TACKLING MICROFIBRES AT SOURCE

Investigating opportunities to reduce microfibre pollution from the fashion industry through textile design and manufacturing innovation

Technical Research Report

FORUM FOR THE FUTURE

Forum for the Future Asia Pacific Limited
Prepared for the UNDP Ocean Innovation Challenge
Contact information: Karen Sim k.sim@forumforthefuture.org

SECTION 1: SUMMARY OF THE TECHNICAL RESEARCH
Authors: Jael Chew, Karen Sim, and Nicole van der Elst Desai from VDE Consultancy

SECTION 2: RESEARCH PLAN AND METHODOLOGY

AND

SECTION 3: FINAL REPORT ON RESEARCH PLAN AND RESULTS
Authors: Junjie Yang, Mengjing Wang, Thomas Fang Mingliang, Xunchang Fei (NEWRI); Jael Chew and Karen Sim (Forum for the Future)

We also thank Nicole van der Elst Desai from VDE Consultancy for co-designing the study's scope, methodology, and flow

Contents

SECTION 1: SUMMARY OF THE TECHNICAL RESEARCH

Executive summary

The Tackling Microfibres at Source project has as its objective “Investigating opportunities to reduce microfibre shedding in the fashion industry through textile design and manufacturing innovation.”

The project identified the research scope and focus textiles and colours, to understand the microfibre shed at the textile manufacturing process, and to establish which step contributes the most shed.

The topic of textile fibres pollution caused by the fashion industry is an emerging one, which means that new information and knowledge emerges regularly; and thus, we incorporated flexibility and adaptability in our approach.

During the research, the COVID-19 pandemic and lockdowns in Singapore and Malaysia disrupted our ability to collect water samples and transport them across borders. We mitigated this by bringing in a Malaysian research lab, UTM, to handle the water samples and transfer them in dry form to NEWRI.

The recommendations for further research are to include all existing measurements to limit the shedding of the textile fibres. Our research found that excluding the data of fibre/lint leads to an incomplete view of the contribution of manufacturing processes to textile fibre shedding.

The proposed fibre shedding profile can serve as a comparison of an existing textile to a preferred textile, i.e. virgin polyester fleece compared to recycled polyester fleece. Virgin polyester, for example, showed a better profile which is useful complementary information for the industry to consider when viewing or developing preferred textiles or textiles assumed to have less environmental impact.

The methodology requires further refinement in terms of testing of the triple sampling and in terms of using different sampling points and sample types, as comparisons are difficult to make i.e. heat setting vs before brush sample; a water sample vs dry sample. It will also benefit from a validation of the correlation between water and dry samples for a more in-depth understanding of the results.

Lastly it should be noted that the results obtained, and conclusions drawn are specific to the facility of Ramatex in Malaysia.

Introduction to the research

This final report describes the **Research** conducted for the project*: Tackling Microfibres at Source: Investigating opportunities to reduce microfibre shedding in the fashion industry through textile design and manufacturing innovation.*

It is important to mention that this research report has been co-created with the input from both the research team at NEWRI and the materials team at Ramatex. Without them, this research would not have been possible.

Also critical to keep in mind is that the topic of textile fibres pollution caused by the fashion industry is an emerging study. It was necessary to be flexible and adaptive in our approach, as new information and knowledge surfaces regularly in the course of our research.

The ongoing COVID-19 pandemic has impacted not only our ways of working but also the practicality of obtaining samples from the factory and transporting these to the research labs.

In this report, you will find the research plan and methodology, the results and findings, and the complete NEWRI research report.

Research plan and methodology

The research consists of four interlinked phases, the first phase of the research is the **Research plan** in which the foundational work is captured, involving the research scope and the identification of which textiles and colours to include.

The focus of the research team was answering the 2 main research questions:

1. *'How to take samples from the processing step in the manufacturing location?'* and:
2. *'How to conduct testing and analysis in the testing location?'*.

The answers to these two questions set the blueprint for the subsequent phases in research.

A visual workflow of the research plan is seen in image 1:

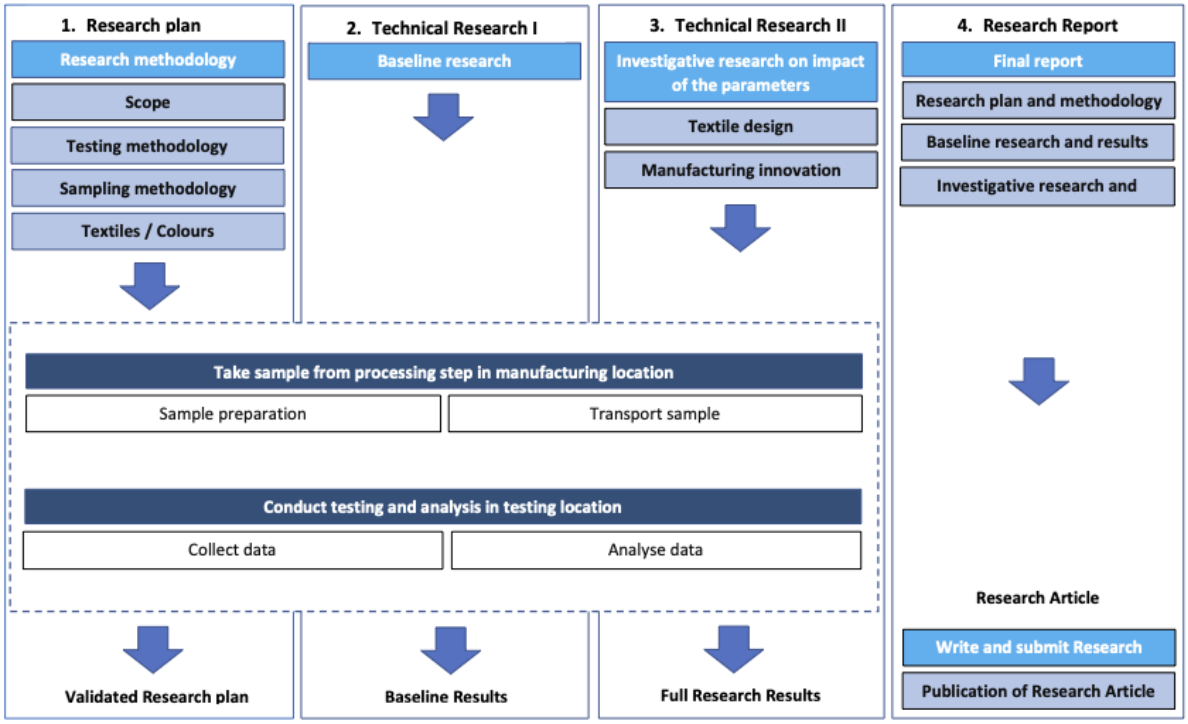


Image1: Research plan workflow

In the **Technical research I**, the Baseline research, the team collected data, and the subsequent analysis provided insights into which of the identified textile processing steps are contributing most to microfibre release within the textile manufacturing facility. In this phase the areas that warrant further investigation are identified.

In the **Technical research II** phase, the Investigative research is conducted. This research phase is an exciting phase as it is an opportunity to understand if implemented changes to either textile design and/or manufacturing innovation has (any) positive impact on the microfibre release of the chosen processing steps for further investigation. The hypothesis is that a reduction of microfibre shed at the upstream manufacturing level will lead to an overall reduction of microfibre at the domestic laundering level.

The research is concluded with this **Final Report** which describes the Research plan & Methodology, the Results & Findings of the Baseline, Investigative and includes Complementary research.

The final deliverable of the research is the publication of the **Research Article**.
The research duration was planned for 12 months, but COVID-19 related restrictions in Malaysia and Singapore continued to have a material impact on the research. The end date for the research was delayed and the technical research phase II was completed in October 2022.

Confirmed Research Plan

Our research focuses on the upstream steps that involve turning raw material into polyester or cotton yarn, which is then made into fabric and undergoes different treatment, colouration and finishing processes.

The **scope** of the research is determined with input from the Ramatex team on the most prevalent **processing steps** and the most high-volume **fabrics and colours** at their facilities in Malaysia.

A visual summary of the baseline research methodology and scope is seen in image 2.

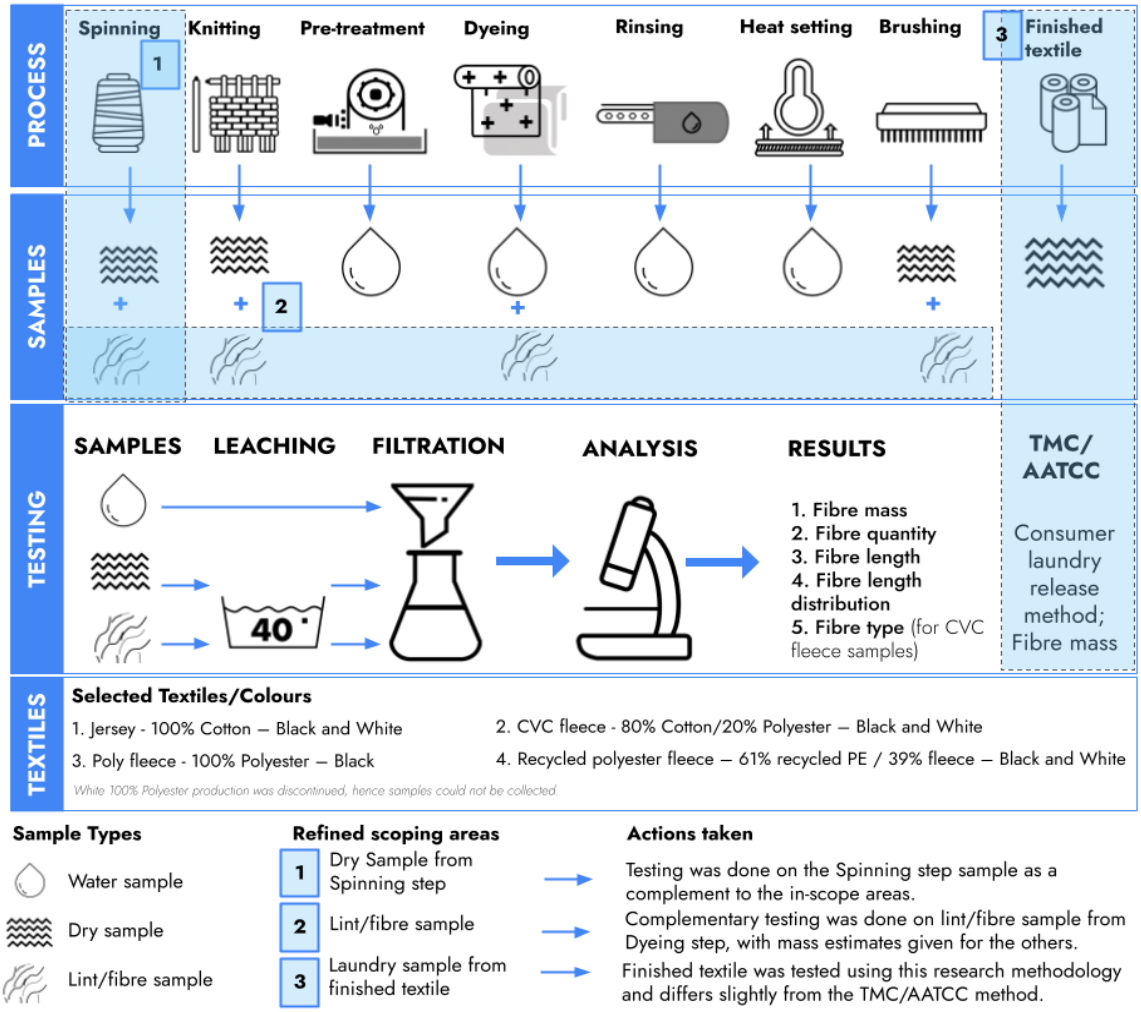


Image 2: Overview of baseline research methodology and scope

Processing steps

As the research looks specifically into microfibre release from processing steps solely within the textile manufacturing facility, at this stage it does not include other factors such as the transport or fate of microfibres.

Processing step	Description
Spinning	The process of producing yarns from the extracted fibres.
Knitting	First, the yarn is put into the knitting machine creels. After that the yarn is passed through the knitting machine where the yarn is converted into knitted greige and it is wound in the roll form.
Pre-treatment	The process of removing impurities from greige and bringing it to a stage where it is more suitable for coloration.

Dyeing	The interaction between a dye and a fibre, as well as the movement of dye into the internal part of the fibre.
Rinsing	The removal of any excess dyes and chemicals.
Heat setting	The coloured textile is subjected to the action of high temperature for a short time to make it dimensionally stable so that the garments made from such fabrics retain their shape.
Brushing	A mechanical finishing process used to raise the surface fibres of a coloured textile.
Finished textile	The final stage of the coloured textile which is wound in the roll form.

Image 3: The manufacturing processing steps that take place at Ramatex facility

Samples

From each step we collected either **dry** or **water samples**. The different types of samples reflect the nature of the process steps. For instance, raw cotton and PET chips go through a spinning process that turn the fibres into yarn, as seen in image 4. Hence, the sample collected from the end product is a dry yarn.

Image 4: left – a Ramatex worker conducting quality checks on spools of polyester yarn. Right: cotton in the process of being spun. Images: Ahmad Iskandar Photography.

In dyeing, water is used in the process and released after completion. Therefore, a water sample is collected for testing. Image 5 captures what happens in the Ramatex dyeing mill.

Image 5: Ramatex dyeing mill Photo: Ahmad Iskandar Photography.

Further to this, certain process steps produce **lint/fibres** that are captured by different devices at Ramatex. A vacuum system captures lint/fibres released to the atmosphere during spinning, knitting and brushing. The jet dyeing tanks used by Ramatex have a built-in auto lint/fibre collection device.

Apart from the wet dyeing lint/fibre amassed inside the device, it is difficult to attribute the vacuum collected lint/fibre using to individual textile types as it is a general collection system operating over extended periods and emptied when full. Hence, we took estimates from Ramatex to obtain an understanding of the total contribution of each of the processing steps.

Image 6 shows the sample types and quantities collected:

Dry samples *As part of the testing methodology, the dry samples are leached to release their fibre content in water*		**Water samples** *Discharge water samples*	
Processing step	**Sample quantity**	**Processing step**	**Sample quantity**
Spinning	Cone – 1 cone per yarn type	Pre-treatment	4 L
Knitting	Greige – 400 gm	Dyeing	4 L
Brushing	400 gm	Rinsing	4 L
Finished sample	400 gm	Heat setting	4 L
Lint/Fibre samples			
Vacuum system; daily averages (generic)		*Built-in auto collection; specific to textile (specific)*	
Spinning	Min. 100 gm	Dyeing	Min. 100 gm
Knitting	Min. 100 gm		
Brushing	Min. 100 gm		

Image 6: Sample types and quantities collected

Testing

To find out the profiles of the microfibres contained, a testing methodology was developed by NEWRI to leach the dry samples, filter the leached samples and the water discharge samples, and analyse the results under microscope.

The results are expressed in weight, quantity and length and distribution of the shedded textile fibres. For the CVC fleece samples comprising cotton and polyester, an additional chemical separation step was performed, resulting in a fibre type.

Image 7 shows the flow of the testing methodology.

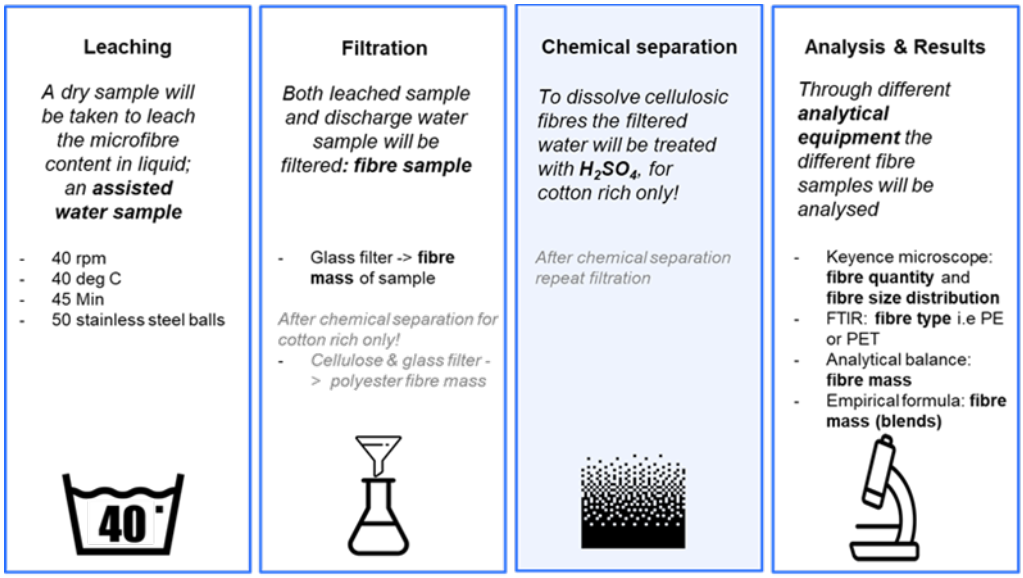


Image 7: Flow testing methodology

Image 8 shows the Testing protocol and obtained Result types including the highlights of some of the key elements and considerations taken in determining the processes and parameters of the testing.

Result type	**Measurement unit**	**Description**
Fibre mass	mg fibre / g textile	The average weight of a fibre. This result type facilitates a link to the industry method used by The Microfibre Consortium (TMC) and American Association of Textile Chemists and Colorists (AATCC), which both solely report on fibre mass.
Fibre quantity	counts/g	To learn about the number of fibres in the sample
Fibre length	µm	To learn more about the size of the fibres, and its distribution.
Fibre length distribution	%	
Fibre type	N/A	To know the raw material which the shedded fibre is made of, especially the stable polymer used. Note this test was applied only to the samples of the CVC fleece – 80% cotton / 20% polyester.
Testing process step	**Key points to highlight**	
1. Leaching	The leaching method is based on the ISO 105-C06 with some modifications. A blank is also processed the same way alongside to indicate any background contamination, and corrections are made when needed.	
2. Filtration	The liquid samples run through a filtration system with vacuum pump using a glass fibre and cellulose filters. Oven-drying is used as a preparation and final step. Using a Keyence Digital Microscope with a VHX Digital Microscope Multi Scan Lens the research institute analysed the fibre residue. The filter image provided the fibre's mass, quantity, length and distribution results.	
3. Chemical Separation	An extra step performed on textiles that are cotton rich to separate from polyester, or other stable polymer. This chemical separation is based in DIN EN ISO 1833-11.	

Image 8: Testing protocol and result types

The research institute adopted a minimum quality assurance measure to ensure qualitative and reproducible results in this research. This includes blank samples, control samples, use of triplicate samples in separate filters, validation of method accuracy, storage and so on.

The standard operating procedures for sampling by Ramatex and the research institutes are detailed in the Research Report created by the NEWRI team (Annex A: NEWRI Research Report).

Textiles

The operating principle of this project is to analyse textiles that are produced and purchased in the highest volumes, or are of importance to the industry, so as to create the greatest impact through this research.

We chose the two most popular colours to understand if this would have an impact on the results. The textile and colour types selected for the baseline research is shown in image 9.

Textile and composition	**Weight** gsm	**Colour**
CVC Fleece 80% Cotton / 20% Polyester	250	Black
	280	White
Jersey 100% Cotton	145	Black
	190	White
Poly Fleece 100% polyester	180	Black
	180	White *
Recycled Poly Fleece 61% recycled Polyester / 39% polyester	180	Black
	180	White
**When operations resumed, Ramatex discontinued production of the white Poly Fleece due to lack of demand. This was indicative of an increasing shift away from virgin to recycled polyester.* *It was not possible to source for old stock from other Ramatex factories. There was the option of a small production run for this study, but the resources used, and the fibres emitted would run counter to the objectives of this project.*		

Image 9: Textiles and colours baseline research

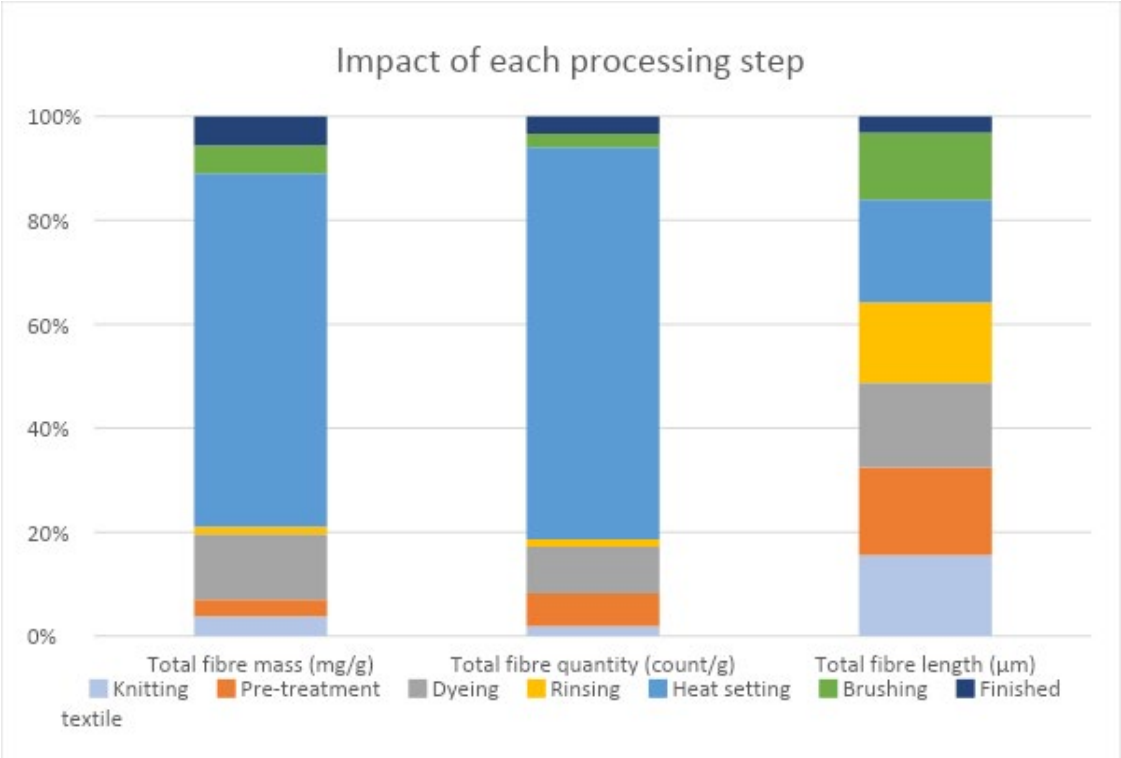
Summary Results & Findings

This novel research into the impact of the different manufacturing processes was an exciting and interesting journey, impacted by the COVID restrictions but also by findings in the baseline which led to an additional section on complementary testing.

The complementary testing provides space to include results in this report that holds value in understanding better the impact and at the same time opportunities to reduce this impact of fibre pollution in the manufacturing stage.

Baseline research results

The objective is to identify the research scope and focus textiles and colours, to understand the textile fibre shed at the textile manufacturing process, and to establish which step contributes the most shed. In the Ramatex facility in Malaysia, the total contribution of the seven identified textiles shows that for each of the different result types – fibre mass, fibre quantity and fibre length – the heat setting was the most impactful processing step.

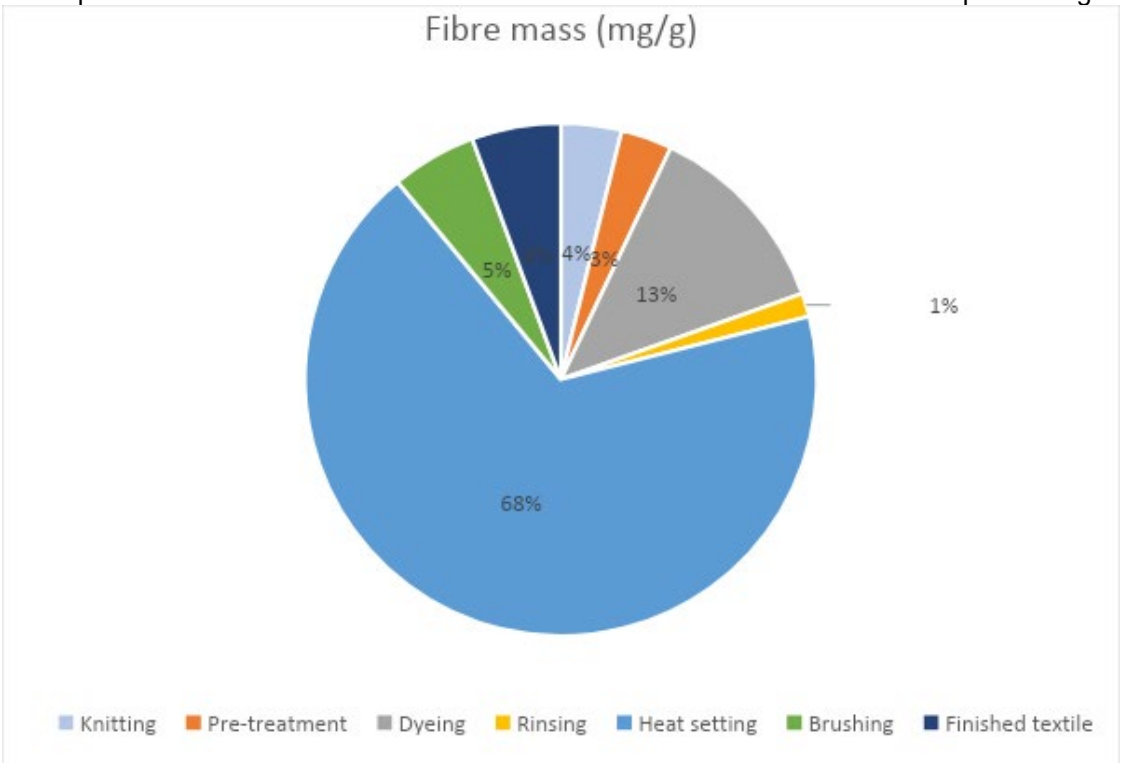


Main testing in baseline phase

The main testing results in the baseline phase are collected for each processing step. It should be noted that for the fleece samples the brushing is the last processing step.

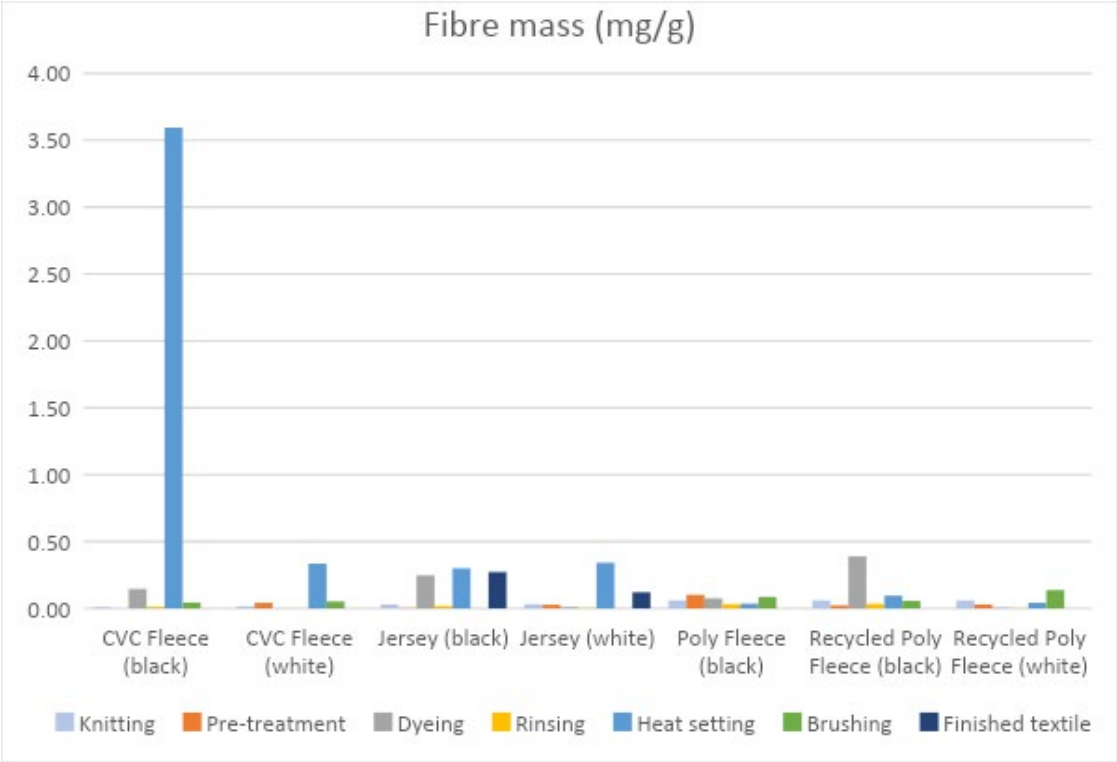
Fibre mass

A comparison between the different fibre mass results obtained for each different processing step:



Following these results, it is recommended to look into the heat setting results to understand it's significant contribution (68%) to the total fibre mass; as a secondary investigation dyeing (13%).

When diving deeper into the different textiles:

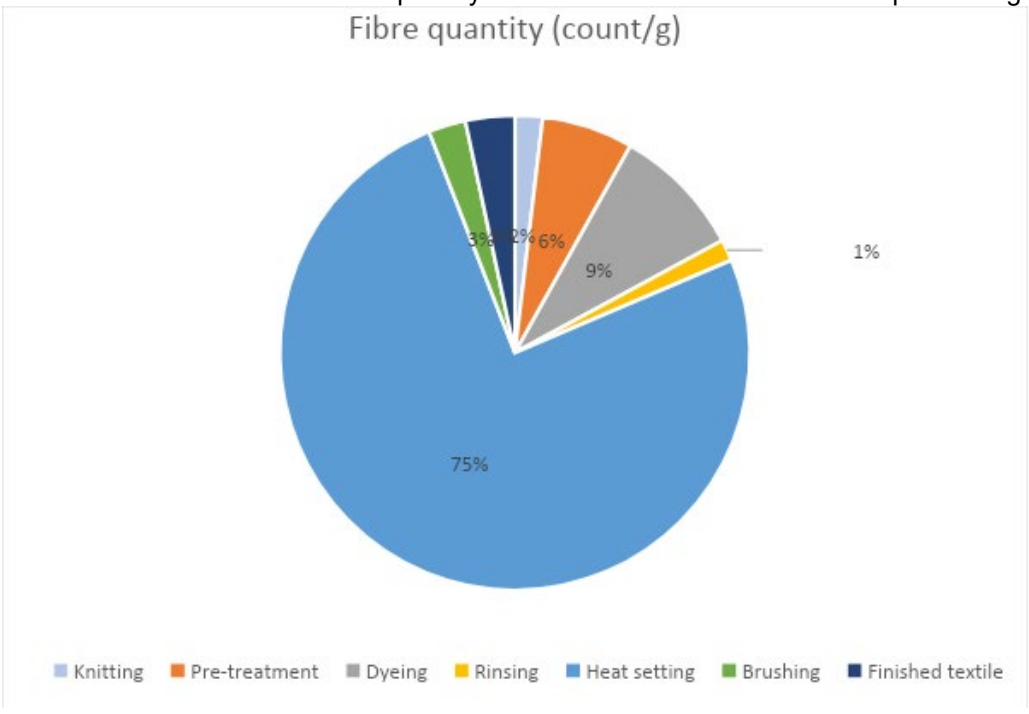


The heat setting results for mass for CVC Fleece black are very high and warrant further investigation. Within the scope of this research, the team decided to keep with using the average of the triplicate results to inform the Technical research II phase (investigative research).

The recommendation for the next step beyond this project is to conduct additional testing on the CVC Fleece (black) with an increased sample size, to validate the findings.

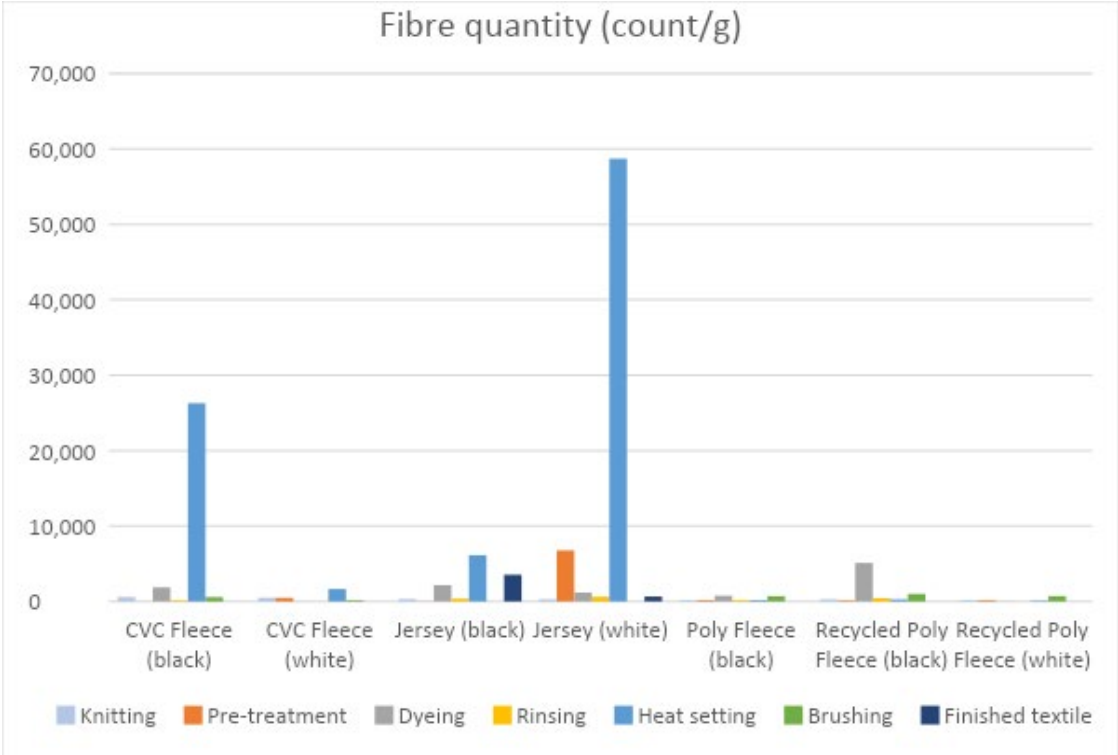
Fibre quantity

A comparison between the different fibre quantity results obtained for each different processing step:



In the view of fibre quantity, again the heat setting process is the larger contributor to the total quantity of fibre shed. It has a similar view as the fibre mass, in terms of being the highest contributing processing step.

When diving deeper into the different textiles:

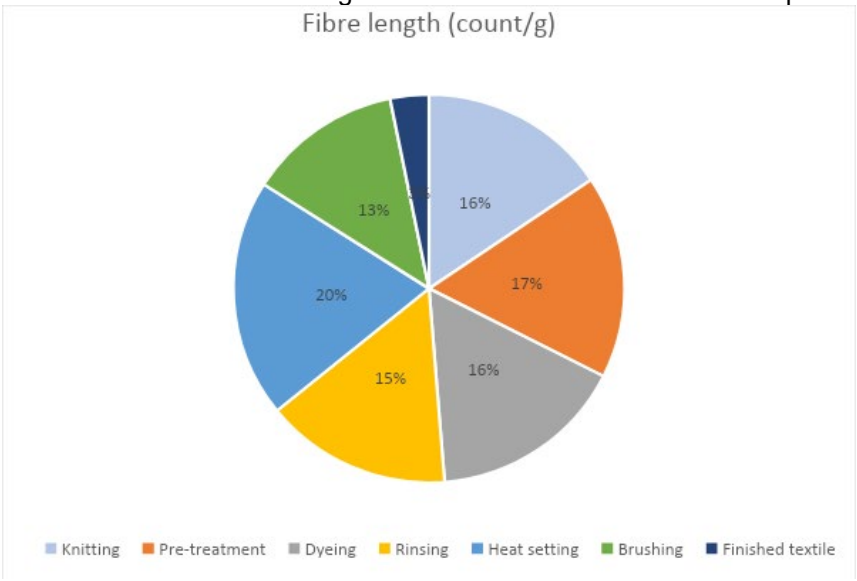


The fibre quantity for both CVC Fleece (black) and Jersey (white) are high; these warrant further investigation as to the cause of this result. Within the scope of this research, the team decided to keep with using the average of the triplicate results to inform the Technical research II phase (investigative research).

The recommendation for the next step beyond this project, is to conduct additional testing on the CVC fleece (black) and Jersey (white) with an increased sample size, to validate the findings.

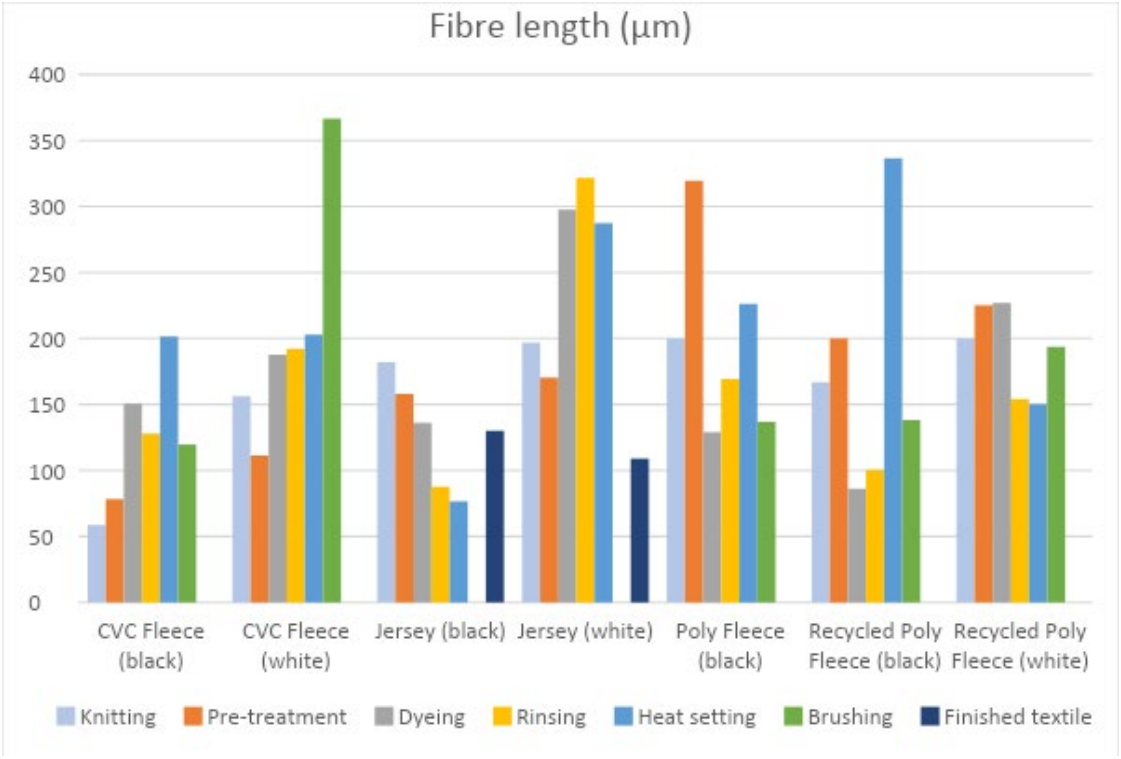
Fibre length

A comparison between the different fibre length results obtained for each different processing step:



The graphs shows that there is no significant process contributing to the fibre length.

On a deeper textile level, the graph shows:

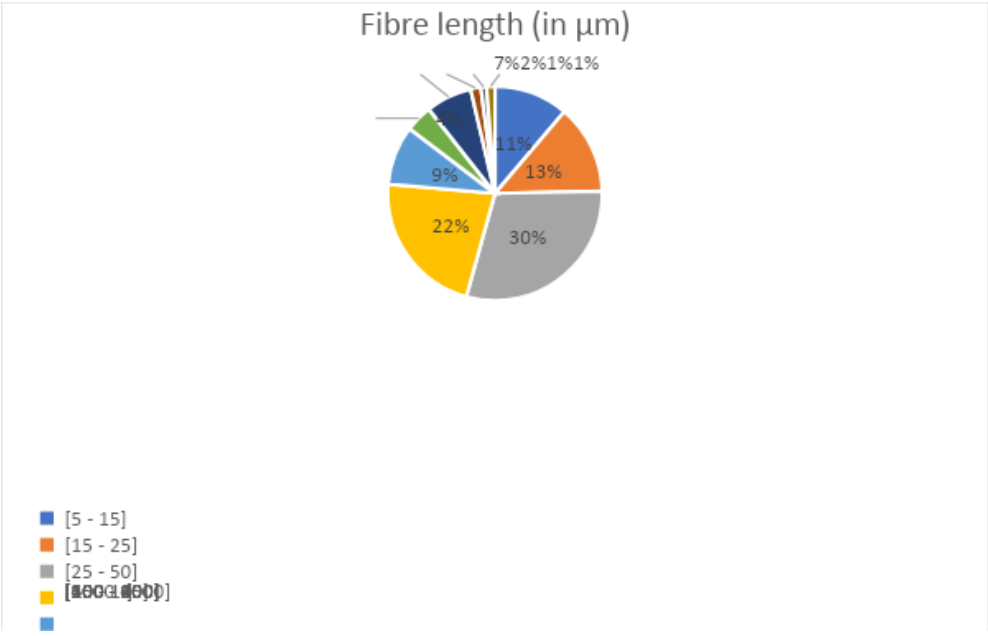


The following observations can be shared as an indication for further research outside of this project:

- CVC Fleece (white) average length for brushing is significantly higher than the other processing step results
- Poly Fleece (black) average length for pre-treatment processing step is higher than the other processing steps
- Recycled Poly Fleece (black) average fibre length is significantly higher for heat setting

Fibre length distribution

The fibre length distribution is an interesting view to understand the length of the fibre shedding for the different textiles in the different processing steps.



The observation is that most of the fibre lengths (µm) are found in the following 4 groups:
[25 – 50] = 30%
[50 – 100] = 22%
[15 – 25] = 13%
[5 – 15] = 11%

This is comparable for each of the different textiles.

Fibre fragmentation profile

The fashion industry has seen a great push towards adopting preferred textiles, as these are believed to have less environmental impact. In these comparisons, textile fibre fragmentation has not yet been included.

As a suggestion for industry, developing new textiles to replace existing ones, it is recommended to **compare the fibre fragmentation profile** of the textiles to assure that the profiles of the new textiles are comparable to the existing ones, or better.

A like-for-like comparison was performed on the Recycled Poly Fleece and the Poly Fleece as the textiles are composed of the same yarn type and weight. It shows that Recycled Poly Fleece (black) has significantly higher fibre mass, and also a significantly higher fibre quantity. The Poly Fleece (black) has a better performance in terms of fibre shedding.

The below data is not including lint/fibre shedding:

Poly Fleece (black) **100% polyester (180gsm)**	**Fibre shedding**		
Processing step	Total fibre mass, average mg/g	Total fibre quantity, average count/g	Total fibre length, average µm
Knitting	0.060	188.067	200.077
Pre-treatment	0.102	174.222	319.683
Dyeing	0.076	763.800	128.913
Rinsing	0.032	190.440	169.147
Heat setting	0.035	182.667	226.313
Brushing	0.087	706.813	136.927
Finished textile*			
Total	0.391	2,206.009	

Recycled Poly Fleece (black) **61% recycled polyester/31% polyester (180gsm)**	**Fibre shedding**		
Processing step	Total fibre mass, average mg/g	Total fibre quantity, average count/g	fibre length, average µm
Knitting	0.060	288.067	166.940
Pre-treatment	0.022	139.422	200.004
Dyeing	0.389	5,108.185	86.165
Rinsing	0.035	444.926	100.391
Heat setting	0.096	276.667	336.515
Brushing	0.057	1,002.650	138.134
Finished textile*			
Total	0.658	7,259.917	

**Note: the final step of the fleece textiles is brushing, hence this data point represents the finished textile.*

This is an important finding. When cross-referenced against available open-source data, some studies have found that rPET fibres shed more than PET fibres, and that rPET knitted fabrics released almost 2.3 times more fibres than the virgin PET fabrics.[1] At the same time, we also found several sources indicating that the key determinants for microfibre shedding in recycled polyester fabrics and virgin polyester fabrics was actually textile construction, rather than recycled fibre content. This is discussed in further detail in the Impact Report (Annex 7 submitted to UNDP).

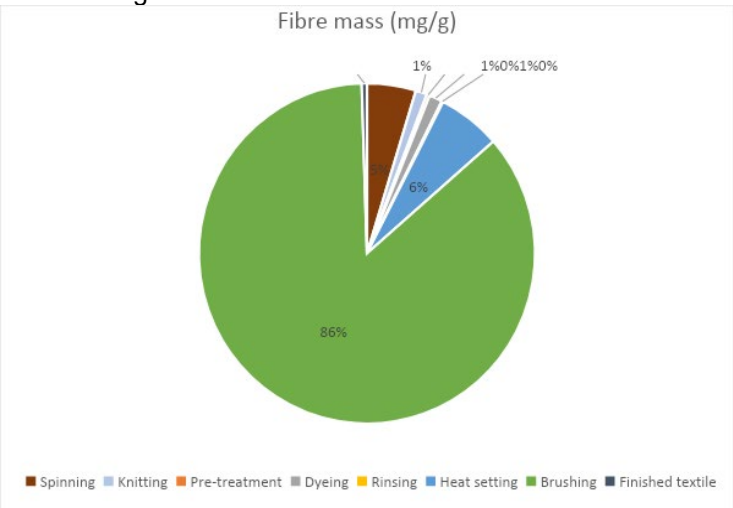
A critical recommendation to the industry is to include **comparing fibre fragmentation profile** in as part of the total environmental impact when suggesting preferred materials.

Complementary testing in baseline phase

Impact of lint/fibre results

One of the learnings is that the textile machinery used by Ramatex and also the processes within its facilities are including extra measurements to limit the shedding of the textile fibres. This learning also impact the fibre shedding profile.

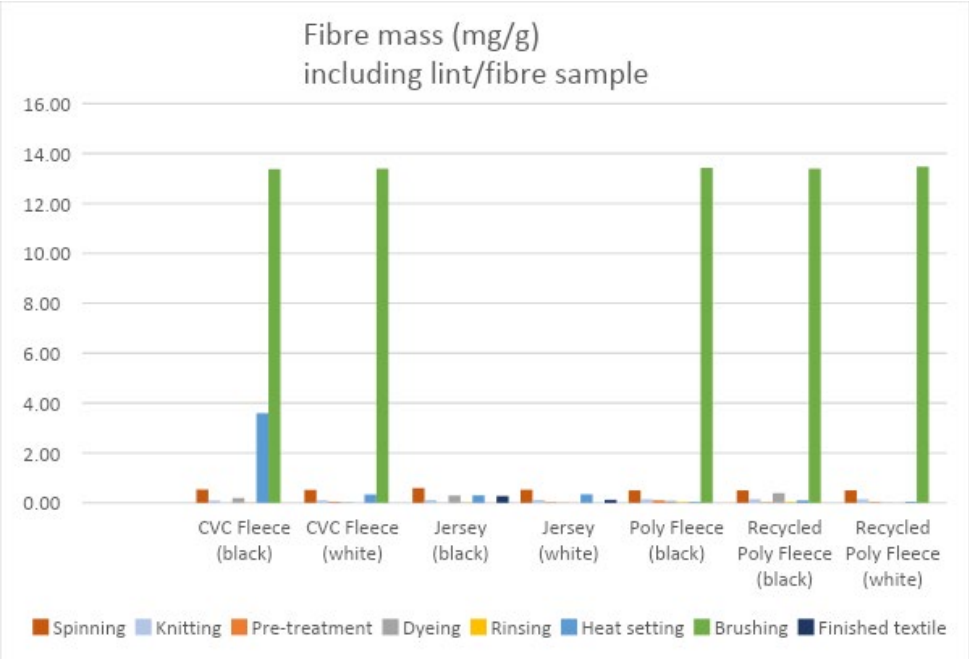
When adding the fibre mass results of the lint/fibre samples on a fibre mass level, a different processing step shows high results: brushing.

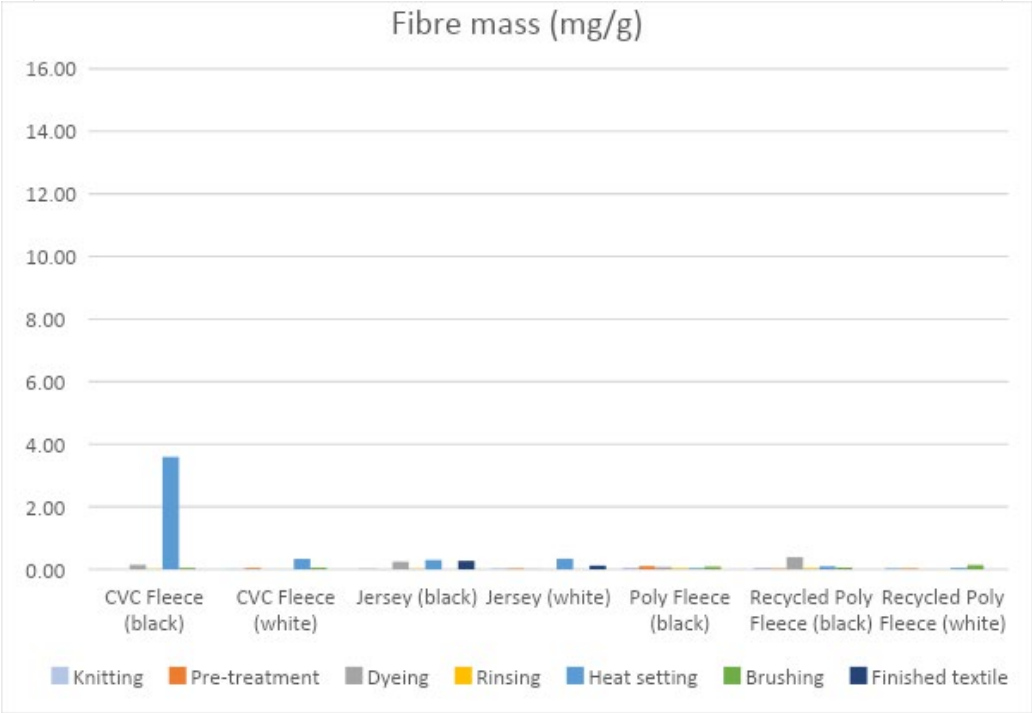


Following these results which include Ramatex's extra measurements to limit the shedding of the textile fibres; it is recommended for further research outside this project to look into the brushing results to understand it's significant contribution (86%) to the total fibre mass; and as a secondary investigation, to also look into heat setting (6%).

When diving deeper into the different textiles:

[1] İlkan Özkan & Sedat Gündoğdu (2021) Investigation on the microfiber release under controlled washings from the knitted fabrics produced by recycled and virgin polyester yarns, The Journal of The Textile Institute, 112:2, 264-272, DOI: 10.1080/00405000.2020.1741760





This data comes from generic shedding captured by vacuum at processing steps: spinning, knitting and brushing; and specific shedding captured by dyeing tank lint collector after each batch.

Excluding the fibre mass data on generic and specific fibre/lint leads to an incomplete view of the contribution of manufacturing processes to textile fibre shedding.

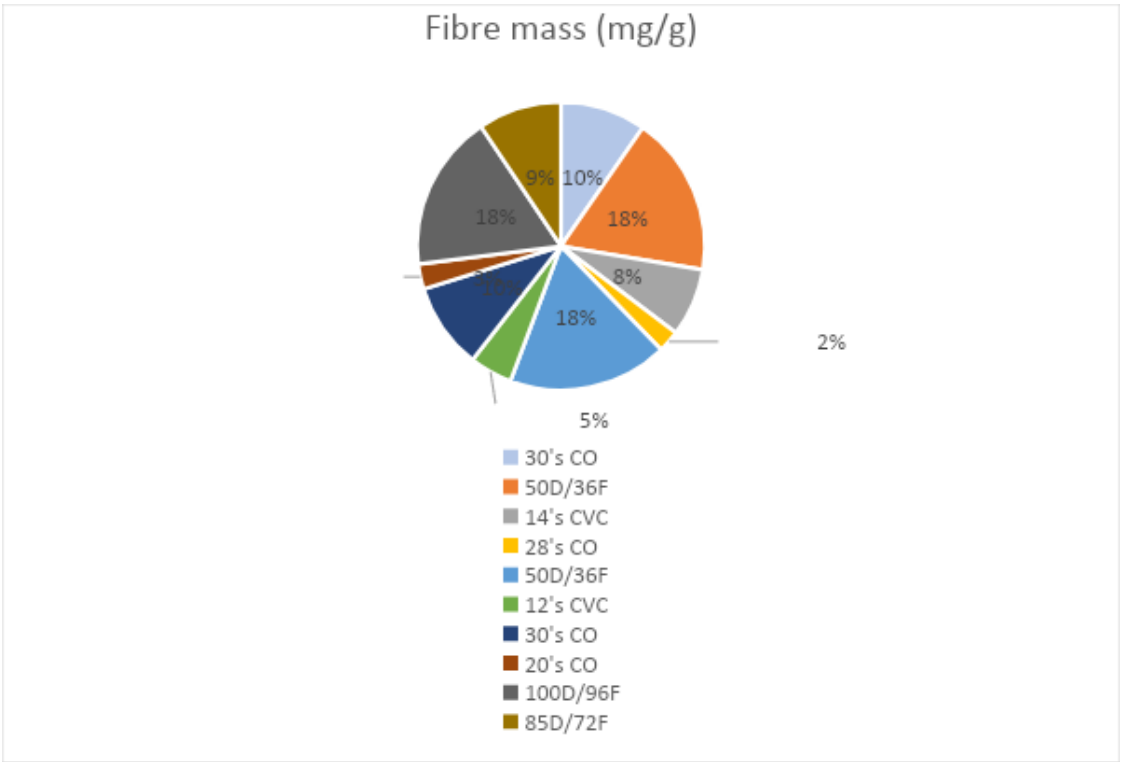
A recommendation for further research conducted in the future is that all measures within a manufacturing facility should be taken into consideration and the data should be used in the overall calculation to understand the total impact on microfibre shed.

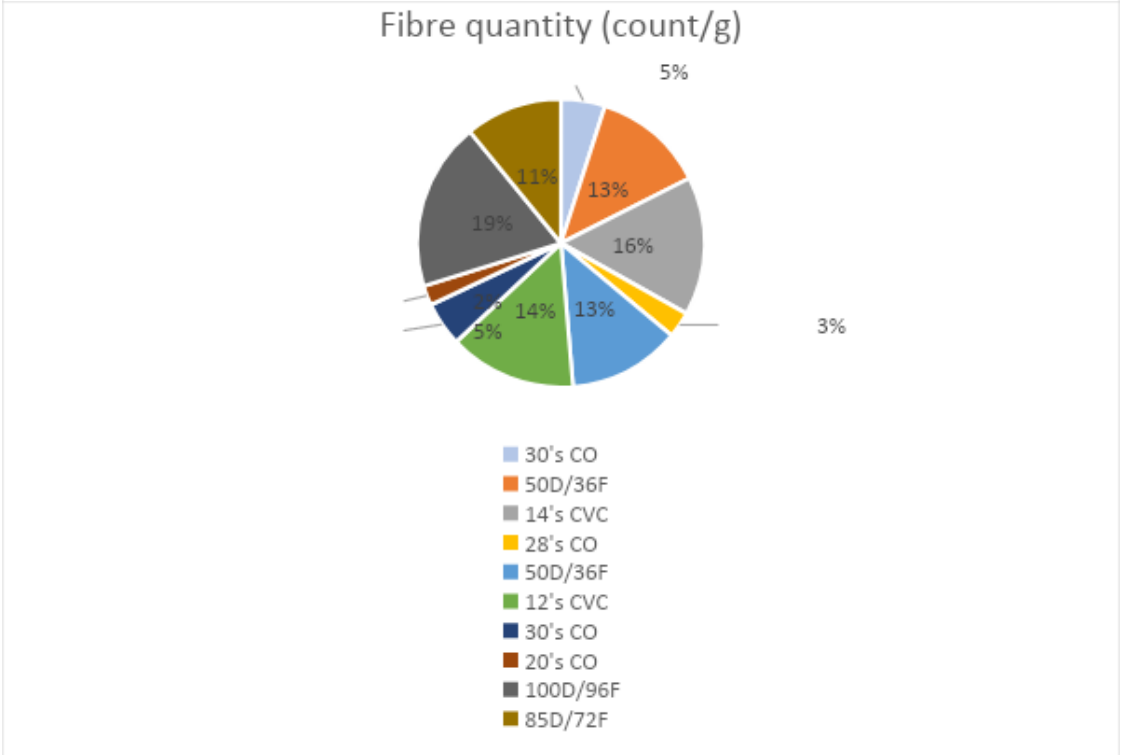
Also, looking at ways to reduce the impact of brushing through design interventions should be considered. A suggested starting point could be to reassess the purpose of brushing textiles, i.e. to create extra softness, to create extra warmth, and for each of these purposes seek alternative solutions and investigate their impact with the objective of reducing fibre shedding.

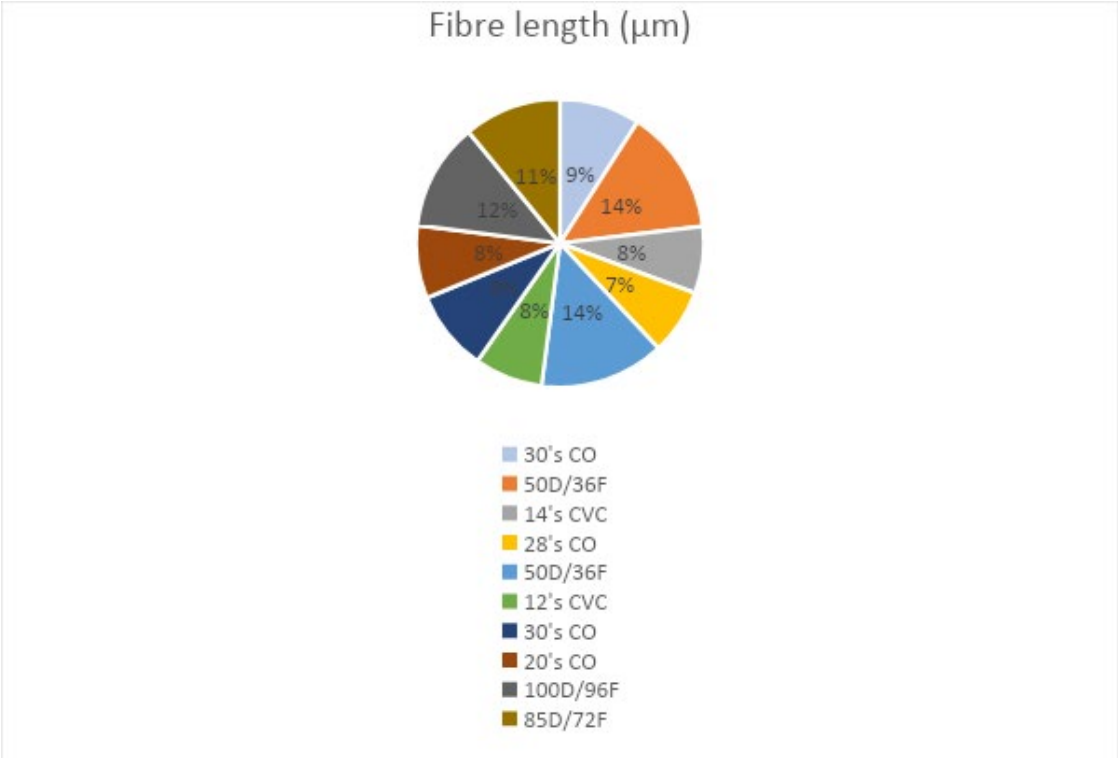
Comparison of different yarns:

For the processing step spinning, the researchers looked at the yarns individually.

It was challenging to attribute the results to the respective textiles in the baseline testing. That said, it still holds value to include this in the report as a play with the ratio of these yarns could hold value in informing the design of textiles and/or considering different quality suppliers as intervention measures that can be explored outside this project.







A recommendation for subsequent research in this area is to include the ratio of the yarns to fully understand the impact of fibre shedding and to look at opportunities to intervene in yarn ratio to reduce microfibre shed.

A challenge arose in identifying which textiles the results of the yarns 100D/96F and 85F/72F belonged to, in relation to the Poly Fleece (black) and Recycled Poly Fleece (black and white). That said, we have presented the data under complementary testing as it is a useful reference for a more complete understanding the different fibre fragmentation profiles in these general yarn types.

Fibre type identification:

The research team set out to look for a way to identify the raw material which the shed fibre is made of, especially the stable polymer used. Note this test was applied only to the samples of the CVC fleece – 80% cotton / 20% polyester. And whilst the results for the stable polymer could be confirmed; there were no results obtained for the contribution of cotton fibre.
The researchers concluded that a Thermogravimetric analysis (TGA) can be used for the composition validation for the raw material of blends containing a cellulose and polymer fibre (Image 10). At the same time it should be noted that this method is not sensitive and cannot be used to detect the leached fibre fragments.

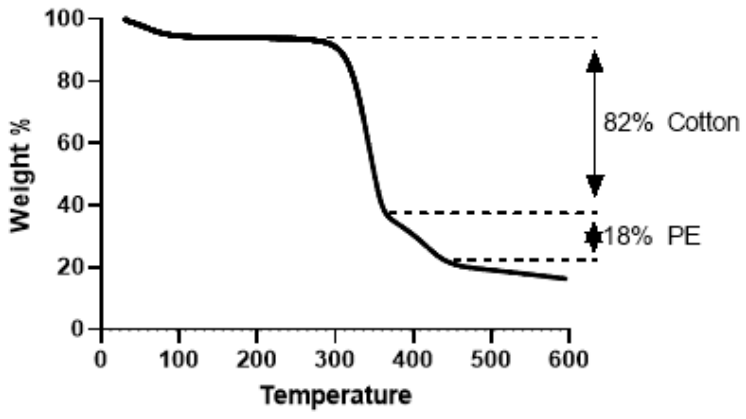


Image 10: Thermogravimetric Analysis (TGA) of the ratio of cotton and polyester

Comparison results wet and dry samples:

During the research, the team conducted testing on a set of samples for which a comparable result might be expected, though this is not the case. This relates to the process steps that take place consecutively: Heat Setting → Brushing → Finished Textile.

The sample *after* heat setting could be anticipated to be comparable to the *before* brushing sample, as it would be at the same point in processing, only as a different sample type (after heat setting producing a wet sample, before brushing being a dry sample) . Similarly, the sample *after* brushing and the finished textile sample are from the same point in processing.

As an example, you can find the results for textile CVC Poly fleece (black):

CVC Poly Fleece (black)					
Comparison	**Processing step**	**Sample type**	**fibre mass (mg/g)**	**fibre quantity (count)**	**fibre length (µm)**
Impact Heat setting process	Heat setting	Water sample	3.592	26,269.200	193.000
	Before brushing	Dry sample	0.155	2,392.333	143.000
Impact Brushing process	After brushing	Dry sample	0.044	579.333	120.000
	Finished textile	Dry sample	0.143	3,699.685	199.000

Since the results are significantly different on fibre mass and fibre quantity it is warranted to conduct further research outside this project to understand what the cause for this is.

For subsequent research it is recommended to further investigate as to what the possible cause is for this. Could it be that a triplicate sample size is too small? Is a comparison between a water sample and a dry sample not the correct methodology to follow? And so on.

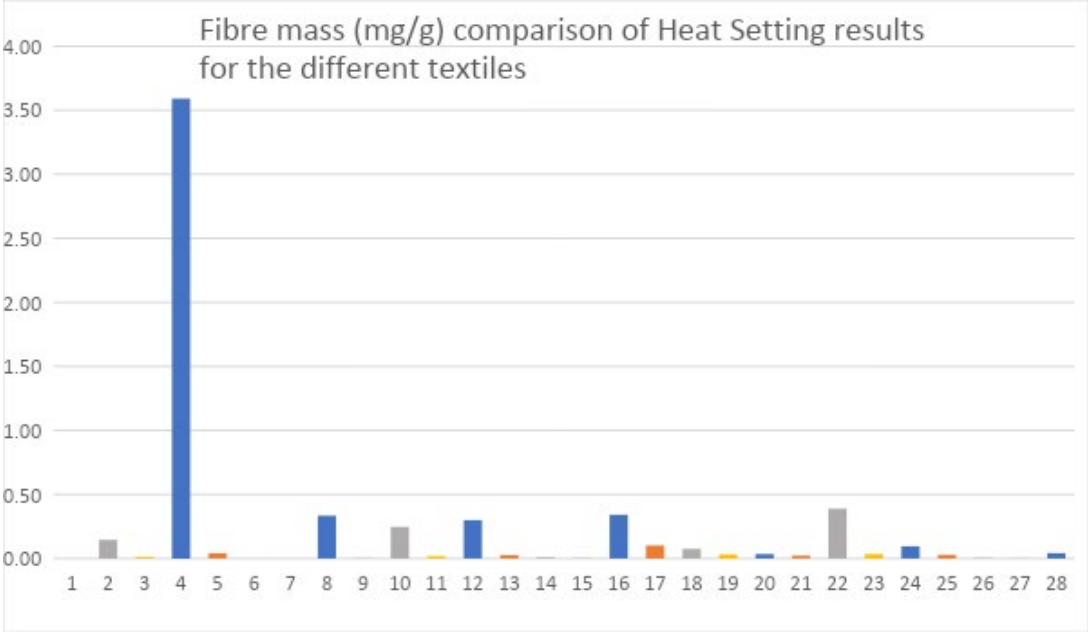
Investigative research results

The investigative research has a focus on fibre shedding mass, the team has chosen to do so as this is in line with most of the existing research, and is the standard measurement used by industry groups such as The Microfibre Consortium and the American Association of Textiles and Colourists.

From the baseline there were three areas that the researchers identified for intervention:

1. Heat Setting
2. Temperature and duration
3. Comparison of other variables on microfibre shed

Heat setting



A trend can be observed for textiles containing a cellulosic fibre; CVC Fleece (black and white), Jersey (white); though for Jersey (black) the dyeing process has a relatively comparable result. The trend is not observed for the polymer fibre containing textiles. The assumption is that the structure of the natural cellulosic fibre, a staple fibre, could possibly be susceptible to loose fibres settling, or 're-attaching' onto the textile; and a polymer fibre, a filament, is unable or less so.

In cotton fibres, modern fibre theory suggests that each cellulose molecule is present within two more crystalline regions of cellulose and held together. Between the crystalline regions in cotton, amorphous unordered regions are found. Voids, spaces and irregularities will occur in these amorphous areas – which allows dyestuffs and chemicals to penetrate readily[2].

This could in turn be indicative of the susceptibility of cotton fibres to the attachment of other elements, like microfibres. Image 11 shows the structure and microscopic view of cotton fibre; image 12 shows the SEM view.

[2] https://www.texcoms.com/wp-content/uploads/2019/06/Textile-Fibres.pdf

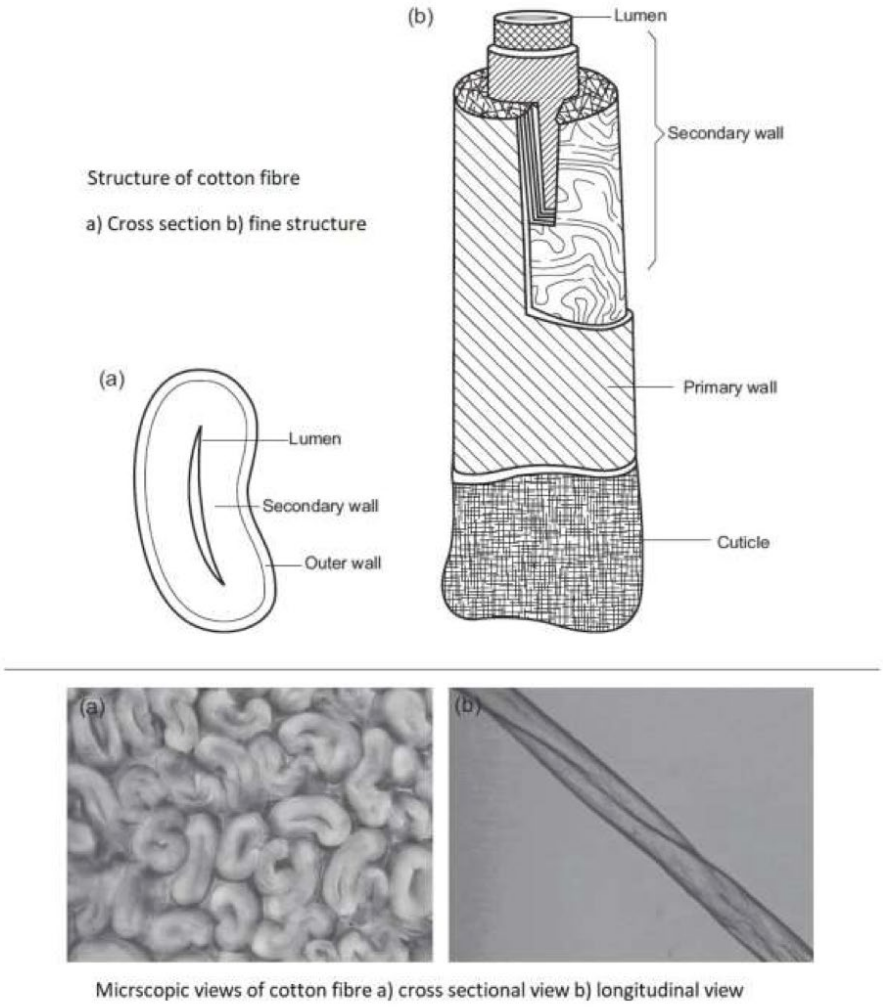


Image 11: Structure and microscopic view of cotton fibre and microscopic view[3]

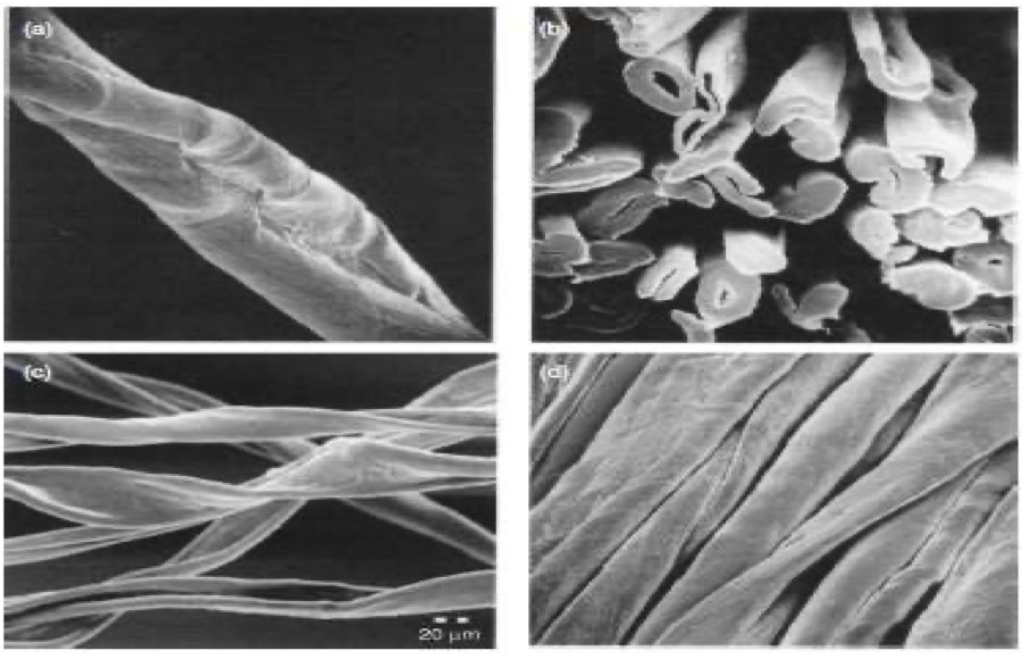


Image 12: SEM view of cotton fibre

[3] https://www.texcoms.com/wp-content/uploads/2019/06/Textile-Fibres.pdf

The polyester molecules of polyester and PET fibres on the contrary tend to pack tightly and held together by van der Waals forces and the chains are fairly stiff and rigid, as seen in image 13. They are highly crystalline unless co-monomers are introduced to disrupt the regularity of the molecular chains – but they are highly resistant to oxidising, biological and chemical agents – apart from hot concentrated acids and bases, with the fibre only melting at 250°C[4].

Image 13: Molecular fibre structure of polyester fibre[5]

We tested the finding that Heat Setting is the greatest contributor of microfibre shed with the Ramatex production staff. From their years of observation on the ground, it was suggested that the microfibre shed at Heat Setting could be a carry-on from the three wet process steps taking place in the same dye tank. This would be the consecutive steps of pre-treatment → dyeing → and rinsing.

To explain this, Ramatex referred to an informal test they ran some years ago. The drainage point of the machine is located at the bottom - after each step the water drains from top to bottom as it sinks through the textile. The textile does not leave the tank. During this time, it is believed that much of the microfibres that have been shed into the water settle onto the construction of the yarn and get trapped inside the fabric, rather than leave with the discharged water after each wet process.
In our secondary research, we came across a similar observation[6] that one disadvantage of the jet dyeing machines was that loose fibres removed from the textile may get redeposited on the textile.

The hypothesis we developed was therefore that we are seeing a 'false positive' result at the after heat setting step, because the pressure applied to the fabric when it passes through the heat setting padder causes the previously-loose microfibres to detach and release as part of the water sample collected.

NEWRI tested this hypothesis in the lab using a sonication bath. They found that the re-attachment of fibres onto textile did indeed take place. Details and the result of this test can be found in Annex A - NEWRI's Research Report.

The information we have gathered from Ramatex and our desktop research leads us to suggest the following:

- Further testing and validation needs to be done, with the following and areas of attention:
    - Taking care to identify as part of the scope the point at which water samples should be collected from the dye tank – as the water stream at the start, middle or end has an impact on the results – and ensure that this is done consistently across all samples and process steps that take place in the dye tank, and finally to collect them on the same day if possible, to minimise the possibility of picking up leftover fibres from other dyeing runs
    - Comparison of rinse water sample + textile sample (after rinse/before heat setting)
    - Comparison of heat setting water sample + textile sample (after heat setting/before brushing)
    - A microscopic image of the after rinse / before heat setting sample to visually see the 'reattached fibreslook at adding a surface enhancement for natural cellulosic fibres i.e. MTIX MSLE; Multiplexed Laser Surface Enhancement "MLSE®" technology[7]

4 https://www.texcoms.com/wp-content/uploads/2019/06/Textile-Fibres.pdf

5 Ibid

6 http://glassfibreindustries.blogspot.com/2013/06/dyeing-of-polyester-and-cotton-blend.html

7 https://mti-x.com/

- Potential areas of research could include investigating other dyeing systems and comparing their impact on microfibre shed.

Temperature and duration

The variables of temperature and duration settings were chosen as the intervention measures for investigating how changes to these settings would reduce microfibre shed.
No specific dyeing formulations were provided - which entail dyestuff and auxiliary chemicals and the duration, points in time, and temperatures at which they are applied – as these are closely guarded trade recipes that form part of the textile supplier's competitive advantage. To respect the partner's preference:

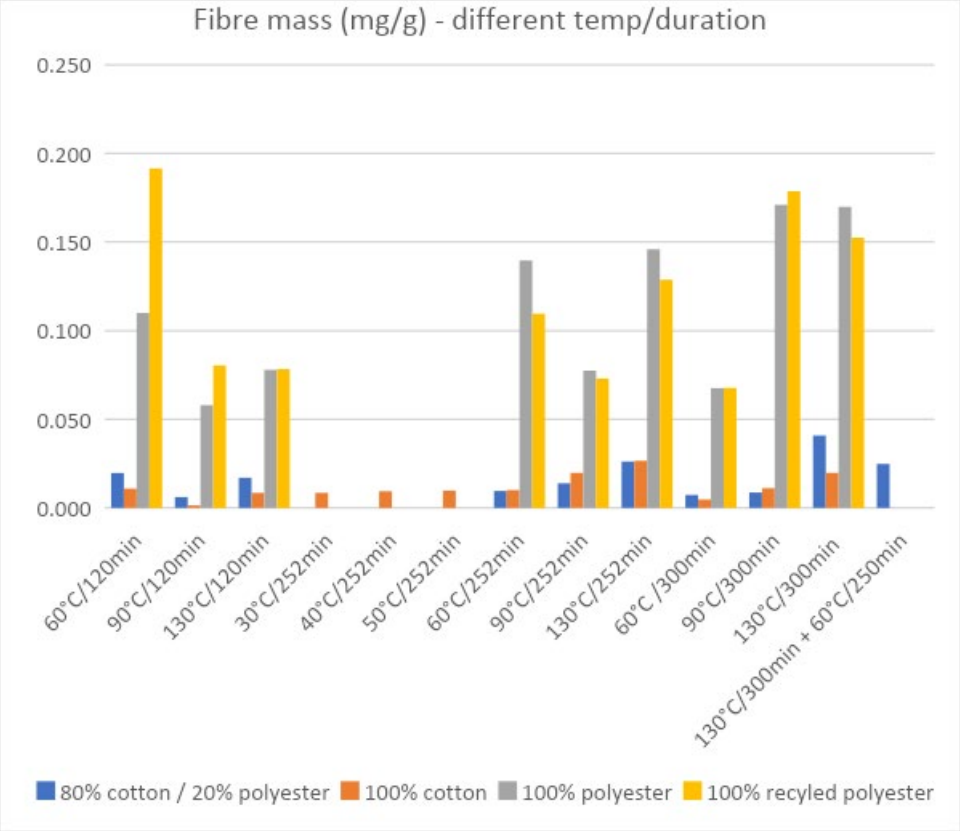
1. Dyestuffs and chemicals as a testing variable were excluded
2. Optimal temperature and duration settings at which dyeing is successfully achieved in the Ramatex dye tank were provided

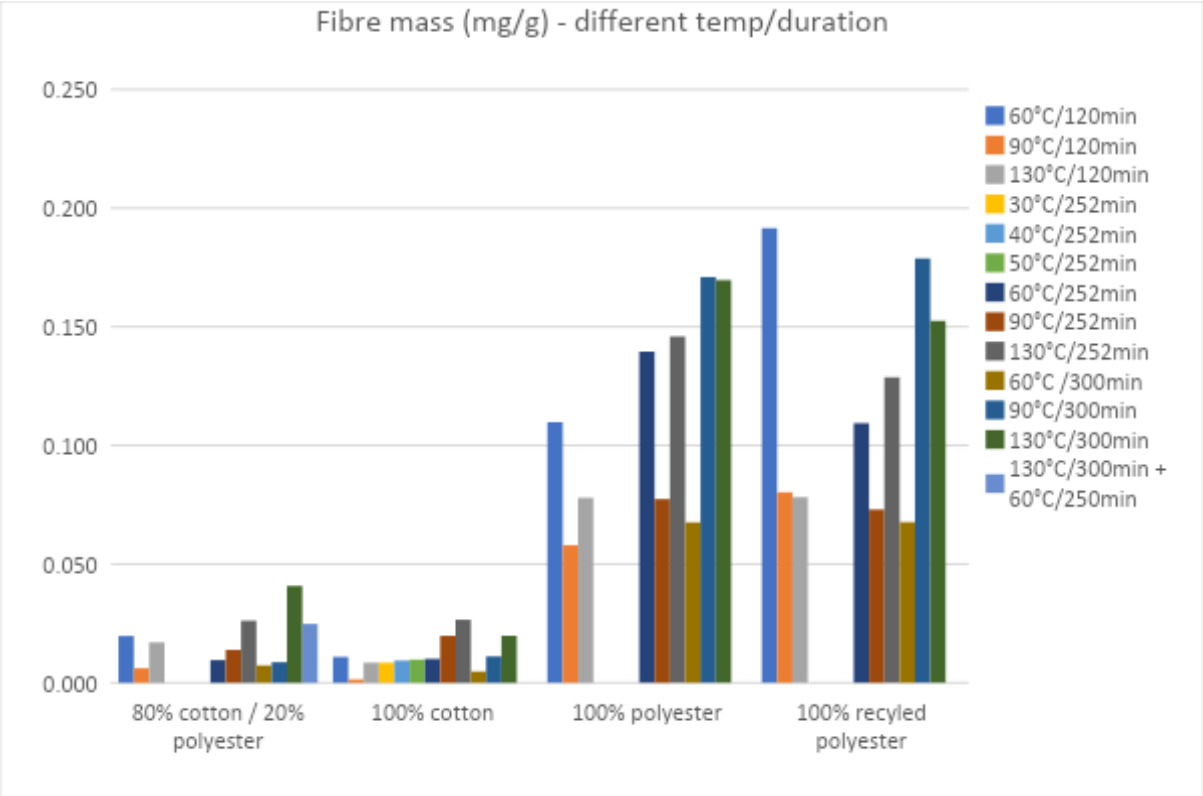
The testing range for the temperature and duration settings for the investigative testing were set up following this. The research team used the following rationale:

a. The provided settings of 130°C and 60°C set the upper and low testing range. We then selected an approximate midpoint of 90°C as the third setting, as a wide range would be able to clearly surface the differences in microfibre shed. We applied these 3 temperatures to all the textiles sampled. On Jersey – 100% cotton we conducted additional tests at 30°C, 40°C, 50°°C to further observe the impact on microfibre shed when temperatures lower than the optimal setting for dyeing cotton were applied.

b. Based on the given settings of 252min and 300min, we picked 120min as a third point of comparison to see if a shorter duration reduced microfibre shed. We applied this range to all the textiles sampled.

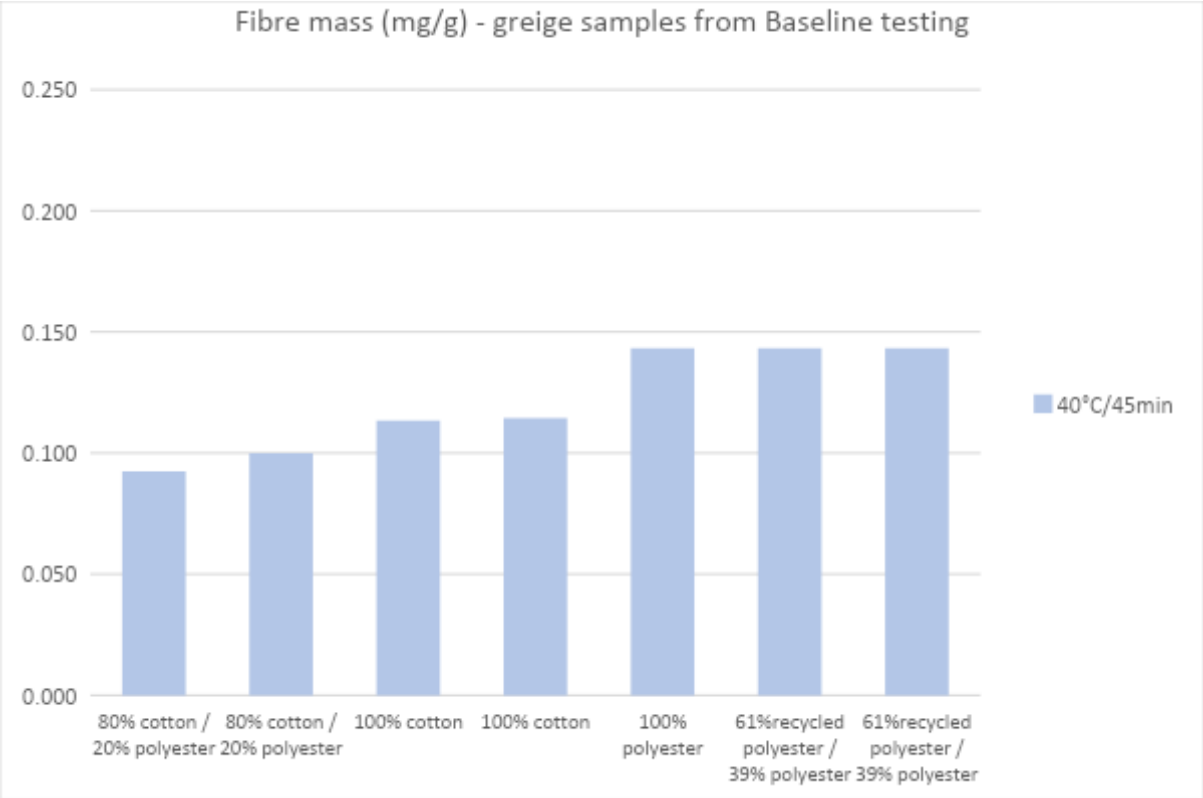
The samples were greige textiles of different composition, and obtained following results as can be seen in below image:





The results obtained in this investigation show that 100% recycled polyester and 100% polyester are shedding most compared to the CVC and 100% cotton greige materials.
From a surface comparison the results appeared to show that 100% recycled polyester and 100% polyester shed the most compared to the CVC and 100% cotton greige materials. This was a surprising outcome in comparison to the results from the baseline research. It should however be noted that a like-for-like comparison of the materials, especially the cotton and polyester, is difficult to achieve due to the differences in construction, yarn types, and so on.

From our available data, we conducted a comparison of the greige / knitting results. It should be noted that the leaching method was used on greige materials, of different composition and the temperature and duration used to leach the fibres is different – 40°C/45min. The results can be viewed in below image:



Comparison of other variables on microfibre shed

During the course of the investigative research Ramatex suggested testing the microfibre shed from yarns constructed using different spinning systems and different compositions of textiles. We conducted preliminary testing on a small range of 7 textiles in greige form and have included the results in this report as a starting point of reference.

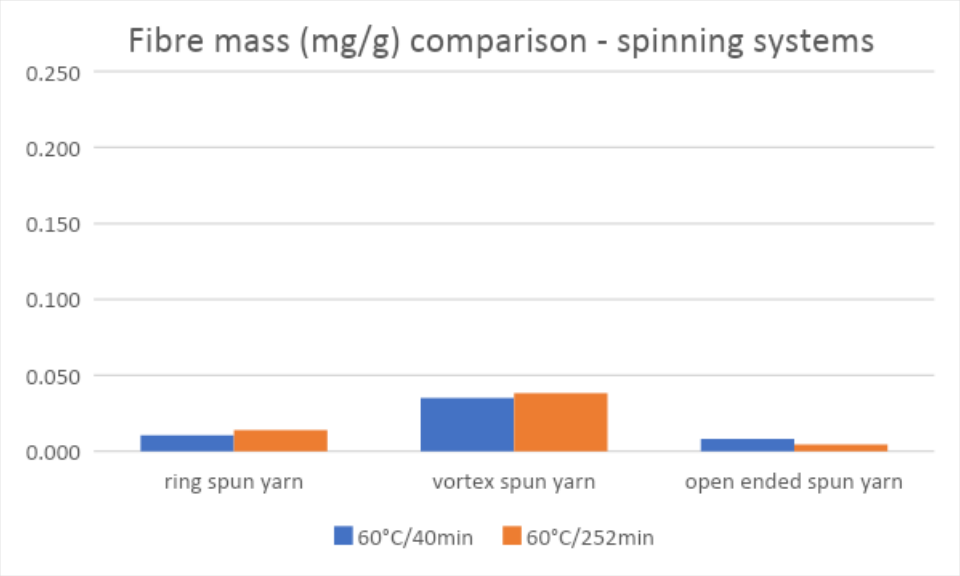
However, we came to the conclusion that this falls outside of the immediate research scope of this research as it will require additional scoping and investigation of the different elements, factors and variables in yarn spinning systems and composition that also influence microfibre shed.

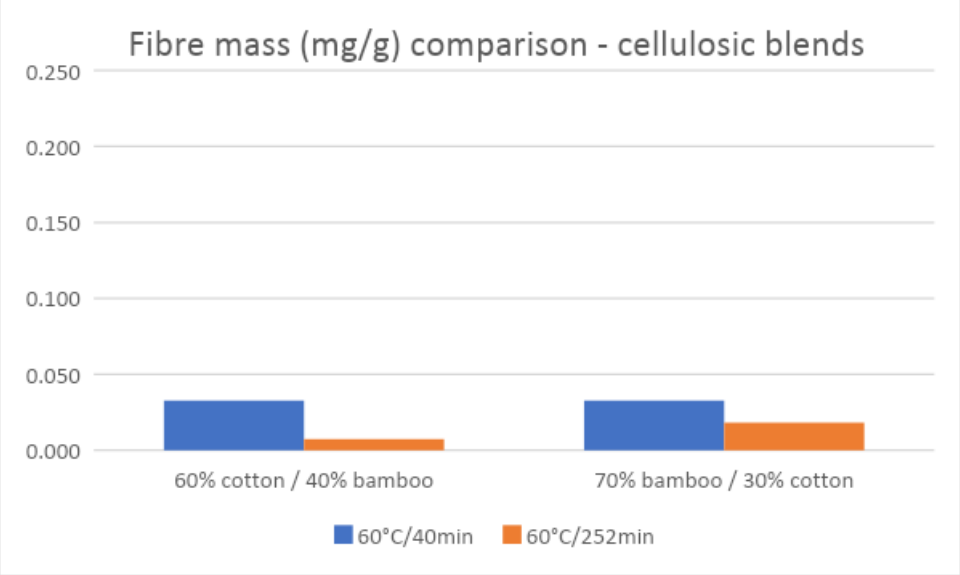
We therefore recommended further research in this area, which is invaluable to informing brands and suppliers' sourcing decisions prior to the manufacturing process. As part of the scope, it will be important for researchers to ensure that they have the full profile of the textile's yarn type, the ratio in which it is used, its construction, weight, and so on. This will allow the pinpointing of a single variable undergoing testing, with all other elements being the same.

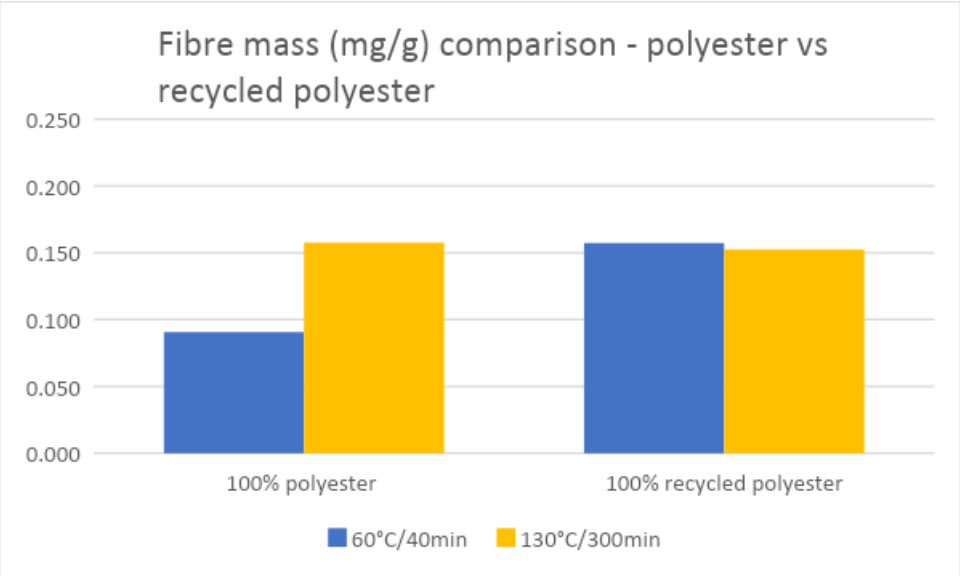
Within this set of complementary testing, the useful insights are obtained from:

1. A comparison of 3 spinning systems used in Jersey - 100% cotton: vortex, open-end and ring
2. The amount of fibre shedding mass from 2 types of cellulosic fibre blends
3. The amount of fibre shedding mass from 2 types of polyester jersey, one made of recycled polyester, and the other virgin polyester

Some of the research questions that arise from our preliminary results include: why does the open end spun yarn shed the least and the vortex yarn shed the most? Why do the filament yarns shed more than the staple yarns contrary to existing literature/research?


Fibre mass (mg/g) comparison - spinning systems
0.250
0.200
0.150
0.100
0.050
0.000
ring spun yarn
vortex spun yarn
open ended spun yarn
60°C/40min
60°C/252min


Fibre mass (mg/g) comparison - cellulosic blends
0.250
0.200
0.150
0.100
0.050
0.000
60% cotton / 40% bamboo
70% bamboo / 30% cotton
60°C/40min
60°C/252min


Fibre mass (mg/g) comparison - polyester vs recycled polyester
0.250
0.200
0.150
0.100
0.050
0.000
100% polyester
100% recycled polyester
60°C/40min
130°C/300min

SECTION 2: RESEARCH PLAN AND METHODOLOGY

Introduction

This report describes the **Research Plan** which is the first deliverable of the research that is underway as part of the project: *Tackling Microfibres at Source: Investigating opportunities to reduce microfibre shedding in the fashion industry through textile design and manufacturing innovation.*

It is important to mention that this research plan has been co-created with the input from both the research team at NEWRI and the materials team at Ramatex. Without them, this research plan would not have been possible.

The topic of textile microfibres and the pollution caused by the fashion industry is an emerging one, which means that new information and knowledge emerges regularly; and thus, flexibility in our approach is needed.

The ongoing COVID-19 pandemic has impacted not only our ways of working but also the practicality of obtaining samples, which has left the team looking for ways to deliver the research plan as completely as possible.

In this report, you will find the research methodology, the research plan and also a short guide to using the research plan.

Research methodology

The research was planned to take place over 12 months from April 2021 to March 2022. **However, with the pause in research due to COVID-19 restrictions in Malaysia and Singapore, the end date for the research was delayed until August 2022**. The research consists of four interlinked phases, as shown in Figure 1:

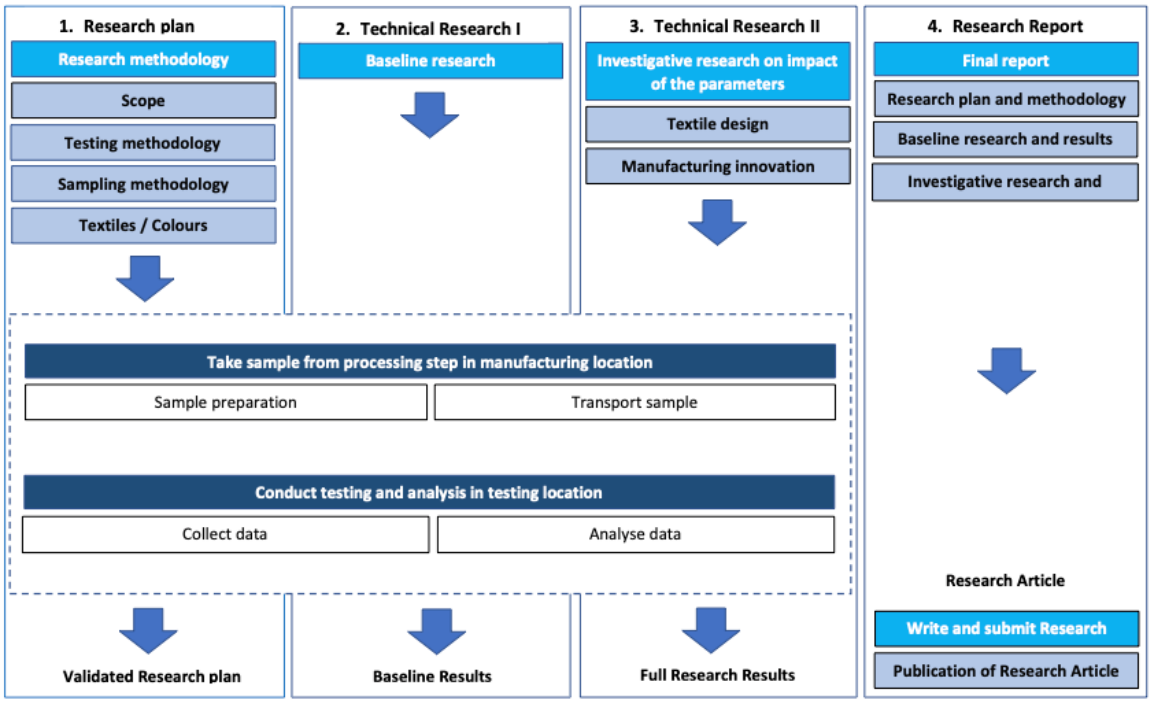


Figure 1: Research Workflow.

A larger image of the schematic workflow of the research can be found in Annex A. Research Workflow.

The first phase of the research is the **Research plan** in which the foundational work is captured like the research scope and the identification of what textiles and colours to include. The focus of the research team was answering the 2 main research questions: ***'How to take samples from the processing step in the manufacturing location?'*** and: ***'How to conduct testing and analysis in the testing location?***'. The answers to these two questions will set the blueprint for the subsequent phases in research (Figure 1: Research Workflow).

In the **Technical research I**, **the Baseline research**, the team will be collecting data, and the subsequent analysis will provide insights into which of the identified textile processing steps are contributing most to microfibre release within the textile manufacturing facility. This is an opportunity in research to further streamline the next phase by identifying which areas in textile manufacturing warrant further investigation.

In the **Technical research II** phase, **the Investigative research** will take place. This phase is an exciting phase as it is an opportunity to understand if implemented changes to either textile design and/or manufacturing innovation has (any) positive impact on the microfibre release of the chosen processing steps for further investigation. This is also the phase where Ramatex suppliers can be brought in to support and possibly test any solutions to reduce microfibre release at the manufacturing level. The hypothesis is that a reduction of microfibre shed at the upstream manufacturing level will lead to an overall reduction of microfibre at the domestic laundering level.

The research is concluded with a **Final Report** which will describe the Research plan & Methodology, the results of the Baseline research and the results of the Investigative research. The final deliverable of the research is the **publication of the research article** and research report.

Research plan progress reporting

Whilst the research plan was underway, the key indicators of sampling and testing methodology were tracked to understand how the research plan was progressing. As this information is no longer critical to the primary reporting, it has been captured in Annex B: Research Plan Progress Reporting.

Scope

The scope of the research has been determined by the input from the Ramatex team on the most prevalent processing steps in their facilities in Malaysia (Figure 2: Processing steps). As the research looks specifically into microfibre release from processing steps solely within the textile manufacturing facility, at this stage it does not include other factors such as the transport or fate of microfibres. The latter may be explored during the development of an impact framework that highlights the intended and unintended social and environmental consequences of the proposed innovations in the Investigative Research phase.

It has to be noted that there are different processing steps for different textile types. Figure 2 shows the revised, and most up-to-date processing steps where samples were collected following the impact of COVID-19 on production schedules at the Ramatex facilities.

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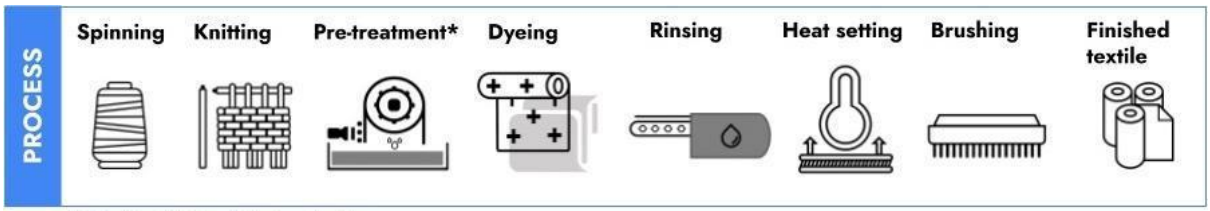


**Initially to be collected from CVC and Cotton Textiles only, subsequently also collected from Polyester textiles.*

Figure 2: Processing steps

The team understood that the more information that could be collected about the microfibres, the easier it should be to identify areas in either textile design and/or manufacturing innovations, in which some of the solutions may be found.

The desire for the project to account for all the microfibres released by these processing steps within the textile manufacturing facility, coupled with the desire to learn as much about these fibres resulted in the following list of result types/key parameters to be included in the research plan:

1. **Fibre mass** - The mass lost before and after steps can suggest how many fibres in total are likely to be generated. This result type was added to facilitate an easy link to the industry method used by The Microfibre Consortium (TMC) and American Association of Textile Chemists and Colorists (AATCC), which both report only on fibre mass.
2. **Fibre quantity** - To learn about the number of fibres in the sample.
3. **Fibre length** and **Fibre length distribution** - To learn more about the size of the fibres and its distribution
4. **Fibre type** - The fibre type informs us about raw material which the microfibre is made of, especially the stable polymer used. Note that this test was only applied to the CVC Fleece 80% cotton / 20% polyester samples.

Regarding fibre mass, at the start of the research, it was known that both The Microfibre Consortium (TMC) and the American Association of Textile Chemists and Colourists (AATCC) were working on its respective methods, though no exact details had been published at that time. One of the third-party labs, Intertek, was contacted to learn if they could help in testing for any of these methods shared a comparison between the methods of TMC and AATCC (Figure 3: Comparison Industry Methods).

	AATCC – fiber fragment	TMC
Trimmed specimen size	100 X 240 mm, 4 specimens	100 X 240 mm, 2 specimens
Filter size	1.6 micron pore size	1.6 micron pore size
Pre-treatment before laundering	Wash once is not required Condition the specimen 21 ± 2°C and 65% ± 5% RH for 4h	Wash once is not required Dry the specimen 50C for 4h
Blank test – accounting any contamination during the test	Required, every 4 specimens	Not required
Laundering liquor	Detergent solution; 0.25% (Detergent, non-ionic, type C13 oxoalcoholethoxylate (7EO) 360 ml, 50 steel balls	360 ml grade 3 water, 50 steel balls
Canister size	1200 ml, 90 X 200 mm	1200 ml, 90 X 200 mm
Laundering condition	40C for 45 mins @ 40+/- 2 rpm	40C for 45 mins @ 40+/- 2 rpm
Rinsing (contents of canister, beaker to collect the rinse water etc)	3 times	3 times
Calculation	Fiber fragment release (g) = W2-W1 Fiber fragment release % = 100 x (W2-W1)/S	Fiber release (g) = 100 X (W2-W1)/S

Figure 3: Comparison Industry Methods

It is important to be aware that both these methods have been developed to support the understanding of the impact of consumer laundry on microfibre fragmentation and release. The testing methodology that was developed in the past few months has looked at these methods to see if there are similarities that can be adopted and at the same time to look at the differences. A consumer product is an assembled product (stitched textiles) and consumer washing includes laundry detergent; neither of these are applicable to a textile manufacturing environment.

The TMC method was developed with the support of the University of Leeds (UoL). The UoL published its methodology in The Journal of the Textile Institute on the study 'Reliable quantification of microplastic release from the domestic laundry of textile fabrics'[Annex J-1] on February 25, 2021.

The TMC method was publicly shared with its members on July 13, 2021 and related information can be found on TMC's website[Annex J-2]. As at January 24, 2022, Forum and Ramatex are full signatories with TMC and will be working towards aligning this research with TMC's test method.

The AATCC method TM212-2021 was released in late August 2021 and provides the global industry with a consistent and uniform test method to follow, addressing discrepancies that have long been a source of confusion for many working to tackle microfibre pollution. The test is applicable to textiles that are expected to withstand home laundering.

In-scope confirmation areas

Throughout this first phase of the research, we came across a few areas that need further clarification; before the baseline research can commence. There are three areas for confirmation on what will be in-scope and what will be out-of-scope.

The three scoping areas that underwent a process of confirmation were:

1. Laundry sample from finished textile

This research has always had the aim to build a link to existing research and testing, most specifically the test methods by TMC and AATCC on the impact of consumer laundry on microfibre fragmentation and release.

We researched several options for this link to be included in research. The following consist of initial options as well as new information obtained from TMC in January 2022. They include:

a. *Testing at an external laboratory*
   This option requires funding; the cost is dependent on the number of samples selected for testing, choice of method and also the choice of third-party laboratory. For quotations, Annex C: Third party laboratory quotations shows a broad comparison obtained in 2021.

b. *Accreditation of our research partner's laboratory*
   A research partner's lab could be accredited for free as long as the lab technicians possess the TMC test method equipment and are able to pass the TMC accreditation test. There will be a cost involved in securing the correct equipment.

c. *Purchasing equipment for research*
   By purchasing the equipment specifically dedicated to the testing for microfibre fragmentation and release it would be a significant investment. Also, this option requires funding of 23,000 USD

d. *Brand partner to conduct testing*
   By bringing in a brand partner, i.e. Nike, it could be requested from this brand partner to perform this test as a contribution to the research. This option is most preferred although discussions are still underway.

We decided to conduct testing on the finished textile using the testing methodology developed in this project. We will continue to engage with TMC on the possibility of conducting additional laundry testing according to the TMC method.

2. Processing step: 'Rinsing'

In the early stages of the research the processing step 'Rinsing' was listed as a processing step after brushing. Later on, the Ramatex team informed us that this rinsing step is actually an incidental step in the manufacturing of textiles. It will only be performed at the special request of its customer.

With the operating principle for this research being to investigate so that the biggest impact can be made, we initially agreed not to include the "Rinsing' processing step at this stage of the research.

The proposed solution that the research team identified was to only include the rinsing step in the third phase of the research; 'Investigative Research', Technical research II.

In late 2021 Ramatex found that there was sufficient volume of available samples from the rinsing step in their actual production plan. As a result, this step is reinstated in our research workflow and Research Plan Visual, and will be included in the reported findings.

3. Processing step: 'Hydro Extraction'

The processing step 'Hydro extraction' was included in the water sample collection and testing between May 2020 and August 2021.

However, in late 2021 Ramatex determined that it was a business need to permanently eliminate this step and reduce the production processing time. As a result, this step is removed from our research workflow and Research Plan Visual.

4. Processing step: 'Pre-treatment'

At the start of the baseline research, it was indicated to us that for the pre-treatment step, only samples from the 100% Cotton and CVC fleece would be collected. However, when collection resumed in 2022 after the COVID-19 lockdowns, Ramatex also sent pre-treatment water samples from the 100% Polyester regular and 100% Polyester recycled.

5. Processing step: 'Spinning'

The results from the processing step 'spinning' prompted a re-framing of this step during the technical review and validation conducted by VDE Consultancy. This is addressed in the summarised and final validated research report, as a complementary testing and analysis to the in-scope areas.

6. Lint/fibre sample

The Ramatex team confirmed that some of the processing steps produce lint/fibre (Figure 4: Lint/fibre sample information) that is being captured by a different range of devices:

- A vacuum system for the processing steps: Spinning, Knitting, Brushing
- A built-in auto lint collecting device for the processing step: Dyeing

The objective of these devices in the process are mostly for quality purposes, i.e., to support an even-dye in the dyeing processing step.

Lint/fibre samples						
Processing steps	**How is lint/fibre captured?**	**What is filter pore size and what material is filter made off?**	**How often is lint/fibre capture device emptied?**	**Where is excess lint/fibre disposed off?**	**How much lint/fibre per batch/process?**	**How many kg of product produced?**
Spinning	vaccum system		collected everyday	sold to third party	about 10kg/day	20000
Knitting	vaccum system		collected everyday	sold to third party	about 10kg/day	120000
Dyeing*	auto lint collecting device	2mm / TBC	once at the end of the cycle	sold to third party	*to be measured*	1500
Brushing	Vacumm		when the fiber bag is full	sold to third party	about 60kg/day	4500
Note: **The lint/fibre from the filter is wet, so it needs drying before packing it.*						

Figure 4: Lint /fibre sample information. Annex D: Lint/fibre sample fibre mass calculation shows the same table with clarity on the line/fibre and product produced per batch/day.

The research team is keen to bring these samples in, as this will give us an understanding of the total contribution of each of the processing steps, including some atmospheric fibres.

The solution at this point is a partial one as we are yet to fully understand, how to sample since the materials are used as consumables, and test:

a. The lint/fibre sample can be captured, though it should be noted that the dyeing lint/fibre samples are wet when captured and require drying before shipment
b. The contribution of the lint/fibre can be calculated per kg of fabric

c. Ramatex materials team is able to track back to which batch of material is being used. But it should be noted that the materials are consumables, therefore there are no physical batches left since it is already being used in the fabric.

One challenge in this project is to estimate the fibre loss during drying processes and the in-built filters in the water discharge. As a proposed solution, we will include the lint/fibre sample from the dyeing processing step for complementary testing and results.

From the others, the intent is to gain an understanding of the total fibre mass lost. Ramatex conducts in-house recording of the estimated percentage of fibre loss in the spinning, knitting and brushing steps and will share this data.

In addition, for the brushing step it is possible to weigh a same-sized cut of the fabric before and after brushing, to better understand the fibre loss. Fabric shrinkage occurs width-wise and not length-wise, hence the size of the cut can be standardised length-wise.

You can find the preliminary results in Annex D: Lint/fibre sample fibre mass calculation.

A graphic has been created to visualise the research plan, including the three areas of scoping for confirmation and its respective proposed solutions (Figure 5: Research Plan Visual).

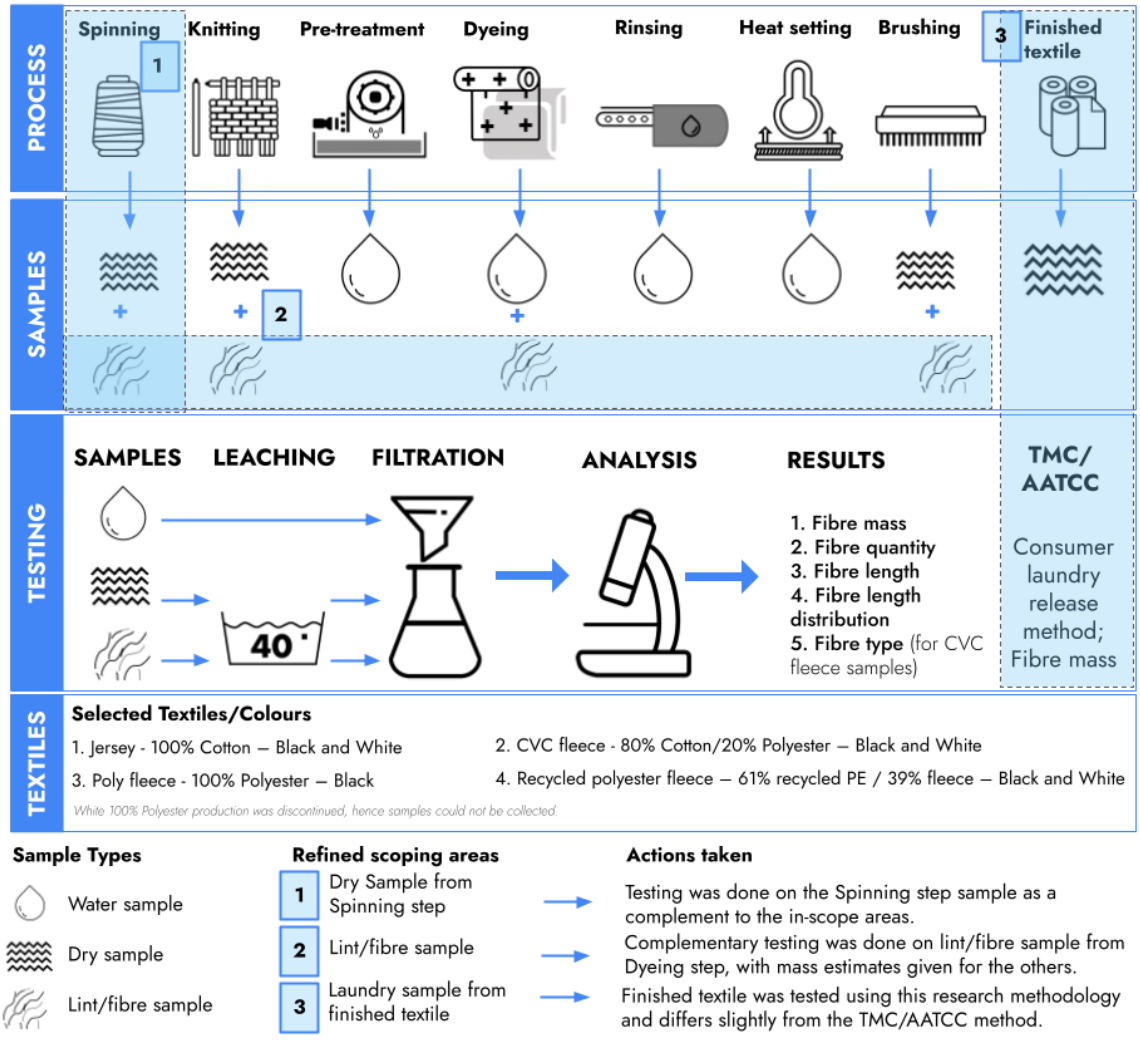


Figure 5: Research Plan Visual

A larger image can be found in Annex E. Research Plan Visual.

Textiles/Colours

The starting operating principle for this project was to analyse textiles that are produced in the greatest volume in order to create the greatest impact (with our research, recommendations, etc).

This research plan also has tried to strike a balance between fulfilling the operating principle and mitigating the challenges and roadblocks that arise from COVID-19.

The selected textiles for this research are the ones produced and purchased in the highest volume and thus create the greatest impact through their study in this research. Also, in any event of the Ramatex manufacturing facility operations being affected by the ongoing COVID-19 situation, these textiles are likely to be first manufactured when the facilities reopen. With the industry increasingly looking at recycled materials to move towards circularity, we have also decided to include recycled polyester.

The selected textiles are:

1. Jersey – 100% cotton

2. CVC fleece – 80% cotton / 20% polyester

3. Poly fleece – 100% polyester

4. Recycled polyester fleece – 61% recycled polyester / 39% fleece

Further, the two most popular colours are selected to understand if colour has a possible impact on the results. **The selected colours are: Black and White.**

The production plan from Ramatex will determine which item number(s), and thus details of the selected textiles will be available for the second phase of the research, Baseline research.

A confirmation of these details will be provided by Ramatex, and you can find the details also on the 'Sample form' when these are sent over for testing and analysis (Annex: F. Template Sample label).

Absence of the 100% Polyester (White) samples in the baseline research testing

Collection and testing paused in late 2021 due to COVID-19 lockdowns. They resumed in Jan - Feb 2022, in sync with Ramatex's production planning. In this period, the brand customers decided not to purchase the 100% Polyester White fabrics. We monitored the situation until Ramatex confirmed in April that it had been put out of production due to lack of demand. This was indicative of an increased shift away from virgin to recycled polyester.

To work around the situation Ramatex tried to source for old stock (dry samples) from its Jordan, Vietnam and Chinese factories, but was unsuccessful. We discussed the feasibility of running a small-batch production for the sole purposes of obtaining the dry and wet samples. We concluded that this was inefficient in time, cost, and operating resources (energy, water, labour, etc) and would contribute to the very issues that this project sets out to address, not least the creation of textile microfibre shed.

We consulted with NEWRI who advised that this would not have a major impact on the validity of the baseline research nor the relevant research outputs. A short scientific justification may be found in Annex L.

Sample methodology

In this section you can read the answer to the first question: *'How to take samples from the processing step in the manufacturing location?'.*

This section provides the information needed for the manufacturing facility to do the sampling; what type of samples to collect, what the collection locations are, how to package the samples and lastly how to transport these to the testing facility.

Types of samples

There are four types of samples recognised in this research (Figure 6: Types of samples):

<table>
<tr><th colspan="6">Types of samples</th></tr>
<tr><td colspan="2">1. Water sample
Samples can be collected at the start and end of the process; in-process water samples</td><td colspan="3">2. Assisted water sample
A dry textile sample will be taken to leach the microfibre content in liquid; an assisted water sample</td><td>3. Lint/fibre sample
Samples can be collected at the end of the day, or at the end of a processing step cycle (dyeing); a lint/fibre sample</td><td>4. Laundry sample
A finished textile sample will be taken to leach the microfibre content in liquid simulating a consumer laundry process; a laundry sample</td></tr>
<tr><td>incoming</td><td>discharge</td><td>Cone*</td><td>Greige</td><td>Finished sample</td><td></td><td></td></tr>
<tr><td colspan="6">Notes:
* cones depending on number of yarns per textile type</td></tr>
</table>

Figure 6: Types of samples

1. Water

Within the manufacturing of textiles there are many processes that are water intensive. Samples where possible are taken from the incoming and outgoing (discharged) water.

- The discharge water, at the dyeing processing step, is taken directly from the pipe and not from the tank as this is safer, since the tank is still hot.
- The processing step hydro-extraction only has discharged water. However, as at November 2021 this processing step is outside of our scope.
- In the final calculations of the results, we decided to focus on the discharged water.

Data that Ramatex will share with NEWRI includes:

a. Volume of entire water in tank and total water used in each wet processing step
b. Recording the quantity of the textile in the tank and calculating the ratio of the fibre collected to water.

2. Assisted water sample

The dry samples, or so-called assisted water samples are from the processing steps: spinning, knitting, brushing and finished textile.

It should be noted that for the textile product type Fleece a brushing sample will be a last step in the textile process, but for Jersey an actual finished sample should be collected after the processing step heat setting.

3. Lint/fibre sample

The Lint/fibre samples are divided in two types as each of these types has a different way of collection.

a. The vacuum system samples are collected at the end of the day, or when the bag is full (spinning, knitting, brushing).
b. The auto lint collection device in the dye tank gets emptied at the end of each dye run (dyeing).

As the lint/fibre sample for the dyeing process is collected after the process, it is therefore a wet sample. For the research this sample will be dried by the team at Ramatex before it can be packaged and shipped to the testing location.

4. Laundry sample

To provide for the 'Laundry sample', samples to be used to test according to the AATCC and/or TMC method, these samples should be taken from the processing step 'finished textile'.

Sampling points/locations

Each processing step has its own types of samples. Below in Figure 7 you can find an overview of each of the processing steps and its sample types that can be sampled, the so-called sampling points.

Sampling points	**Types of samples**						
	1. Water sample		***2. Assisted water sample***			***3. Lint/fibre sample***	***4. Laundry sample***
Processing steps	Incoming	Discharge	Cone*	Greige	Finished sample		
Spinning			X			X	
Knitting				X		X	
Pre-treatment**	X	X					
Dyeing - chemistry	X	X					
Dyeing - rinsing	X	X				X	
Heat setting	X	X					
Brushing					X	X	
Finished textile					X***		X
Control	X						
Notes:							
**Cones: depending on number of yams per textile type*							
***Pre-treatment: only for CVC & 100% Cotton*							
****Non-fleece samples (i.e. Jersey) also need a finished textile to be tested.*							

Figure 7: Sampling points

A larger image of this can be found in Annex: G. Sampling points.

For the lint/fibre sample, it should be noted that it is proposed that the spinning/knitting and brushing samples will not be actually sampled; only the results of the fibre mass per kg calculation is included (Annex D: Lint/fibre sample fibre mass calculation).

The only lint/fibre sample to be sampled and tested in this research is from the dyeing processing step.

Sample collection

The Ramatex materials team members in the manufacturing facility will collect the samples from the respective processing steps.

The sample taker is to wear nitrile gloves and a polyamide monofilament lab coat to avoid any cross contamination and thus possible pollution of samples (Figure 10: Sample packaging instructions).

The following quantities are agreed to be collected for each of the different sample types:

- *Water samples (discharge)*
  A minimum of 4 litres per sampling point is requested.
- *Dry samples (assisted water sample, lint/fibre sample, finished textile sample)*
  - Cone - 1 cone per yarn type
  - Greige/ knitting sample – 400gm
  - Brushed – 400gm
  - Finished textile - 400gm
  - Lint/fibre sample - minimum 100 gram

Each sample needs to be accompanied by a “Sample label” which you can find an example of in Annex: F. Template: Sample label.

In addition, the NEWRI team created a set of standard operating procedures in November 2021 to increase the rigor of the collection, handling and filtration of the **water samples**. This can be found in Annex L: NEWRI Progress Report: Standard Operating Procedures.

Sample packaging

To assure that the samples do not get contaminated the NEWRI team has identified the following preferred packaging instructions for the different sample types.

Also, same as with the sample taker, whomever packages the samples is to wear nitrile gloves and a polyamide monofilament lab coat to avoid any cross contamination and thus possible pollution of samples.

Containers for wet samples

The NEWRI team has investigated which containers are best to use to avoid any pollution of the samples taken. A PET bottle, for example, cannot be used as recycled polyester has the same raw material and thus it cannot be guaranteed that the microfibres found are from the processing step investigated or from the container it was packaged and shipped in.

The NEWRI team indicated early on in the research to use the glass type Erlenmeyer, though its shape caused concern. An alternative glass bottle was looked into with a glass bottle top though after much consideration this was also declined as an option.

The NEWRI team ultimately decided on the use of a poly propylene (PP) bottle.

Packaging for dry samples

Whilst finding the optimum between existing processes taking place within the Ramatex facility, the guiding principle for the packaging of the dry samples (cone, greige, brushed and finished textile and the lint/fibre samples) is to wrap the individual sample in aluminium foil and seal this in a plastic bag.

The sampling and packaging instructions are identified in figure 8:

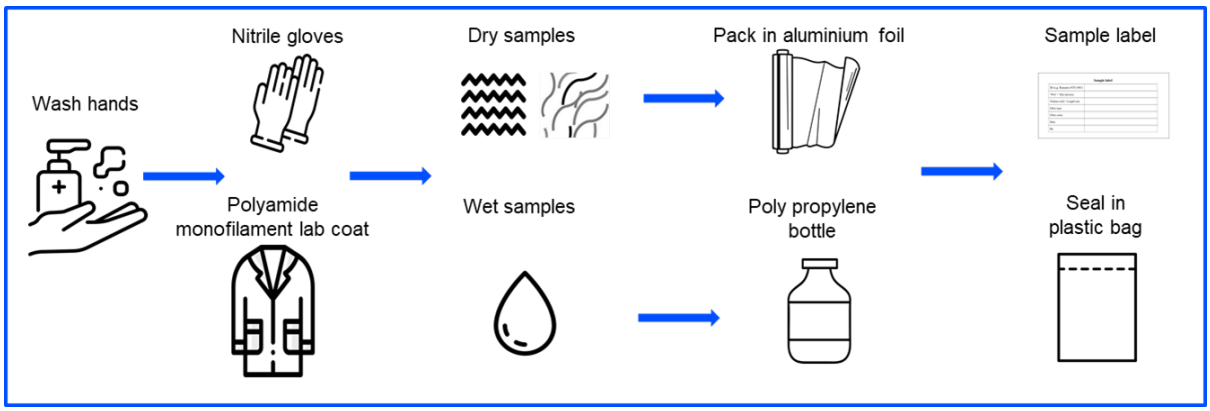


Figure 8: Sample packaging instructions

Sample shipments

The shipments of the samples are for the most part taken care of by the Ramatex team.

Export

The Ramatex team will work with its own teams to arrange for export documentation for the samples needing to be shipped to Singapore for research purposes.

Logistics

As with the export arrangements, the Ramatex team supports the logistics part of the transportation of the samples.

Import

For the import of dry textile samples (cones, greige, brushed, finished and lint), Ramatex has its own transportation and logistics teams to provide for this.

The import of the water samples into Singapore proved to be more difficult as it is unprecedented for Ramatex to bring in water samples across the border. In addition, COVID-19 restrictions meant that Ramatex facilities had to be shut for a few months with no sample taking possible. Additional COVID-19 measures from NEWRI's side were also put in place to make cross-border transport of wet samples more challenging.

To overcome these challenges, we are working with a partner laboratory in Johor – Universiti Teknologi Malaysia (UTM) that will assist in the testing of the water samples. Located near the Ramatex facility, the UTM team will support the preparation and filtration of the wet samples so that they can then be sent to NEWRI as dry samples for the testing to take place accordingly.

Testing methodology

The second main question to answer in this research plan, as mentioned in the introduction is: *'How to conduct testing and analysis in the testing location?'.* This section will answer this.

Due to the COVID-19 pandemic lockdown, in the period between August to October 2021 the Ramatex team was only able to collect the dry samples and unable to collect water samples. As a result, the NEWRI team was unable to conduct any validation of research into what would be the best testing methodology for the water samples.

Ramatex restarted the collection of water samples for CVC Fleece and Jersey on 24 November 2021. During the period that COVID-19 restrictions on cross-border transport remain in force, water samples will be sent to UTM for filtration before reaching NEWRI as dry samples for testing.

The dry and water samples for 100% Polyester and Recycled Polyester were only able to be collected in January to February 2022. A complete validation of the research plan will be presented for those once the results are out.

A visual has been created to show the flow of the testing methodology (Figure 9: Flowchart Testing methodology).

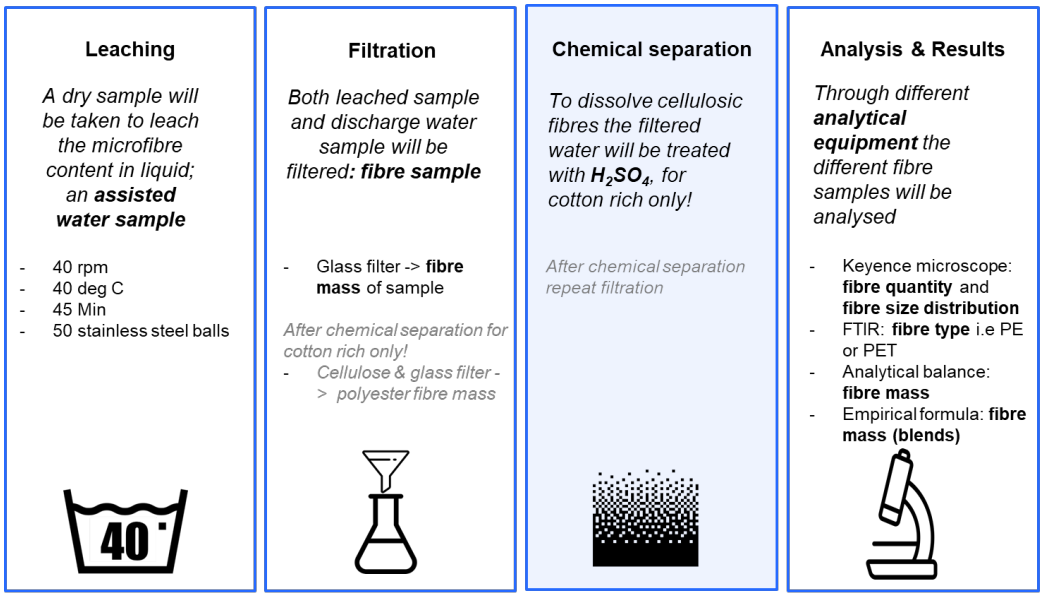


Figure 9: Flowchart Testing methodology

A larger image of can be found in Annex H: Flowchart Testing methodology

Sample receipt & handling

NEWRI will inspect all samples upon receipt against the information provided on the respective 'Sample label'. Any deviation or clarifications will be communicated to the project manager within 24 hrs of receipt. The samples will be accepted for testing once all clarifications are received.

Dry samples

The dry samples are cut to a weight of 10 gram, folded once in half. These samples were re-packed in aluminium foil and sealed in plastic bags until transferred to the lab for testing and analysis.

Sealing the edges: the team discussed the best way to prepare the dry samples for testing and considered stitching (as per industry methods on consumer laundry), and glue. It was initially decided against any of these preparation methods as it would not be representative of the adopted textiles in this research. Subsequently, the NEWRI team found that they needed a way to seal the edges as testing proceeded, and utilised glue. These textiles from Ramatex are circular knit, the edge therefore is a cut edge. The team has proposed to use the naked cut edge for the leaching process as it mimics the manufacturing facility's setting.

Water samples

The request for the water samples in sampling is 4 litres, though it was said that only 2 litres is needed for testing.

Test methods

NEWRI has developed a methodology for both the leaching of the dry samples and the subsequent filtration to be able to answer how many fibres the sample consists of, what the fibre length is, what the fibre source of raw material is, and also what the total fibre mass is.

Leaching

After sample preparation the next step is to extract the fibres from the dry samples into a liquid. The methodology for extracting these fibres has been based on the ISO 105- C06 with some modifications.

The NEWRI team used the following parameters for the fibre extraction method:

- Equipment: laboratory rotator
- Temperature: 40 degrees Celsius
- RPM: 40
- Duration: 45 minutes
- Glass vessels
- Liquid: 200 ml of DI water
- Additional mechanical action: 50 steel balls, 6 mm diameter
- Sample size: 10 g of yarn/textile/lint/fibre

This step gets repeated 3 times for each of the identified textiles/colours for this research.

In addition, a vessel without sample content but with 200 ml of DI water is used as a blank to account and correct for any potential background. This step is part of the quality systems in place at NEWRI.

The next step is to:

- Remove sample
- Wash sample thoroughly with ultrapure water from wash bottle
- Separate the steel balls and rinse with DI water
- Collect all liquids and dilute volume to 300 ml

➔ Assisted water sample

Filtration

Use the liquid from the ‘assisted water sample’ to perform the next step: filtration.

NEWRI methodology for filtration** – run liquid sample through following filtration system:

- Filtration system with vacuum pump
- Filter: glass fibre + cellulose filter*
- Pore size glass filter: 5 µm

➔ Cellulose filter with fibre residue

* *This research uses two colours of cellulose filter, black and white, to achieve vast contrast. This means that for a light-coloured sample the black filter is to be used and for a dark coloured sample the white filter.*

** *UTM will submit a formal report to NEWRI.*

As a preparation step for calculation the cellulose filter was first oven-dried at 50 degrees Celsius for 12 hours; take weight (= Fm1).

In the last step the ‘Cellulose filter with fibre residue’ needs to be dried:

- Fibre residue on cellulose filter inside aluminium protective case
- 24 hours

➔ Dried fibre residue

The ‘dried fibre residue’ subsequently gets oven-dried at 50 degrees Celsius for 12 hours; take weight (= Fm2).

Calculation: Fibre mass = (Fm2 - Fm1)

To image the fibre residue on the filters a Keyence Digital Microscope with a VHX Digital Microscope Multi Scan Lens was used:

- High res 96 dpi
- Typically, 30-50 individual images are needed to create whole filter image
- Add appropriate scale bar for later processing with open-source software

This filter imaging analysis obtains the following types of results:

- Fibre quantity
- Fibre size distribution

Analysis by Keyence Microscope: Fibre quantity and Fibre size distribution

Chemical separation

For textiles that are cotton rich an extra step is needed to achieve a chemical separation from polyester, or other stable polymer, and cotton. This chemical separation is based on DIN EN ISO 1833-11.

Separation:

- Create a ‘Cellulose filter with fibre residue’
- Add 20 ml concentrated sulphuric acid (75% p.a., KB Bernd Kraft)
- Incubate for 1 hour at 50 degrees Celsius
- Check to make sure all cellulose filter has disappeared

➔ Non-cellulose fibre residue solution

In the next step this solution needs to be neutralised:

- Add: 50 ml ammonia solution (25% p.a., Emsure® Merck)

➔ Neutralised non-cellulose fibre residue solution

The subsequent step is to filter the neutralised solution:

- Pre-weight filter: glass fibre
- Pore-size: 0.45 µm
- Diameter: 4.5 cm
- Dry in oven: 1 hour at 80 degrees Celsius
- Condition in climate chamber

➔ Dried non-cellulose fibre residue

This fibre residue was further screened using a Keyence Digital Microscope method to identify the fibre quantity of the non-cellulose fibres.

Analysis by Keyence Microscope: Fibre quantity and Fibre size distribution

By using the Fourier Transform Infra-Red spectroscopy (FTIR) the fibre type is confirmed.

Analysis by FTIR: Fibre type

The weight of the fibres released by the polyester or other stable polymer is calculated using the following equation:

Calculation Fibre mass: $$Grams\ of\ microfibers\ /\ gram\ fabic = C \times \left(\pi \times \frac{D^2}{4} \times L\right) \times \rho$$

where C is the number of microfibres released from 1 gram of fabric; D is the average diameter of the fibres; and ρ is the density of the fabric.

Quality systems

The NEWRI team understands the need for qualitative and reproducible results in the research. Therefore, in this research the following minimum quality assurance measures are in place:

Reproducibility

For one selected sample, triplicate samples were filtered through separate filters, imaged, and analysed for both fibre size distribution and released fibre mass.

The used samples for quality assurance purposes should only be taken from the same wash vial, assisted water sample. This way both the fibre size distribution and the fibre mass are expected to be the same. Experimental triplicates may have a certain amount of variability due to differing amounts of fibre release from the dry sample.

Any variation in the QA/QC sample set, therefore, depicts uncertainty arising from the sample processing and/or image processing only.

Blank

In addition, one procedural blank without any dry sample inside but processed the same way alongside to indicate any background contamination. The final reported values will be blank corrected.

Method accuracy

Furthermore, we also prepared one pre-spiked dry sample without known mass to validate the accuracy of the method.

Control sample

A control sample is taken from the incoming tap water used in the wet processing steps. Only one control sample is required as Ramatex uses a shared, single water source.

Cold storage

It was noted that around 4 °C would be the optimum temperature to minimise bacteria growth in the water samples. It should avoid zero/below 0 °C storage as there would be expansion in the bottle as a result of the liquid freezing

During the time between sample collection and courier pick-up, Ramatex will store the water samples in a refrigerator at around 4 °C. The recommended maximum storage period is 6 hours. However, this is dependent on production and courier schedules, and is not immune to delay.

The project team is aware that under such circumstances, the samples may be stored in the fridge for longer than 6 hours. Ramatex notes that the tap water sourced is itself treated (such as with chlorine), which helps minimise bacteria growth.

Upon arrival at UTM, the water samples will be stored in a cold room at 4 °C as the filtration work progresses.

Others

UTM will record the humidity and temperature of the laboratory when performing the filtration and weighing, and state this in the measurement data sheet for referencing.

At Ramatex, there are dedicated tanks for black coloured and white coloured fabrics. After every cycle, washing of the tank takes place.

For the lint collection from the dye tank after every batch is processed and sample is taken, the filter will be cleaned, ensuring no crossing of fibres.

Further to this, at the first instance of completing each wet process, Ramatex will collect the discharged water at once to minimise the settling down of particles in the water.

Reporting requirements

For the communication of the test data/test results this research requires this to be submitted to the research team and the larger Forum team.

It is important that the integrity of the test data for this research is at its highest possible standard. This will help the research make informed choices in the research phases to come.

A test results template has been developed for each of the processing steps as a guide for what should be used at all times (Annex I. Template Test result

SECTION 3: FINAL REPORT ON RESEARCH PLAN AND RESULTS

1. Summarised research plan

1.1. Schematic flowchart of samples treatment and analysis for both dry and wet textile process

The dry and wet samples were obtained from a textile factory, RAMATEX Group. The 4 most prevalent processing steps was selected, including After Scouring, After Dyeing, After Rinsing and Heat Setting. The wet samples were water effluent from selected process steps. The dry samples were greige fabric, finished fabric, yarn type.

A schematic of fabric processing and experimental wash procedures/conditions is shown in Figure 1.

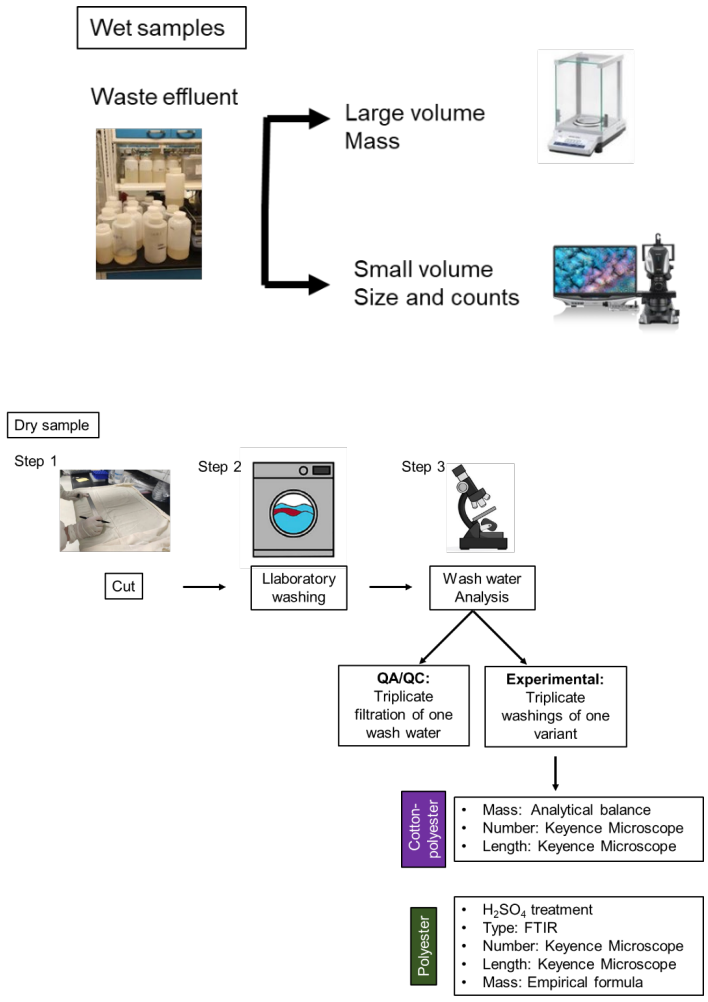


Figure 1. Schematic of experimental design for both wet (above) and dry (below) samples and wash water analysis in terms of mass, fibre numbers, composition, and size distribution.

1.2. Sample collection and contamination prevention

To minimise contamination, all laboratory glassware was cleaned prior to use, oven dried and stored in a dust-free environment. Glassware was kept covered to prevent airborne contaminations [1]. Glassware was kept covered to prevent airborne contaminations. In contrast to reported studies, no cotton lab coat was used as personal laboratory equipment. Instead, a lab coat made of a polyamide monofilament commonly used for surgical gowns was worn as it is designed to have a low-lint surface [1]. Additionally, nitrile gloves were worn during the wash experiments, and samples were exposed to the ambient environment only during the brief transfer from the filter to the Petri dishes [2]. Due to the transport issue, the wet samples were collected into a sealed polypropylene bottle [3], and then transferred to the lab for microfibre analysis.

Figure 2. Schematic of experimental design for packaging the dry sample

1.3. Pre-treatment of dry samples: sealing method (Step 1)

Dry samples were cut to a weight of 10 g, folded once in the half. Using a textile glue (Skinotex, Weniger) the corners and edges were sealed to prevent fibre loss. For this purpose, the textile was immersed into the adhesive by 0.5 mm on each side [1]. The prepared specimens were air dried overnight in a dust free environment prior to washing. The samples were then packed in aluminium foil and sealed in plastic bags. These were transferred to the lab for analysis.

1.4. Standardised Washing Procedure for dry samples (Step 2)

Due to the fact that we cannot collect the fragment debris of the dry samples during industrial production, we use a washing method to extract the fragments from those dry samples before and after the engineering treatment. The washing procedure was carried out using a laboratory washing test for industrial laundry processes and based on the ISO Standard 105-C06:2010, with some modifications for these specific solutions [4, 5]. A laboratory rotator (Figure 3) was operated at 40 rpm with glass vessels. Temperature was controlled at 40 °C. The entire experimental washing lasts for 45 min. For each washing sample, every 10 g of fabric was

placed inside the vessel with 200 mL DI water, depending on the experimental conditions and instrument detection limit. To simulate the stress produced during a normal washing cycle and release the maximal number of fibres, 50 stainless steel balls (Ø 6 mm) were also introduced in the samples to provide additional mechanical wear during the wash cycle [4]. In common practice, the washing experiment was repeated three times for each type of fabric samples unless stated otherwise. Experiments with results of large variation would be repeated more than three times for better data quality. After washing, the samples were removed and rinsed with ultrapure water from both sides using a wash bottle. The steel balls were separated from the washing liquid and rinsed with DI water. All washing liquids were collected, and the solution was diluted to a total volume of 300 mL prior analysis measurements. In addition, analyses were made on the liquid obtained from the vessel without fabrics used as blanks to account and correct for the potential background of the washing agent and the background variability [6]. This standard washing protocol is used throughout the baseline research and the complementary investigation phase for the quantification of microfiber shedding.

Figure 3. Photograph of the laboratory rotator.

1.5. Chemical Separation of Polyester and Cotton Fibres (Step 3)

In this project, we aim to identify the chemical composition of the fibre fragments. Due to the large background of cellulose, the samples were treated with sulphuric acid based on DIN EN ISO 1833-11 to achieve a chemical separation of polyester or other stable polymer and cotton. Using the sulphuric acid treatment, the cotton component is hydrolysed, whilst polyester or other stable polymer is left over [1]. The cellulose filter with trapped fibre fragment were incubated in 20 mL concentrated sulphuric acid (75% p.a., KB Bernd Kraft) for 1 h at 50 °C until all the filter disappears. The solution was further neutralised with 50 mL ammonia solution (25%, p.a., Emsure®Merck). The samples were further filtered with one pre-weighted 0.45 µm glass filter (Ø 4.5 cm) and dried for 1 h at 80 °C before being conditioned in the climate chamber. The weight gain was determined if there was significant reading change in the balance. The number of fabric fragment on the filter was further screened using Keyence Microscope method and the material type can be confirmed by Fourier Transform Infra-Red spectroscopy (FTIR) as detailed below [7]. The spectra obtained were compared to a spectral database of synthetic polymers.

In the case of the water sample collected from the factory, the suspensions were filtered through a cellulose acetate filter (MFS membrane filters; Advantec MFS, Dublin, CA, USA) with a pore size of 0.45 µm and a diameter of 45 mm under vacuum conditions. The filter was

carefully rinsed by 100 mL DI water and left to dried for 15 min at 50 °C in a glass Petri dish. Then, 20 mL sulphuric acid was added, and the suspension was incubated for 1 h at 50 °C. The samples were carefully shaken in regular time intervals of approximately 20 min. After the treatment, the acid solution was neutralised. The entire liquid was transferred to 150 mL water while stirring and the ammonia solution was added until a pH-value of 7 was reached. Each sample was diluted to a total volume of 300 mL and cooled down to room temperature prior to analysis.

1.6. Wash Water Filtration, and Filter Imaging (Step 3)

The filtration system consisted of a vacuum pump pulling the wash water through a glass fibre 5 µm pore sized filter (Ø 4.7 cm). The filters were then left to dry for 24 hour inside an aluminium protective case that reduced possible airborne residue and contaminants. The white colour filter paper will be used for the dark colour fabric to achieve the vast contrast. Similarly, the black colour of the filter paper was used to analysis the white colour of the fibres if there is any.

The quantity of material released from the fabrics during washing was calculated using the following equation:

$$Mass\ of\ \ Fiber\ released = (Fm2 - Fm1)$$

Where Fm1 is the oven dry mass (50 °C, 12 hours), in grams, of the filter prior to testing; Fm2 is the oven dry mass, in grams, of the filter assembly after testing (including any material collected by the filter),

A Keyence Digital Microscope System with a VHX Digital Microscope Multi Scan Lens was then used to image the filters. The image captured with the microscope was a high-quality resolution image of 96 dpi, which was a large composite image digitally stitched together from multiple snapshots. Typically, 30~50 individual images were needed to image the whole filter, and the individual images were automatically aligned by the microscope computer software to give one single image of the filter as a final result. An appropriate scale bar was placed on the composite image for later processing with the open source software ImageJ.

Depending on the wash condition, the volume of wash water filtrated was either 50, 100, or 200 mL. This can be decided during the preliminary test. This variation depended on the quantity of fibres that can be observed on the filter during the filtration step to ensure an optimal quantity of fibres were deposited for analysis. Either too many or few fibres would create extensive overlapping or invisible of individual fibres, thus (1) making fibre identification more difficult or (2) causing fibre mass underestimation using image analysis.

1.7. Filter Image Processing Fibre Length Distribution, and Fibre Mass Calculations (Step 3)

Three metrics were obtained from the analysis, (1) fibre number, (2) fibre length distribution, and (3) fibre mass. In all cases, images were uploaded in the .tiff format into the ImageJ

software with their native pixel resolution of 96 dpi. To obtain the fibre number and fibre length distribution, all individual fibres were counted manually over the entire filter for all standard wash condition filters. Using a digital tablet and stylus, lines were drawn on top of the fibres. The scale was set using the imbedded scale bar on the image and applied to lines drawn on the fibres. Lengths of individual fibres were exported to Excel for further data processing.

To determine the mass of polyester or any other stable polymer microfibres released from synthetic fabrics, all the fibres after acid treatment were counted on the filter, the known fibre diameter and density were then used to calculate the mass of fibres on the filter. The fibre length distribution was converted to a volume distribution (using the known fibre diameter) and subsequently to a mass distribution when multiplied by the fibre density. By integrating this mass distribution, the mass of fibres was calculated. The weight of microfibres released from synthetic fabrics could be estimated using an empirical formula [8]: Further, the weight of cotton microfibres released or the mass of stable polyester microfibres could be calculated using the following equation. Meanwhile, those estimated values can be further compared with the mass calculated from filter weighing in previous steps.

$$Grams\ of\ microfibers\ /\ gram\ fabic\ = C \times \left(\pi \times \frac{D^2}{4} \times L\right) \times \rho$$

where C is the number of microfibres released from 1 gram of fabric; D is the average diameter of the fibres; and ρ is the density of the fabric.

1.8. Quality Assurance/Quality Control Experiments (Step 3)

Re-producible fibre quantification (in terms of both fibre size and fibre number) was of utmost importance for the success of the method and thus QA/QC experiments were performed in order to test the reliability of the wash water filtering, filter imaging, and data processing sequences [5]. For one selected sample, triplicate samples were filtered through separate filters, imaged, and analyzed for both fibre size distribution and released fibre mass. While experimental triplicates may have a certain amount of variability due to differing amounts of fibre release from the textile swatch, because these QA/QC samples originated from the same wash vial, both the fibre size distribution and the fibre mass should be the same. Any variation in the QA/QC sample set, therefore, depicts uncertainty arising from the sample processing and/or image processing only. In addition, we have one procedural blank without any fabric inside but processed the same way alongside to indicate any background contamination. Furthermore, we also prepared one pre-spiked fabric without known mass to validate the accuracy of the method.

1.9. Microfibre reattachment measurement

For the testing fabric, we used the material with the most microfibre shedding, the black-coloured 80/20 CVC Fleece 250gsm). We created enough microfibres from the fleece using simulated laundering based on the standardised washing procedure for dry samples (1.4),

measured suspended microfibre content in the water, rinsed fleece again in a sonication bath, and measured the suspended microfibre content in the water (Figure 4).

We measured, compared, and analysed the microfibre re-attached to the fleece. Specifically, 10g of black fleece fabric was washed inside the washing vessel with 200 mL DI water, depending on the experimental conditions and instrument detection limit. After washing, each fabric sample was drained, and the washing water was filtered by following the filtration method in 1.6, for the measurement of microfibre shedding. Further to this, each fabric sample was then rinsed in a 200 mL DI water in a sonication water bath for 1 hour at room temperature (25°C). The rinsing water was filtered and measured for microfibre re-attachment.

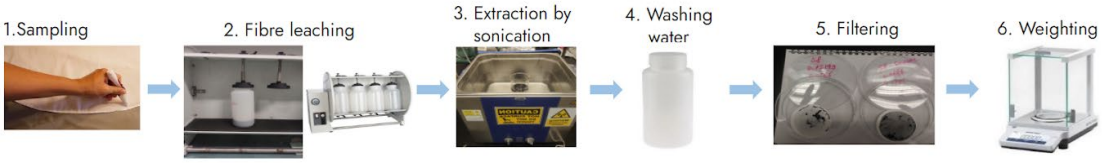


Figure 4. The experimental setup of the microfiber reattachment measurement.

1.10. Measurement of temperature impact to microfibre shedding in dyeing process

The dyeing process is the interaction between a dye and a fabric, as well as the movement of dye into the internal part of the fabric. Temperature and duration are the key controlling factors. The 4 types of fabrics, including 80/20 CVC Fleece, 100% Cotton Jersey, 100% Polyester, and 100% Polyester - Recycled were selected to investigate the impact on microfibre shedding from temperature and processing duration. It should be noted that while the baseline research used a recycled polyester blend (61% recycled PE, 39% polyester fleece), the investigative research used 100% recycled polyester in testing the temperature and duration impact on microfibre shedding.

The temperature and duration settings that Ramatex provided is based on what is applied to the fabric in the full duration that is it in dye tank. This was provided as follows:

i. Polyester fabric: 130°C for 300 min

ii. Cotton fabric: 60°C for 252 min

iii. 80/20 cotton/polyester fabric: 130°C for 300 min followed by 60 °C for 252 min.

In the experiment of measuring temperature impact on microfibre shedding, 10g of fabric sample was rinsed in 200 mL DI water in glass beakers. A range of heating temperatures (60°C, 90°C, 130°C) was applied to the samples respectively. Following the instructions from Ramatex, polyester samples were heated for 300 minutes. Cotton samples were heated for 252 minutes. Finally, 80/20 samples were heated for 552 minutes. After heating, we collected the rinsed water and measured the microfiber shedding following the methods in 1.6.

1.11. Measurement of duration impact to microfibre shedding in dyeing process

In the experiment of measuring processing duration impact on microfibre shedding, a range of heating durations (120 min, 252 min, 300 min) was applied to the samples respectively. Following the instructions from Ramatex, polyester samples were heated at 130°C. Cotton samples were heated at 60°C. Finally, 80/20 samples were heated at 130°C and 60°C, consecutively. After heating, we collected the rinsed water and measured the microfibre shedding following the methods in 1.6.

2. Preliminary results

2.1. The characterisation of composition and structure in raw fabric material

Thermogravimetric analysis (TGA) has been used in the analysis of chemical composition in one fabric sample received[9]. Thus, the result found the mix has a ratio of 82% cotton and 18% polyester (Figure 5). As told by the factory, a 20/80 polyester-cotton fabric was chosen. This result from TGA is roughly consistent with the provided parameters. Therefore, TGA can be used for the composition validation for the raw material for the following selected fabric samples when needed. However, this method is not sensitive and cannot be used to detect the leached fibre fragments (Data not shown).

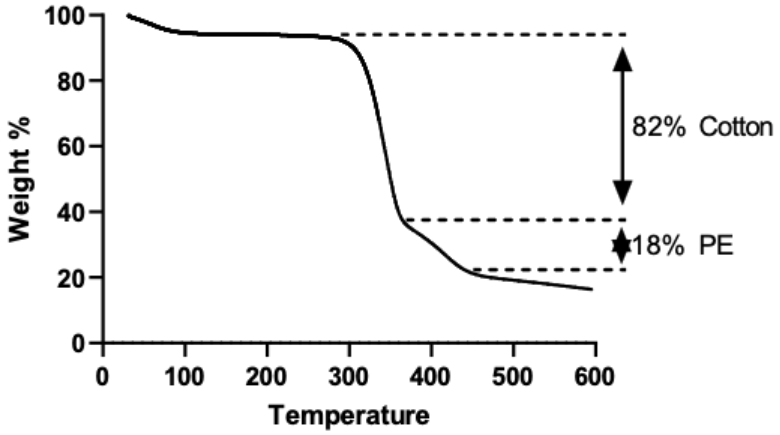


Figure 5. Thermogravimetric Analysis (TGA) of the ratio of cotton and polyester.

Separating polyester and cotton fibres from a mixture by concentrated sulphuric acid was investigated first conducting the experiments on the textile surface. If the chemical treatment conditions were appropriate, a complete degradation of the cotton component was expected. Figure 6 shows the textile surface characterisation before and after sulphuric acid treatment. Indeed, surface changes were observed after chemical incubation, indicating a more aligned structure in the SEM-image. Besides optical changes, the haptic properties turned to a smoother surface. Both observations point out characteristic properties of synthetic fibres. This result was consistent with the weight loss of 81 % after the acid treatment (see Figure 7), concluding that the cotton component was fully removed. As a control experiment, one sample of 100% cotton fabric was exposed to sulphuric acid. In fact, no fabric was left. These

experiments confirmed the hydrolysis of cotton under sulphuric acid incubation whilst polyester remains. In addition, we further verified the polyester fabric as polyethylene terephthalate (PET) using FTIR (Figure 8).

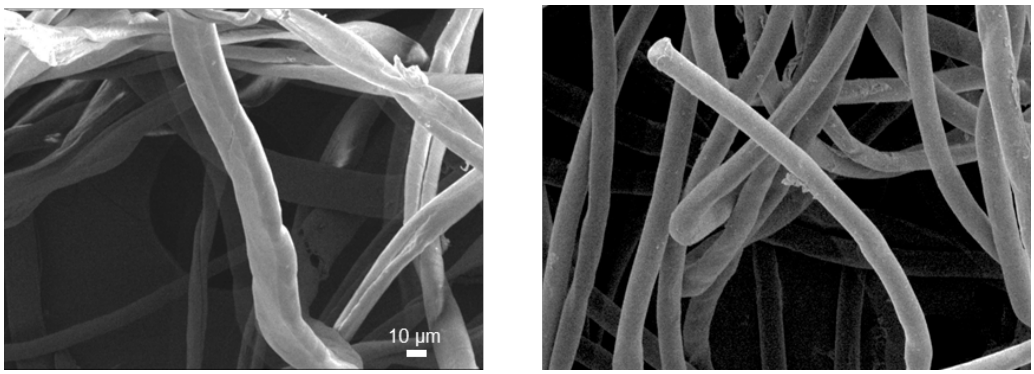


Figure 6. SEM-images of a 20/80 polyester-cotton intimate blended fabric. (Left) Surface of the untreated fabric (Right) Fabric surface after treatment with concentrated sulphuric acid. It shows a more aligned structure and a smoother surface after sulphuric acid incubation, confirming the removal of cotton.

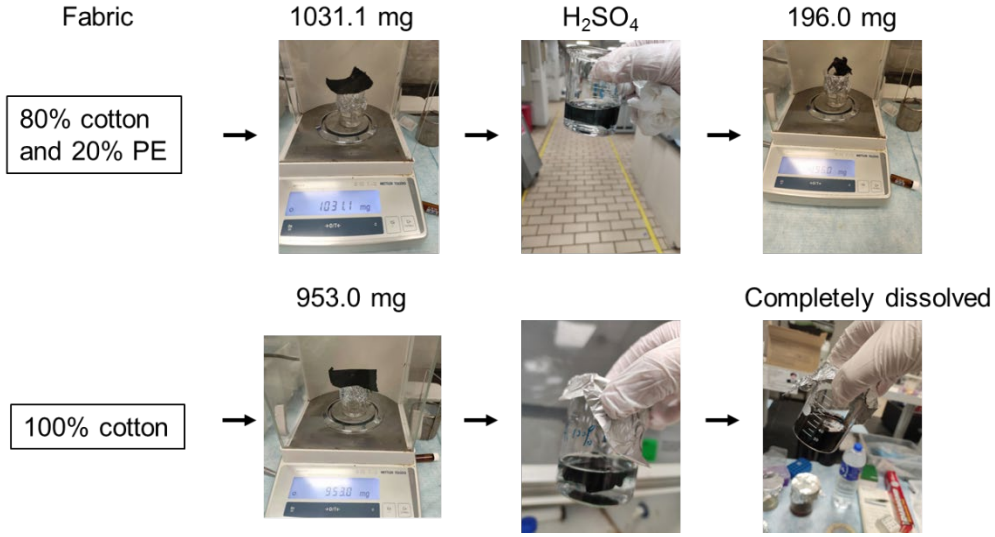
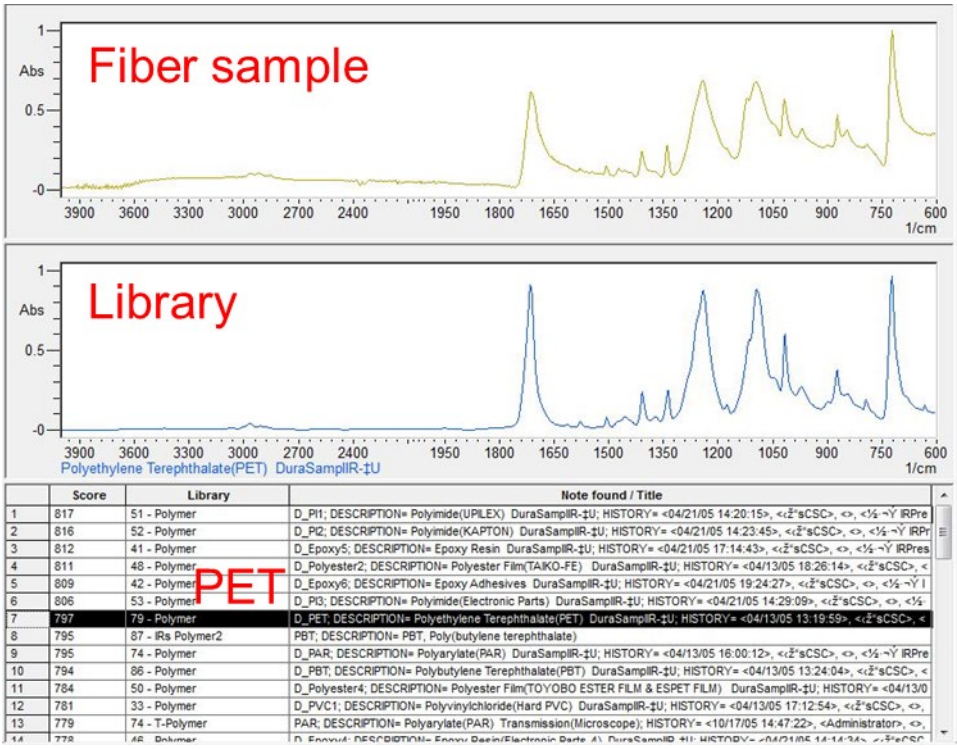


Figure 7. Weight of 20/80 polyester-cotton blended fabric and 100% cotton before and after sulphuric acid treatment.



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2	816	52 - Polymer	D_PI2; DESCRIPTION= Polyimide(KAPTON) DuraSamplIR-‡U; HISTORY= <04/21/05 14:23:45>, <‹Ž"sCSC>, <>, <½·¬Ý IRPr
3	812	41 - Polymer	D_Epoxy5; DESCRIPTION= Epoxy Resin DuraSamplIR-‡U; HISTORY= <04/21/05 17:14:43>, <‹Ž"sCSC>, <>, <½·¬Ý IRPres
4	811	48 - Polymer	D_Polyester2; DESCRIPTION= Polyester Film(TAIKO-FE) DuraSamplIR-‡U; HISTORY= <04/13/05 18:26:14>, <‹Ž"sCSC>, <
5	809	42 - Polymer	D_Epoxy6; DESCRIPTION= Epoxy Adhesives DuraSamplIR-‡U; HISTORY= <04/21/05 19:24:27>, <‹Ž"sCSC>, <>, <½·¬Ý I
6	806	53 - Polymer	D_PI3; DESCRIPTION= Polyimide(Electronic Parts) DuraSamplIR-‡U; HISTORY= <04/21/05 14:29:09>, <‹Ž"sCSC>, <>, <½·
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8	795	87 - IRs Polymer2	PBT; DESCRIPTION= PBT, Poly(butylene terephthalate)
9	795	74 - Polymer	D_PAR; DESCRIPTION= Polyarylate(PAR) DuraSamplIR-‡U; HISTORY= <04/13/05 16:00:12>, <‹Ž"sCSC>, <>, <½·¬Ý IRPre
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Figure 8. FTIR spectrum of microfibres after acid digestion.

2.1. Scanning electron microscope (SEM), FT-IR, and microscopic imaging of microfibres

To figure out which method works best for this project, we have conducted a few imaging methods using one trial sample, including scanning electron microscope (SEM), FT-IR, and microscopic imaging. First of all, the SEM method was performed on the fibres released from the textile after washing. SEM is a trustable tool, but it is time and cost consuming. Here, 28 individual images were taking using SEM and some representative ones are shown in Figure 8. The minimum measurable length of a fibre with this analytical setup was determined to be ~20 µm, suggesting the nanoscale of fibre fragment is not a concern for this project. The limitation of following KEYENCE microscope is 0.5 µm and SEM result showed that the microscope can be a good choice for this experiment. This usage of microscope has also been reported in other studies [5, 10]. Further, this length also corresponds to the minimum number of pixels that could definitively be considered a fibre when manually counting or when using the filter coverage/binary analysis method, which was between 2 and 5 pixels depending on the saturation of the particular image.

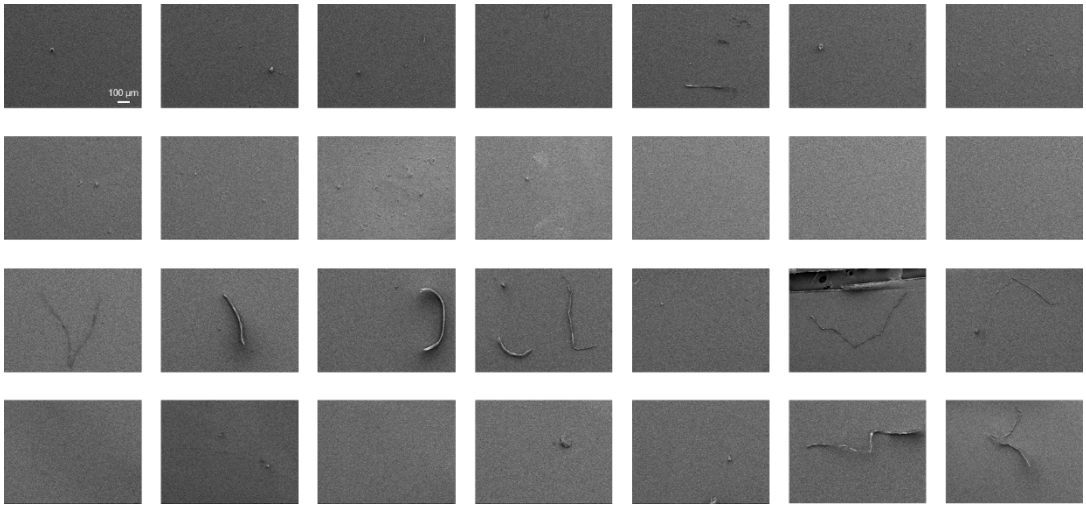

Figure 9. SEM images of the fibres released from the textiles after washing.

Next, to examine the validity of KEYENCE microscope, a Keyence Digital Microscope System with a VHX Digital Microscope Multi Scan Lens was used to image the filters. As shown in Figure 9, 24 individual images were taken to image half of the filter, and the individual images were automatically aligned by the microscope computer software to give one single image of the filter as a final result.

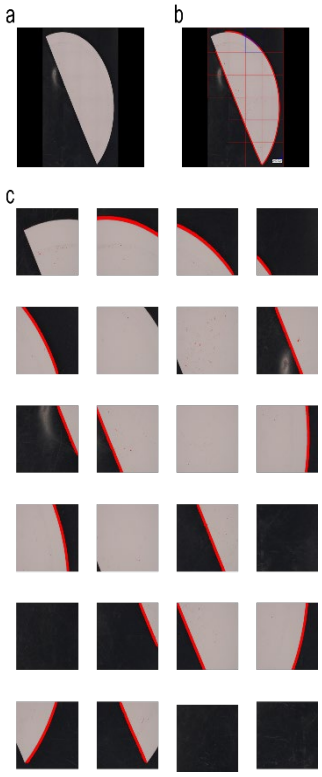

Figure 10. **a** Snapshot of a filter image for textiles, **b** A large composite image digitally stitched together from multiple snapshots, and **c** Individual images of the filter by the KEYENCE microscope.

The size distribution (Fibre length distribution histograms Figure 9, summary of data Figure 10) and mass (Figure 10) of microplastic fibres shed from textiles washed under DI H_2O was

further determined by the microscope. The result showed that a large quantity of microfibres have the size of ~50 µM with a wide size range. Similar fibre length release profiles and the mass of fibres shed were found, compared to the results of SEM. This substantiates the results obtained by the KEYENCE microscope is acceptable.

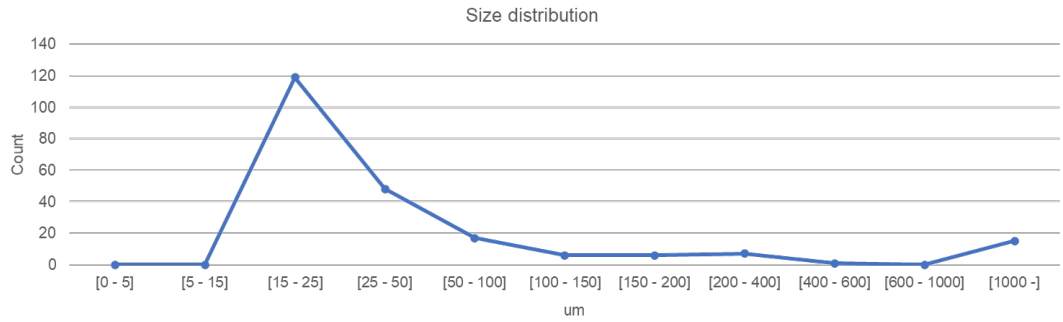

Figure 11. Microfibre size distribution released from washing in DI H_2O. The sample was filtered with a 1.6 µm filter (Ø 4.5 cm) and imaged using KEYENCE microscope.

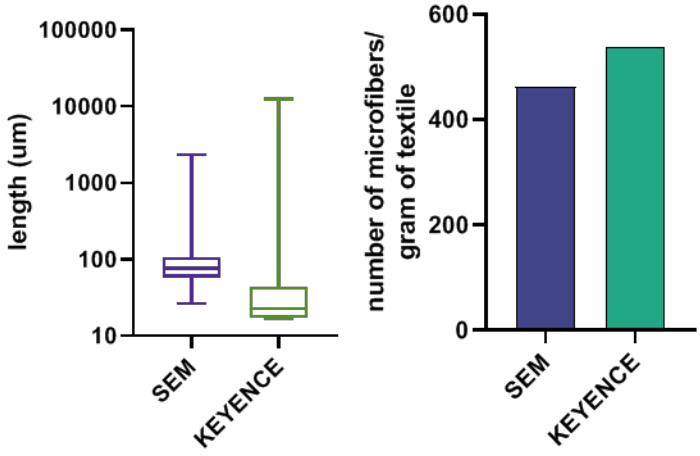

Figure 12. Microfibre size distribution (left) and calculated average mass (right) of microplastic fibres released from washing in DI H_2O obtained from SEM and KEYENCE microscope.

To validate the performance of “acid-wash+FTIR” imaging in identifying the polymer type, FTIR auto imaging was applied. As shown in Figure 11, attenuated total reflectance Fourier transform infrared (ATR-FTIR) analysed the microfibres collected in the glass filter paper and revealed that they were PET (Figure 11). In contrast, without acid treatment, the high background of cellulose prevented the identification of this type (data not shown), suggesting the proposed method works well to identify the polymer type.

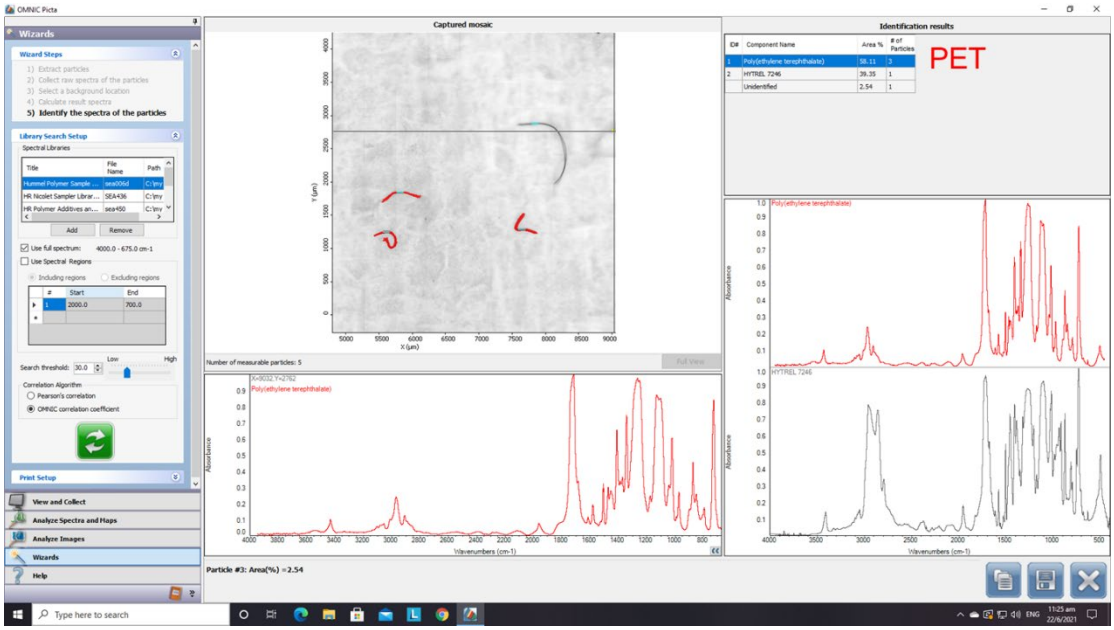

Figure 13. Attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectra of microfibre from the textiles after washing.

2.2. Quality Assurance and Quality Control Studies.

We explored two facets of quality assurance and quality control in these studies; i.e., the capture efficiency of fibres suspended in the wash water onto the filter and fibre analysis on the filter, in terms of size distribution and total mass. Among them, the size distribution was analysed using SEM. As mentioned above, we have pre-spiked 2.9 mg manually cut fibres into the DI water. The weight calculated from the filter mass difference is 0.1 mg, suggesting the capture efficiency is very high and almost all the fragments can be trapped on the top of the filter. For the same wash sample, we have conducted the measurement by taking liquid aliquots three times. The result showed that the size and calculated weight is very comparable for all three measurement. Those result verified that the analytical techniques we used were consistent in providing similar released fibre size and mass metrics (Figure 13). In addition, method blanks of DI H_2O were analysed for presence of fibres but too few (<3 per filter) were detected to image the entire filter in our system, suggesting the contamination from the operation is acceptable.

Figure 14. Glass fibre filters have a relatively high efficacy for capturing the polyester fragmentations.

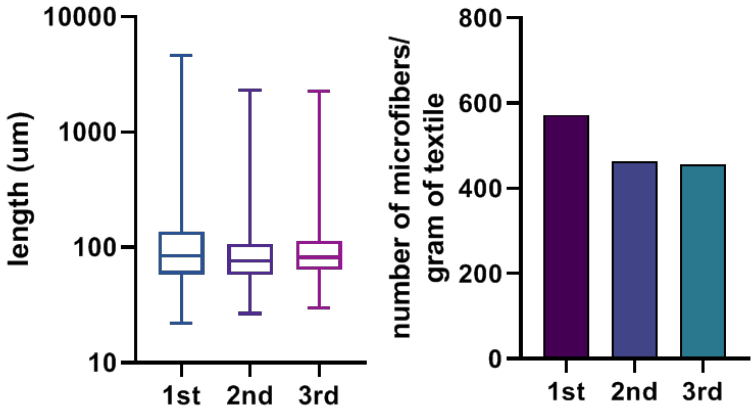

Figure 15. QA/QC of triplicate filters from a subset of wash water. Analysis of the fibre length distribution in DI H_2O. Fibres measured on different filters are shown in different colours. Since these are aliquots of the same wash water, this data does not represent the variability of the washing procedure between different fabric swatches but rather the variability associated with sample processing, filter preparation and filter analysis.

3. Standard Operating Procedures (SOP)

3.1. SOP for Ramatex collection of wet samples

1. **Note**: The dry and wet samples should be from the same batch to make the result more comparable. Write down the total mass or quantity of the textile processed.
2. Wear glove and lab coat as provided.
3. **Clean the bottles before collection:** Before collecting wet samples, we need to wash the new bottles. As shown in figure 14 below, we need to follow the following steps to wash the bottles 1) Add 200 mL clean tap water (e.g., no rusty debris) to the bottle then cap it; 2) Shake the water around 5 cycles in the bottle then dump. 3) Repeat washing one more time. 4) Remove excess water. 5) Leave these bottles open to air dry with bottom up.

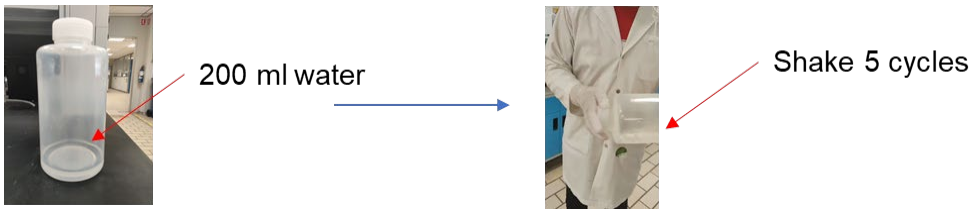

Figure 16: Steps for washing of bottles.

4. **Cleaning the tank:** For the batch that are going to be used for the wet sample, it will be good to clean the tank thoroughly to remove any background contamination.
5. **Wet sample collection:** Collect the discharged water ASAP when the particular process is done and ready for discharge to minimize settling down of particles (Danny's advice). Take 4 L from each process.

6. For those processes that **have filter in the discharge of the tank**, collect the **filtered fibres** on top of the lint. Dry the filtered fibres at ~50 °C. Wrap up the dry fibres using the aluminium foil, which are further labelled and stored in the plastic bag.
7. **Sample storage and delivery:** For the collected sample, it should be best to be stored in one 4°C refrigerator if the waiting time is more than 6 hours to minimize the bacteria growth. It will be good to ship all the samples to UTM on the same day after the completion of one batch collection.
8. **Control sample:** Collect one control sample by using 4 L tap water (the same water for the textile manufacturing process) for each batch.

3.2. SOP for UTM handling and filtration of wet samples

Dry sample

1) Yarn – one cone of about 200gram each.
2) Greige fabric – 400 grams
3) Finished Fabric – 400 grams

Wet Sample

1) Control – 4L
2) Pre-treatment – 4L
3) Dyeing
   - After Dyeing – 4L
4) Hydro extraction – 4L
5) Heat Setting – 4L

Standard Operation Procedures (SOP) of Wet sample in UTM

Material and Setup

Figure 17: Filter setup; Volumetric graduated cylinder; cellulose acetate (CA) membranes (5.0 µm pore size); Aluminium foil; petri dish; labels

Steps (see Figure 18 for illustration):

1. After receiving wet sample, store the samples in the cold room (4 °C).

2. Before process the samples, wear nitrile gloves and polyamide monofilament lab coat, and recover the wet samples to room temperature.
3. Clean the vacuum filtration apparatus with DI water.
4. 5 µm cellulose acetate (CA) filter is dried at 55 °C for a minimum of 8 h; let it reach the RT and weight is recorded (for white coloured textile, use black filter; for others, use the white colour).
5. Install the filter on the filter assemble and rinse with 50mL DI water.
6. Filter sample for microfibre mass calculation: Shake the water in the bottle well before pouring into the graduate cylinder. Take 500 ml of wet sample into the graduate cylinder, which is then filtered onto a 5 µm CA filter. Rinse the graduated cylinder with 100 ml water and then filtered. For each wet sample (i.e., 4L for each wet sample), triplicate samples were filtered through separate filters (i.e, 1,500 mL from the 4L will be used).
7. Size and numbers: The procedure mostly follows Step 6. However, 100 ml of wet sample was filtered onto a 5 µm CA filter, and rinsed with 20 ml water. For each selected sample, triplicate samples were filtered through separate filters.
8. After filtration, the filters were dried overnight at 55 °C in a clean petri dish. The weight is recorded. Filters together with the petri dish were enclosed in aluminium foil, labelled and transferred to NEWRI.
9. Between two samples, the filter setup needs to be cleaned by washing with DI waters.
10. To make sure the weight measurement is comparable between NEWRI and UTM, UTM will ship three unused filter with recorded weight and NEWRI will use it as caliber to calibrate the weight accuracy.

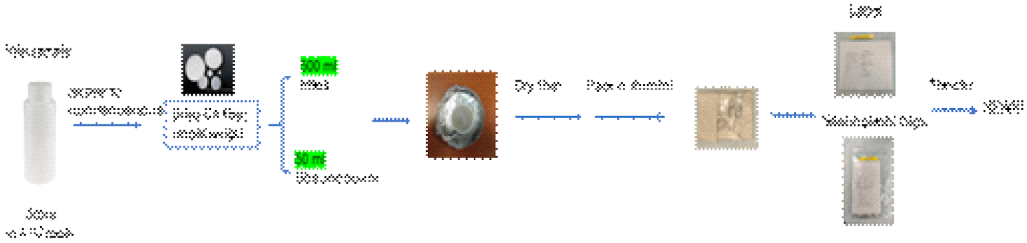

Figure 18: Illustration of the standard operation procedures (SOP) of wet sample in UTM

4. Selection of images from sample collection and analysis

4.1. Wet sample collection from Ramatex facility

Collection of wet samples from black-coloured textile by Ramatex manufacturing facility staff wearing glove and lab coat (Figure 19).

Figure 19. Collection of wet samples from black-coloured textile.

Figure 20. Collection of wet samples from white-coloured textile by Ramatex manufacturing facility staff wearing glove and lab coat

4.2. Wet sample handling and filtration at UTM facility

Figure 21. Semi-micro balance calibrated prior to usage

Figure 22. Filtration apparatus is cleaned with tap water then de-ionized water followed by acetone. The apparatus is then dried and rinse with de-ionized water again before filtration work. The filtration apparatus is cleaned after each filtration work

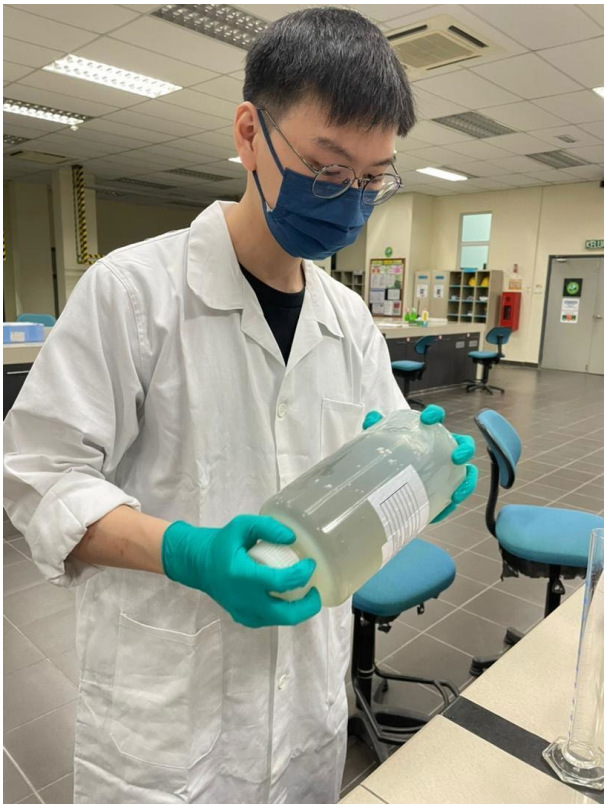

Figure 23. Samples are mixed vigorously before filtration

Figure 24. Desired sample volume is being measured using graduated cylinder

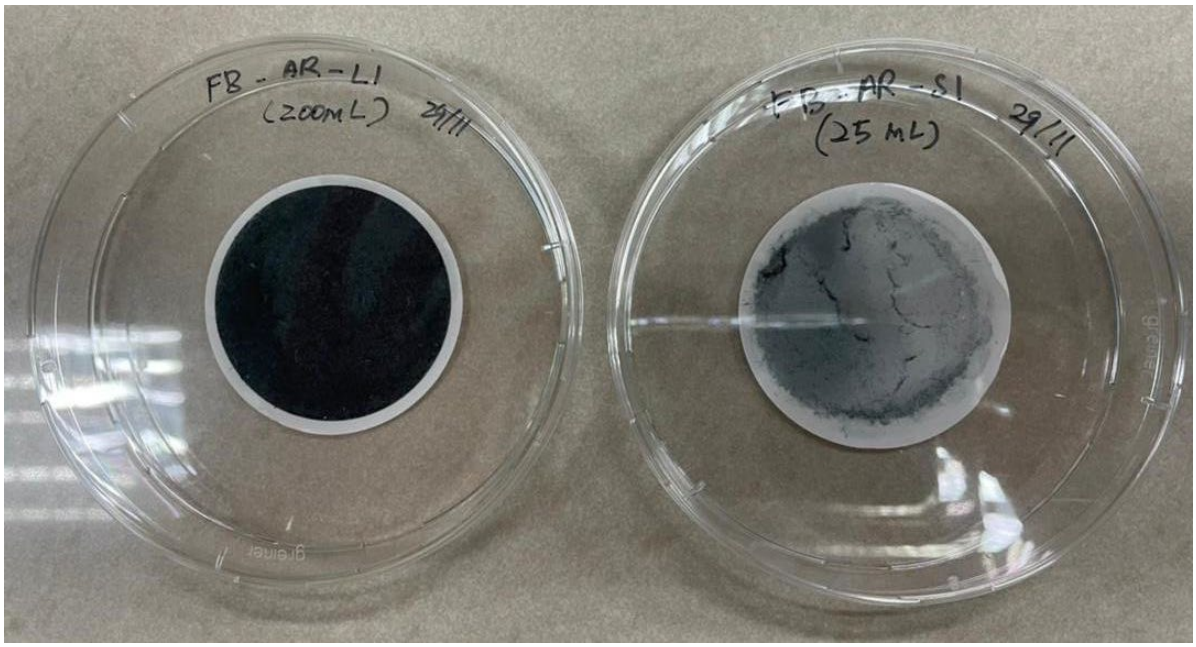

Figure 25. Fleece (Black) - After Rinsing (L: Large volume (mass calculation); S: small volume (size and number)

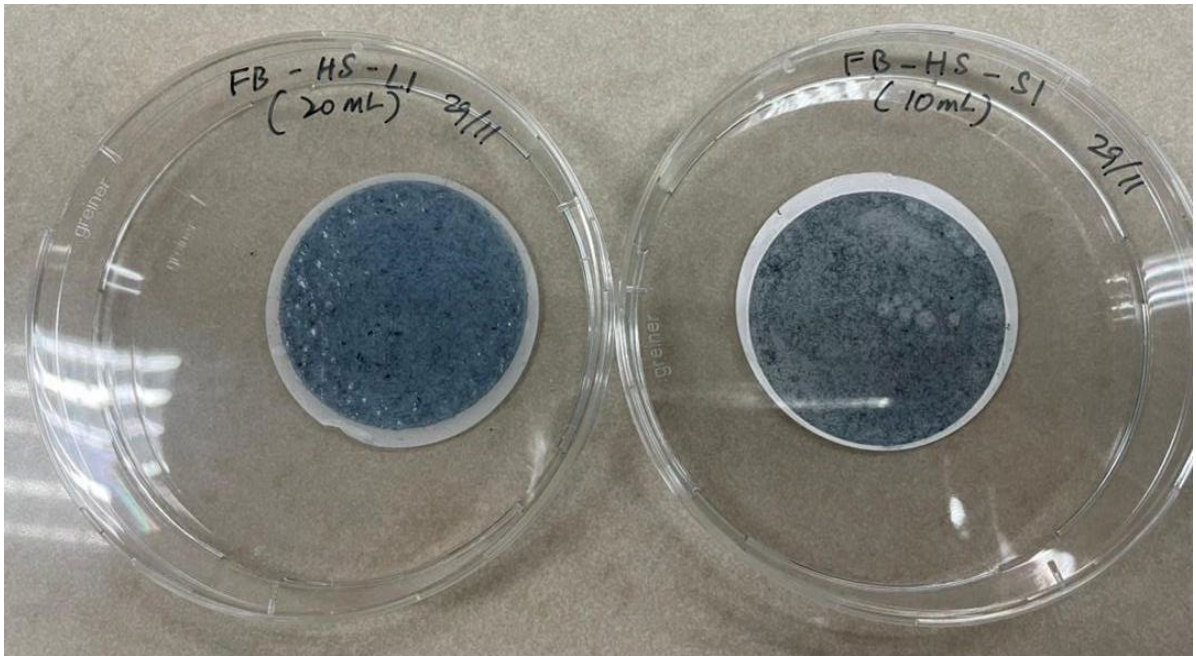

Figure 26. Fleece (Black) - Heat Setting

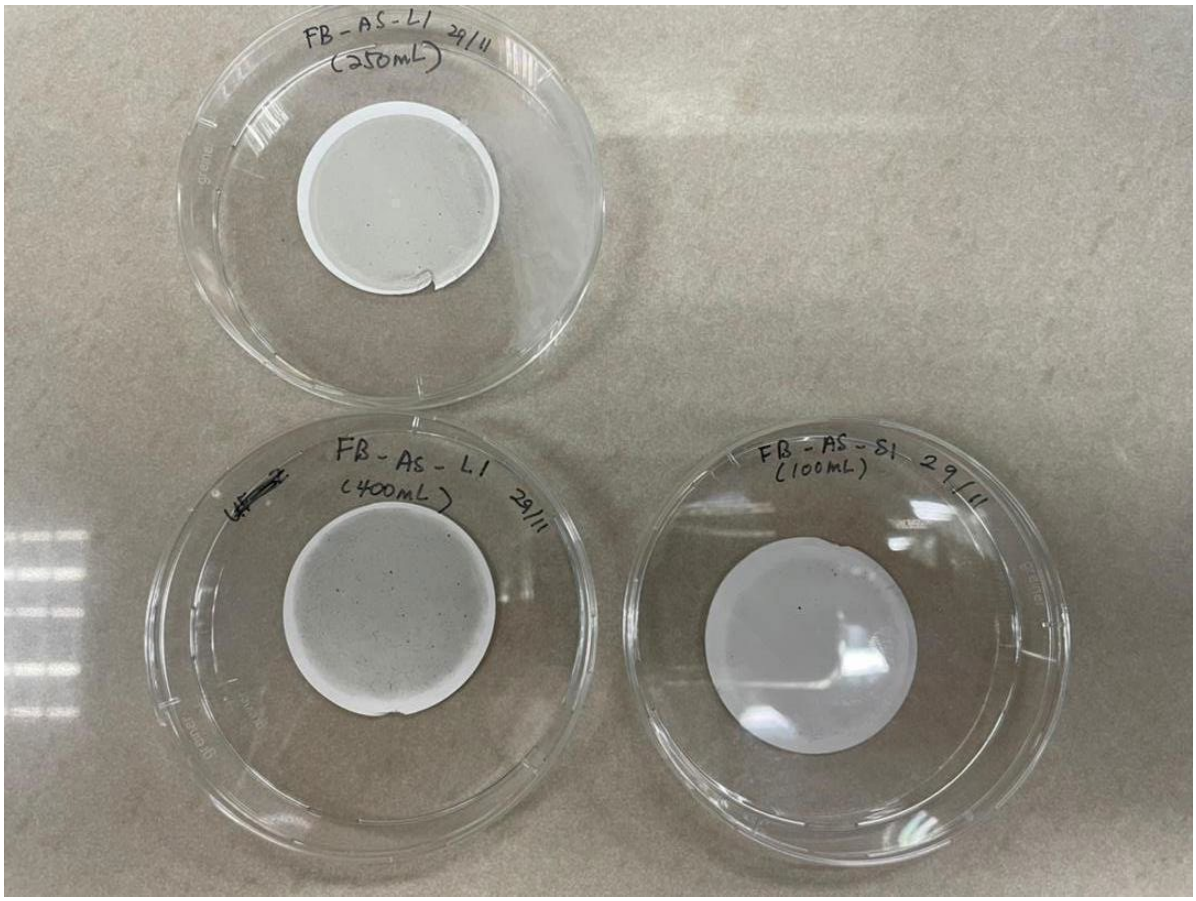

Figure 27. Fleece (Black) - After Scouring

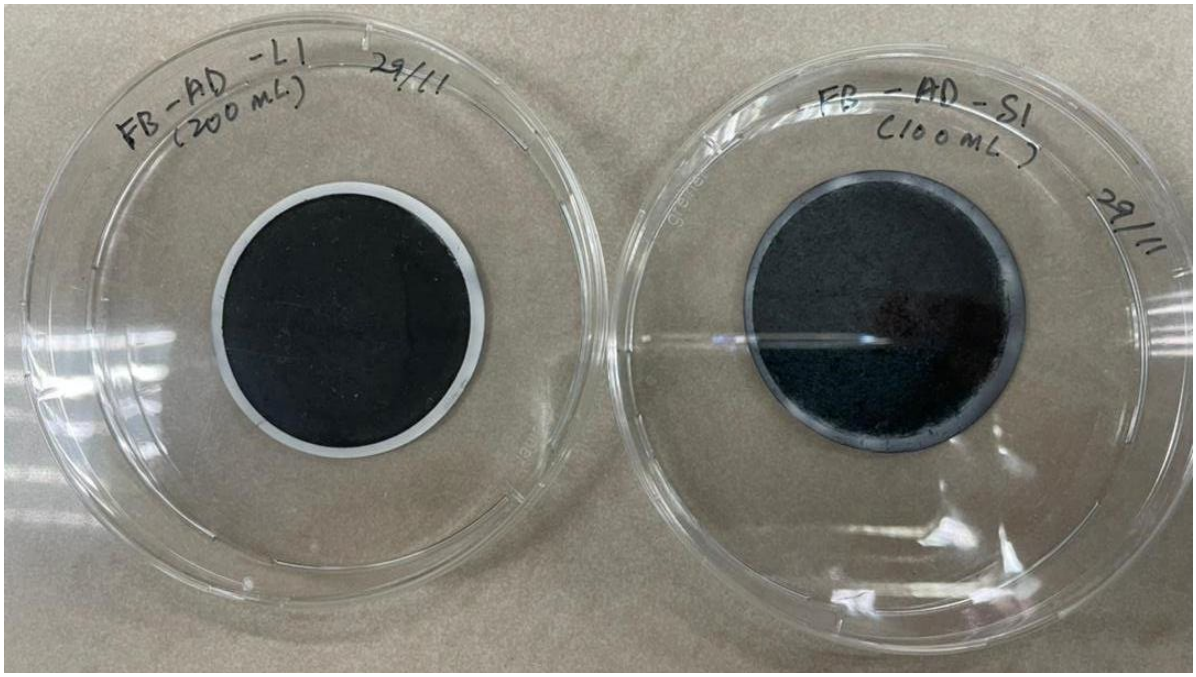

Figure 28. Fleece (Black) - After Dyeing

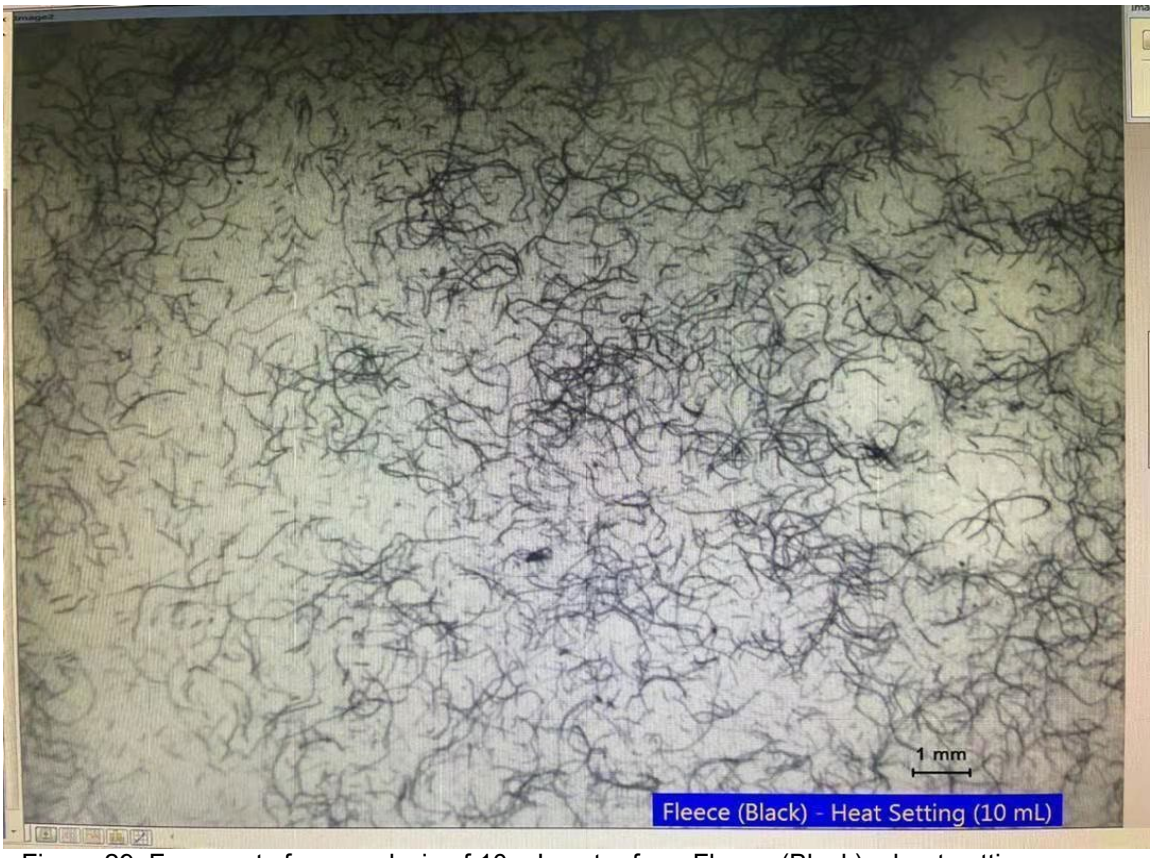

Figure 29. Fragments from analysis of 10 mL water from Fleece (Black) – heat setting process

5. Analysis of wet samples and dry samples

5.1. Summary

Microfibres are a common type of microplastic. One known source of microfibres to the environment is from domestic laundry, which can release thousands of fibres into washing machine effluent during every wash. However, there is still a lack of information on the direct release of microfibres from manufacturing process steps solely within the textile manufacturing facility. Here, we assessed the microfibres shed from the 4 most prevalent processing steps (i.e., After Scouring, After Dyeing, After Rinsing, and Heat Setting) during the production of Fleece, Cotton Jersey, 100% Polyester and Recycled Polyester textiles. Among all the variables tested, the heat setting process appeared to mostly promote the total mass and number of microfibres released, though the overall microfibre length profile remained similar, with the vast majority of fibres ranging between 15 and 400 µm in length. Further, the fibre released from fleece black-coloured textile water samples were higher than that of the other six textiles. A chemical separation of blended fibres released from fleece (black and white) revealed that >80% of the microfibres were cotton. The preliminary result of the release from Recycled Polyester showed the least fibre emission. In addition to the wet samples, we further simulated home washing under controlled laboratory conditions to test the fibre leaching potential from dry samples (i.e., greige fabric and finished fabric). The quantitative data suggests that more microfibres were released from finished fabric than greige fabric. Furthermore, greige fabric made of different raw material showed different fibre leaching potential, suggesting that material selection can be an important factor to control fibre release and warrants further investigation. Overall, the fibre released during production processes can be several-fold or orders of magnitude more than the ones released from finished product during domestic washing. In sum, our baseline study strongly showed that fibre leaching from production processes are step-dependent and material-specific, highlighting that proactive solutions and suggestions are required and possible to avoid and reduce the release of microfibres among the textile industry's manufacturing process steps.

5.2. A short scientific justification on the absence of 100% Polyester (White)

The baseline experiments have investigated microfibre release by fleece, cotton jersey, blended fleece and recycled polyester (61% recycle PE and 39% cotton). As outlined earlier, we could not collect samples from the 100% Polyester White as it had gone out of production and could not be sourced from other channels. Ample evidence from these four types of fabrics indicated that the black textile has more pronounced fibre mass compared to white textile. It is likely because black dyeing process is a lot lengthier compared to white and more damage is done on the fabric, regardless the types of fabrics. Further investigations on microfibre release of the 100% polyester do not necessarily provide additional information for the conclusion drawn from the baseline investigation. Therefore, taking out 100% white polyester

will not invalidate the baseline research.

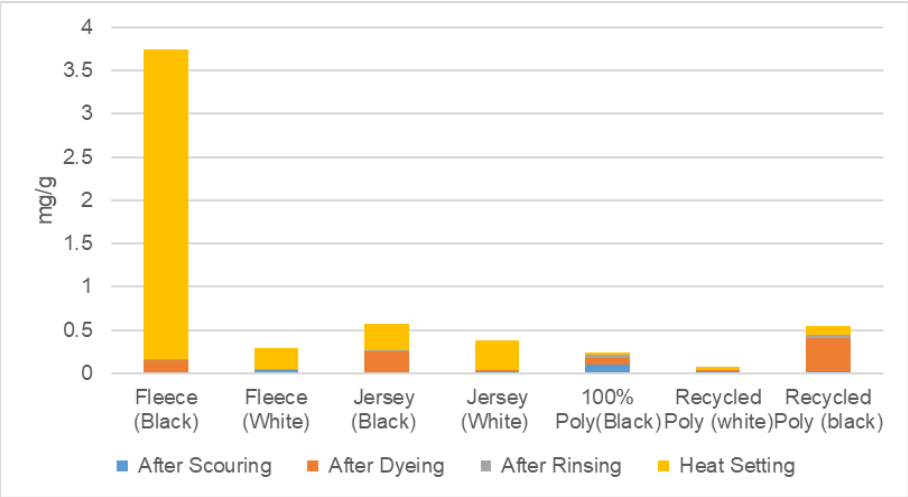

Figure 30. Total mass of microfibres released from all wet processes for each type of fabric textile.

5.3. Mass of microfibres released from wet processes

Here, we analysed microfibres released from the most prevalent processing steps, including “After Scouring”, “After Dyeing”, “After Rinsing” and “Heat setting”, during the production of 7 types of fabric (black Jersey, white Jersey, black Fleece, white Fleece, black 100% Polyester, black Recycled Polyester, white Recycled Polyester). The 4 most prevalent processing steps of these 7 fabric types all promote the release of microfibres (Figure 31), with noticeable differences in the values (Figure 32). The “heat setting” process of black Fleece, and black Jersey showed a higher generation of microfibres per mass of fabric washed, which was followed by the “after dyeing process”. The “heat setting” process of white Jersey, white Fleece, and white Recycled Polyester also show a higher generation of microfibres per mass of fabric washed; however, it was followed by the “after scouring” process. In addition, the released microfibres of black 100% Polyester and black Recycled Polyester were dominant in the “after scouring” process and “after dyeing” process, respectively.

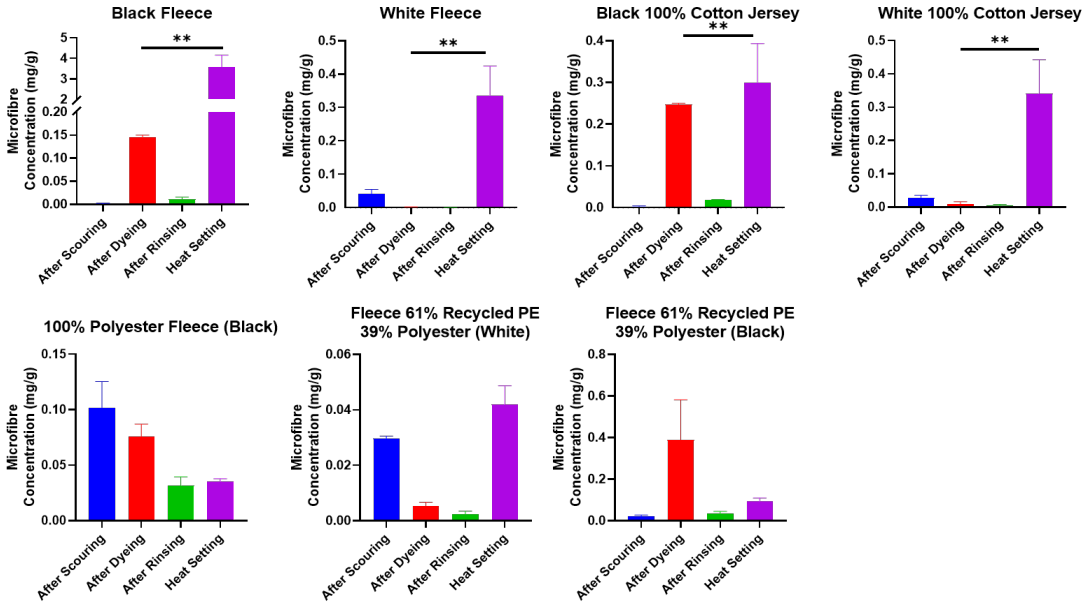

Figure 31. Effect of different processes on the mass of microfibres released during production. The error bars represent the standard error of the mean, N = 3 for all samples. *p < 0.05, **p < 0.01 (Student's t test).

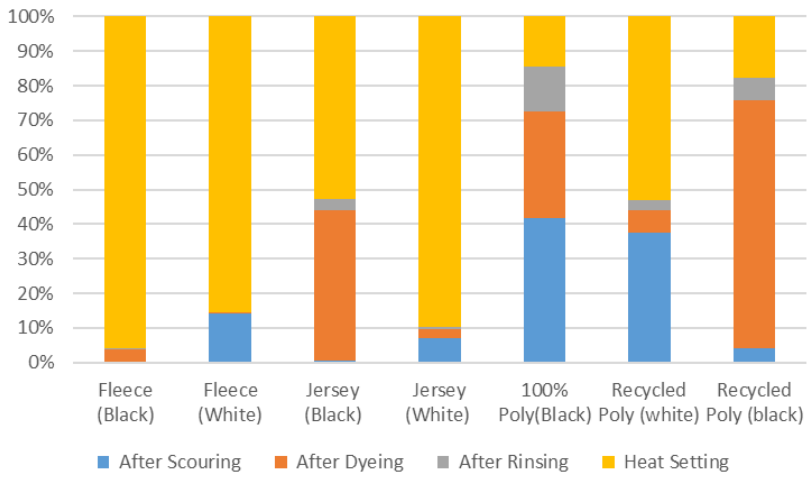

Figure 32. Distributions of microfibres in the wet samples from different fabrics. The percentage was calculated from the mass percentage by each sample.

Interestingly, our result showed that white Fleece released the most fibres in the "after scouring" process (Figure 33). Notably, a considerable mass of microfibres were released from the white fabric. This is likely attributed to the fact that the scouring process acts as a cleaning process. The white fabric has to be cleaned thoroughly to be further whitened without any further delicate dyeing treatment. Thus, it is possible to cause more damage on white fabrics during scouring in relation to the black coloured fabric, though it is currently not clear why the black 100% Polyester has a high emission in the "after scouring" step. In contrast, the black Recycled Polyester released the most fibres in the dyeing and rinsing processes (Figure 33). This is likely attributed to the black dyeing process taking a much longer time compared to the white and likely causing more damage to the fabric. Black fleece released the most

fibres in the “heat setting” step. This is attributed to the fabric going through a bath vat that has water and chemical treatment before going through the drying heat. As mentioned above, the dyeing process for the black Fleece takes the longest time; thus, it is highly likely that there are a lot of loose fibres that are still attached to the fabric after the dyeing process, and they could be completely released during further treatment in the “heat setting” step. Overall, the black-coloured Fleece shows the highest total mass of microfibres (mean 3.75 mg/g) released from the wet process, and the white-coloured Recycled Polyester shows the least total mass of microfibres (mean 0.079 mg/g) released from the wet process (Figure 33).

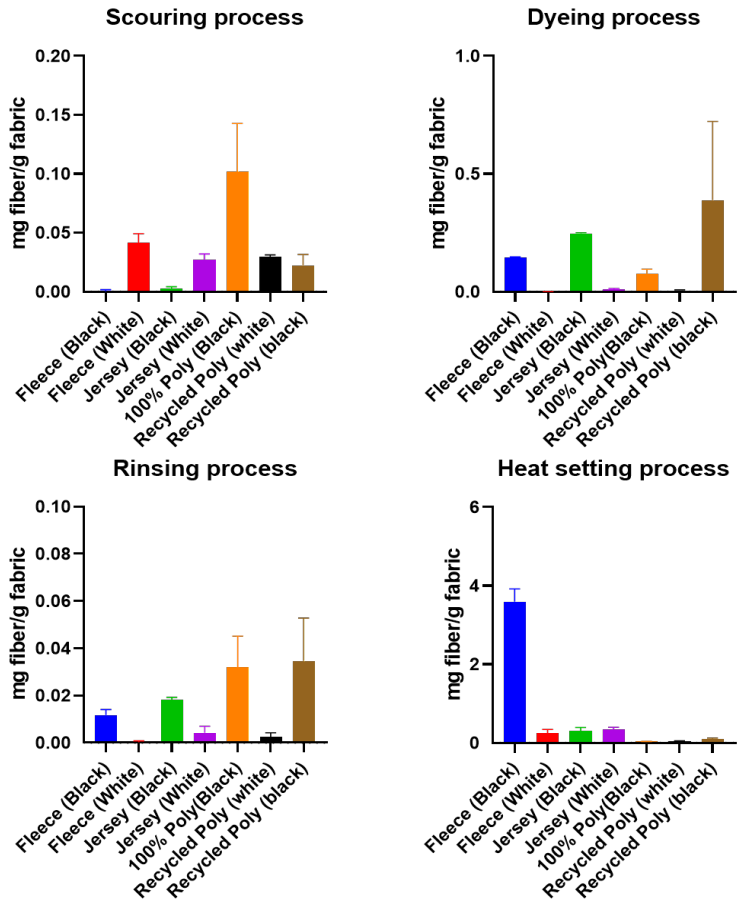

Figure 33. Effect of different treatment processes on the mass of microfibres released during production. The error bars represent the standard error of the mean, N = 3 for all samples.

5.4. Number of microfibres released from wet processes

In general, the number of microfibres released from the wet process steps present a similar trend in the mass of microfibres released (Figure 34). The “heat setting” step is the major contributor in the wet samples of the black Fleece, white Fleece, black Jersey, and white Jersey. The number of microfibres released from this step of the black Fleece, white Fleece, black Jersey, and white Jersey were 26,269 ± 893, 1,610 ± 99, 6,315 ± 1,060, and 56,468 ± 23,741 /g textile, respectively. The “dyeing” process is the major contributor in the wet samples

from the black 100% Polyester Fleece, and black Recycled Polyester (61% recycled PE 39% poly fleece). The number of microfibres released from these two fabrics were 1,311 ± 190, and 5,924 ± 624 /g textile, respectively. The scouring process is the major contributor in the wet samples from the white Recycled Polyester (61% recycled PE 39% polyester fleece). The number of microfibres released from this fabric was 318 ± 48 /g textile.

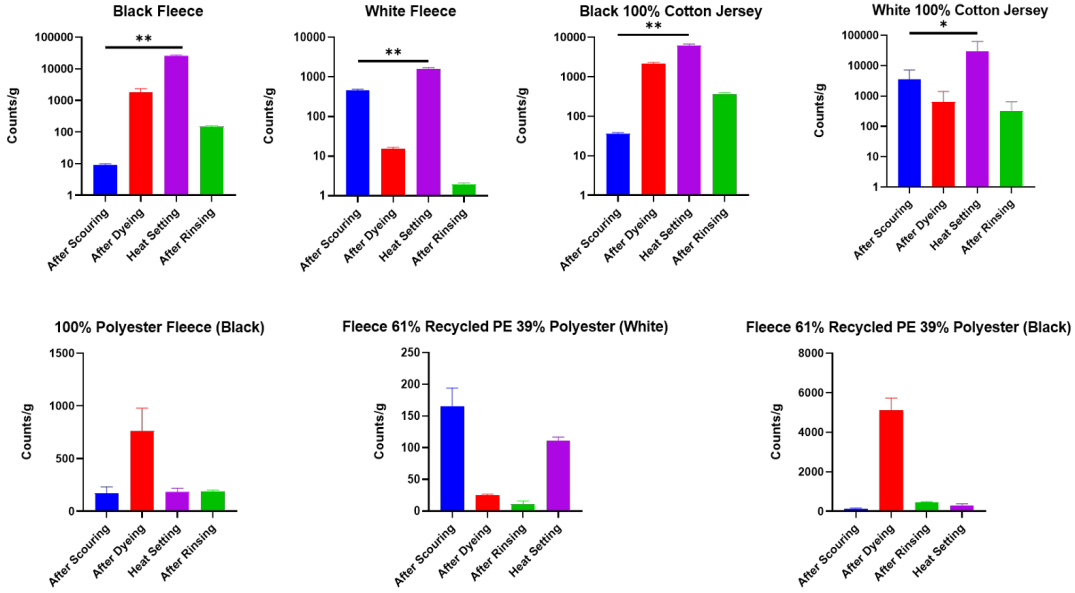

Figure 34. Effect of different treatment process on the number of microfibres released during production. All the concentration was normalised by the mass of the fabric produced. The error bars represent the standard error of the mean, N = 3 for all samples. *p < 0.05, **p < 0.01 (Student's t test).

5.5. Length of microfibres released from wet processes

The length of the microfibres is shown in Figure 35. Appearing to release the smallest sized microfibers were the black Fleece at the "after scouring" step (78 ± 7.09 µm), the white Fleece at the "after scouring" step (110 ± 3.54 µm), and the white Jersey at the "after scouring" step (170 ± 31 µm). Appearing to release the largest size microfibres at the "after scouring" step were: the black Jersey (158 ± 23 µm), the black 100% Polyester Fleece (320 ± 74 µm), and the white Recycled Polyester (61% Recycled PE, 39% Poly Fleece) (255 ± 41 µm). Appearing to release the largest size microfibres at the "heat setting" step were: the Black Fleece (201 ± 19 µm), and the Black Recycled Polyester (61% Recycled PE 39% Poly Fleece) (337 ± 53 µm). Overall, there was little variability between the fibre release profiles for these textiles, with the vast majority of fibres ranging between 15 and 400 µm in length (Figure 36).

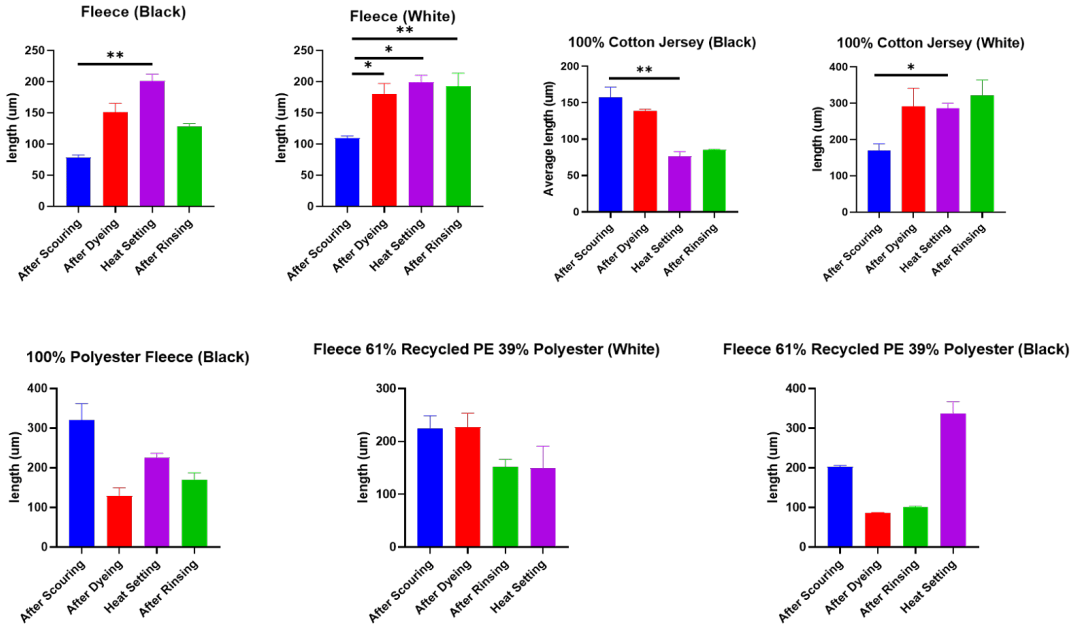

Figure 35. Effect of different treatment processes on the average length of microfibres released during production. The error bars represent the standard error of the mean, N = 3 for all samples. *$p < 0.05$, **$p < 0.01$ (Student's t test).

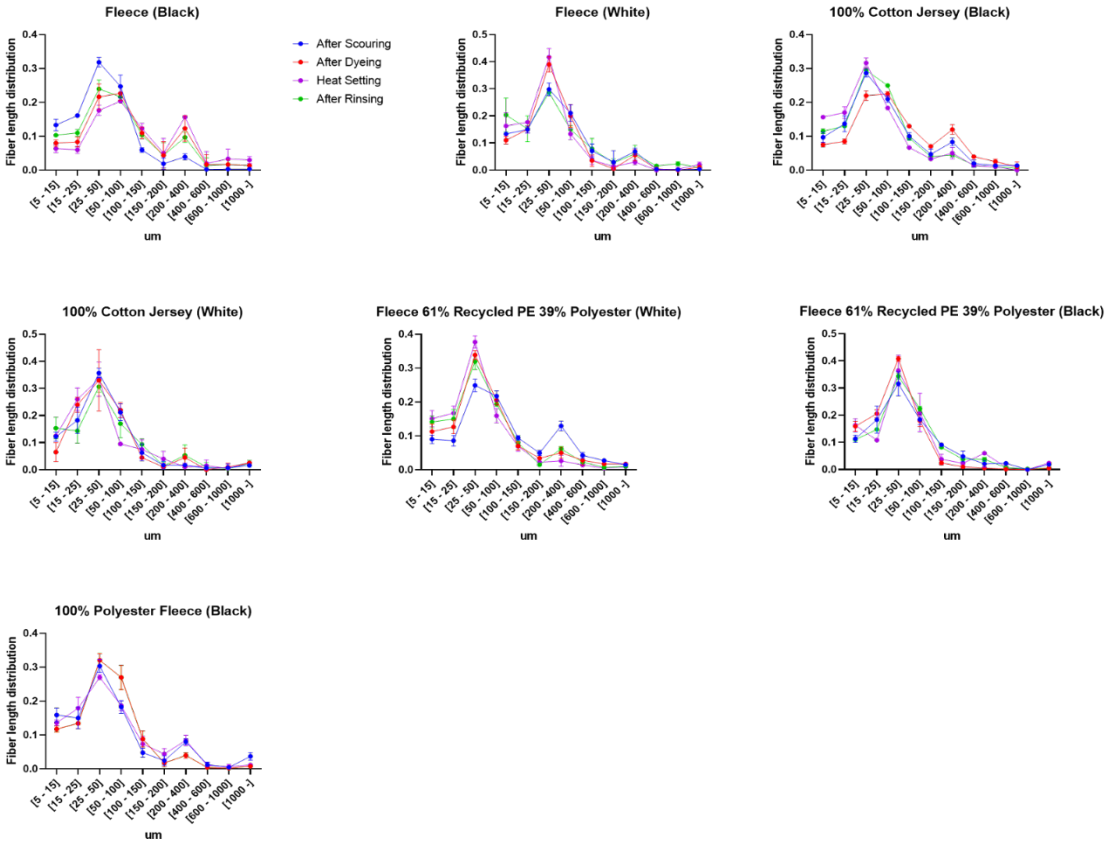

Figure 36. Effect of different treatment processes on the length profiles of microfibres released during production. The error bars represent the standard error of the mean, N = 3 for all samples.

In addition, we conducted the step of separating polyester and cotton fibres from blended fabrics using sulphuric acid. The fibre number from the polyester-cotton blend fabric was significantly decreased after sulphuric acid treatment (Figure 37). The mean length of

microfibres from the “after dyeing”, “after rinsing”, and “heat setting” steps of the blended fabrics also showed an obviously decreasing trend. The remaining mass of fibres were then calculated and >80% of the microfibres released in blended fabrics were found to be cotton (Figure 38).

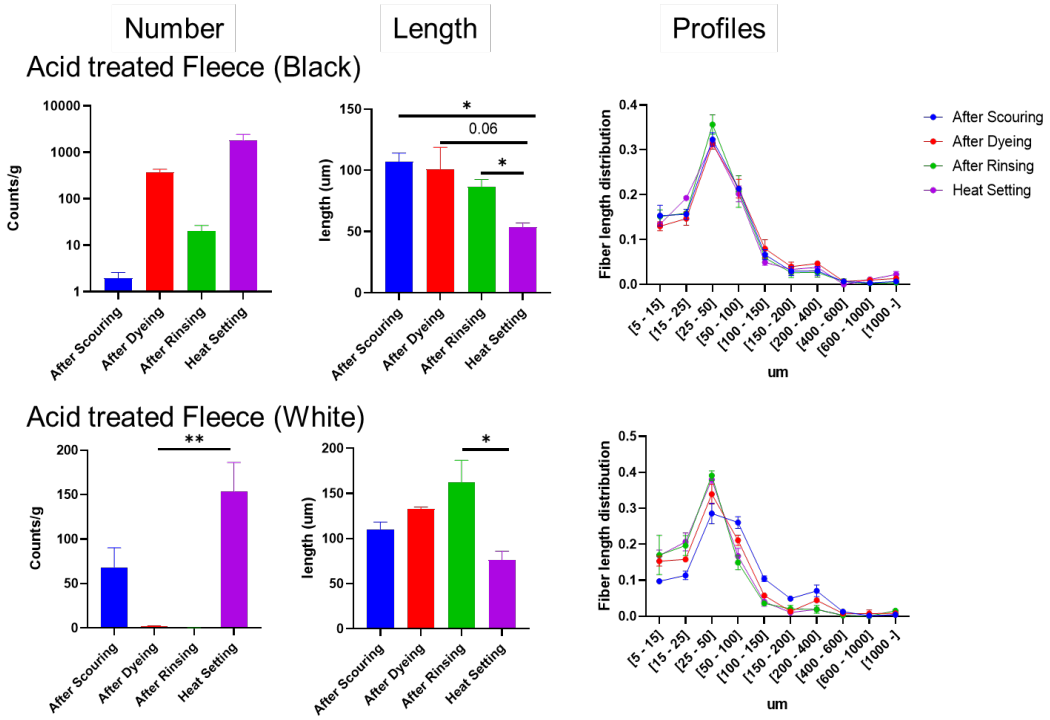

Figure 37. The number, length, and length profiles of released microfibres from wet samples after sulphuric acid treatment.

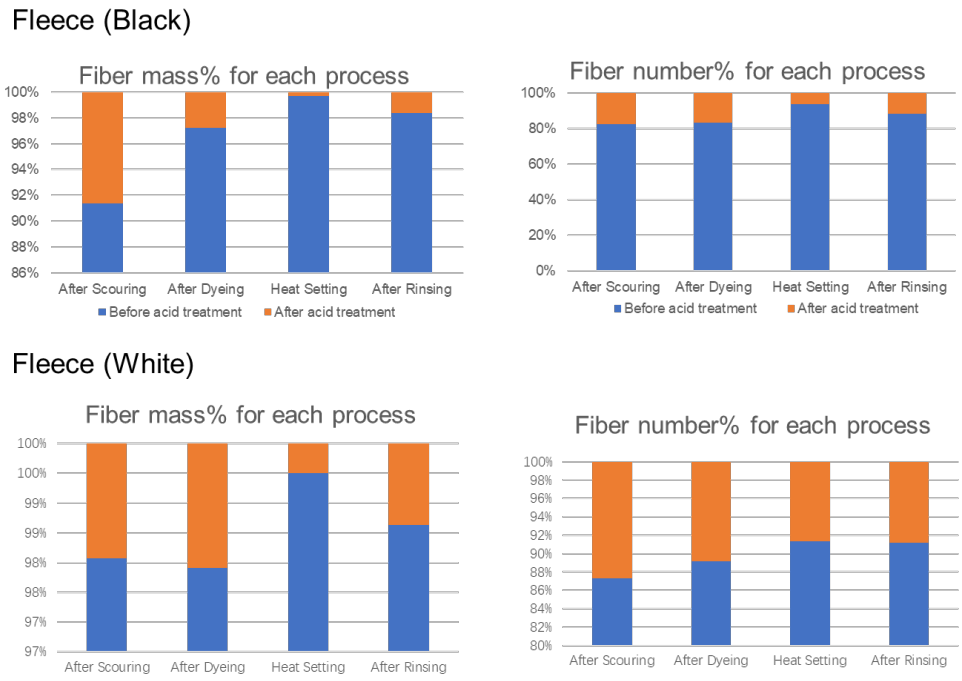

Figure 38. The weight percentage of cotton and polymer in the microfibre released blended fabrics during the production.

5.6. Microfibres released from greige and finished fabric.

We further provided quantitative data regarding the mass, size, and number of the microfibres released from dry samples using simulated home washing under controlled laboratory conditions. The mass (Figure 39) and number (Figure 40) of microfibres released from finished fabric were higher than that of greige fabric. This was attributed to the fact that the greige fabric is the product just after the knitting process and no further harsh physical or chemical treatment processes are applied to it as yet. Thus, it is expected that not much physical or chemical damage has been done to the greige. Meanwhile, there was also little change in the size of the microfibre profiles released from these textiles (Figure 41 and 42). In addition, after the treatment of blended fabrics by sulphuric acid it was found that the fibre number from blended fabric showed significant decrease (Figure 43). The remaining mass of fibre was also then calculated, and the result showed >90% of the microfibres released blended fabrics were cotton (Figure 44).

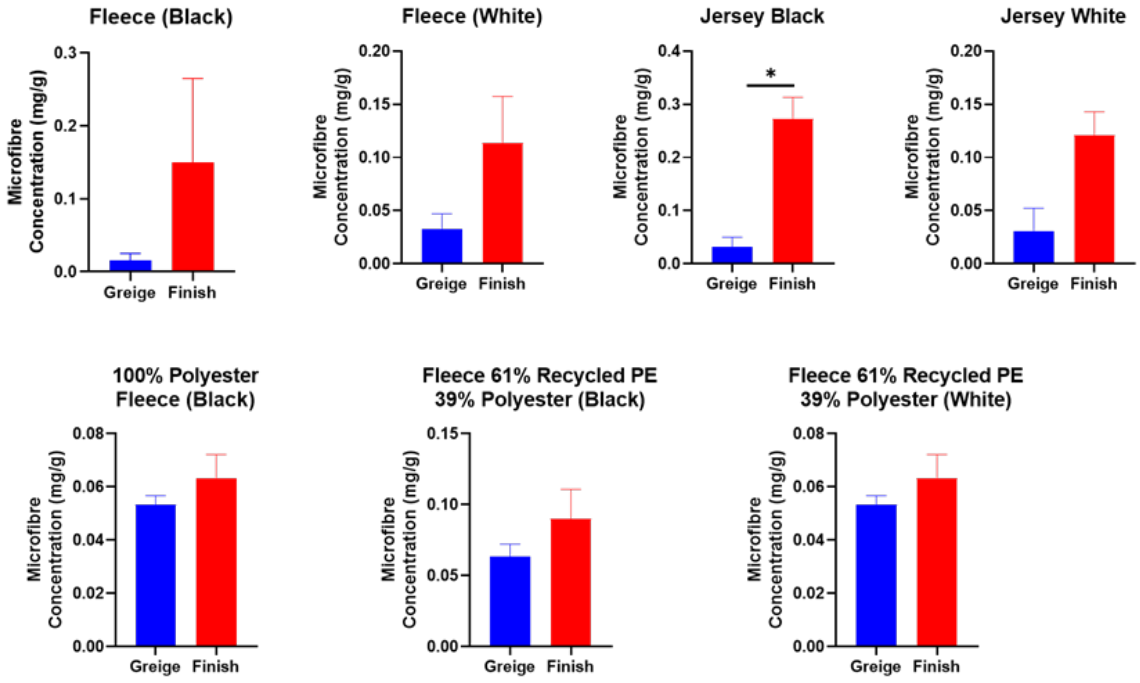

Figure 39. The mass of released microfibres from greige and finished fabric.

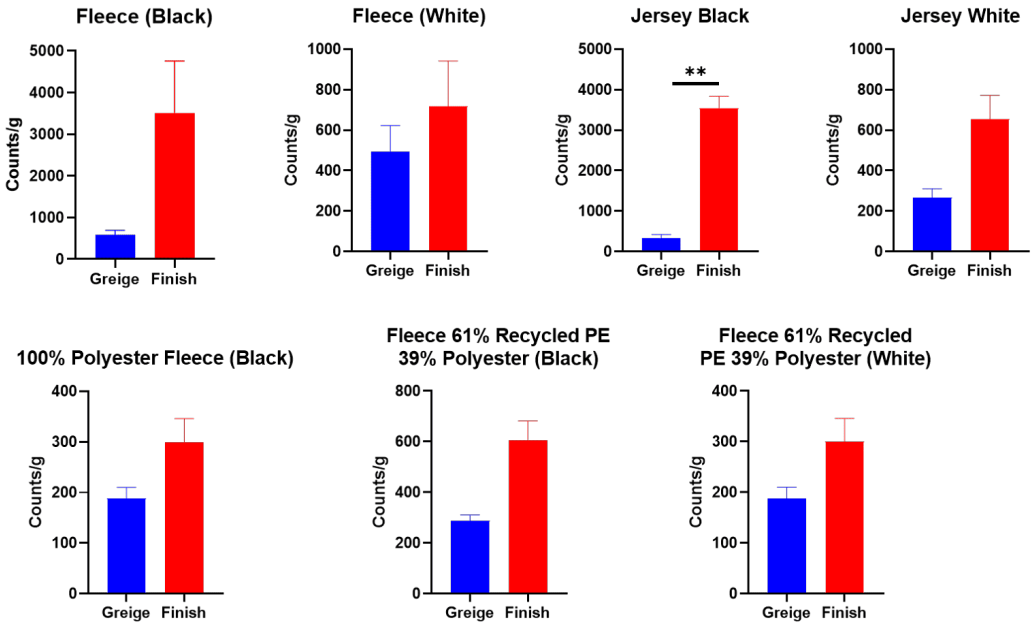

Figure 40. The number of released microfibres from greige and finished fabric.

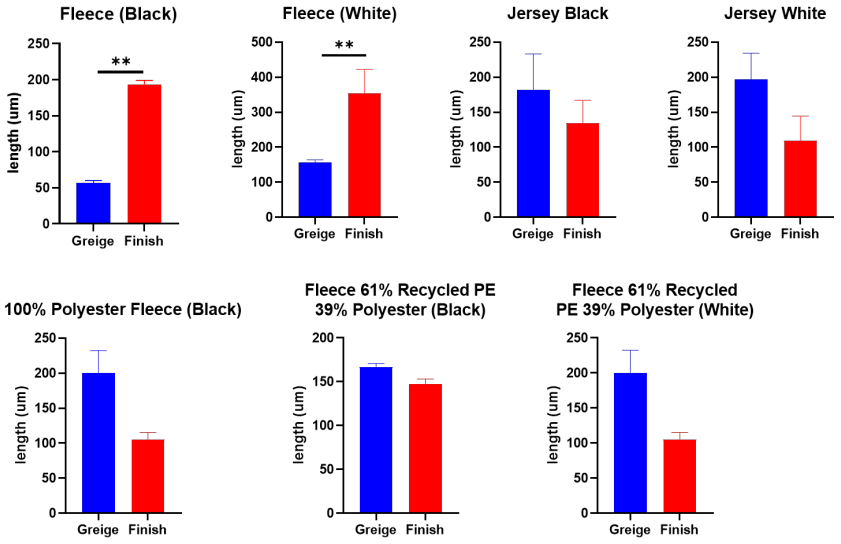

Figure 41. The length of released microfibres from greige and finished fabric.

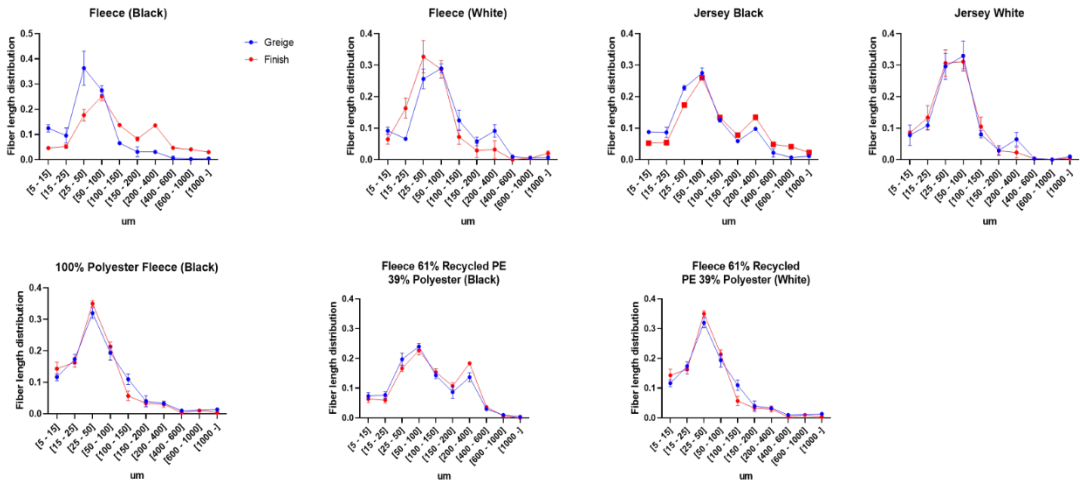

Figure 42. The length profiles of released microfibres from greige and finished fabric.

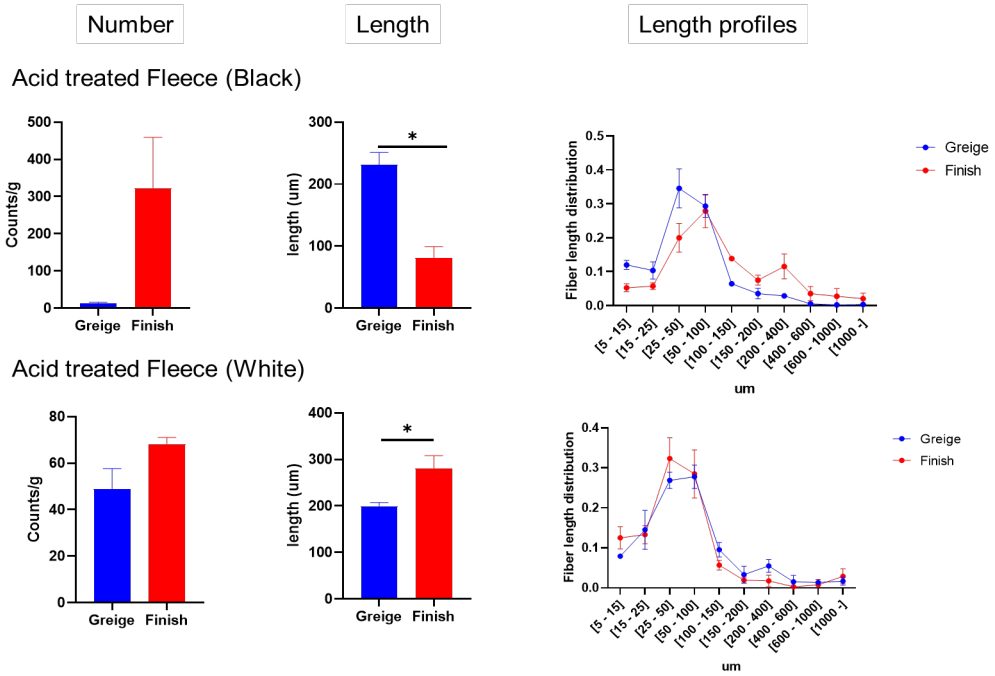

Figure 43. The number, length, length profiles of released microfibres from dry samples after sulphuric acid treatment.

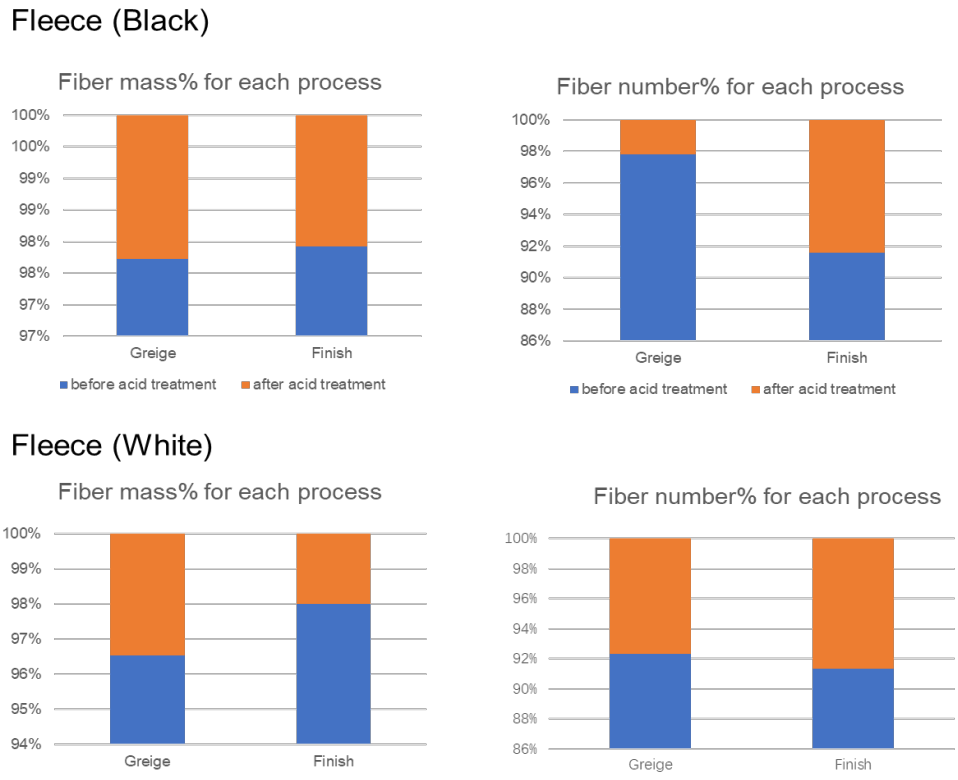

Figure 44. Chemical Separation revealed the amount of cotton microfibre released blended fabrics.

5.7. Comparison of the number and mass concentration of released microfibres from wet and dry samples.

The present study showed that the mass and number of microfibres (per g of textile) released

from black Fleece during wet processes was approximately 2 and 1 order of magnitude higher than that from the finished samples (Figure 45 and 46), respectively. Besides the white Recycled Polyester (61% recycled PE 39% Poly Fleece) fabric, the mass and number of microfibres (per g of textile) released from the other textiles during the wet processes was about 1 order of magnitude higher than that from the finished samples. Therefore, the microfibres released from the wet process steps cannot be ignored. Moreover, the risks they pose should be assessed and managed in appropriate ways. Notably, among the selected 7 fabrics, black Fleece released much more microfibres than the others, while Recycled Polyester released the least. These results indicate that different textile materials have different potential impact on the fibres released during textile production. For example, black Fleece may not be an ideal choice while recycled polyester could be recommended to reduce microfibre contamination.

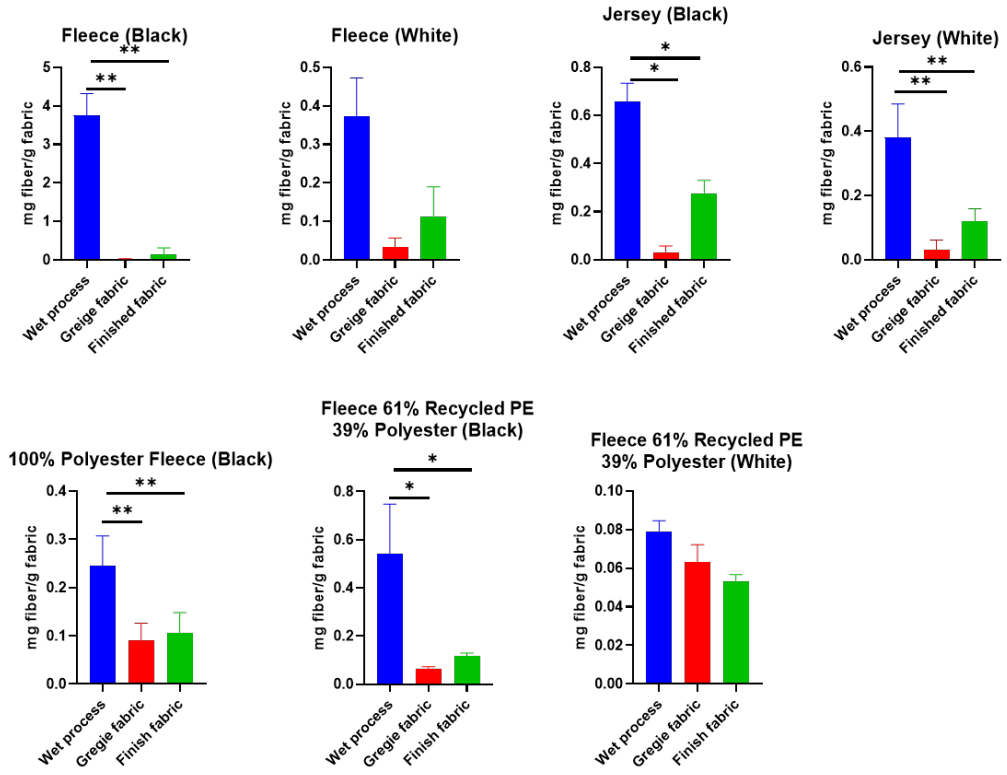

Figure 45. The mass of microfibres released from wet processes, greige and finished fabric.

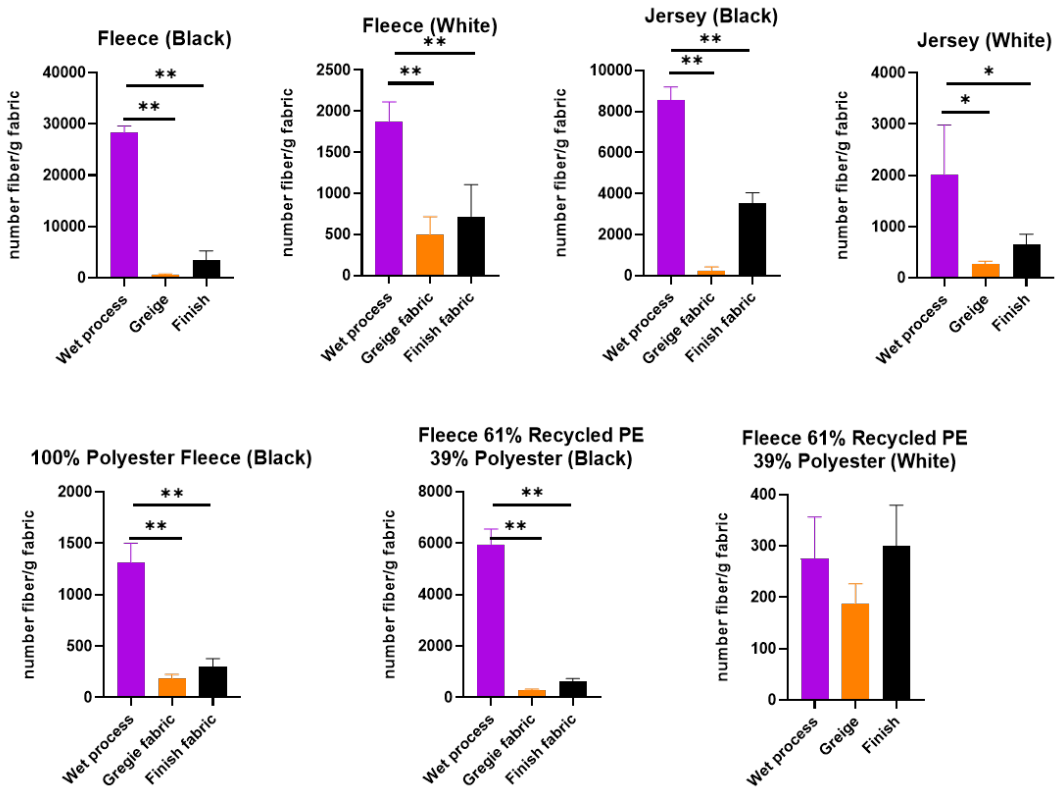

Figure 46. The number of microfibres released from wet processes, greige and finished fabric.

5.8. Comparison of the mass concentration of released microfibres from the dyeing lint samples.

These samples were taken from the lint collection system located inside Ramatex dye tanks. The current study showed that the mass of microfibres (per g of textile) released from lint samples during dyeing processes of black Fleece (0.0488 mg/g) and black Jersey (0.047 mg/g) was approximately 2 times of that from the white Fleece samples (0.01868 mg/g, Figure 47). Besides the white Fleece fabric, the mass of microfibres (per g of textile) released from the lint in the dyeing process of the white Jersey fabric was about 20 times and 10 times lower than that from the black Fleece or Jersey, and white Fleece, respectively. The lint from the black coloured textile samples consisted higher fibre content. It could be due to the longer time in black dyeing process, which caused more damage to the fabric. These results indicate that more microfibres will be generated with longer dyeing processing time and that lint collection has little impact on retaining the microfibres from the dyeing solution before it is passed out as effluent.

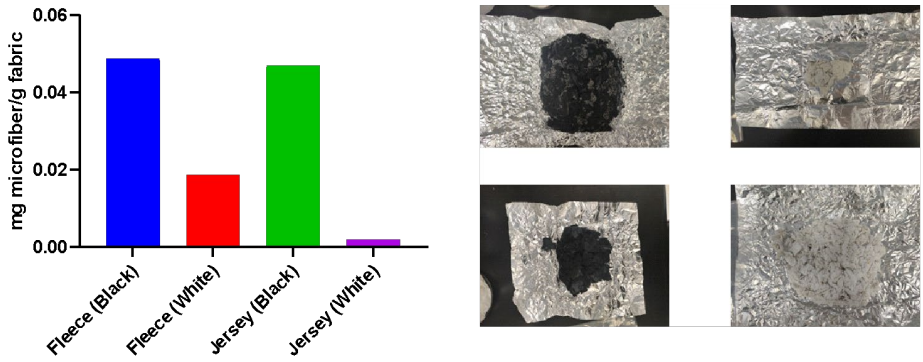

Figure 47. The mass concentration of released microfibres from lint samples after dyeing

5.9. Comparison of the number and mass concentration of released microfibres among different yarn samples.

We compared the microfibre release among different yarn samples. We observed the most microfibre release in mass from 50D/36F yarn type (Figure 48). Meanwhile, it's have found that the black fleece textile made from this yarn type also released the most amount of microfibers. Besides 50D/36F, 100D/96F released higher numbers of microfibres than the rest of the yarn types. In addition, the average length of the shed microfibres is around 100 um for all yarn types except 50D/36F, 100D/96F, and 85D/72F, which is around 150 um. The microfibre length distribution is almost similar among all yarn types. We further investigated the microfibre release of three yarn types that shed the most microfibres, 12's CVC, 14'3 CVC, and 50D/36F (Figure 49). After acid treatment to remove cotton of each yarn type, we still observed higher number of microfibre release from 50D/36F yarn type. However, the fibre length release profiles are similar among the three yarn types. Overall, the results indicate that most yarn types share similar microfibre release profiles besides 50D/36F. The average length of shed microfibre by 50D/36F yarn type is larger than the rest of yarn types, which could be due to the polyester component of the yarn type. The selection of different yarn types for fabric production should be more careful. For example, the application of 50D/36F may not be an ideal choice regarding the microfibre contamination.

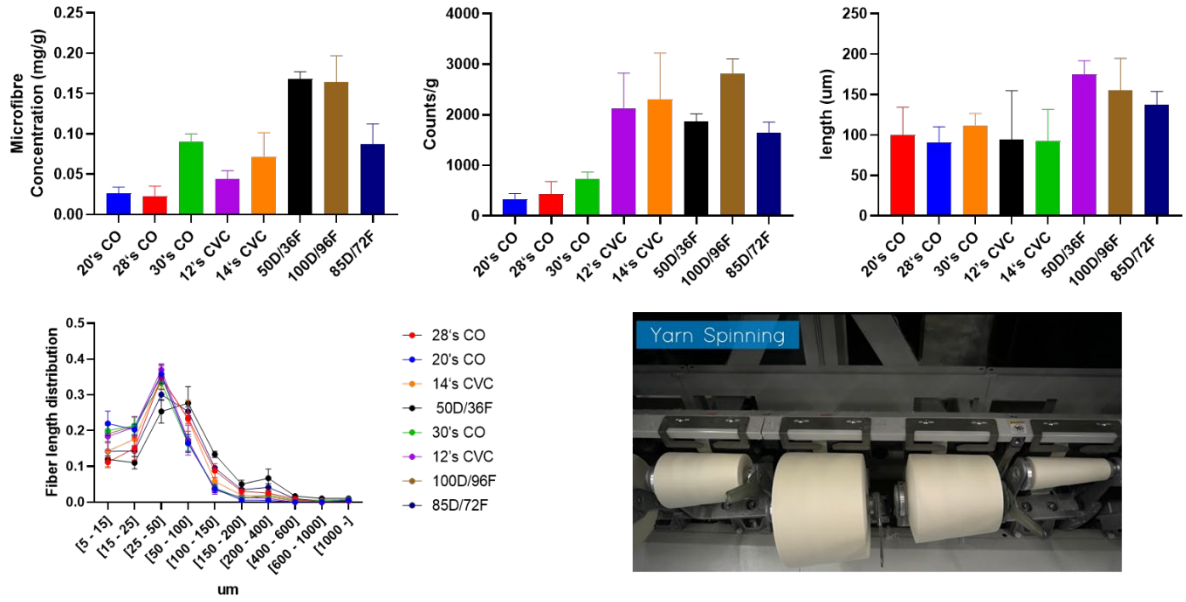

Figure 48. The number, length and mass of microfibre release for different yarn types.

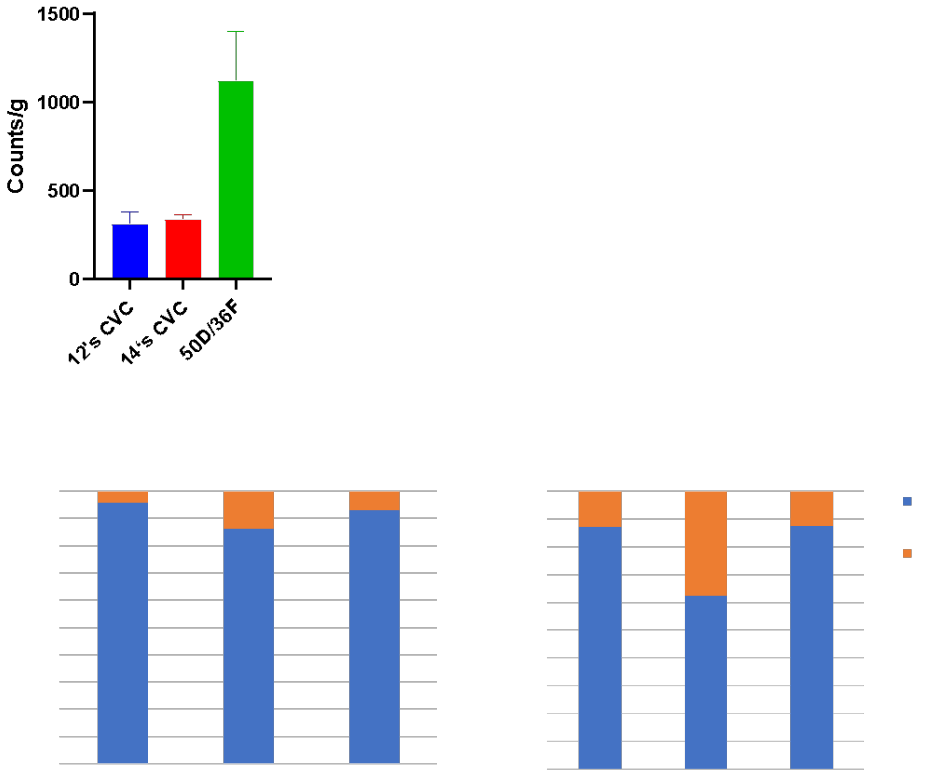

Figure 49. The number and fibre length of the acid treated fleece of three yarn types.

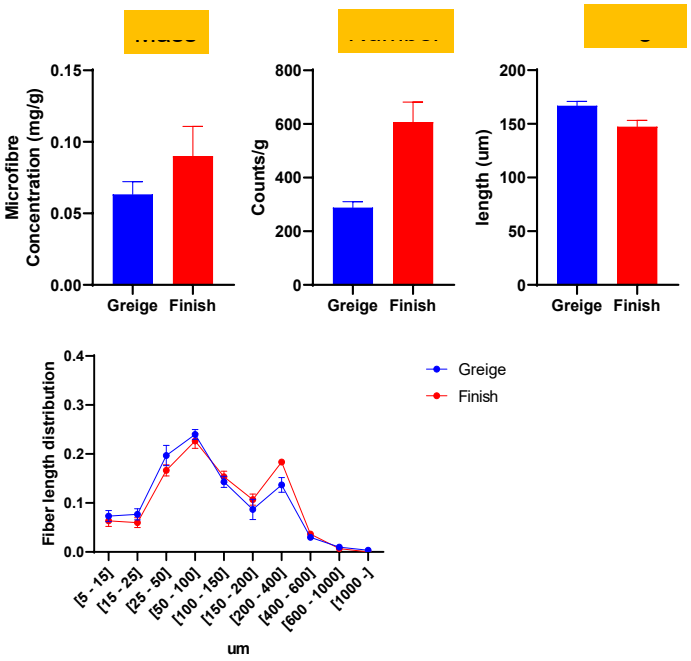

Figure 50. The number, length and mass of microfibre release for 100% PE fleece (black)

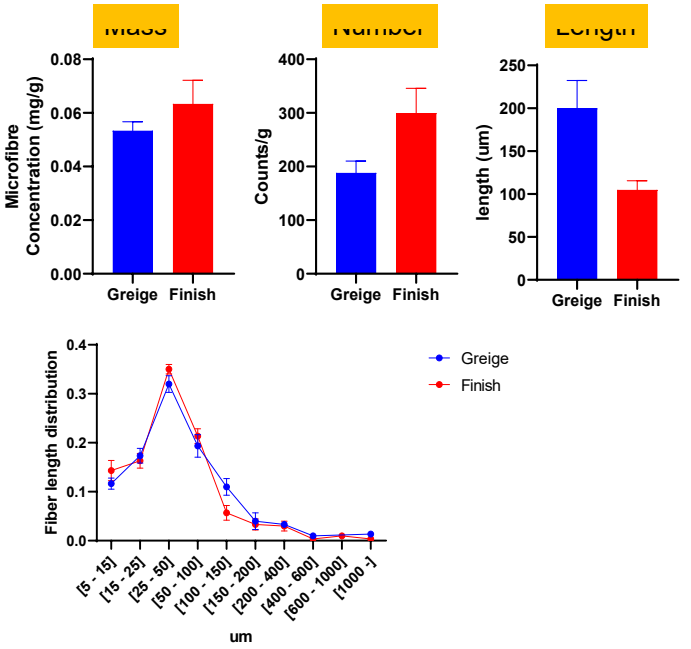

Figure 51. The number, length and mass of microfibre release for 61/39 PE fleece (White–100D/96F, 85D/72F)

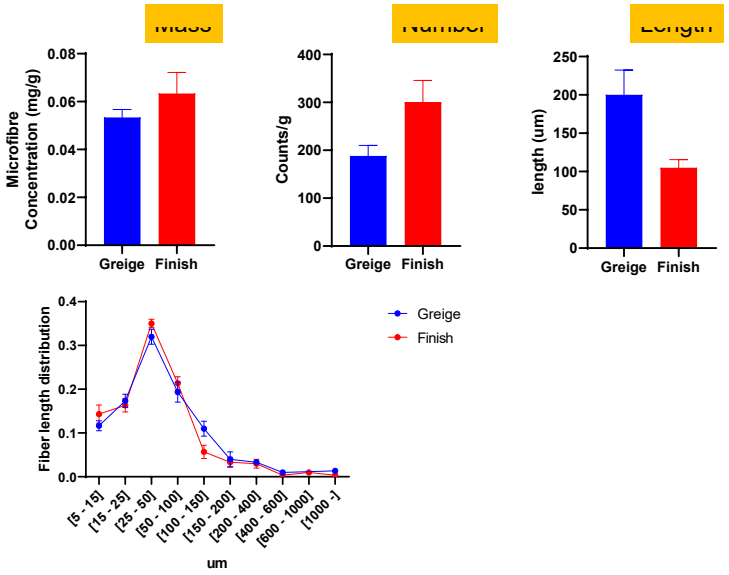

Figure 52. The number, length and mass of microfibre release for 61/39 PE fleece (black–100D/96F, 85D/72F)

5.10. Comparison of the number and mass concentration of released microfibres from fabrics before brushing and after brushing.

The present study showed that the mass and number of microfibres (per g of textile) released from fabric before brushing was approximately 1 to 3 times higher than that from the samples after brushing (Figure 53), respectively. Therefore, the microfibres released from fabrics before brushing cannot be ignored. Notably, among the selected 5 fabrics, 61% polyester recycled, and 39% polyester fleece white released more microfibres than the others, while 100% polyester fleece released the least. In addition, the increment of microfibre shedding of 100% polyester fleece before brushing and after brushing was lower than the rest of the samples, while the increment of CVC 80/20 fleece was higher than the others. Further, the fibre length profile of 61% polyester recycled and 39% polyester fleece before brushing was shifted when compared to that after brushing. Microfibres of a longer length were released more often in the fabrics after brushing. Overall, these results indicate that most microfibres are released in the fabrics before brushing. More interventions should be explored before brushing to reduce microfibre contamination. Also, different materials have different potential impact on the fibres released before and after brushing. The methods for alleviating microfibre shedding could be varied for different materials.

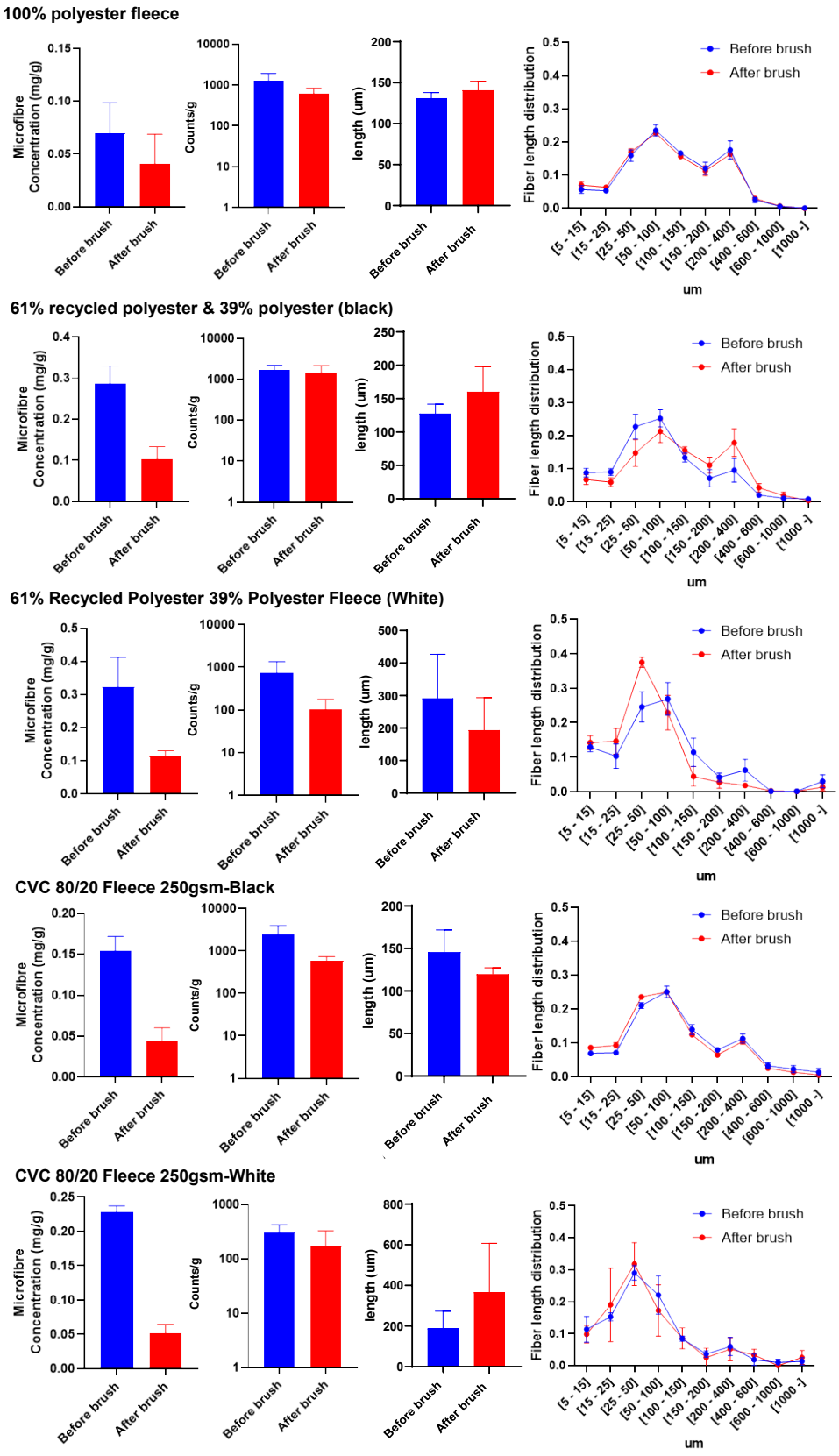

Figure 53. The number, length, and mass concentration of released microfibers from fabrics before brushing and after brushing.

5.11. The assessment of microfiber reattachment on the fabric in dyeing process.

As indicated by our previous study, the microfibre shedding in the heat setting process was more significant than those in other processes. It could be due to the loose fibre reattaching on the textile after the dyeing process. Therefore, it was necessary to conduct an assessment on microfibre reattachment on the fabric during the dyeing process. We selected the black-coloured 80/20 CVC fleece samples for this assessment because they released the largest amount of microfibres in the wet process and the heat setting process (Figures 30 & 45). We extracted the microfiber from reattachment by the sonication in water bath. The control experiment verified that there was no significant impact on the microfiber shedding from sonication. Therefore, sonication can be used to extract the microfiber on the fabric surface. The results from the assessment of microfibre reattachment showed that there was a large amount of microfibre shedding by the black 80/20 CVC fleece samples, with the maximum amount of microfibre release being 1.39 mg per gram of textile (Figure 54).

Further to this, we observed a substantial amount of microfibre reattachment on the fabric. The largest amount of microfibre reattachment on fabric samples was 0.86 mg per gram of textile. Our result indicated that microfibre released during the dyeing process reattached to the fabric substantially and could be released to the environment during the post-dyeing processes. The large amount of microfibre shedding in the heat setting process was partly from the microfibre reattachment. The result also indicated that the dyeing process could be the process where the largest amount of microfibre shedding is generated. An optimisation of the dyeing process is recommended to alleviate the microfibre shedding.

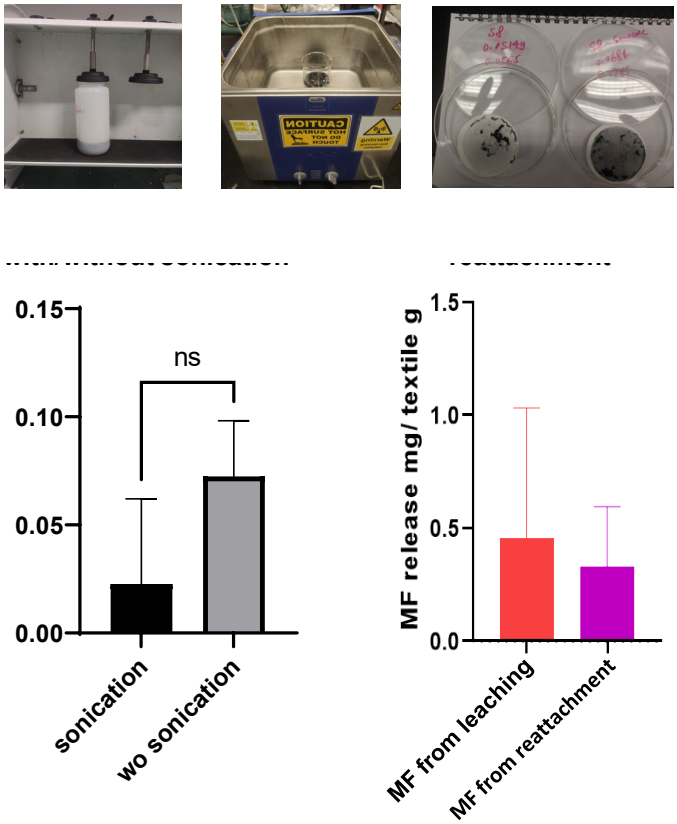

Figure 54. The microfibre released in washing water and microfibre reattached on the black coloured 80/20 CVC fleece samples.

6. Investigation phrase of microfibre shedding

6.1 The impact of temperature on microfibre shedding in the dyeing process

The microfibre shedding in the dyeing process is substantial. The previous study showed that the dyeing process might bring more damage to fabric than other processes. The result also indicated that improvement of the dyeing process can alleviate microfibre shedding. The dyeing process is the interaction between a dye and a fabric, as well as the movement of dye into the internal part of the fabric. The processing temperature and duration are the key controlling factors. For the 100% recycled polyester, the microfiber shedding at 130°C was lower than those at 60°C. For the 100% regular polyester, microfibre shedding increased slightly along with the increment of processing temperature (Figure 55). However, there is no significant differences in the numbers of microfiber shedding between low temperature and high temperature. However, for 100% Cotton, the temperature promoted the release of microfibres. There was a significant increment in the mass of microfibres released when textiles were heated at a higher temperature. The microfibre shedding of 100% cotton jersey increases when temperature increases and reaches the highest number at 130 °C. The trend of 100% cotton also was observed in the 80/20 Cotton/PE and the microfiber increased along with the increment of the temperature. Moreover, the results of microfiber shedding in the 2-step dyeing consecutive progression indicates that dual dyeing temperature and time did not seem to have significant impact on the total emission of fibre shredding. The result indicates that a lower temperature is recommended for microfibre pollution control.

The results indicated that 100% Cotton and 80/20 Cotton/PE suffer more damage under high temperatures. Therefore, the dyeing process for 100% cotton and 80/20 Cotton/PE is recommended to proceed under a lower temperature. Based on current testing the dyeing temperature for 100% Cotton is recommended to be as low as 60°C. For 80/20 Cotton/PE, which is dyed in the 2-step consecutive progression, the recommendation is to maintain 60°C throughout. We will be continuing our investigation on the cotton textiles at temperatures lower than 60°C, which is the factory setting used by Ramatex.

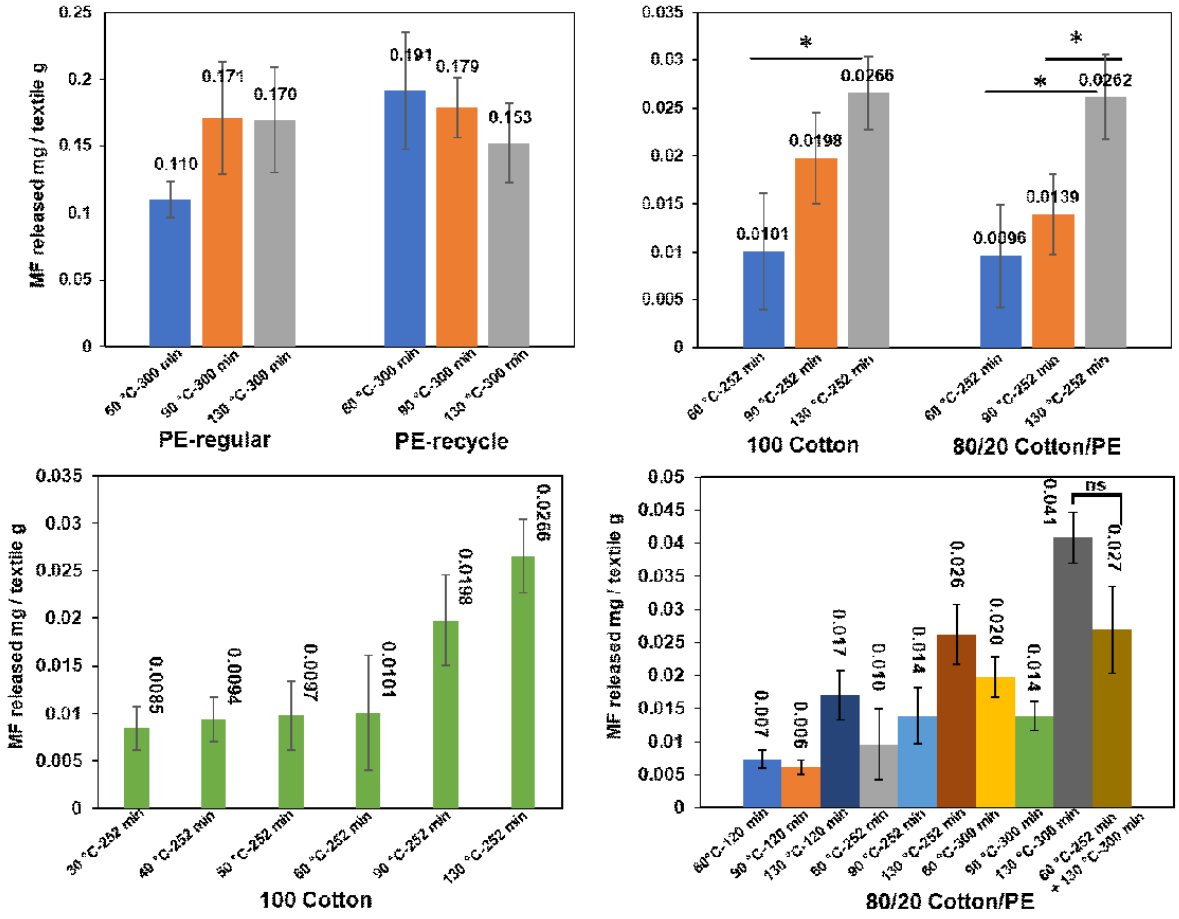

Figure 55. The microfibre shedding for PE-regular (60°C,90°C,130°C for 300 min), PE-recycled (60°C,90°C,130°C for 300 min), Cotton (60°C,90°C,130°C for 252 min), and 80/20 cotton-polyester (60°C,90°C,130°C for 300 min).

6.2 The impact of processing duration of the dyeing process on microfibre shedding

Different processing times were applied to the four types of samples. In addition, 80/20 cotton/PE requires a 2-step dyeing process which is the total of the length of time taken to dye polyester, followed by cotton. The results indicated that dual dyeing temperature and time does not seem to have significant impact on the total emission of fibre shredding. The results showed that microfibre shedding for all tested fabrics increased as the processing time increased. Fabrics suffered more damage as they were processed for a longer time. Various materials require different amounts of time to be processed at the present industrial level for dyeing. Within the testing range of dyeing temperature and duration, the dyeing settings for all 4 tested fabrics are recommended to be: PE regular (90 °C-120 min), PE recycle (60 °C-120 min), 80/20 Cotton/PE (90 °C-120 min), Cotton (90 °C-120 min), as showed in Figure 56.

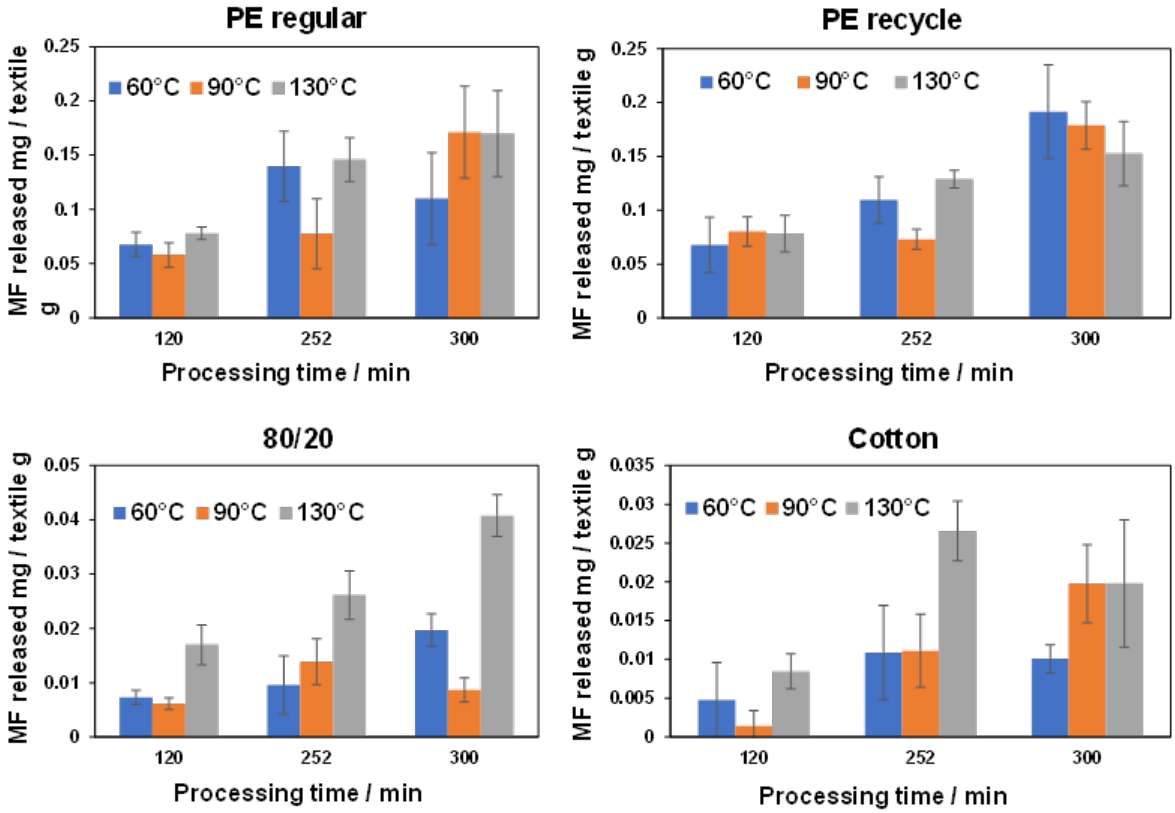

Figure 56. The microfibre shedding for PE-regular, PE-recycle, cotton, and 80/20 fleece with different processing time (120, 252, 300 min).

6.3. Investigating the impact of yarn types on the microfibre shedding as a complement to the in-scope areas

The continuing investigation focus on yarn type impact on microfibre shedding by the modified standard washing method and the simulation test of dying process. First of all, the 100% cotton with open yarn construction and ring yarn construction shed the least of microfibre, as shown in Figure 57 of the standard washing testing. For cotton and cotton blended material, the cotton-bamboo with vortex yarn construction shed less microfibre than 100% cotton with vortex yarn construction. In the simulated test of dyeing process, the 100% cotton with open yarn construction shed the least microfibre than all materials.

By standard washing method, polyester recycle and polyester regular shed more microfibre than 100% cotton and cotton-bamboo blended material. The recycled polyester and regular polyester both shed the similar number of microfibers. In the simulated test of dyeing process, the recycled and regular polyester shed the most of microfiber than the other cotton and cotton blended material. These results showed that the 100% cotton with open yarn construction and ring yarn construction shed the least amount of microfiber than other cotton fabrics by the standard washing method. Meanwhile, in the simulated test of the dyeing process, the recycled and regular polyester shed much more microfibre than cotton-based fabrics and PE-blended fabrics. However, there is not significant differences regarding the temperature impact

on both polyester materials. Overall, the results from standard washing method and the simulated dyeing process both indicate that 100% cotton with open and ring yarn construction is recommended for application regarding the control of microfibre pollution.

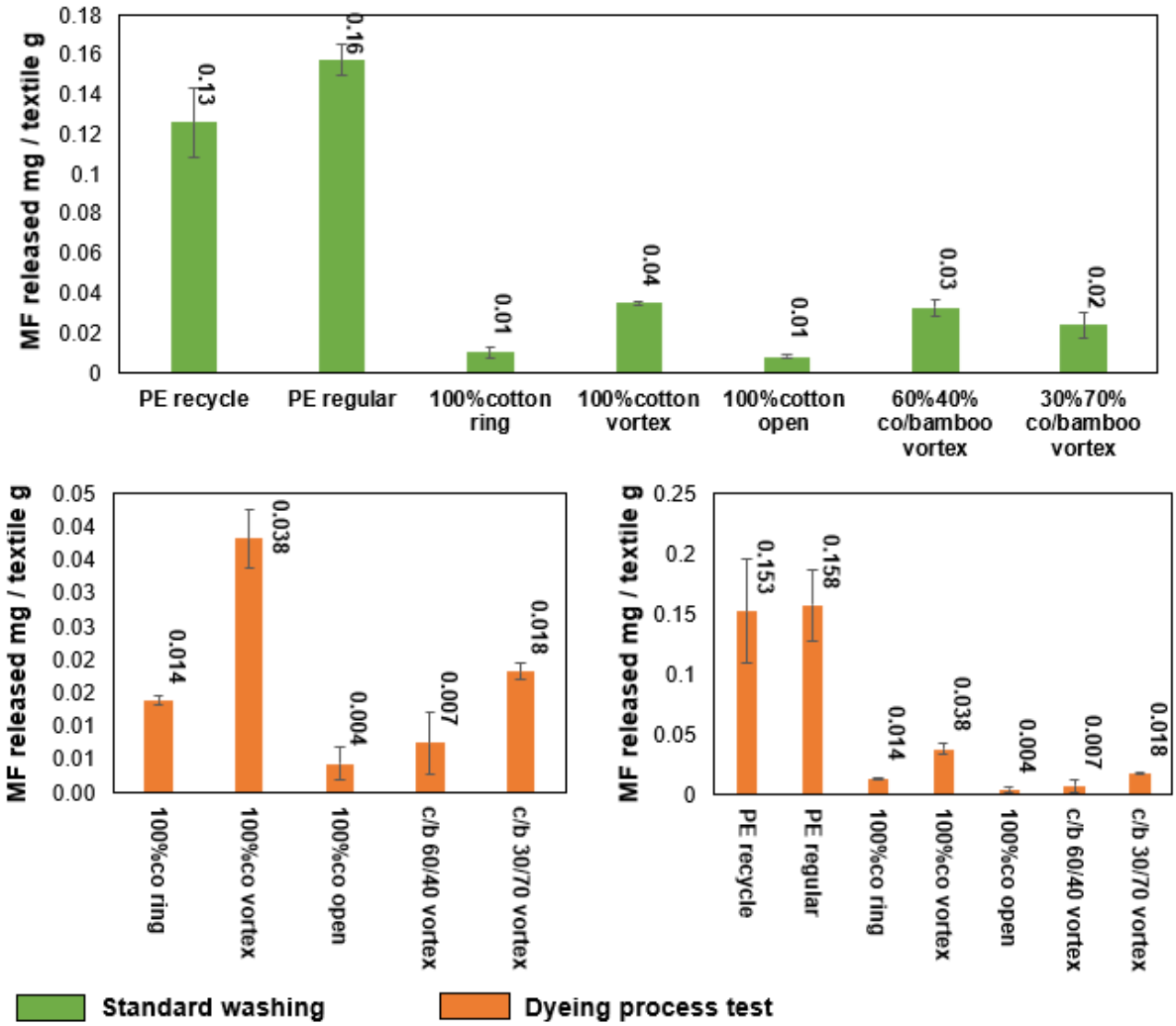

Figure 57. The microfiber shedding regarding different yearn types.

6.4. Environmental impact of implementing the optimised dyeing settings.

With an estimated 20% of all water pollution coming from textile treatments including coloration procedures, the textile industry is one of the leading environmental polluters. Conventional textile dyeing processes are responsible for 17% to 20% of the world's water pollution[11-12]. One tonne of fabric dyed using traditional methods might pollute up to 200 tonnes of water. Salt, dye, detergents, peroxides, and heavy metals are just a few of chemicals of high concentrations found in the highly coloured effluent generated during the textile manufacturing process[13-15]. Implementing the optimisation of the dyeing settings can reduce the water consumption and pollution by shortening the processing duration and increasing the bath ratio in dyeing.

Apart from the pollution from dyes, most of the microfibres found in the ocean are released from textile industries. Microfibres not only endanger aquatic organisms and animals, but also

further pose a great threat to human health. Microfibres are as small in size as plankton but of greater problem because of their higher liberations. Ingestions of microfibre by aquatic species such as zooplankton, coral, fish, crabs, mussels, whales, and many others are reported, which take planktons as a chief food source[16-18]. The ingestion of microfibre leads to a number of harmful effects to organisms, including decreased feeding ability, abnormalities in reproduction, decreased nutrition, and poor health resulting in liver toxicity in fish. Further, microfibres have been shown to absorb, carry, and retain pollutants[19-20]. Pollutants attached to the microfibres are absorbed by the organisms consuming these microfibers and ultimately are absorbed by humans. The bioaccumulation of pollutants along the food chain can cause stomach damage, lung inflammation and infection, and endocrine disruption[21-22].

Considering these harmful effects brought by microfibres, the biodegradability of fabrics should be considered. Natural fabrics such as cotton can be biodegradable in the environment and thus create less harm. However, synthetic fabrics such as polyester have shown low biodegradability because polyester is less susceptible to fragmentation of the structure by hydrolysis and bacterial adherence in polyester fabrics was low compared to cotton[23-24]. Although numerous methods have been proposed for management of microfibres of low biodegradability, it is critical to limit the generation and release of microfibres from the source[25]. As a primary source of microfibres, the dyeing process in textile industries shed a larger number of microfibres than other processes[26]. To optimise the dyeing settings is to reduce microfibre pollution by limiting microfibre shedding during the process.

In addition to water pollution and consumption, the energy needed to heat water and dry textiles is another concern pertaining to the impact of the dyeing process. The type of machine, the actions, and the dyeing conditions all affect the amount of energy needed to produce textiles[27-28]. This energy is primarily produced by burning fossil fuels, which adds to the carbon footprint. According to the Intergovernmental Panel on Climate Change of the United Nations, the textile industry is responsible for 10% of worldwide greenhouse gas (GHG) emissions[29]. Air emissions from heating processes, such as those from boilers that emit acetic acid, formaldehyde, nitrogen, and sulfur oxides, were recognized as the textile industry's second-biggest pollution issue. Therefore, it is important to carefully consider factors that directly affect the process, such as temperature, pH, bath ratio, and equipment. Implementing the optimal dyeing temperature and duration is the key step to reduce energy consumption and air emissions caused by textile dyeing.

7. Conclusions

The present study demonstrated that large number of microfibres can be shed from 7 types of synthetic fabrics during production, among which the “heat setting” process released the highest number of microfibres, and the dyeing process in black-coloured textiles also promoted the release of microfibres from tested fabrics. In contrast, the scouring process of white fabric is also seen to be able to produce more fragments due to the violent whitening process. Different textile materials also have different potential to leach microfibres during production. Moreover, fabrics tend to generate more microfibres during production than that of domestic laundry processes. To reduce or eliminate microfibres from effluent before entering the aquatic environment, the contribution of microfibres from the wet processes should be given attention and the selection of fabric type also should be considered.

Moreover, the investigation study demonstrated that many microfibres can be reattached to the fabrics during the dyeing process, leading to the substantial microfibre shedding in the post-dyeing processes, including the “heat setting” step. Optimisation of the dyeing process can be a key role to limit the microfibre shedding during the production processes. The damages to both synthetic and natural fabrics during the dyeing process can be alleviated by using a shorter processing duration. Specifically, fabrics containing natural fabrics tend to shed more microfibres at a higher temperature, compared to its shedding at a low temperature. However, recycled synthetic fabrics tend to be less affected by the temperature regarding microfibre shedding. To reduce the microfibres releases, the optimisation of the dyeing settings should be given more attention.

8. Recommendations

We hence propose a few recommendations for future studies. For one, we need to potentially explore how yarn, colour and finished fabric type impact microfibre shedding, by collecting more dry samples for additional yarn types and other finished fabric types. Second, we can further investigate how to optimise production parameters to observe whether microfibre release can be minimised from other types of less commonly used dyeing processes e.g. fibre dyeing or cold-pad dyeing, compared to traditional dyeing. In addition, we need to estimate how much fibres can be released from the textile industry at a global scale and estimate the overall impact to necessitate the wastewater treatment of production wastewater.

Here, the present study demonstrated that a large number of microfibres can be shed from these 11 types of fabrics during production, among which the “heat setting” process released the highest number of microfibres, and the dyeing process in black-coloured textiles also promoted the release of microfibres. In contrast, the scouring process of white fabric is also seen to be able to produce more fragments due to the violent whitening process. Different textile materials also have different potential to leach microfibres during production. Moreover,

fabrics tend to generate more microfibres during production than that of domestic laundry processes. To reduce or eliminate microfibres from effluent before entering the aquatic environment, the contribution of microfibres from the wet processes should be given attention and the selection of fabric type also considered.

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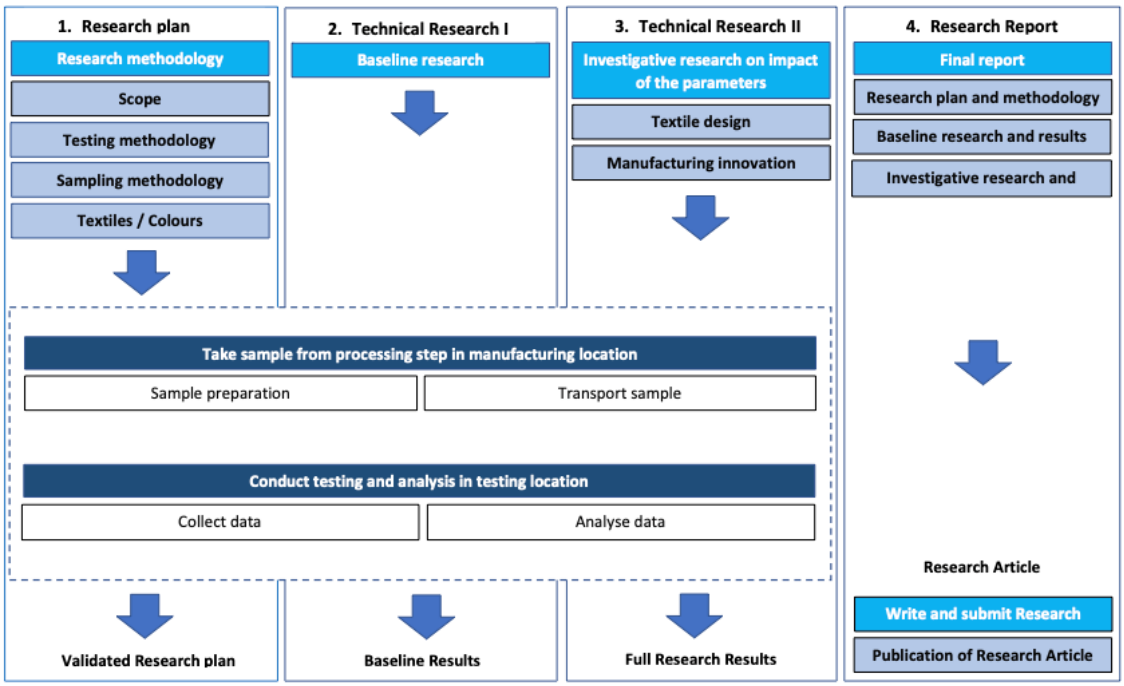
Annex

A. Research Workflow



B. Research plan progress reporting

The indicators for each of the methodologies are listed in the figure below titled Key indicators Research plan reporting:

Testing methodology	Sampling methodology
Types of samples	Sample receipt & handling
Sampling points/locations	Test methods
Sample collection	Quality systems
Sample containers	Reporting requirements
Sample shipments	

Key indicators Research plan reporting

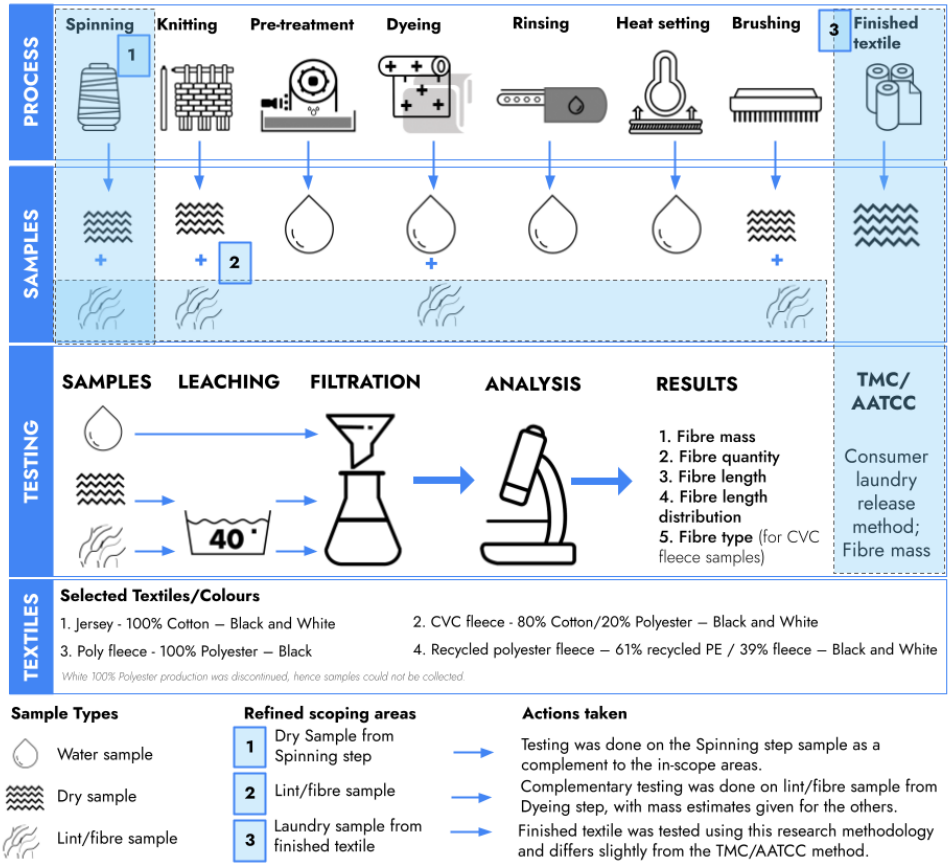
C. Third party laboratory quotations

Lab name	Testmethods			Info
	TMC	AATCC	Other	
BV	260 USD / sample	x		Two locations will be available to conduct testing: China and Vietnam. Only TMC method is released, hence no costing information on the AATCC method at this point.
TUV SUD	x			Sits on the TMC guidelines task team; testing some samples. Permission needed to share any analysis details as the analytical method development is funded by one of the brands and TUV SUD is under NDA with brand + TMC.
SGS	943 USD / sample	385 USD / sample	x	TMC and AATCC can be testing in HK/TW/Shanghai; Other testing options: Full-scale testing: Surface area measurement on final product (98 USD/sample; HK only) // Full-scale testing: Microfibre release (565 USD; HK only). In addition they charge for: Technical Consultation per project 300 USD (if needed)
Intertek	1 wash 110 USD / 3 wash 330 USD	x	x	HK and TW have been testing: TMC, AATCC and client testing methodology. Indicated "Both test methods have their own limitation, but AATCC is recommended." This may confirm why most labs are sharing additional testing options to requested AATCC/TMC.
Note the above cost are not including shipment cost!				

D. Lint/fibre sample fibre mass calculatio

Lint/fibre samples calculation				
Sampling points	**How much lint/fibre per batch/process? (kg)**	**How many kg of product produced?**	**Day / batch**	**Calculation of lint/fibre release per kg (= mass)**
Spinning	10	20000	day	0.00050
Knitting	10	120000	day	0.00008
Dyeing	TBC*	1500	batch	lint/fibre mass/kg of product produced
Brushing	60	4500	day	0.01333
** To take out after dyeing process of identified textiles - dry - and then weigh and ship to NEWRI.*				

E. Research Plan Visual



F. Template Sample label

Sample label

ID (e.g. Ramatex-NTU-001)	
'Wet' / 'Dry'process	
Volume (ml) / Length (m)	
Fibre type	
Fibre color	
Date	
By	

G. Sampling points

Sampling points	**Types of samples**						
	1. Water sample		***2. Assisted water sample***			***3. Lint/fibre sample***	***4. Laundry sample***
Processing steps	Incoming	Discharge	Cone*	Greige	Finished sample		
Spinning			X			X	
Knitting				X		X	
Pre-treatment**	X	X					
Dyeing - chemistry	X	X					
Dyeing - rinsing	X	X				X	
Heat Setting	X	X					
Brushing					X	X	
Finished textile					X***		X
Control	X						

Notes:

**Cones: depending on number of yarns per textile textile type*

***Pre-treatment: initially from CVC and Cotton Textiles only, subsequently also collected from Polyester textiles.*

****Non-fleece samples (i.e. Jersey) also need a finished textile to be tested.*

H. Flowchart: Testing methodology

Leaching

*A dry sample will be taken to leach the microfibre content in liquid; an **assisted water sample***

- 40 rpm
- 40 deg C
- 45 Min
- 50 stainless steel balls

Filtration

*Both leached sample and discharge water sample will be filtered**: fibre sample***

- Glass filter -> **fibre mass** of sample

After chemical separation for cotton rich only!

- *Cellulose & glass filter -> polyester fibre mass*

Chemical separation

*To dissolve cellulosic fibres the filtered water will be treated with **H_2SO_4**, for cotton rich only!*

After chemical separation repeat filtration

Analysis & Results

*Through different **analytical equipment** the different fibre samples will be analysed*

- Keyence microscope: **fibre quantity** and **fibre size distribution**
- FTIR: **fibre type** i.e PE or PET
- Analytical balance: **fibre mass**
- Empirical formula: **fibre mass (blends)**

I. Template Test results

TEST RESULTS TEMPLATE	1. Water sample							
	Incoming				*Discharge*			
PROCESSING STEP	*A. fibre quantity*	*B. fibre size distribution*	*C. fibre type*	*D. fibre mass*	*A. fibre quantity*	*B. fibre size distribution*	*C. fibre type*	*D. fibre mass*
Spinning								
Knitting								
Pre-treatment*	X	X	X	X	X	X	X	X
Dyeing - chemistry	X	X	X	X	X	X	X	X
Dyeing - rinsing	X	X	X	X	X	X	X	X
Heat Setting	X	X	X	X	X	X	X	X
Brushing								
Finished textile								

Notes(s):

**Pre-treatment: initially from CVC and Cotton Textiles only, subsequently also collected from Polyester textiles.*

TEST RESULTS TEMPLATE	2. Assisted water sample			
PROCESSING STEP	*A. fibre quantity*	*B. fibre size distribution*	*C. fibre type*	*D. fibre mass*
Spinning	X	X	X	X
Knitting	X	X	X	X
Pre-treatment*				
Dyeing - chemistry				
Dyeing - rinsing				
Heat Setting				
Brushing	X	X	X	X
Finished textile	X	X	X	X

Notes(s):

**Pre-treatment: initially from CVC and Cotton Textiles only, subsequently also collected from Polyester textiles.*

TEST RESULTS TEMPLATE	3. Lint/fibre sample			
PROCESSING STEP	*A. fibre quantity*	*B. fibre size distribution*	*C. fibre type*	*D. fibre mass*
Spinning				X
Knitting				X
Pre-treatment*				
Dyeing - chemistry				
Dyeing - rinsing	X	X	X	X
Heat Setting				
Brushing				X
Finished textile				

Notes(s):

**Pre-treatment: initially from CVC and Cotton Textiles only, subsequently also collected from Polyester textiles.*

TEST RESULTS TEMPLATE	4. Laundry sample for testing according to AATCC/TMC method			
PROCESSING STEP	*A. fibre quantity*	*B. fibre size distribution*	*C. fibre type*	*D. fibre mass*
Spinning				
Knitting				
Pre-treatment*				
Dyeing - chemistry				
Dyeing - rinsing				
Heat Setting				
Brushing				
Finished textile				X

Notes(s):

**Pre-treatment: initially from CVC and Cotton Textiles only, subsequently also collected from Polyester textiles.*

References

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J. Short Guide to using the Research Plan

Step 1: Identify in manufacturing facility:

- Processing steps and respective sample types
- Textiles/colours

Step 2: Take samples from processing steps in manufacturing facility:

- Follow sampling collection and sample type formats and relevant quality systems, including refrigerating water samples in the time between collection and pick-up
- Prepare the sample packaging documents

Step 3: Prepare samples for shipment by packaging samples:

- Water samples in plastics containers, in plastic bag– 4 litres each
- Dry samples wrapped aluminium foil packaged in plastic bag – 1 cone per yarn type, 400gm greige, brushed and finished textiles
- Pack each sample with the respective sample label document

Step 4: Transport sample to testing facility:

- Organise export documentation
- Arrange for logistics to test lab
- If needed organise import license

Step 5: Receive samples and prepare for testing:

- Within 24 hours review samples received
- Only accept samples when there is no deviation / unclarity
- Follow preparation format and relevant quality systems, including storing water samples in a cold room at 4 °C.

Step 6: Conduct testing and analysis in testing location:

- Assisted water samples and Lint/fibre samples to be leached into water
- All samples to be filtered
- Filtered fibres to be analysed or calculated for: fibre mass, fibre quantity, fibre length and length distribution, fibre type
- Input data in test results overview

Step 7: Analyse test results:

- Which of the processing steps has the most impact on microfibre release of the tested textiles/colours?
- What conclusions can you draw from the data? Can it be linked to textile design? Or can it be linked to manufacturing processing?