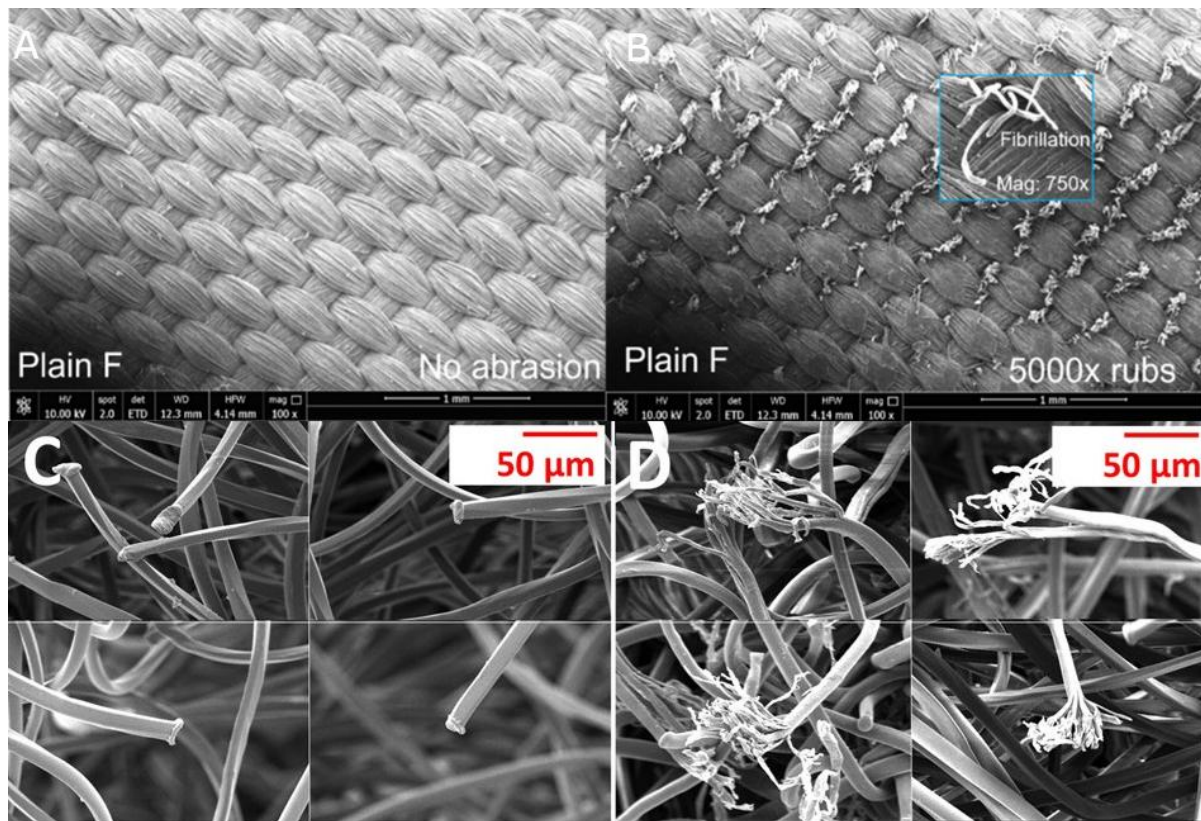


Figure 14: SEM images performed by Yang et al. [54] (A&B) and Nowack et al. [55] (C&D): (A) 100x magnification of non-abraded plain woven polyester filament yarn fabric surface, (B) abraded plain woven polyester filament yarn fabric surface, (C) 2000x magnification of solid fibre ends of non-abraded fleece polyester fabric, and (D) 2000x magnification of fibrillated fibre ends of abraded fleece polyester fabric.

1.4 FILTRATION AND MICROFIBRE ANALYSIS

Although there are some exceptions, such as De Falco et al. [46] who used a peristaltic pump, the majority of the researchers used a vacuum pump to assist the filtering [12], [44], [45], [68]. However, many of these studies differ in the type of filter used and the method of MF quantification.

1.4.1 Filter type and pore size

De Falco et al. [29] used subsequent filters with a decreasing porosity to filter wastewater originating from washing commercial clothes in a domestic washing machine, and investigated the number and length of the MFs on the filters. They used filters with a pore size of 400, 60, 20 and 5 µm and found that the most considerable fraction of shed MFs was retained by the filters with a pore size of 60 µm, which is visible in Figure 15. Additionally, the large variation in filter use among researchers is visible in Table 2.

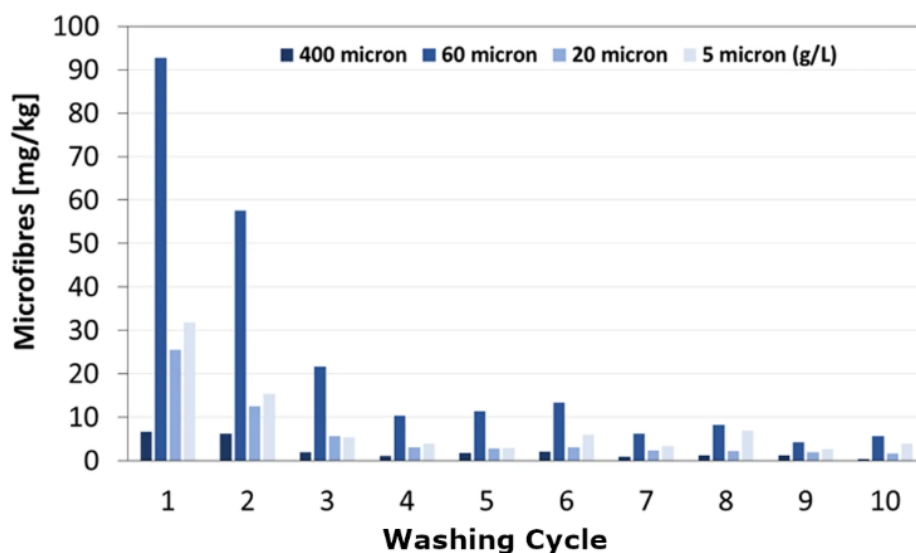


Figure 15: Microfibrils recovered on filters with 400, 60, 20 and 5 µm pore sizes across multiple washing cycles by De Falco et al. [29].

Table 2: Literature comparison of the chosen filter type and pore size of microfibre release studies.

Article	Filter type	Pore diameter (µm)
Belzagui et al. [68]	Polyamide filters (Millipore NY20)	20
Cai et al. [52]	Cellulose nitrate membrane (GE Whatman)	0.45
De Falco et al. [46]	PVDF filters (Durapore, Merck Millipore)	5
De Falco et al. [29]	Not mentioned	400, 60, 20, 5
Hartline et al. [57]	Nitex nylon filters	333, 20
Hernandez et al. [44]	Whatman paper filters	0.45
Napper et al. [11]	Whatman No. 4 paper filters	20-25
Petersson et al. [45]	Glass fibre filter VWR (Cat. No. 516-0887)	1.2
Pirc et al. [60]	Removable stainless steel filter	200
Yang et al. [28]	PTFE membrane filters	5
Mahbub et al. [65]	Cellulose paper filters	50, 7.5, 2.5
Almroth et al. [47]	Glass filters (Whatman GF/C)	1.2
Berruezo et al. [51]	Glass filters	1.2
Yang et al. [72]	Polycarbonate cyclopore membrane (Whatman)	2
Sillanpää et al. [27]	Type HC Millipore	0.7

1.4.2 Quantification of microfibrils

The MF release from fabrics can be determined by measuring the difference in mass or by counting the MFs visible on the filter. Gravimetric measurements are less labour-intensive and time-

consuming than counting the fibres [12], [44]. However, particles that are not MFs are also weighed and reported as MFs, which can influence the results [68]. Nevertheless, a similar problem occurs when the MFs are counted because manual counting leaves considerable potential for counting errors, certainly when the fibres overlap in a 3-dimensional spaghetti-like structure [12]. Additionally, it is often deemed too time-consuming to analyse the whole filter area, resulting in only a selected area of the filter where the MFs are counted. This assumes that the MFs are distributed homogeneously on the filter, which might not always be the case [12].

De Falco et al. [46] used SEM analysis to determine the number of MFs on the filters. They took 21 electron micrographs along two orthogonal diameters of the circular filter, visible in Figure 16(A), of each filter sample. Every micrograph represents a rectangular area (A_r) of the filter surface. This method was chosen to make sure that the centre and borders of each filter were observed. The number of MFs in each micrograph was manually determined with ImageJ software. Finally, the method was validated by using an extended counting method where 121 micrographs were analysed, as shown in Figure 16(B).

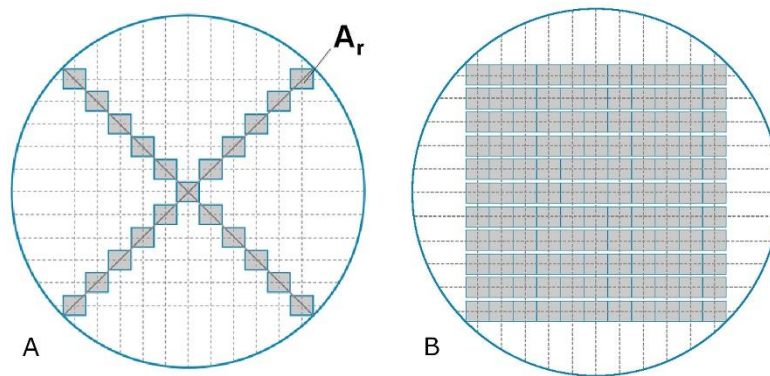


Figure 16: Schematic representation by De Falco et al. [46] of: (A) Position of the acquired micrographs (A_r) along the filter used in the counting method; (B) Positions of the 121 acquired micrographs of the filter in the extended counting method to validate the counting method.

The disadvantage of SEM analysis is that only a small part of the filter can be analysed per round [42]. This is why many researchers use optical microscopy to count the MFs on the filters [11], [37], [38], [41], [42], [44], [45], [47], [52], [53], [60], [68]. Yang et al. [28] based their method for counting the MFs on De Falco et al. [46]. However, they used an OLYMPUS SZ 61 optical stereomicroscope for observation. Additionally, they decided that they would only count the MF if more than half the volume was located in the grid. Belzagui et al. [68] also used a stereomicroscope for visual counting. They decided to change the background depending on the colour of the garment to increase the visibility of the MFs, a similar approach to Hernandez et al. [44], who used white filters for black MFs to make identification of the fibres easier.

The reported MF release rates from various studies were compared and are represented in Table 3, which also includes the type of textile, washing machine type, material, and washing conditions applied.

Table 3: Literature comparison of reported MF release rate.

Article	Textile type	Cutting method	Washing machine type	Abrasion tests	Material	Washing conditions	MF release reported
Browne et al. [9]	Commercially available garments	/	Front-load washing machine	NO	Polyester	40°C, 600 rpm	>1900 fibres/garment
Sillanpää et al. [27]					Polyester; cotton	40°C, 75 min, 1200 rpm	340 mg or 223,000 MFs/wash (polyester); 809 mg or 973,000 MFs/ wash (cotton)
Pirc et al. [60]					Polyester	30°C, 15 min, 600 rpm	0.0012 wt%
Belzagui et al. [68]					Polyester	Ambient temperature, 15 min, 1000 rpm	175 MF/g or 30 000 MF/m ²
Napper et al. [11]	Plain weave and double knit jersey	Scissor cut and sewed	Front-load		Polyester; 65% polyester/35% cotton blend	30 and 40°C, 75 min, 1400 rpm	496 030 MFs/ 6kg polyester; 137 951 MFs/ 6kg polyester-cotton
De Falco et al. [46]		Scissor cut and sewed	Linitest		Polyester	40°C, 45 min	6,000,000 MFs/ 5kg
Almroth et al. [47]		Laser cut	Gyrowash		Polyester	60°C, 30 min	9 ± 7 MFs/ 100cm ² fabric (knit); 1177 ± 135 MFs/ 100 cm ² fabric (fleece)
Cai et al. [52]	Various types of weave and knit	Laser cut or scissor cut	Gyrowash	YES	Polyester	40°C, 45 min	370 MF/g (Laser-cut plain woven filament yarn)
Yang et al. [54]		Laser cut	Gyrowash		Polyester	40°C, 45 min	4 900-640 000 MFs/g; 0-350 000 Fibrils/g

Chapter 2: Objective

The first goal of this study was to investigate the microfibre (MF) release of plain woven textile fabrics during washing in a laboratory washing machine, with a focus on the influence of material properties and fabric wear. The fabrics were always a blend of conventional polyester yarns in the warp direction, with conventional polyester, biodegradable polyester, recycled polyester, staple fibre polyester, entangled fibre polyester, or cotton yarns in the weft direction. To simulate different types of stress, two sets of experiments were conducted: one involving repeated laundering cycles only, and a second that combined laundering with an abrasion cycle in each iteration. This dual approach enabled a detailed comparison of microfibre release under distinct conditions, with surface analysis and MF quantification employed to assess the extent of the inflicted damage.

Secondly, the microfibre release of conventional polyester and cotton-polyester fabrics was determined in a domestic laundry machine to compare the microfibre release in a lab-scale washing machine with the release in washing machines used by households.

The final goal of this study was to determine whether there are nanoparticles present in the wash water effluent after filtering by studying the particle size distribution.

Chapter 3: Materials and Methods

3.1 METHODOLOGY: SCHEMATIC REPRESENTATION

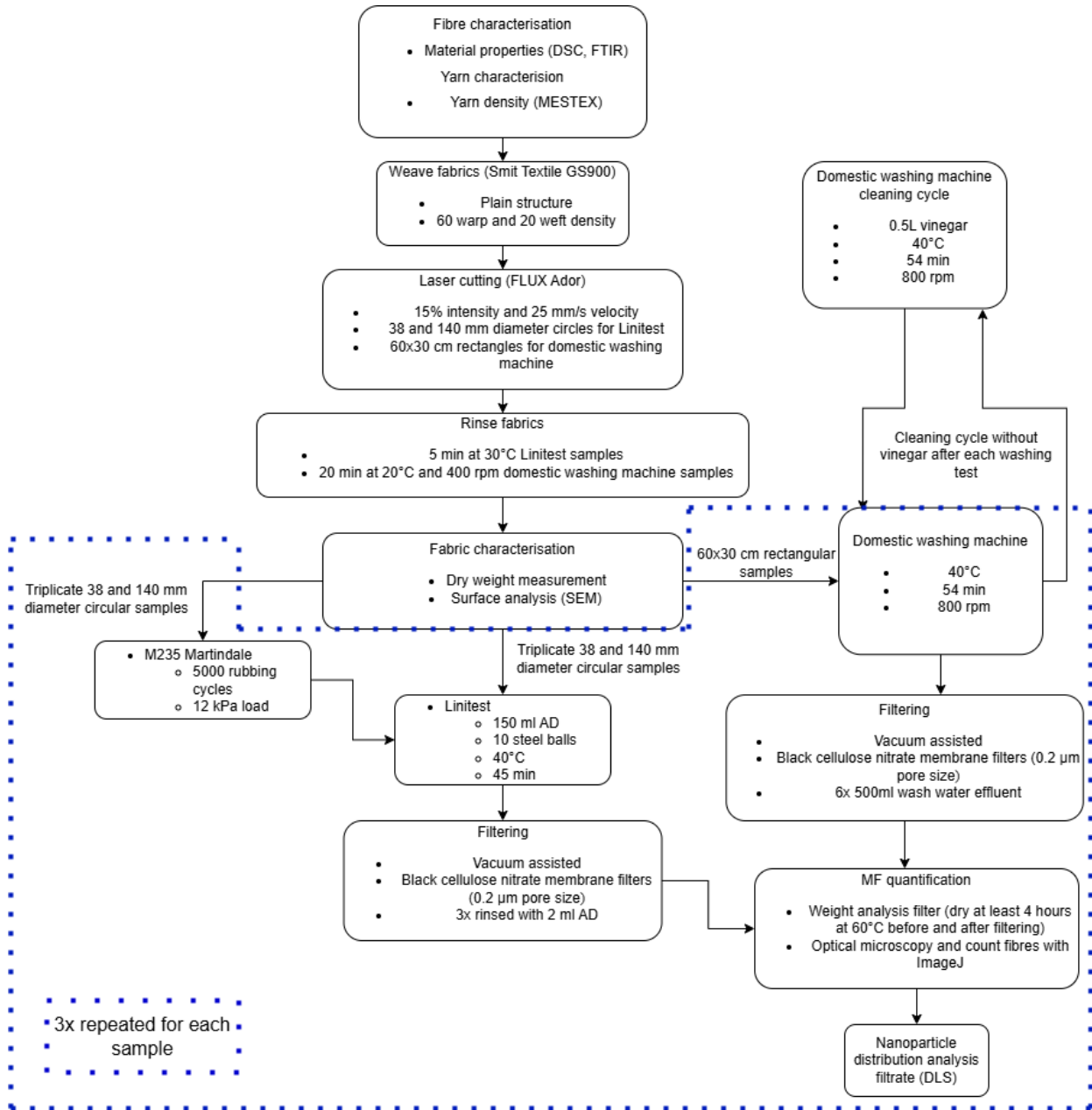


Figure 17: Schematic representation of the experimental workflow.

3.2 SAMPLE PREPARATION

3.2.1 Materials

The provided yarns consisted of conventional polyester, biodegradable polyester, recycled polyester, staple fibre polyester, entangled polyester, and cotton fibres. The specific material characteristics were unknown. However, they were investigated using Fourier Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC).

Fourier transform infrared spectroscopy (FTIR)

The University of Alicante executed the FTIR analysis in Attenuated Total Reflectance (ATR) mode. They used the Jasco FTIR 4700 IRT 5200, with ATR PRO ONE as an accessory, and chose a resolution of 4 cm^{-1} . The FTIR analysis provides insight into the material's chemical composition.

Differential scanning calorimetry (DSC)

The University of Alicante also performed the DSC analysis. The instrument type used was a DSC250, and the test was conducted in a nitrogen environment with a flow rate of 50 ml/min. The starting temperature was set at 30°C, while the ramp was set at 10°C/min until a temperature of 300°C was reached. Afterwards, the temperature was decreased at a rate of 10°C/min to 30°C. Finally, the temperature was increased a second time by 10°C/min until it reached 300°C. Controlled cooling is essential to define the thermal history of the sample before the second heating cycle.

3.2.2 Yarn characterisation

The linear density of the yarns was determined by measuring the length of the yarn and the weight corresponding to the length. The yarn needed to be placed under the correct tension to ensure reproducibility over multiple tests. This was done using the ISO 2060:1994 [73] standard method on the MESTEX.

Table 4: Yarn materials and corresponding densities.

Yarn	Yarn density (Tex)
Conventional polyester	18.1
Biodegradable polyester	17.4
Recycled polyester	17.1
Entangled fibre polyester	33.1
Staple fibre polyester	40.2
Cotton fibre	19.4

3.2.3 Fabric manufacturing and characterisation

The fabrics were woven on the Smit Textile GS900. A plain woven structure was chosen for all the fabrics, which are represented in Table 5.

Table 5: Fabric characteristics with yarn types, weft and warp density, fabric structure, and fabric density.

Sample	Warp yarn type	Warp density (yarns/cm)	Weft yarn type	Weft density (yarns/cm)	Structure	Fabric density (g/m ²)
N	Conventional polyester	60	Conventional polyester	21	Plain	150.4
S		60	Staple fibre polyester	20		208.6
T		61	Entangled polyester	21		189.7
R		59	Recycled polyester	21		151.6
B		60	Biodegradable polyester	21		151.6
C		61	Cotton	20		158.1

The following abbreviation structure was chosen to clarify the fabric type, the number of washing cycles, and the number of abrasion cycles: x_yR_zL. The “x” represents the sample type, the “y” the number of abrasion cycles, and the “z” the number of washing cycles. For example, N_1R_1L is made from conventional polyester yarns, underwent 1 abrasion cycle, and 1 washing cycle.

Cotton concentration polyester/cotton blend fabric

Firstly, two samples were cut to approximately 1g in weight. Secondly, the samples are dried in the oven for 2 hours at 100°C. Afterwards, they are placed in a bowl with silica, which ensures a moisture-free environment, and cooled for an hour. When the samples are cooled down, they are weighed and placed in an Erlenmeyer flask to which 200 mL of sulfuric acid (75%, 1.7 g/l) is added to ensure that all the cotton dissolves. The Erlenmeyer containing the fabric and the sulfuric acid solution is placed into a water bath at 50°C for 40 minutes. Afterwards, the solution is filtered to capture the polyester fibres. During the filtering process, all the polyester fibres must be removed from the Erlenmeyer, so sulfuric acid is used to rinse the Erlenmeyer and pass all the remaining fibre fragments onto the filter. To make sure that the sulfuric acid solution is completely removed from the polyester fibres, 0.5 L of water is used to rinse the fibres on the filter. Finally, the filter containing the polyester fibres is again placed in the oven for 2 hours at 100°C to dry the fibres before they are weighed. After weighing, it was determined that the polyester/cotton blend fabric contains 27 wt% cotton.

3.2.4 Scanning electron microscopy analysis

The textile samples were observed by Scanning Electron Microscopy (SEM), using a Field Emission Microscope FESEM (ULTRA 55, ZEISS). Firstly, the surface of each sample is coated with a layer of gold and palladium for 30 seconds, to make the surface conductive. The coating was performed with a BAL-TEC SCD 005 Sputter Coater. Finally, the samples were analysed with an acceleration voltage of 2 kV.

3.2.5 Fabric cutting and edge sealing

The fabric samples for the tests in the Linitest washing machine were cut with a FLUX Ador laser cutter into circles with a diameter of 140 mm and 38 mm, according to the ISO standard 12947-2:2016 [56]. The laser was set at an intensity of 15% and a velocity of 25 mm/s. The laser cutter with the cut samples is illustrated in Figure 18.



Figure 18: Flux Ador laser cutter preparing the samples for the Linitest with a diameter of 140 mm and 38 mm.

The samples for the tests in the domestic laundry machine were also cut with the laser cutter. However, the dimensions of these fabrics were 30 cm x 60 cm, shown in Figure 19.

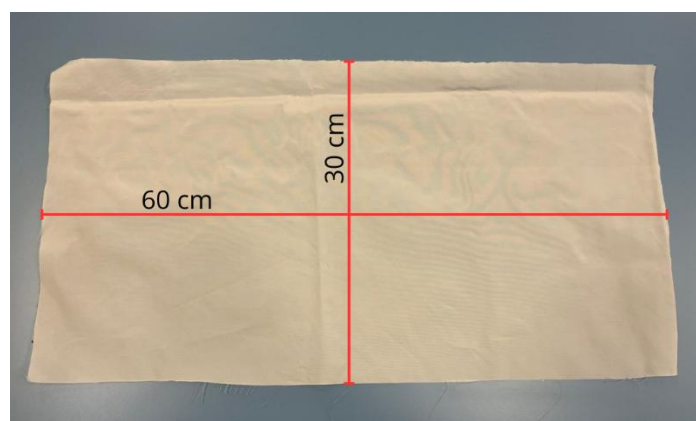


Figure 19: Laser-cut test fabric prepared for domestic laundry with indicated dimensions.

3.2.6 Contamination mitigation

During all tests, nitrile gloves and a cotton lab coat were worn to reduce the MF contamination originating from the operator. Additionally, the fabrics prepared for the Linitest are rinsed for 5 minutes in 150 mL of distilled water at 30°C to remove the MFs that could be trapped inside the fabrics due to the weaving process. The fabrics prepared for the domestic laundry testing were rinsed by using the “rapido” program for 20 min at 20°C and 400 rpm. Afterwards, the fabrics are dried in the oven at 60°C for more than 4 hours to remove moisture. Finally, the fabrics are weighed to determine the dry weight.

3.3 LAUNDRY AND ABRASION

3.3.1 Abrasion testing to simulate fabric wear

The abrasion tests were performed with the M235 Martindale, shown in Figure 20. The wear of fabrics was simulated by applying 5000 rubbing cycles with a load of 12kPa, which are the same parameters as those used by other researchers who studied the influence of abrasion with the Martindale on the release of MFs [39], [54], [55]. The rotation speed was 47.5 rpm.



Figure 20: Martindale testing to simulate abrasion.

3.3.2 Washing procedure

Laboratory-scale washing machine (Linitest)

Each washing vial contained the prepared circular sample fabrics of 38 mm and 140 mm diameter, 150 ml of distilled water and 10 steel balls. The steel balls are added to simulate abrasion during the washing process. The washing was performed in a Linitest laboratory-scale washing machine for 45 minutes, and the temperature was set at 40°C, which is similar to other studies investigating the MF release of polyester and cotton fabrics [9], [27], [30], [42], [46]. The prepared vials and washing machine are shown in Figure 21.



Figure 21: Washing vials containing the test samples, 150 ml of distilled water and 10 steel balls (left), and the Linitest laboratory washing machine (right).

Front-load washing machine

The domestic laundry tests were performed in a Samsung front-load washing machine on the “LANA” (wool) program, which takes 54 minutes, applies a temperature of 40°C, and a rotation speed of 800 rpm, as illustrated in Figure 22. Firstly, a rinsing cycle with 0.5 L of vinegar, without the addition of test fabrics, was performed to remove lime and remaining fibres from the washing machine and effluent pipe. Afterwards, another rinsing cycle was applied to make sure that all the vinegar was removed before starting the washing test.

Each washing cycle, apart from the blank reference cycle, included five fabrics of the same type (polyester or polyester/cotton blend), each measuring 30 cm x 60 cm in dimensions. A rinsing cycle on the same program was introduced after each test to ensure that all remaining MFs were removed, which could otherwise contaminate the next washing test.



Figure 22: Washing machine set on the wool program (54 min; 40°C; 800 rpm) with the cotton test fabrics inside to simulate domestic laundry conditions.

3.4 FILTERING AND MICROFIBRE QUANTIFICATION

3.4.1 Filtering procedure

The water residue inside the vials was filtered by utilising black cellulose nitrate membrane filters with a pore size of 0.2 μm and a diameter of 47 mm. Additionally, a vacuum pump was used to increase the filtering speed, and a sieve was placed on top to capture the steel balls, which is illustrated in Figure 23.

The entire content of the Linitest vials was poured onto the filter. Secondly, the vials were rinsed 3 times with 2 mL distilled water to remove all remaining MFs inside the vials, and this rinse water was also filtered. Finally, the filters were placed inside glass Petri dishes after filtering to avoid contamination from the lab environment.



Figure 23: Vacuum-assisted filtering setup.

In the case of the domestic washing machine, all effluent water was captured into three different containers of 25 L. These containers were thoroughly shaken before a 1 L sample was taken from each container. This 1 L sample was divided into two samples of 0.5 L to avoid overpopulation of MFs on the filter surface, which could otherwise interfere with accurate MF counting.

3.4.2 Microfibre quantification

Gravimetric analysis

The Petri dishes containing the filters were opened and placed in the oven at 60°C and 50% ventilation for at least 4 hours to dry. Afterwards, they were placed in the desiccator for at least 30 minutes to cool down to room temperature. Finally, the gain in mass was determined by comparing the mass of the filter before and after filtration.

Optical microscopy analysis

The MFs on the filters were analysed with the Leica SAP0 optical microscope, linked with a Flexacam C3 camera, visible in Figure 24(A). There was a need to find a balance between the reproducibility and reliability of the MF quantification of the filter's content and the time it would take to count them. It would take too long to count all the MFs on the whole filter surface, so it was necessary to select specific areas. Multiple areas on the filter were chosen to minimise the influence of the inhomogeneous MF distribution. These areas are the green rectangular zones illustrated in Figure 24(B). Each area includes two squares of the filter grid. The first area is located in the centre (C), while the other four areas are located at the edges (L; T; R; D). It is important to note that these are not the edges of the filter surface but the edges on the inside of the magnetic closing piece that was placed on the filter, which is the brown object on top of the edges of the filter, visible in Figure 24(B). The surface of the filter that was exposed to the washing effluent was always clearly visible under the microscope due to the particles and MFs deposited on it, making identification of the counting areas possible. This was also possible for the blank tests, where MFs and particles were still present due to contamination.

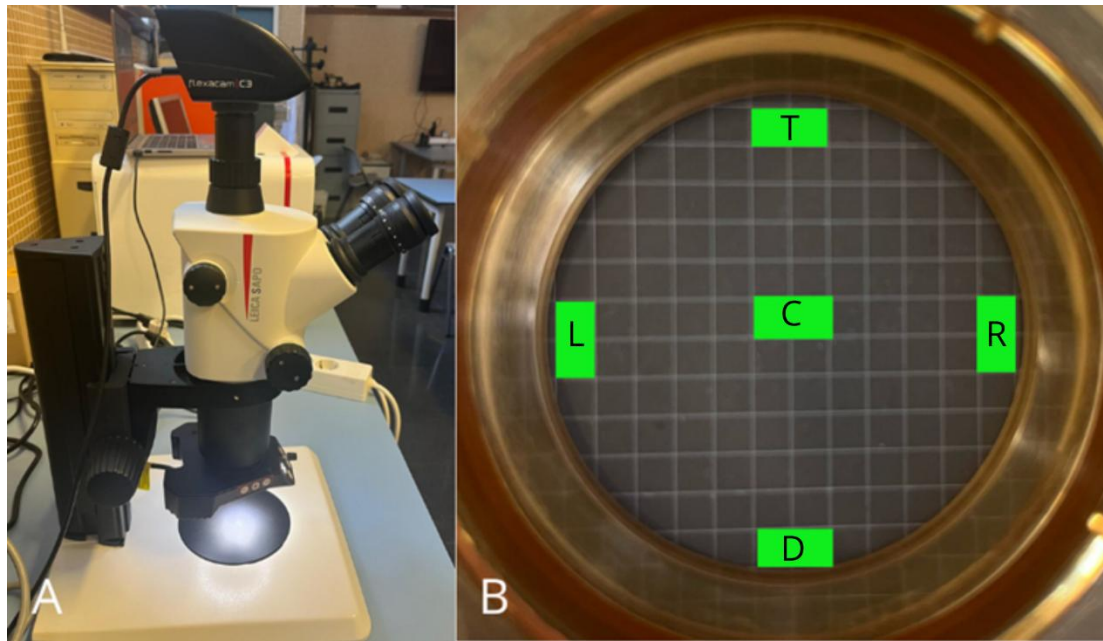


Figure 24: (A) Leica SAP0 optical microscope linked with a Flexacam C3 camera; (B) Filter surface with the green rectangular zones indicating the MF counting areas: centre (C), left (L), top (T), right (R), and down (D).

Firstly, the images were processed with ImageJ to increase the visibility of the MFs by enhancing the contrast, which is illustrated in Figure 25(A, B). Afterwards, the MFs inside the predetermined counting zones, including the MFs that were located on the edges of the zones indicated by the white grid, were counted by labelling them manually in ImageJ with the multi-point function, which is illustrated in Figure 25(C). However, only white yarns were used to weave the test fabrics, meaning that all the fibres with a different colour do not originate from the test fabrics. These coloured MFs are indicated with yellow arrows in Figure 25(C), where it is visible that they are not counted because they are considered as contamination. Additionally, fibres with a length exceeding 5 mm were also not counted because they are not considered as MFs, such as the fibres indicated with the purple arrows in Figure 25(C).

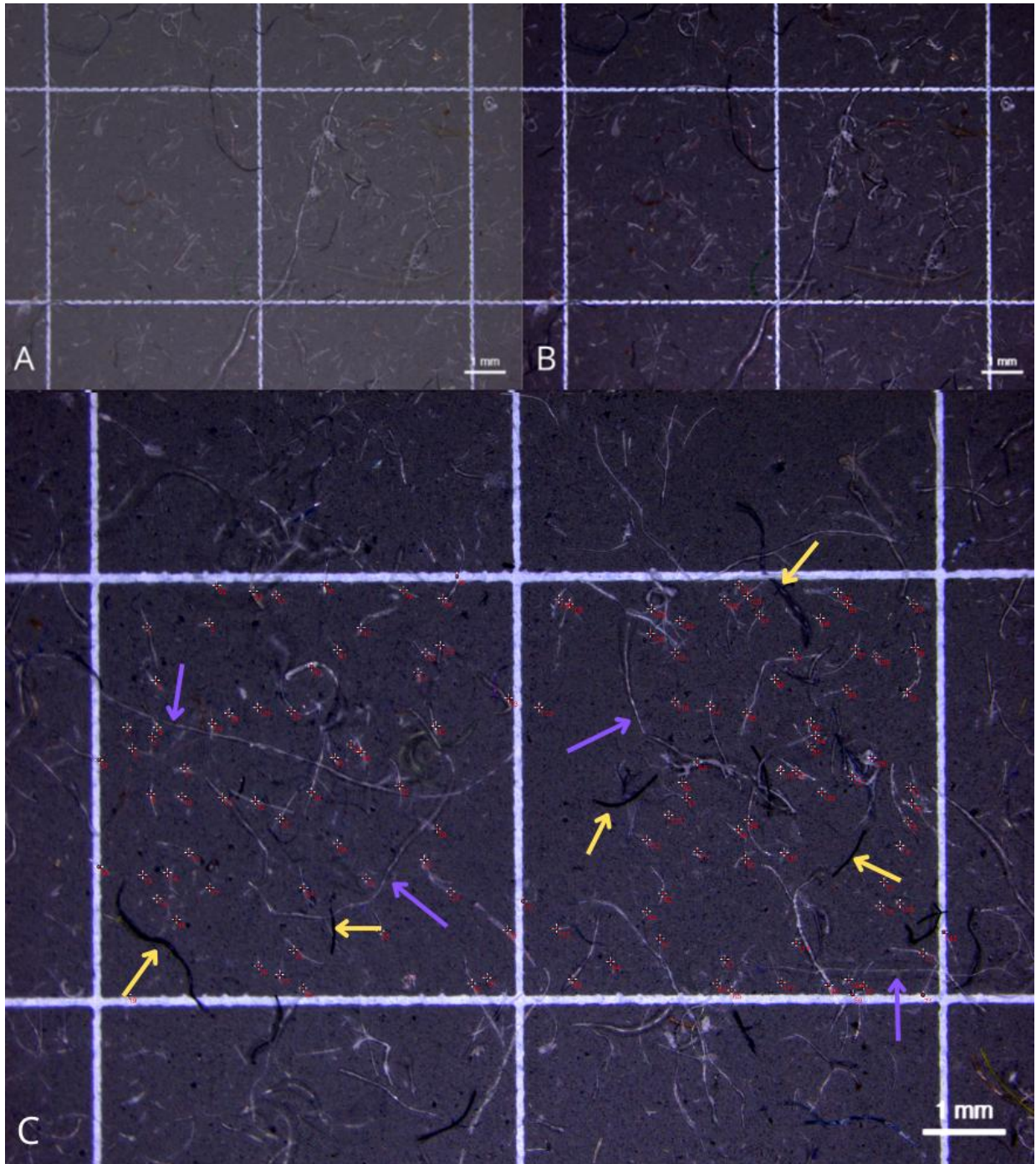


Figure 25: Image taken with the optical microscope: Before modifications with ImageJ (A); With enhanced contrast (B); After counting the MFs in ImageJ (C), with the yellow arrows indicating MFs due to contamination that were not counted, and the purple arrows indicating the fibres that are not considered as MFs because they are longer than 5 mm and thus not counted.

3.4.3 Dynamic Light Scattering analysis

Dynamic Light Scattering (DLS) was used to analyse the size distribution of the nanoparticles present in the aqueous filtrate obtained after filtering. Each sample was analysed in triplicate to ensure reproducibility. The DLS measurements were performed using the ZETASIZER Nano Series with polystyrene cuvettes, at a fixed scattering angle of 173° and a temperature of 25°C . The ZETASIZER Nano Series with an inserted polystyrene cuvette is illustrated in Figure 26.



Figure 26: ZETASIZER Nano series DLS device with inserted polystyrene cuvette, containing a filtrate sample.

Chapter 4: Results and Discussion

4.1 MATERIAL CHARACTERISTICS

4.1.1 Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra confirmed that all the tested samples are polyesters, as illustrated in Figure 27, with the indicated characteristic peaks. The peaks corresponding to the aromatic and aliphatic C-H bonds are located between 2850 and 3000 cm^{-1} , the strong, sharp peak at 1712 cm^{-1} corresponds to the C=O stretching of an aromatic ester, and the two characteristic C-O stretching bands between 1300 and 1100 cm^{-1} confirm the presence of ester linkages [74]. Additionally, the C=C aromatic stretch band at 1504 cm^{-1} confirms the presence of an aromatic structure. Finally, the strong, sharp peak at 720 cm^{-1} corresponds to the CH₂ bending, characteristic of the CH₂ present in the aromatic structure and the ethylene group present in PET [74].

The absorption intensity of the entangled polyester is significantly lower than the others. However, this is the case across the entire spectrum, which is likely due to a difference in testing conditions rather than chemical composition. On the other hand, the recycled polyester shows a clear difference in the intensity and width of the peaks corresponding to the C-O bands. Additionally, the peaks in the region indicated with the cyan coloured measurement line in Figure 27, are higher and broader in the case of the recycled polyester in comparison to the others. This may be caused by the presence of a greater variety in C-O linkages, originating from degradation processes [75]. These degradation processes result in chain scission, leading to the formation of different esters and ethers that contribute to the increase in intensity and width of the peaks in the C-O stretching region [75]. Finally, there is no significant difference visible between the conventional, biodegradable and staple fibre polyester.

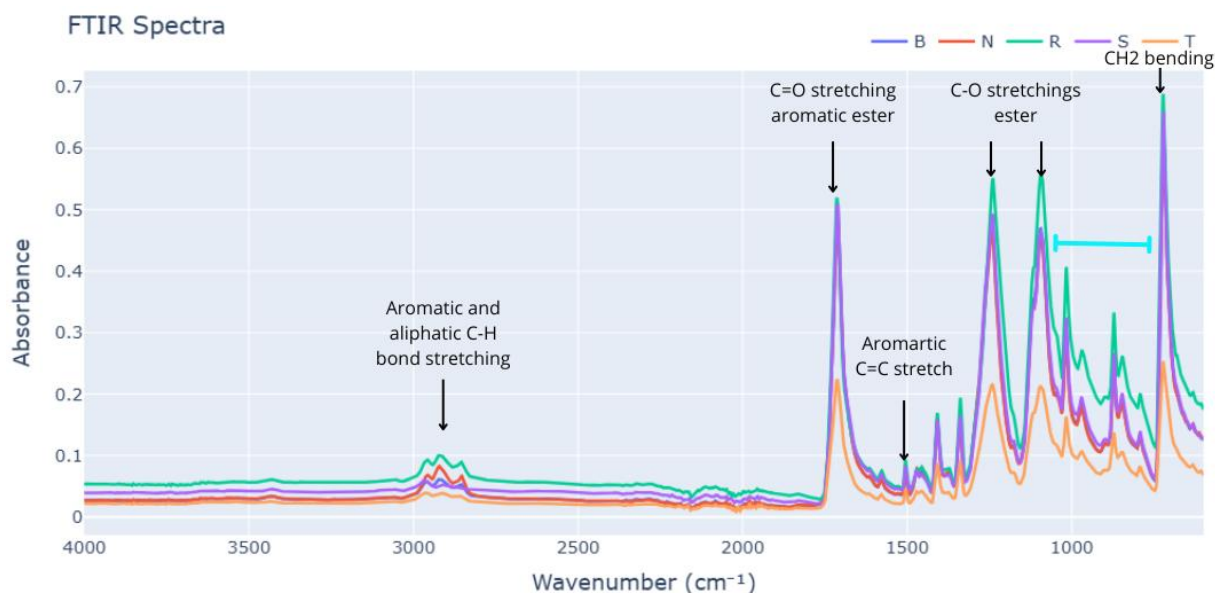


Figure 27: ATR-FTIR spectra of biodegradable (B), conventional (N), recycled (R), staple fibre (S) and entangled (T) polyester.

4.1.2 Differential scanning calorimetry

The DSC curves of the biodegradable (B), conventional (N), recycled (R), staple fibre (S), and entangled (T) polyester are illustrated in Figure 28. Firstly, it is remarkable that there are no glass transition temperatures visible on the DSC curves of any of the tested samples. This may be due to an error in the testing because the DSC curve of PET should contain an endothermic peak in the interval ranging from 60°C to 80°C, representing the glass transition [76].

The melting temperature of the recycled polyester is significantly lower than that of the other tested samples, which indicates the presence of shorter molecular chain lengths due to degradation effects during the mechanical recycling. The DSC curves of the other samples are similar and show no significant differences.

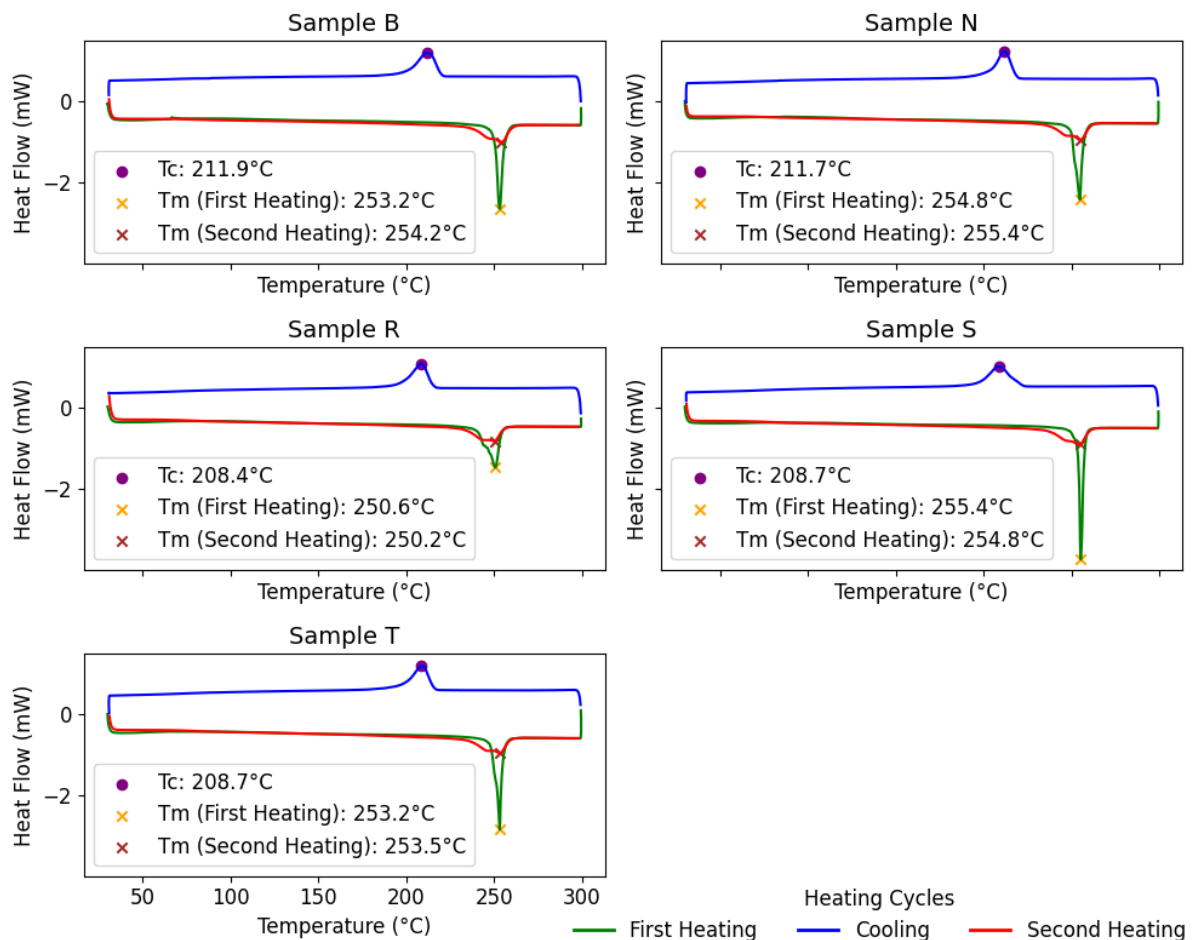


Figure 28: DSC curves of the biodegradable (B), conventional (N), recycled (R), staple fibre (S), and entangled (T) polyester. The crystallisation temperature (T_c ; purple circle) is indicated on the cooling cycle (blue), and the melting temperatures (T_m ; yellow and red crosses) are displayed on the first heating cycle (green) and second heating cycle (red).

4.2 SURFACE ANALYSIS

4.2.1 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) analysis was used to investigate the evolution of the fabric structure through several critical stages of the sample fabric's life cycle: after weaving and laser cutting, after washing, and after abrasion. Imaging the fabric after weaving provided an insight into the fibre

behaviour and possible presence of loosely bound fibres. Additionally, by investigating the laser-cut edges, the influence of the thermal treatment on the structural characteristics of the fibres at the edges could be analysed. SEM analysis after washing allowed the evaluation of surface damage and fibre breakage. Finally, the fabrics were examined after the first and third abrasion cycles to assess the effect of wear on the fabric integrity. These analyses provide an insight into the structural changes that may contribute to the MF release.

SEM analysis of the fabric structure

SEM images of the fabrics after weaving and rinsing, illustrated in Figure 29, show that the biodegradable, conventional, recycled, and entangled polyester fabrics have a compact structure without detached fibres. In contrast, there is a small number of partially detached fibres visible on the surface of the cotton-polyester blend, while a large number of these fibres were visible on the surface of the staple fibre fabric. This may be due to the shorter fibre length of the staple fibre yarns, which makes the fibre ends more prone to detach from the yarn and fabric structure. However, this could also indicate that the fibres from the weaving process, which were not sufficiently interlaced, are still present, suggesting that the rinsing procedure was unable to remove these partially detached fibres.

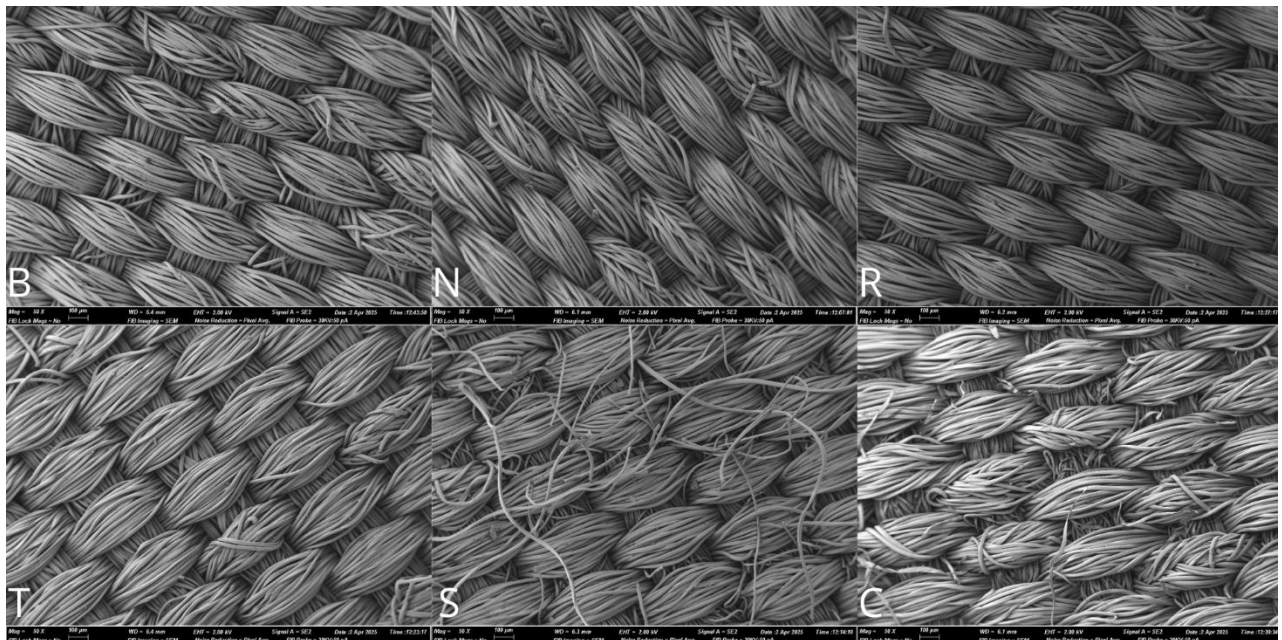


Figure 29: SEM images at x50 of the woven sample fabrics: biodegradable (B), conventional (N), recycled (R), entangled (T), staple fibre (S) polyester, and cotton-polyester (C).

Figure 30 illustrates that the partially detached fibres on the surface of the staple fibre polyester fabric were removed after the first washing cycle. This could lead to a higher MF release during the first washing cycle compared to subsequent washes. However, the polyester-cotton blend fabric showed no significant reduction in disorganised fibres after the first washing cycle, suggesting a more gradual MF shedding behaviour.

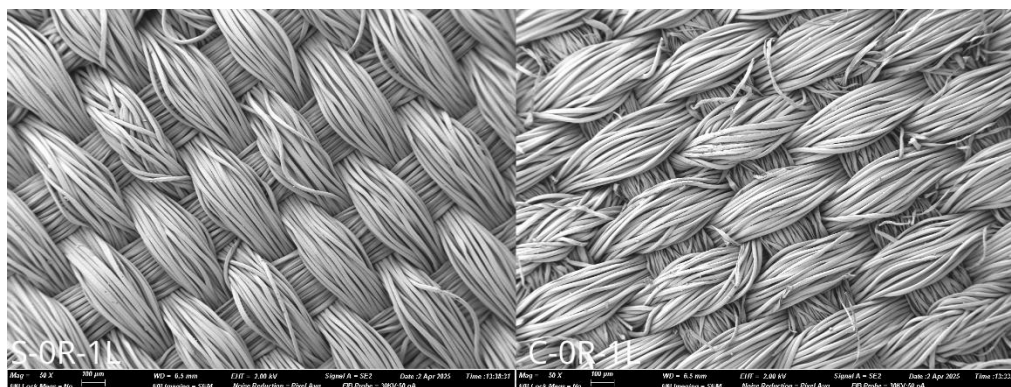


Figure 30: SEM images at x50 of the staple fibre polyester (S-OR-1L) and cotton-polyester (C-OR-1L) test fabrics after one washing cycle.

SEM images of the fabric surfaces after one and three combined washing and abrasion cycles, illustrated in Figure 31, show the presence of damage across all samples. The biodegradable polyester fabric (B-1R-1L) already shows considerable surface damage after the first cycle, including frayed and partially detached fibres. This indicates a lower mechanical resistance, which could result in an increased tendency to release fibres during laundering after abrasion. This degradation becomes more clear after the third cycle, where many fractured fibre remnants are visible, especially at the yarn intersections of the woven fabric structure. This indicates that many fibres broke and detached from the surface, further strengthening the likelihood of MF shedding.

However, the recycled polyester fabric (R-1R-1L) shows a lower level of surface damage after the first cycle, with a higher level of alignment of the fibres and a lower number of detached fibres in comparison to the biodegradable polyester fabric (B-1R-1L). Although there are some detached fibres and fractured fibre remnants visible after the third cycle (R-3R-3L), the overall integrity of the recycled polyester fabric appears to be better than that of the biodegradable polyester (B-3R-3L).

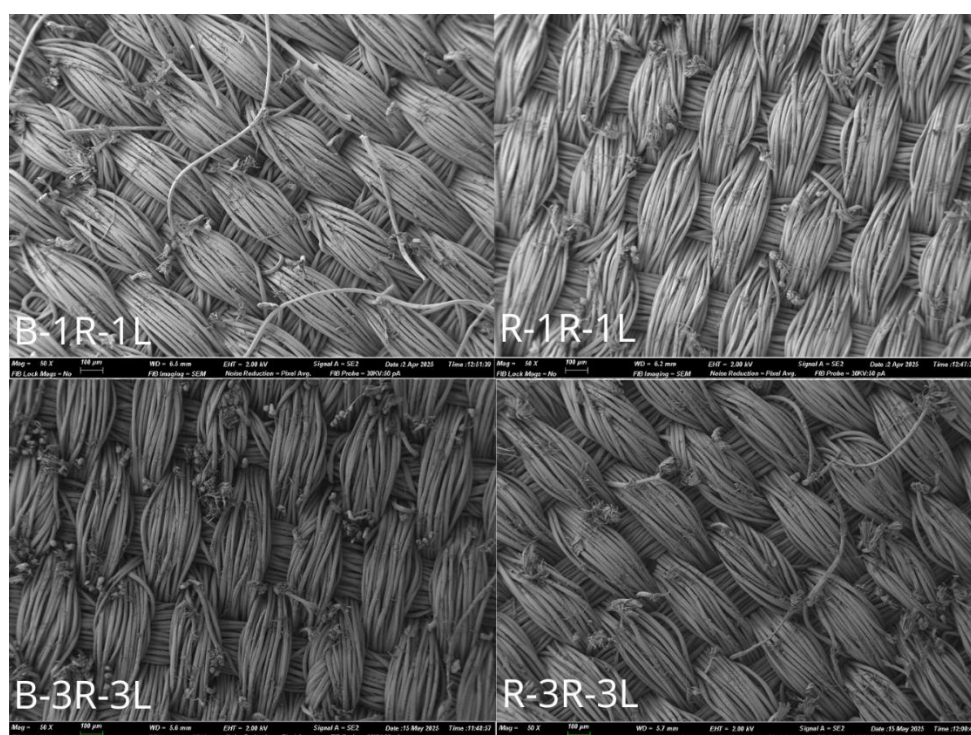


Figure 31: SEM images at x50: Biodegradable polyester (B-1R-1L) and recycled polyester (R-1R-1L) test fabrics after one abrasion and washing cycle; Biodegradable polyester (B-3R-3L) and recycled polyester (R-3R-3L) test fabrics after three abrasion and washing cycles.

SEM fibril formation analysis

As discussed in [Chapter 1:1.3.4](#), fabric wear can result in fibrillation of the fibres. This is visible in Figure 32, which shows the split fibre ends at the surface of the biodegradable and recycled polyester fabrics after three abrasion and washing cycles. These split fibres can release fibrils, which are visible on the filter's surface in Figure 32(FILTER_S-1R-1L), where the fibrils are indicated with the white arrows and the MF is indicated with the red arrow. The fibrils can be identified due to their visibly lower diameter and length compared to the MF. Although it is visible on the SEM that these fibrils are formed, it was not possible to quantify them in this study because these fibrils are not visible with the optical microscope.

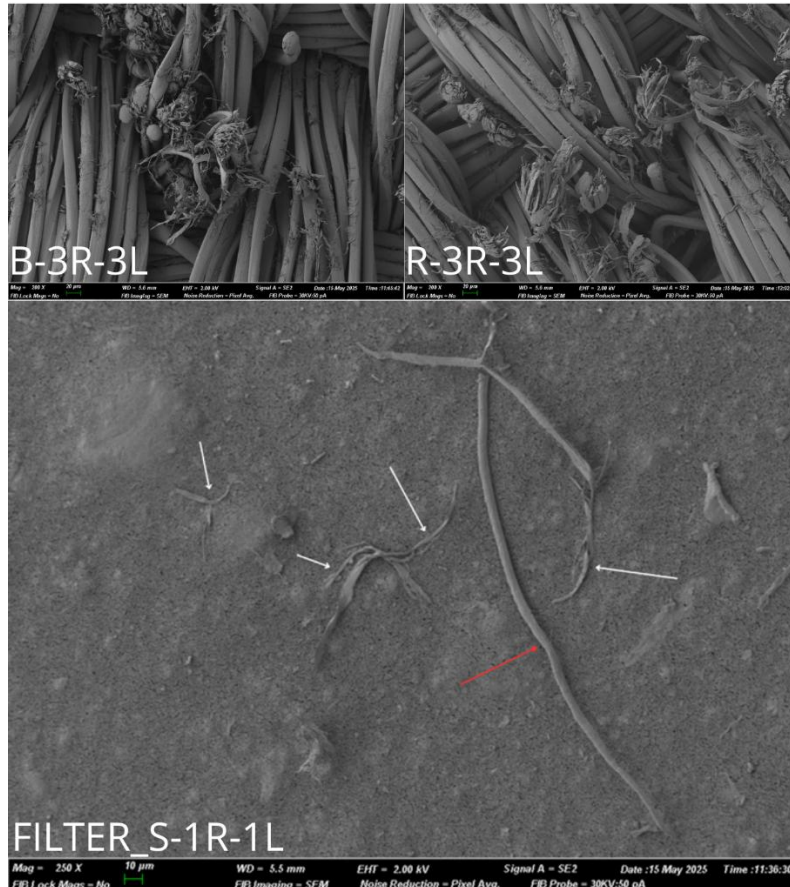


Figure 32: SEM images at x200 showing the split fibre ends at the surface of the biodegradable polyester (B-3R-3L) and recycled polyester (R-3R-3L) fabrics after 3 abrasion and washing cycles; SEM image at x250 showing the surface of the cellulose nitrate filter used to filter the wash water effluent of the staple fibre polyester fabric after 1 abrasion cycle. The red arrow indicates the MF, while the white arrows indicate the fibrils.

SEM analysis of the laser-cut edges

The SEM images of the laser-cut fabric edges, illustrated in Figure 33, show that the fabrics behave differently when exposed to the thermal treatment of the laser cutting process. Firstly, the staple fibre polyester fabric (S) has a compact, smoothly fused edge, which indicates good thermal sealing. Secondly, the recycled polyester fabric (R), which has an approximately 5°C lower melting temperature, shows a less uniform but more spread-out molten layer. This irregular melting behaviour could help encapsulate fibre ends and reduce MF release from the cut edge. However, there are more cracks visible on the edge's surface, indicating thermal damage, which could make the structure more brittle and increase the risk of breakage. In contrast, the SEM image of the cotton-polyester fabric (C) shows that the cotton fibre bundles are partially exposed because the molten polyester warp yarns

only partially cover the cotton weft yarns. This partial melting may prevent some fibres from slipping out, which could result in a lower MF release compared to scissor-cut fabrics. However, the polyester-cotton fabric could still have a higher tendency to release MFs at the edges under mechanical stress compared to fabrics made only from polyester.

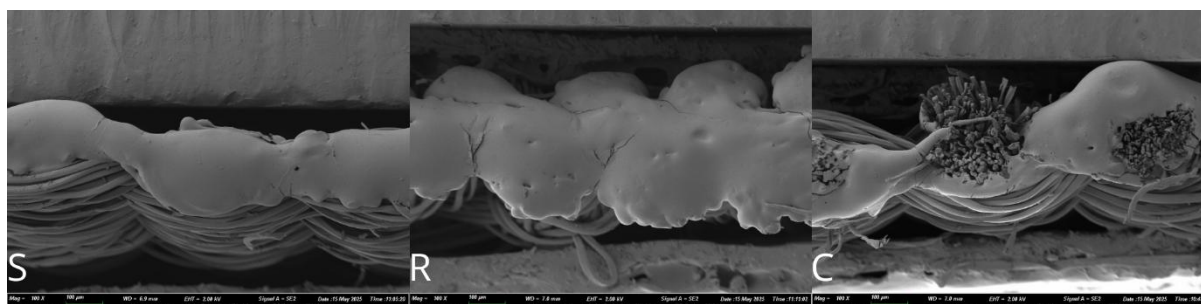


Figure 33: SEM images at x100 of the laser cut fabric edges of the staple fibre polyester (S), recycled polyester (R), and cotton-polyester (C) fabrics.

4.3 MICROFIBRE QUANTIFICATION

4.3.1 Quantification of microfibres based on gravimetric analysis

The mass gain of the cellulose nitrate filters was analysed by measuring the dry mass before and after filtering the wash water effluent. The results of these measurements are shown in Figure 34 and Figure 35, where high variations in mass gain are observed, and some filters even lost mass after filtering. The inconsistencies can be attributed to several factors. Firstly, the analytical balance has an accuracy of 0.1 mg, which is relatively low compared to the small quantities of microfibres typically retained on the filters. For example, Pirc et al. [59] reported that garments release 0.0012 wt% of MFs. Given that most of the samples used in this study have a mass of approximately 2.5g, this means that the fabrics used in this study should only shed 0.03 mg of MFs. This is below the threshold of the analytical balance, meaning it is undetectable by the balance. Secondly, the cellulose nitrate filters themselves may also contribute to the variability. The filters may degrade, lose additives or gain moisture due to their hygroscopic nature. Finally, these measurements were performed under standard laboratory conditions, where ambient humidity, static charge, and air drafts can affect the precision of such sensitive measurements. Therefore, it was not possible to obtain accurate and repeatable data with gravimetric analysis in this study.

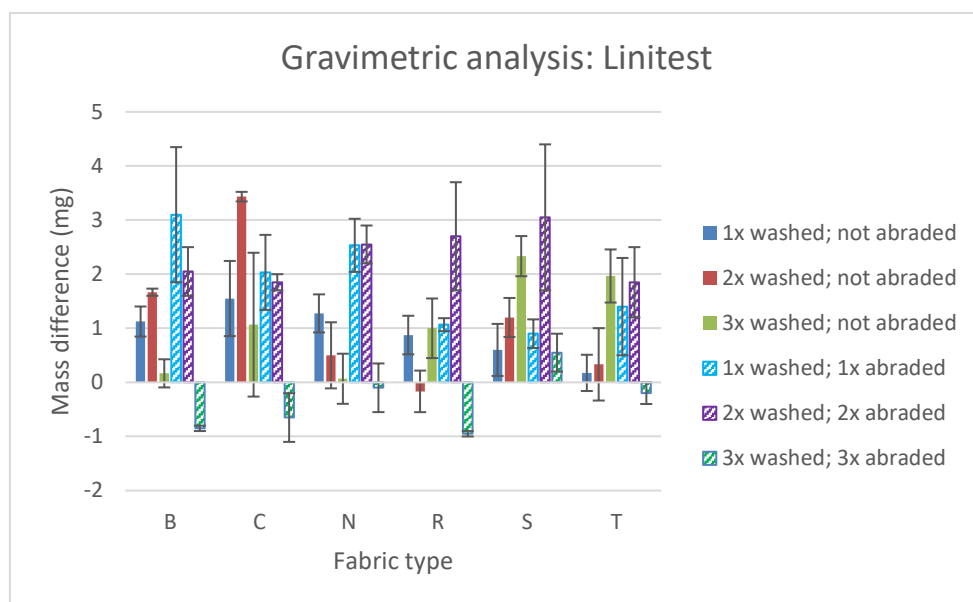


Figure 34: The mass difference of cellulose nitrate filters before and after filtering the wash water effluent of the fabrics washed in the Linitest. The samples include: biodegradable polyester (B), cotton-polyester (C), conventional polyester (N), recycled polyester (R), staple fibre polyester (S), and entangled polyester (T) after varying washing and abrasion cycles.

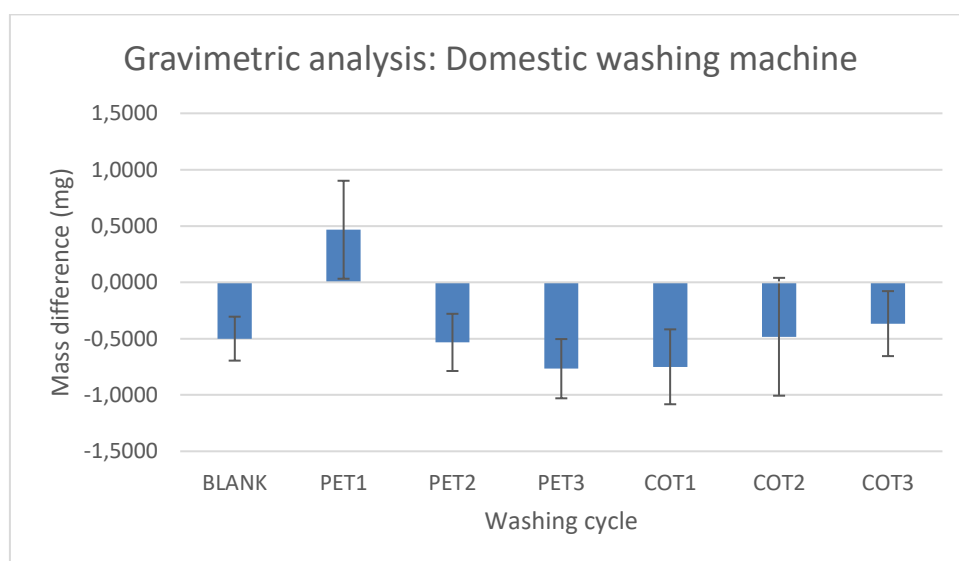


Figure 35: The mass difference of cellulose nitrate filters before and after filtering the wash water effluent of the fabrics washed in the domestic washing machine. The samples include: a blank cycle without fabric (BLANK), the first (PET1), second (PET2), and third (PET3) washing cycles with conventional polyester, and the first (COT1), second (COT2), and third (COT3) washing cycles with cotton-polyester fabrics.

4.3.2 Quantification of microfibrils based on optical microscopy

Laboratory-scale washing machine (Linitest)

The MFs retained on the filter surface were observed with the optical microscope and quantified. The results of the unabraded fabrics washed in the Linitest are illustrated in Figure 36, where it is visible that the MF release is always significantly lower in the third washing cycle compared

to the first and second. Apart from the staple fibre polyester fabrics, the MF shedding stays constant in the second washing cycle, when compared to the first cycle.

Secondly, it is visible that the cotton-polyester fabrics release significantly more MFs compared to polyester fabrics. This is in agreement with multiple studies stating that natural fibres tend to shed more MFs due to their irregular shape and shorter lengths compared to synthetic fibres such as polyester [2], [27], [28], [29]. Additionally, Haap et al. reported that most of the MFs released from a cotton-polyester blend fabric originate from cotton. However, the staple fibre polyester also has shorter fibres. Still, it behaves similarly to the other polyester fabrics, suggesting that the length of the fibres is potentially not the reason for the significantly higher MF release of the cotton-polyester blend. The SEM analysis of the laser-cut edges (Chapter 4:4.2.1) showed that the edges of the cotton-polyester blend fabric were only partially covered, increasing the potential for fibres to slip out of the structure under mechanical stress. Additionally, Periyasamy et al. [8] reported that natural fibres such as cotton tend to release more MFs during washing compared to synthetic fibres because they swell due to their hydrophilic nature, which increases the possibility of water entering the amorphous regions and reducing the intermolecular interactions between the fibres.

The data represented in Figure 36 also shows that there is no significant change in MF release between the first and second washing cycle for the majority of the fabric types. However, the MF release of the staple fibre polyester fabric decreased after the first washing cycle. This can potentially be attributed to the partially detached fibres present on the fabric's surface observed during the SEM analysis (Chapter 4:4.2.1). Additionally, the SEM analysis of the staple fibre polyester fabric's surface showed that these detached fibres were no longer present after the first washing cycle, further strengthening the hypothesis that these fibres with loose fibre ends are responsible for the decrease in MF release after the first washing cycle.

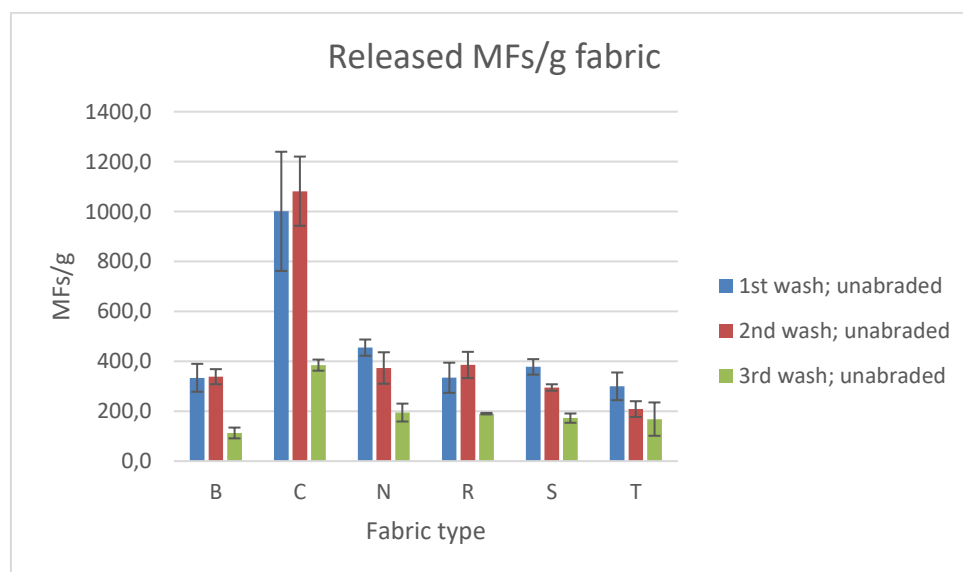


Figure 36: Released number of MFs/g of fabric during the first (blue), second (red), and third (green) washing cycles in the Linitest washing machine. The fabric samples include: biodegradable polyester (B), cotton-polyester (C), conventional polyester (N), recycled polyester (R), staple fibre polyester (S), and entangled polyester (T).

Figure 37 illustrates the number of MFs released per gram of fabric during washing, including three abrasion and washing cycles. Firstly, the graph shows that the error margins of the biodegradable polyester, conventional polyester, and entangled polyester fabric's first cycle are very high, suggesting that these fabric types could have high variations in fabric damage after the first abrasion cycle.

There is no significant change in MF release observed between the three abrasion and washing cycles for the biodegradable and conventional polyester fabrics due to the high error margins.

However, the MF release of the cotton-polyester fabrics increased during the second cycle, and then decreased again in the third. The recycled polyester fabrics, on the other hand, shed considerably fewer fibres during the first washing cycle after one abrasion cycle, aligning with the observations performed with the SEM (Chapter 4:4.2.1), where there were more damage and partially detached fibres present on the surface of the biodegradable polyester fabric compared to the recycled polyester fabric. Additionally, there was visibly more surface damage after the third abrasion cycle, which already suggested that the MF release would be higher in the third cycle compared to the first cycle.

Unfortunately, there is no error margin present for the second cycle of the staple fibre polyester fabric because there was only one sample left. This renders the data unreliable for deriving relevant information from the insufficient set of measurements. Finally, the error margin of the first cycle of the entangled fibre polyester fabrics was so considerable that the results could not be used for comparison with the other cycles. However, this was not the case for the second and third cycles, where the MF shedding during the third cycle was significantly lower than in the second.

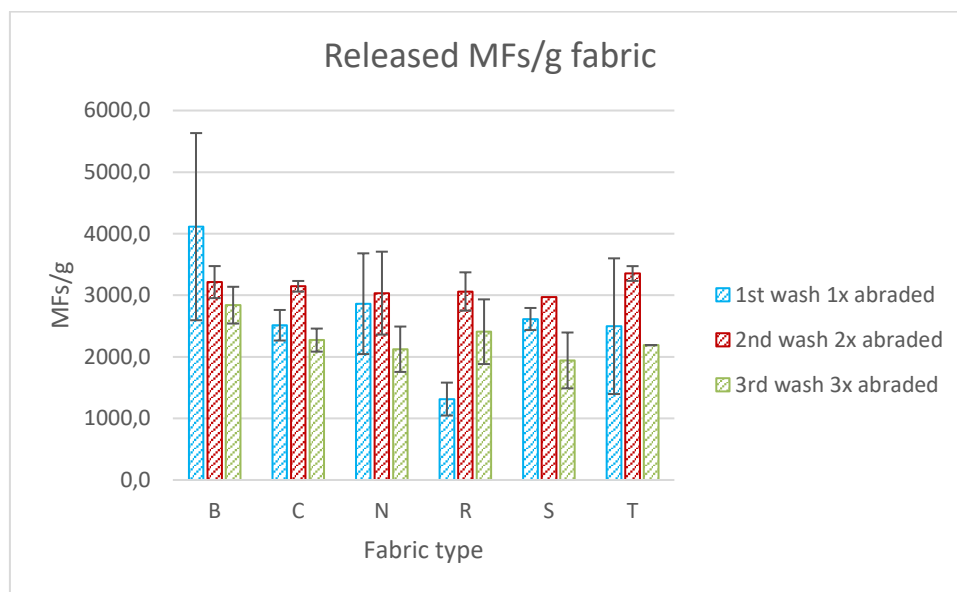


Figure 37: Released number of MFs/g of fabric during the first (blue), second (red), and third (green) washing and abrasion cycles. The fabric samples include: biodegradable polyester (B), cotton-polyester (C), conventional polyester (N), recycled polyester (R), staple fibre polyester (S), and entangled polyester (T).

The average release of MFs for unabraded and abraded fabrics is illustrated in Figure 38, where there is a significant difference visible between the fabric treatments. The abraded fabric samples release significantly more MFs compared to the unabraded fabric samples. This is probably due to the damage inflicted by the abrasion, which is clearly visible on the SEM images (Chapter 4:4.2.1). It should be noted that unabraded samples were washed three times consecutively, whereas abraded samples underwent three abrasion and washing cycles, in which abrasion was applied before each wash. The higher MF release in the abraded samples thus reflects the cumulative effect of repeated surface wear combined with washing.

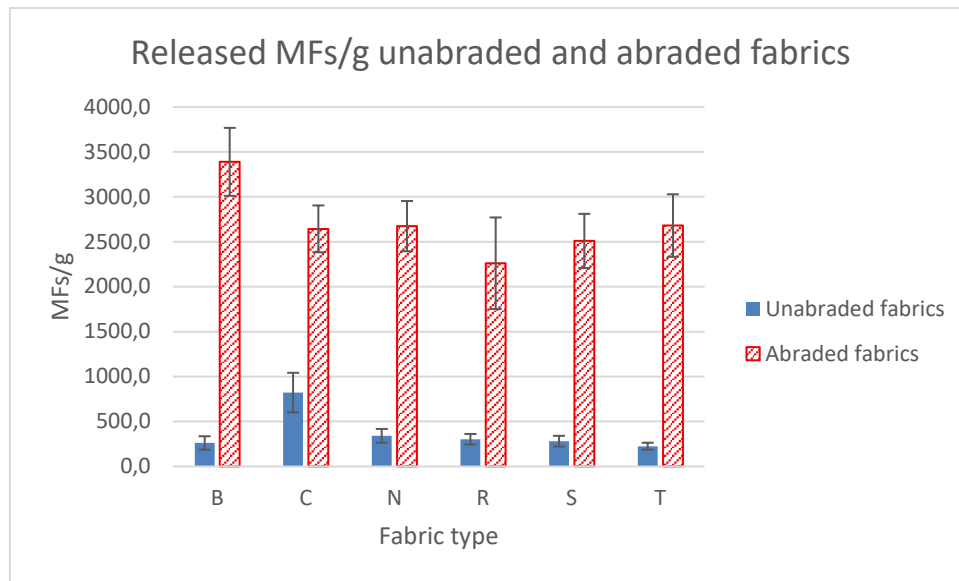


Figure 38: Average MF release over the 3 studied cycles of the unabraded (blue) and abraded (red) samples. These samples include: biodegradable polyester (B), cotton-polyester (C), conventional polyester (N), recycled polyester (R), staple fibre polyester (S), and entangled polyester (T).

Reported microfibre release compared to the literature

Table 6 represents the average MF release measured over three test cycles, both with and without abrasion. Since all the fabrics are blends using conventional polyester warp yarns, it is difficult to find comparable data in the literature due to variations in blending ratios and the lack of material specifications, such as the recycled content in the recycled polyester yarns. However, comparison with literature data for conventional polyester can still be relevant. Cai et al. [52] reported that laser-cut, plain woven fabrics of polyester filament yarns shed 370 MF/g of fabric, which is similar to the 341 ± 77 MFs/g reported in this study. In contrast, Yang et al. [54] reported that polyester textiles shed 4900 to 640,000 MFs/g of textile after 5000 rubs with the Martindale. This is significantly higher than the 2673 ± 280 reported in this study. This discrepancy could be attributed to differences in counting methods: Yang et al. [54] used SEM to analyse the filter surface and count the MFs, while this study used optical microscopy. It is possible that SEM enabled the detection of MFs that are not visible under an optical microscope.

Table 6: The average reported microfibre release over three washing and abrasion cycles of the non-abraded and abraded biodegradable (B), conventional (N), recycled (R), staple fibre (S), entangled (T) polyester, and cotton-polyester (C) fabrics.

Sample type	Released MFs/g fabric (non-abraded)	Released MFs/g fabric (abraded)
B	262 ± 74	3389 ± 378
C	822 ± 220	2643 ± 261
N	341 ± 77	2673 ± 280
R	303 ± 58	2261 ± 510
S	282 ± 60	2509 ± 302
T	226 ± 39	2680 ± 348

Domestic washing machine

Figure 39 shows that there was no significant difference in MFs found in the wash water effluent of the blank tests and the tests with fabric. This indicates that a substantial amount of contamination remained inside the washing machine and tubes, suggesting that the rinsing procedure was insufficient to remove this contamination. Additionally, this data also indicates that more fabric is required to investigate the MF release of fabrics inside a domestic washing machine. Only 133g of polyester and 137g of cotton-polyester fabric were used, while the washing machine uses approximately 50L of water, diluting the MFs released by the fabrics too much to be detectable. This makes it impossible to detect any differences between washing cycles or types of fabric.

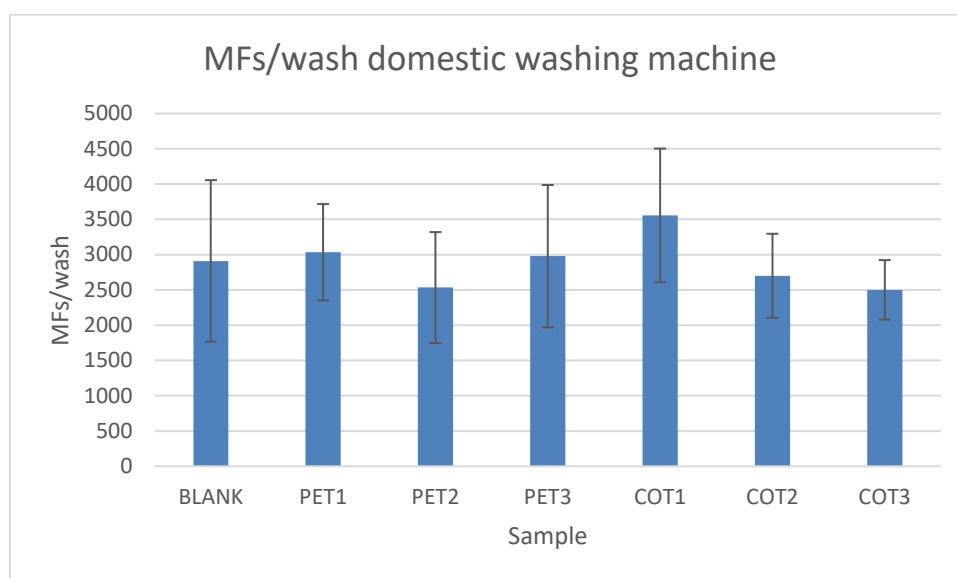


Figure 39: The number of released MFs/wash in a domestic washing machine. The samples include: a blank cycle without fabric (BLANK), the first (PET1), second (PET2), and third (PET3) washing cycles with conventional polyester, and the first (COT1), second (COT2), and third (COT3) washing cycles with cotton-polyester fabrics.

4.3.3 The influence of contamination on the microfibre quantification

Figure 40 illustrates how contamination can influence the quantification of the microfibrils when optical microscopy is used. The majority of the content that is retained on the filter after filtration of the wash water effluent originating from the Linitest washing machine has the morphology of a fibre. There is visible contamination; however, the majority of this contamination consists of fibres of a different colour. These fibres can easily be neglected during the microfibre counting process and are not accounted for. However, this is not the case for the content retained after filtration of the wash water effluent of the domestic washing machine, where the majority of the contaminants are small white particles. This is illustrated in Figure 41, where some of these particles are indicated with yellow circles. Additionally, the majority of the fibres present on the filter's surface used for the filtration of the empty domestic washing machine's effluent are white, making it very difficult to distinguish them from the fibres released by the fabrics. This increases the counting error significantly and reduces the reproducibility of the counting method. Quantification of the MFs released from fabrics during washing in a domestic washing machine would require a perfectly rinsed domestic washing machine or a more in-depth analysis, where the diameter and length of the MFs are determined. Then the MFs would only be counted when their diameter and length fall within a predefined range. However, this was beyond the scope of this study.

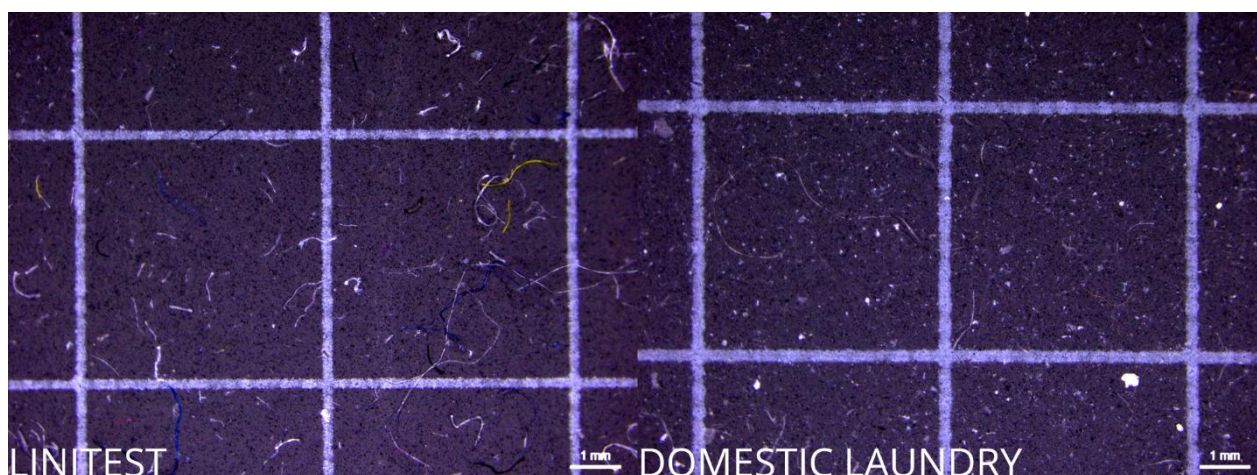


Figure 40: Images of the cellulose nitrate filter surface after filtration of wash water effluent originating from the Linitest (left) and the domestic washing machine (right), taken with the optical microscope.

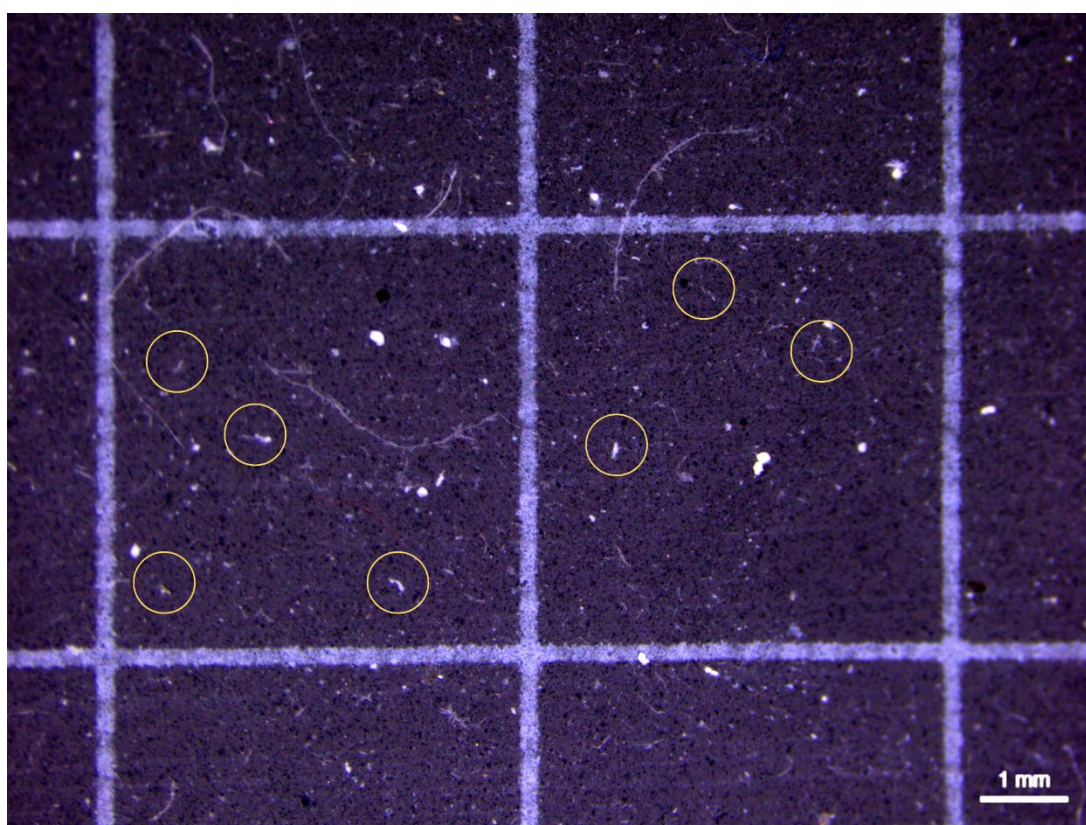


Figure 41: Image of the cellulose nitrate filter after filtration of the wash water effluent originating from an empty washing machine cycle (blank cycle), with some of the small contaminants indicated with yellow circles.

4.3.4 Environmental impact

Boucher et al. [7] reported that each person consumes an average of 6.1 kg of synthetic fibres yearly for apparel. This value was used to scale the MF release data obtained in this study for conventional polyester fabrics. Only the data from samples subjected to washing cycles were considered, excluding those that also underwent abrasion. This choice was made because an abrasion

cycle was applied after every washing cycle, and three abrasion cycles would probably overestimate the fabric wear under typical consumer use.

Assuming that the consumer would wash each garment twice, and based on the MF release measured during the first two washing cycles of conventional polyester fabrics, an estimated $5,049,699 \pm 248,796$ MFs per capita would be released over the first two initial washes.

A second scenario was considered in which the manufacturer would pre-wash the fabrics before they reach the consumer. In this case, the estimated MF release of the consumer decreases to $3,463,125 \pm 544,491$ per capita over the first two initial washes. This suggests a potential opportunity to reduce household MF emissions. However, this would only be environmentally beneficial when the manufacturer can correctly treat the prewash effluent water and prevent these MFs from entering the environment.

4.4 DYNAMIC LIGHT SCATTERING ANALYSIS

The results of the Dynamic Light Scattering (DLS) analysis, illustrated in Figure 42, represent the average size and dispersity of the nanoparticles present in the filtrates of the wash water effluents after filtration with the cellulose nitrate filters. The sample containing only distilled water (blank) and the filtrates of the polyester fabrics washed in the Linitest (PET) have an average nanoparticle size of approximately 150 nm and a moderate-to-high polydispersity index (PDI) ranging from 0.2 to 0.5. This PDI is used to measure the heterogeneity of a sample based on particle size, with a low PDI indicating a narrow size distribution [77]. The DLS analysis suggests that the particles present in the wash water filtrate of the cotton-polyester fabrics washed in the Linitest (COT-PET) have an average size of approximately 600 nm. Additionally, the measured PDI is higher compared to the blank and the polyester samples, indicating a broader size distribution. Finally, all the samples of the domestic washing machine effluent have a high average particle size and very high PDI. This correlates with the observations made using the optical microscope of the filter used for the domestic washing machine effluent (Chapter 4:4.3.3), where many small, non-fibrous particles were present. This may also be the case on the nanoscale, which could be the reason why the PDI is so high for these samples. The samples of the domestic washing machine are likely to be significantly influenced by contamination, making the samples originating from the Linitest more reliable for investigating the nanoparticle release from the fabrics.

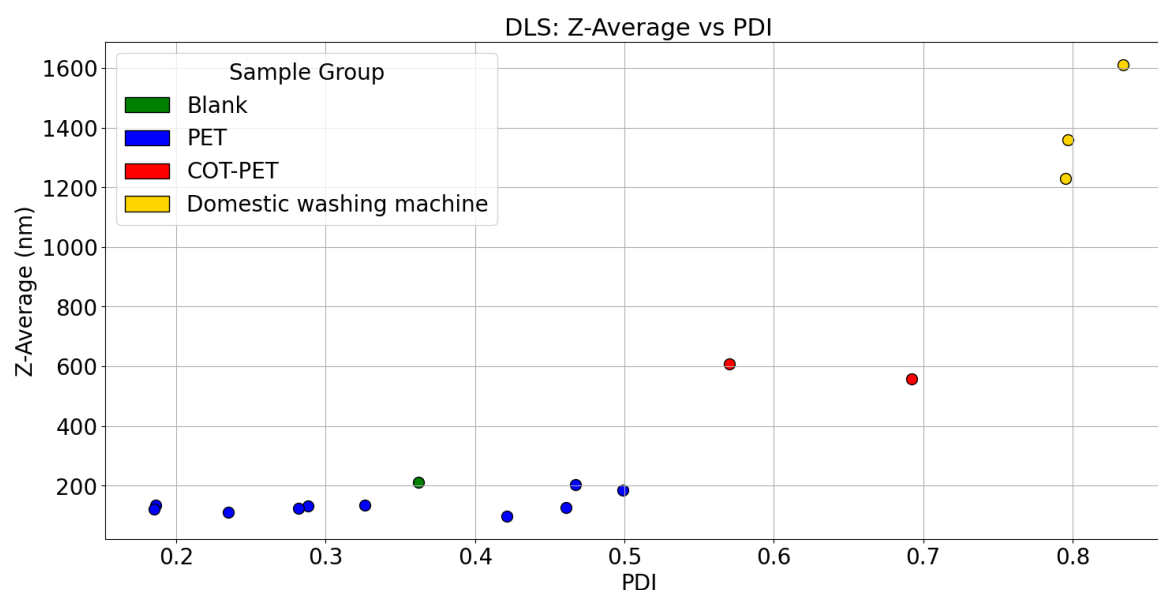


Figure 42: Dynamic Light Scattering (DLS) results showing the relationship between the average particle size (Z-average) and the polydispersity index (PDI) of the wash effluent filtrates. The blank

sample (only distilled water) is indicated in green, the wash effluent filtrates of the polyester fabrics washed in the Linitest in blue, the cotton-polyester blend fabrics washed in the Linitest in red, and the filtrates of the wash water effluent from the domestic washing machine in yellow.

Chapter 5: Further research possibilities

Although this study provides valuable insights into the MF release of fabrics, numerous possibilities remain for further research. Firstly, other studies have already reported that fabric structure and washing parameters can significantly affect the release of MFs. In this study, a plain woven fabric was chosen, and the washing conditions were kept constant. Changing the fabric structure by using different weaving or knitting patterns, and adjusting the washing parameters such as temperature, rotation speed, washing load, and detergent use, can provide a more comprehensive view of the MF shedding behaviour of fabrics under real-world conditions. Additionally, only the MF release of fabrics was investigated, while households will wash complete garments. Investigating the MF release of commercially available garments would offer the possibility of comparing the MF shedding rates of different textile types. Secondly, all the fabrics were laser-cut. However, it could be very interesting to compare the obtained MF release rates with those of scissor-cut fabrics.

Thirdly, the MFs were manually counted in this study. However, it would save a lot of time if this could be automated through artificial intelligence (AI). The manual approach was chosen in this project because many images with manually indicated MFs would probably be necessary to train the program, putting the creation of a MF counting model out of the scope of this study. Although there are multiple studies, such as those performed by Javier Lorenzo-Navarro et al. [78], Usov et al. [79], and Galata et al. [80], showing that the quantification of MFs can be automated, it would be necessary to modify their setup to apply it in this study. This is due to variations in the size and visibility of the MFs, as well as other differences, such as the background, which depends on the filter type used. For example, the model used by Usov et al. is trained to identify particles, while this study focused solely on MFs. However, it could be possible to use the images made with the optical microscope in this study to train a MF counting model.

Finally, the nanoparticle distribution in the washing effluent was investigated in this study. However, it would be more relevant to perform a qualitative and quantitative assessment of these particles. This would provide insight into the nature of these particles and the concentration present in the wash water filtrate.

Chapter 6: Sustainability analysis

6.1 UNITED NATIONS SUSTAINABLE DEVELOPMENT GOALS

The MF release from textiles represents a global environmental and societal challenge, influencing human health, biodiversity, water quality, and climate change. Furthermore, there is a need for societal cooperation to address this issue. The United Nations Sustainable Development Goals (SDGs), illustrated in Figure 43, can be used as a framework to incorporate strategies that take these environmental and societal challenges into account. The SDGs, which were incorporated in this study, are discussed in this sustainability analysis.



Figure 43: Illustration of the Sustainable Development Goals [81].

6.1.1 Goal 3: Good health and well-being

As already mentioned in Chapter 1:1.1.4, humans are constantly exposed to MFs by their environment, and these MFs are already found inside the human body [8]. Although these MFs can enter vital organs of living organisms such as fish, and have also been shown to be able to carry toxic pollutants, there is still a need for further research into the effects these MFs have on human health [14], [16], [18], [23], [24]. However, it is self-evident that the MF intake of humans should be avoided as much as possible to maintain good health and well-being for everyone.

6.1.2 Goal 6: Clean water and sanitation

Peng et al. [82] reported that wastewater treatment plants (WWTP) can remove 95 to 99% of microplastics from the wastewater. However, despite this high level of efficiency, Talvitie et al. [83] reported that on average 4.9 (± 1.4) of fibres and 8.6 (± 2.5) of particles per litre remain in the water.

Eventually, this can result in approximately 65 million particles per day, as estimated by Murphy et al. [84]. This data indicates that it remains a challenge to remove MFs and microparticles from wastewater, demonstrating the importance of reducing the release of MFs investigated in this study.

6.1.3 Goal 12: Responsible consumption and production

As mentioned in Chapter 1:1.1.2, Browne et al. [9] reported that a higher number of MFs was found at shorelines in densely populated areas. Additionally, many of these MFs originate from domestic laundry [7], [9], [11], [12], [85]. Many studies showed that the MF release decreases over sequential washes [27], [58], [60], [61], [62], [63], [64], [65]. This was confirmed in this study, where the MF release of the third washing cycle was significantly lower than that of the first and second washing cycles. Consumers should be aware of this and adjust their purchasing habits accordingly. The quality of the textiles is highly relevant because this study has shown that damage due to mechanical stress and wear significantly increases the MF release during washing. To conclude, consumers should be encouraged to adopt more sustainable practices by purchasing fewer, but higher-quality, clothes.

6.1.4 Goal 13: Climate action

Polyester is the most common fibre material used for textile production, with a demand that is expected to increase in the following years [41]. Polyester is produced from non-renewable petrochemicals, making it unsustainable [41]. However, polyester is a thermoplastic polymer, offering the possibility to recycle it by remelting it [41]. This makes the production process partially circular, reducing waste and resource consumption. Additionally, this study showed that the MF release from various conditions of the recycled polyester fabric was very similar to that of the virgin polyester fabrics, with the recycled polyester even releasing fewer MFs during washing after the first abrasion cycle. This suggests that recycling polyester not only saves resources and reduces waste but also has no negative impact on the MF release of the textiles.

6.1.5 Goal 14 & 15: Life below water and on land

As previously mentioned in Chapter 1:1.1.3, many MFs are present in aquatic and terrestrial environments, affecting all organisms. These small particles accumulate in organisms, can penetrate biological barriers, and carry toxic chemicals. This study not only investigated the MF release of fabrics during washing, but also the nanoparticle distribution in the wash water effluent after filtration to investigate the presence of nanoparticles. The small size of these nanoparticles enables them to enter vital organs and accumulate there.

6.1.6 Goal 17: Partnerships for the goals

The contamination by MFs is a global problem and is expected to increase due to the increasing population [86], signifying the need for collaboration in the fashion industry. Projects such as *Tackling Microfibres at Source*, where Ramatex Group, Nanyang Environment and Water Resources Institute (NEWRI), and Universiti Teknologi Malaysia (UTM) collaborate to reduce the MF release during manufacturing, are an example of a workspace where insights and methods can be co-developed to accelerate change and innovation [85], [87]. This collaboration is facilitated by *Forum for the Future*, which brings these manufacturers together and creates a space for the people involved to co-create [85], [87].

Chapter 7: Conclusion

The investigation of the material characteristics by applying FTIR and DSC analysis showed that the chemical composition and thermal behaviour of the studied polyesters was very similar apart from the recycled polyester, which showed clear signs of degradation by having broader peaks with a higher absorbance intensity at the C-O stretching vibrations and a lower melting temperature compared to the other polyesters.

Gravimetric analysis proved unsuitable for quantifying microfibre (MF) release. Literature review confirmed that the mass of retained MFs is often below the detection threshold of analytical balances. Accurate weight-based MF quantification demands controlled conditions and highly sensitive equipment.

MF release from unabraded fabrics decreased significantly after the third washing cycle for all material types. Cotton-polyester blends released substantially more MFs than pure polyester fabrics. SEM analysis revealed exposed cotton fibre ends at the laser-cut edges, only partially covered by molten polyester, which likely contributed to this release. Additionally, cotton's hydrophilic nature may promote fibre swelling and loosening, further facilitating MF shedding. Specifically, the conventional polyester fabric released an average of 341 ± 77 MFs per gram of fabric over three washing cycles without abrasion. Although only one study with comparable materials, sample preparation, and washing conditions could be identified, the agreement between results suggests that the experimental procedure used in this study is reliable.

Abrasion followed by washing increased the MF release, though high variation between replicates limited definitive conclusions for some fabrics. Notably, cotton-polyester fabrics reached maximum MF release after the second cycle. Recycled polyester released the fewest MFs after the first cycle, correlating with minimal surface damage seen under SEM compared to, for example, biodegradable polyester. SEM also confirmed that abrasion caused fibre splitting, which is believed to result in fibrillation. These fibrils were visible on the filter surface but too small to be quantified using optical microscopy.

MF release during domestic washing could not be assessed reliably due to contamination. Filters from a wash cycle without any fabric captured as many MFs as those with test fabrics. This suggests residual contamination in the washing machine and tubing. The small load (five fabrics, approximately 130 g) was insufficient for accurate measurement in the wash volume (approximately 50 L). Additionally, contaminants from the domestic washing machine included fibres and particles that were difficult to distinguish from test MFs under optical microscopy, thereby increasing counting error. In contrast, contaminants from the Linitest were mostly coloured fibres, which were easier to exclude. Future studies should define a counting protocol based on fibre length and diameter thresholds to improve accuracy. These findings highlight the importance of thorough rinsing, sufficient washing load, and a standardised counting method when studying domestic MF release.

Finally, Dynamic Light Scattering analysis revealed that polyester fabrics washed in the Linitest released nanoparticles similar in size and distribution to distilled water. In contrast, data from the cotton-polyester fabrics suggests that larger nanoparticles are present with a broader distribution in size, which may be related to the differences in fibre characteristics of the cotton. On the other hand, samples collected from the domestic washing machine showed markedly larger and highly polydisperse particles, which could be primarily driven by contamination from the washing machine environment.

This study demonstrates that recycled polyester performs as well as virgin polyester in terms of MF shedding and durability, with no observed increased release and even a lower shedding rate in one

test case. This strengthens its potential as a viable option for more sustainable textile production. Additionally, the laser-cut edge of the polyester fabric was sealed very well, possibly due to its lower melting temperature, which prevented fibre exposure. This property of the recycled polyester could potentially be used to encapsulate or protect the exposed fibre ends of the cotton yarns through the development of a suitable fabric design.

Moreover, the findings presented here suggest that pre-washing textiles at the manufacturer could substantially decrease the MF release during the early stages of consumer use. However, this is only a viable option when manufacturers can effectively filter the fibres out of the wash water effluent and prevent the MFs from entering the environment.

This research was conducted under controlled laboratory conditions. However, real-world environments may influence the MF shedding of textiles differently due to factors such as sunlight exposure, microbial activity, and fluctuations in moisture and temperature levels. Future research could investigate the MF release rate of complete garments and examine the impact of real-world environmental factors on the MF release rate of these garments. Moreover, textiles are not only used in garments but also in other applications, such as fishing nets, geotextiles, and agricultural coverings, where they are exposed to different conditions. Future research could investigate the MF release behaviour of complete garments and functional textiles under these various environmental factors.

Chapter 8: Reflection on prewash feasibility

This study highlighted that reducing the microfibre (MF) release into the environment could be achieved by integrating a prewashing step with an efficient on-site MF capturing and processing system. Although this study focused specifically on quantifying the MF release from textiles rather than treating the wastewater, insights from wastewater treatment studies can serve as an example of how manufacturers could integrate removal technologies. For instance, Sturm et al. [88] investigated the application of a two-stage removal process, which is illustrated in Figure 44. The study involved the agglomeration-fixation of microplastics and a fixed-bed Chemical Oxygen Demand (COD) adsorber, treating wastewater from a plastic manufacturer, and reported a reduction in microplastic loads of nearly 98% and a COD reduction of $94.3 \pm 8.9\%$. This not only prevented microplastics from entering waste water treatment plants (WWTPs), but also offered the possibility to reuse the water, making the process more sustainable and reducing the carbon footprint [88]. If a similar approach could be applied to textile manufacturing, the prewash effluent could be treated and reused or safely discharged after removing the MFs.

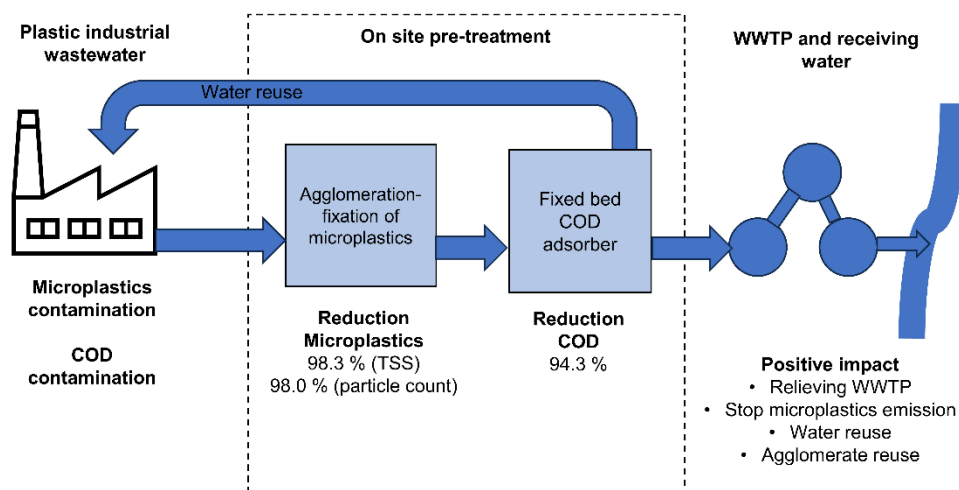


Figure 44: The graphical abstract used in the study of Sturm et al. [88], representing the two-stage on-site removal of microplastics and chemical oxygen demand (COD) process.

However, adapting this strategy for the textile industry presents significant challenges. Industrial laundry processes consume vast volumes of water [89], necessitating a substantial scale-up from pilot tests, such as those conducted by Sturm et al. [88], where only 1 m^3 of wash effluent was processed per test. Additionally, while prewashing textiles would reduce the number of MFs released during the initial wash in households and decrease the number of MFs entering the environment by processing them, it would also increase water and energy consumption due to the extra steps involved, as well as raise costs due to the need for advanced equipment. A comprehensive assessment of these trade-offs, including a detailed cost-benefit and life-cycle analysis, is essential to determine whether the environmental benefits would outweigh the added resource inputs.

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Appendices

Appendix A: Gravimetric analysis data

Table 7: Data gravimetric analysis of the cellulose nitrate filters used before and after filtration of the Linitest washing effluents.

Sample	Weight sample (g)	Abrasion cycles included	Weight gain filter (mg)		
			First cycle	Second cycle	Third cycle
B1	2.4604	Yes	5.5	/	
B2	2.446		1.3	2.3	-0.9
B3	2.4586		2.5	0.5	-0.8
B4	2.4712	No	1.3	/	
B5	2.4493		0.3	1.8	0.6
B6	2.4485		1.5	1.6	0.2
B7	2.4585		1.4	1.6	-0.3
C1	2.5424	Yes	3.2	/	
C2	2.5205		0.8	2.0	-1.1
C3	2.529		2.1	1.7	-0.2
C4	2.5262	No	0.1	/	
C5	2.5221		2.3	3.3	3.6
C6	2.5322		0.7	3.6	0.5
C7	2.5261		3.1	3.4	-0.9
N1	2.4469	Yes	2.5	/	
N2	2.4337		3.4	2.2	-0.2
N3	2.4383		1.7	2.9	0
N4	2.4371	No	1.1	/	
N5	2.4378		1.3	0.9	-0.7
N6	2.4596		2.2	1.3	0
N7	2.4342		0.5	-0.7	0.9
R1	2.4562	Yes	1.0	/	
R2	2.4457		0.9	1.7	-0.9
R3	2.4496		1.3	3.7	-1.0
R4	2.4467	No	1.7	/	
R5	2.4629		1.1	-0.6	0

R5	2.4604		0.7	-0.5	1.1
R7	2.4526		0.0	0.6	1.9
S1	3.435	Yes	0.8	/	
S2	3.4274		1.4	1.7	0.9
S3	3.4129		0.5	4.4	0.2
S4	3.4671	No	1.9	/	
S5	3.4614		0.3	1.0	1.6
S6	3.4279		-0.4	1.9	2.8
S7	3.4477		0.6	0.7	2.6
T2	3.0846	Yes	2.3	1.2	-0.4
T3	3.0943		0.5	2.5	0
T4	3.0818	No	0.1	/	
T5	3.1419		0.0	1.4	2.6
T6	3.1116		-0.5	0.5	1.0
T7	3.1039		1.1	-0.9	2.3
Blank	/	/	5.5		
Blank			1.3		
Blank			2.5		
Blank			-0.9		
Blank			-3.2		
Blank			1.5		
Blank			-2.3		
Blank			-1.2		
Blank			-0.5		

Table 8: Data gravimetric analysis of the cellulose nitrate filters used before and after filtration of the domestic washing machine effluents.

Sample	Sample weight	Weight gain filter (mg)		
		First cycle	Second cycle	Third cycle
PET	133.3	1.3	-0.3	-0.3
		0.8	-0.8	-0.6
		-0.5	-0.5	-2
		2	-0.5	-0.5
		-0.6	-1.5	-0.9
		-0.2	0.4	-0.3
COT-PET	137.1	0.4	-0.1	-1.1
		-0.4	1.5	-0.3
		-0.2	-0.2	0
		-1.6	-0.3	0.8
		-1.6	-1.9	-0.6
		-1.1	-1.9	-1
Blank	/	-0.7		
		0		
		-0.6		
		-0.3		
		-1.3		
		-0.1		

Appendix B: Optical microscopy analysis data

Table 9: Number of microfibrils counted in every counting zone on the cellulose nitrate filter after filtering the Linitest wash water effluent of the tested samples.

Sample	Abrasion cycles included	First cycle					Second cycle					Third cycle				
		Counting zone (MFs/square)														
		C	D	L	R	T	C	D	L	R	T	C	D	L	R	T
B1	Yes	120.5	79.5	74	65	59.5	/									
B2		27.5	45.5	25.5	33.5	25	47	56	57.5	77.5	58	64.5	64	60.5	58	67.5
B3		160.5	126	157.5	116	127.5	67	71	82	73	57	49.5	52	58.5	51	45
B4	No	12	5.5	10.5	9.5	7	/									
B5		4	8	11.5	7	9	6	11.5	8.5	5.5	8.5	0.5	1.5	4	2.5	5
B6		8.5	5.5	6.5	6.5	5	4	5	10	7.5	4	3	3.5	4	1	2
B7		4	4	5	2.5	3	4	6	7	7	7.5	1	0.5	0.5	3	2
C1	Yes	50	42	61.5	44.5	51	/									
C2		46.5	60	40.5	39	37	46.5	56.5	48	77	88	41	53.5	57	57.5	45
C3		64	49	59.5	62.5	75	71.5	86	59	59	59.5	45.5	43	54.5	30	43
C4	No	15.5	20	5.5	26	27	/									
C5		36	23	35	22.5	39.5	15	14.5	25.5	21	15	7	10.5	6	7	6.5
C6		23	22	26.5	21	32	20	21	16	26	22.5	5	15	8	9.5	7
C7		10	8	7.5	5	9,5	19	13	44	30	33.5	7.5	8	9	7	6.5
N1	Yes	24	26	27	23.5	23	/									
N2		71	63	64	97.5	60.5	52.5	39.5	45	46	52.5	32	41.5	42	32.5	27
N3		90.5	82	69	58	79	71	83.5	71	83.5	61.5	44	51.5	50	52.5	51
N4	No	8	6	14	10.5	11	/									
N5		9.5	10	6.5	7	5	7	10	7	4	9.5	3.5	4.5	5	5	6.5
N6		8.5	9	7	7	11	4	7.5	4	8	3	4	6.5	3.5	5.5	2
N7		4.5	20	10	8.5	9	7	15	9.5	10	6.5	1.5	2.5	0.5	4	4
R1	Yes	31	18.5	35	25	36.5	/									
R2		15	14.5	23.5	16.5	10.5	82.5	51.5	66.5	52.5	85	55	62	64	58.5	54.5
R3		33.5	39.5	28	31	38	45	62.5	60,5	52	56	44,5	30.5	39.5	27	47.5
R4	No	4	5.5	3	5	3	/									
R5		7	7.5	6.5	8	15	8	11	14	7	8	2.5	4	6	5.5	1.5

R5		3	4	3.5	8	7.5	6.5	6	9	6.5	11	4	5	4	3	3.5
R7		6	8	10.5	11	8.5	6	6.5	5	4	8	3.5	4	3	5	3
S1	Yes	102.25	69.5	69.5	72	80	/									
S2		/										52.5	47	42.5	32	35
S3		71	70	65	61.5	73	85	86.5	86	85	73	61	71.5	68.5	60	74
S4	No	5.5	12	12.5	10	8.5	/									
S5		17.5	9.5	8	11	7	5	9	18	10	3.5	2	6.5	3	3.5	6
S6		16	14	11.5	18.5	5.5	7	12	9	5.5	6	1	5	5.5	6.5	4.5
S7		7	9	12	13	5.5	6.5	8.5	8	7	10	4	5	3	8.5	9
T2	Yes	59	65	100	126	105	82.5	92.5	87	79	98	53	60	50	74	40
T3		38	39	22	52.5	25.5	73	97	71.5	81.5	87	55.5	45	54	59	64
T4	No	7	7	5.5	10	14.5	/									
T5		3	2.5	4	3.5	4.5	6	6.5	5	6	8	1.5	3.5	1.5	2.5	4
T6		7.5	23.5	8	7.5	2	7.5	4.5	9	3.5	5.5	5.5	10	4.5	8.5	10
T7		11	10	8	4	9.5	1.5	4.5	5.5	3.5	3.5	2	2.5	3.5	3	2
Blank		1	2	2	1	1	/									
Blank		3	1.5	1	1.5	2										
Blank		0.5	1	2	1.5	0.5										

Table 10: Number of microfibrils counted in every counting zone on the cellulose nitrate filter after filtering the domestic washing machine wash water effluent of the tested samples.

Sample	First cycle					Second cycle					Third cycle				
	Counting zone (MFs/square)														
	C	D	L	R	T	C	D	L	R	T	C	D	L	R	T
PET	35	51	55	34	61	34	46.5	53.5	54	30	57.5	54	45	48	53.5
	38.5	34	20	35	50.5	32.5	45.5	37	42	32.5	42	48.5	49	48	57
	9	11.5	20	25.5	32	8.5	4.5	12.5	7	9.5	11.5	15.5	18	16	6
	14	17.5	20	31.5	19.5	12	14.5	22	15	12.5	17	22	19	11	10.5
	5.5	12	5	40.5	10	4	16	6	9.5	6.5	6	10	7.5	7.5	8
	13	15	9.5	16	5	9	15.5	7.5	14	9.5	10	11.5	7	5.5	10.5
COT-PET	62	67.5	51	58.5	51	16.5	22	21.5	25	18.5	24	32	27.5	28	34
	37.5	39	48.5	43.5	49	51	39	66.5	33	34	33.5	33	20.5	36	41
	6	4	17	7	12.5	8	6	11	7.5	12.5	11.5	17	18	16.5	22
	21	22.5	44.5	36	24	22	25	14	18	25	20.5	22	16	23	9
	9	14	3.5	13	13	10.5	15.5	9.5	13	36	10.5	11.5	19	14	14.5
	27	21	14	28.5	29.5	15	22	26.5	19	21	7.5	11	11.5	16	14.5
Blank	52.5	54.5	72	61	61	/									
	40	51.5	43.5	51	40										
	8	11	8.5	7	11.5										
	7	15	13.5	19	17										
	7.5	7	2.5	16.5	0.5										
	2.5	4.5	8	10.5	11.5										

Appendix C: DLS data

Table 11: DLS Z-average and PDI.

Sample	Z-AVERAGE (nm)	PDI
BLANK	211.7	0.362
N1_1R_1L	135.6	0.186
N4_OR_1L	183.6	0.499
R1_1R_1L	133.9	0.326
R4_OR_1L	97.2	0.421
T1_1R_1L	131	0.288
T4_OR_1L	125.8	0.461
S1_1R_1L	202.5	0.467
S4_OR_1L	110.2	0.235
B1_1R_1L	121.1	0.185
B4_OR_1L	124.7	0.282
C1_1R_1L	607	0.57
C4_OR_1L	558.5	0.692
BLANK_WM	1611	0.834
PET_WM	1359	0.797
COT_WM	1231	0.795

Appendix D

Table 12: DLS size distribution spectra.

