POLITECNICO DI TORINO

Master's Degree in Environmental and Land Engineering

Climate Change

Master's Degree Thesis

Reducing Microplastic Fibre Release from Polyester Fabric through Plasma Pretreatment

Supervisors

Prof. Rossana Bellopede

Dr. Raffaella Mossotti

Dr. Sinem Hazal Akyildiz

Candidate

Baris Alp Sevgi

July, 2025

Table of Contents

List of Figures

List of Tables

Abbreviations

EU	European Union
FTIR	Fourier Transform Infrared Spectroscopy
NaOH	Sodium Hydroxide
SEM	Scanning Electron Microscope
mg/L	Milligrams per Liter

Abstract

The increased use of synthetic textiles, particularly polyester, raised environmental concerns due to the release of microplastic fibres during domestic laundering activities. This study focuses on the effect of surface treatments on the microplastic fibre release behaviour of polyester fabrics after multiple washing cycles.

Five fabric types were tested: untreated (raw) polyester and four pre-treated samples with 0.6% and 1.2% sodium hydroxide (NaOH) and chitosan solutions. Washing trials were conducted in accordance with ISO 4484-1:2023 protocol. Filtration and drying processes are carried out after the first, third, and fifth washes. Quantification is made by assessing the mass and count data of microplastic fibres collected on glass fibre filters. In addition, fibre lengths were examined to assess distribution patterns and morphological modifications.

The results indicate that pre-treatment significantly influences microplastic fibre release. Both NaOH and chitosan treatments, led to reductions in total fibre count and mass loss compared to the raw polyester sample. Surface modifications were observed using FTIR and SEM analysis.

These findings show that surface treatments are capable of decreasing microplastic fibre shedding from polyester textiles, providing a viable and scalable solution for controlling microplastic pollution from domestic laundry. The study contributes to efforts in textile engineering and environmental protection by highlighting effective pre-treatment options for reducing microplastic fibre release at the source.

1. Introduction

1.1 Textile Waste and Microplastic Pollution

1.1.1 Synthetic Textile Waste

Recently, production and consumption of synthetic materials increased globally. Directly, this situation caused an increase in synthetic waste generation. These petroleum-based materials are more durable and resistant compared to environmentally friendly materials. Synthetic materials are being widely used in the packaging, automotive, construction, and textile industries. Among these, textile industry covers a large share due to the use of synthetic materials in clothing and industrial applications.

The textile industry is one of the largest consumers of synthetic polymers. In 2019, synthetic fibres covered approximately 63% of global fibre production, with polyester alone representing over 50% of the total (Shen et al., 2020; Belzagui et al., 2021). Also, polyamide (nylon) and acrylic are other commonly used synthetic fibres due to their strength, elasticity, and low cost. Since these fibres provide low production cost and better performance, they are widely used in the textile industry (Rosa et al., 2024).

From 2000 to 2020, global fibre production nearly doubled. Reaching approximately 113 million tons, with synthetic fibres leading the growth. Polyester production alone, almost tripled in this period, being the most produced textile fibre in the world (Rathinamoorthy & Balasaraswathi, 2024; Shen et al., 2020).

In the European Union, over 5.8 million tons of textile waste are generated annually, with nearly 60% as synthetic materials such as polyester and nylon (European Environment Agency, 2019). A majority of this waste originates from post-consumer. These wastes are either incinerated or sent to landfill due to limited recycling possibilities (Shen et al., 2020).

The use of synthetic textiles is not limited with casual wear. These fibres are widely used in sportswear, technical textiles, home furnishings, medical textiles, and automotive interiors due to their lightweight nature and moisture resistance (Allen et al., 2024). Even though they have many benefits, the increasing production and consumption of synthetic textiles cause major pollution problems (Periyasamy, A. P., & Tehrani-Bagha, A., 2022b).

1.1.2 Microplastic Release Mechanisms

Microplastics are one of the most dominant forms of plastic pollution. They can occur in various forms such as fibres, fragments, and beads. Microplastic fibres' sizes vary between 1nm and 5 mm in length (GESAMP, 2015; Frias & Nash, 2019). Microplastic fibres generally shed from synthetic garments composed of polyester, polyamide (nylon), and acrylic (De Falco et al., 2019).

The release of microplastic fibre occurs on different stages of fabrics lifecycle such as manufacturing, usage, washing, drying, disposal (Napper & Thompson, 2016). Mechanical stress, chemical degradation, and thermal effects are primary causes of fibre detachment during the laundering process (De Falco et al., 2018).

Also, the textile structure and laundering conditions are essential. Mechanical abrasion is caused by friction between garments and leads to detachment of surface fibres. The condition and age of the garment also plays a critical role in this process. Older fabrics tend to shed more due to surface degradation (Shirvanimoghaddam et al., 2020).

Textile parameters such as yarn twist, fibre length, and production techniques (weaving or knitting) significantly affect shedding. Knitted fabrics are generally more prone to fibre loss due to their looped configuration. Woven fabrics with short-staple fibres, and they tend to instant shedding than continuous filaments (Pirc et al., 2016). Production factors like yarn tension and fabric density also affect the resistance of fibres.

Detergent use in the washing can cause strong mechanical agitations. This can increase the breakage and release of fibre fragments from textiles. (Periyasamy, A. P., & Tehrani-Bagha, A., 2022b). Thermal degradation reduces fabric integrity and elevates fibre shedding. Though less studied, tumble drying has also been identified as a potential contributor to airborne microplastic fibre emissions, an environmental concern parallel to wastewater release (De Falco et al. 2019).

Finally, fabric type significantly affects microplastic fibre release rates. Polyester compared to polyamide, tends more to shedding, possibly due to its lower hydrogen bonding capacity and surface cohesion (Rathinamoorthy & Balasaraswathi, 2024). Recycled polyester generally sheds more than virgin polyester. Probably because it contains shorter molecular chains, and during mechanical recycling structural imperfections occurs (Pirc et al., 2016). It has been discovered that some coatings and surface treatments, such as silicone or plasma modifications,

assist to reduce fibre release by smoothing fibre surfaces and increasing surface resistance (De Falco et al., 2018).

The washing machine design also affects the shedding, front-loading machines shown less shedding compared to top-loading models (Hartline et al., 2016).

Once released from the textile, microplastic fibres pass into wastewater. Primary and secondary filtration at the treatment systems capture a significant fraction, but up to 40% of microplastic fibres may still pass the treatment and enter aquatic environments (Boucher & Friot, 2017).

Released microplastic fibres can be examined through various techniques. With microscopic examinations such as SEM, it is possible to see the rough and broken surfaces (Monira et al., 2023). By using another method such as FTIR, identification of microplastic fibres is possible based on infrared peaks (Xu et al., 2019).

1.1.3 Environmental and Health Effects

Synthetic microplastic fibres' persistent behaviour and fast dispersion create serious ecological harm. Synthetic microplastic fibres originate from variety of sources, this makes them very dangerous. They can rapidly spread through terrestrial, aquatic, and atmospheric environments due to their smaller size, lower density and resistance to degradation (Tatsii et al., 2023) Studies confirm that, they have been detected in aquatic ecosystems, urban runoff, and wastewater (Akyildiz et al., 2022).

In aquatic systems, microplastic fibre can be found in both surface and deep levels. Many marine species such as planktons, mussels, shrimps, fishes are ingesting these fibres. This ingestion often causes less growth or reproduction. According to studies, synthetic fibres have been found in the digestive systems of up to 60% of sampled fish (Henry et al., 2019).

Microplastic fibres also can be found in soils, mainly caused by the land application of treated wastewater sludge. During the wastewater treatment process, large part of microplastic fibres retain in the sludge and then dried to use in agricultural activities as fertilizer. A report indicated that sludge applied in the EU introduces up to 1.2×10^{13} microplastic fibres into soil annually (Zubris & Richards, 2005). These transferred fibres can alter the soil properties which might affect the aeration and water retention capacity. The accumulation in soil also affects the activity of microorganisms and can reach to roots to damage the plants.

Atmosphere is also a highly affected environment. Indoor use of synthetic textiles in clothing, carpets, furniture, curtains cause high emission of microplastics to the air and lowering the indoor air quality. An urban monitoring study found that microplastic fibre content indoors reached up to 275 fibres per square meter per day (Dris et al., 2017). Outside, with the help of wind, the microplastic fibres are carried in the air. This phenomenon caused the microplastic presence in the remote zones such as Arctic even there is no local textile sources.

Microplastic fibres create environmental risk because they often contain dyes, protective chemicals and coatings. Once microplastic fibres have been released to the environment, these additional materials can mix with soil, water or air. On the other hand, microplastic fibres are able to adsorb certain pollutants from the environment. Laboratory analyses shown that synthetic fibres recovered from marine environments contain heavy metals and organic pollutants (Koelmans et al., 2016). Both of these capabilities of microplastic fibres make them a serious pollutant source.

Microplastic fibres being a risk to environment also affects the human health. Through ingestion, inhaling, and physical contact, humans often get exposed to microplastic fibres. A recent analysis indicated that an average adult may ingest between 39,000 and 52,000 microplastic particles (mostly in microplastic fibre form) per year (Cox et al., 2019). While most of these particles are disposed out of human body, a minor part can remain in the tissues.

Inhalation of microplastic fibres is one of the most continuous exposure sources. Its low visibility makes it a dangerous source. Humans can be exposed to microplastic fibres during everyday activities such as making the bed, getting dressed, folding, vacuuming. Some studies conducted in the occupational environments in textile facilities have shown chronic exposure to synthetic dust and fibres. Through this exposure, respiratory system issues are observed such as coughing, irritation, and reduced lung function (Pauly et al., 1998). Animal studies using fish (zebrafish) and small invertebrates show liver damage, gut inflammation, and behavioural changes following fibre ingestion or waterborne exposure (Choi et al., 2018).

There are concerns on nanoscale plastic formation from microplastics, which are smaller in particle size and pass through cellular membranes and can reach internal organs that microplastic fibres cannot (Geiser et al., 2005). Currently, nanoscale plastic detection methods are not widely used or completely reliable, however it can be seen as a serious issue and further research should be conducted.

1.2 Determination of Microplastic Fibre Release from Fabrics During Washing

A standardized evaluation of the microplastic fibre release is essential to assess the environmental impact of synthetic textiles and produce mitigation strategies. Laboratory tests provide these standardized methods to investigate quantity and quality of the microplastic fibres released compared to variable and inconsistent real-life washing conditions. In this chapter, the internationally standard procedures for simulating domestic laundering, collecting and analysing microplastic fibres, processing and interpreting the result data are introduced.

1.2.1 Standardized Procedure for Textile Washing Trials

A standardized and repeatable method is required to measure microplastic fibre release from textiles. As until now a standard method was not available and therefore laboratories and researchers relied on their own methods such as scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR) or gravimetric analysis. The main problem was that, to obtain all the necessary information on microplastics, multiple techniques had to be used in combination. In this regard, the lack of uniformity has pushed the scientific community to collaborate with national (UNI), European (CEN) and international (ISO) standardization bodies to develop standard methods for the quantification and identification of textile microplastics. Then the currently used standards UNI EN ISO 4484 series have been created.

This method insured consistency, reliability, and scientific acceptability of the results. After increase of hazard risk of microplastic fibre release and studies related to this field International Organization for Standardization (ISO) released the ISO 4484 series in 2023, to provide a framework for using in the textile sourced microplastic fibre shedding studies and experiments.

According to ISO 4484 standards, microplastic fibres have length of 300 nm ≤ × ≤ 15 mm and a length/diameter ratio higher th 3. Standards include three parts. ISO 4484-1:2023 specifies the procedure of material loss determination of textile products during domestic washing. In this part, sample dimensions, washing temperatures, water amount, machine settings are mentioned. ISO 4484-2:2023 describes the methodology for collecting, analysing, and characterizing the microplastics released during these washing trials. Fibre counting, polymer identification, and mass-based quantification are also components of this part. ISO 4484-3:2023 widens the testing protocol to finished textile end-products, allowing the use of same

methodology across various sample types. The main objective of these standard series is to simulate the domestic washing in the laboratory environment and minimizing the external sourced variability.

According to ISO 4484-1, sample fabrics are typically cut to a standard size. The temperature of the fabrics is set to 50 ± 3°C in the oven and 65 ± 4% relative humidity for 24 hours to stabilize moisture content and eliminate variability. After conditioning, samples are weighed on a calibrated analytical balance, and the dry weight is recorded before the washing trials started.

Washing process is carried out using standardized laboratory washing devices such as Gyrowash or Linitest. These machines can simulate the mechanical agitation of a domestic washing machine while maintaining control over temperature, rotation speed, and duration. The washing temperature used in the trials is generally 40 ± 2°C, and no detergent is used unless different conditions are needed. Containers used for washing trials containing 360 ml demineralised water with 50 steel balls. Water hardness also needs to be standardized; usually grade 3 demineralised water (specified in ISO 3696) is used to reduce interactions between fibres and minerals. Before washing cycles, containers must be pre-heated for approximately 10 minutes. According to standards, washing duration is 45 minutes and number of cycles are defined by the test objective. In many cases, one, three, or five cycles are done to evaluate both initial and cumulative fibre release. The wastewater of washing is collected after for filtration and analysis.

For the analysis, ISO 4484-2 describes the protocols for filtration, fibre quantification, and polymer identification. The water used in the washing is filtered using glass fibre filters or membrane filters with a pore size typically between 1.2 and 5 μm. Filters are dried in controlled conditions in the oven at 50±3 °C and weighed before and after filtration to determine the mass of material loss and released material. These values can be associated with fabric mass loss, filter mass gain, and fibre count.

ISO 4484-2 also suggests particle level analysis in addition to mass data and the counting the fibres by optical microscope, μFTIR (micro-Fourier Transform Infrared Spectroscopy), MICRO Raman. Filters need to be inspected fibres should be counted by using visual or digital method on imaging software such as ImageJ. Grid overlaying might help and counting of representative areas can be extrapolated to determine overall microplastic fibre count. Length and diameter analysis would be also helpful to understand the microplastic fibre morphology

and assess the microplastic release during mechanical stress of washing process. μFTIR or Raman spectroscopy methods are used to verify the polymer composition of the fabric and extra materials possibly caused by the treatment process. These techniques are useful to separate natural and synthetic fibres.

The results can be reported in two types of formats: mass loss per unit textile weight (e.g., g/g) and number of fibres released per sample. These two format allows interpretation of both environmental risk assessments and product lifecycle studies. Fibre count and characterization is more relevant for ecological impact, while mass data is more suitable for assessing mechanical durability or treatment efficiency.

ISO 4484-3 allows the measurement of released microplastic fibres during washing, from finished textile end products and home textile end products such as garments, towels, or furniture. These samples can be challenging, additional steps in testing might be needed to analyse these types of products. However, this part of the standard is valuable since the real-life products are being used in the experiments.

To ensure accuracy and repeatability, ISO 4484 also recommends the use of blank controls, which is washing without a fabric sample. They support the detection of potential external contamination. According to the standard, all testing processes must be performed under clean lab conditions to prevent airborne fibre contamination. Filter handling, drying, and imaging should be conducted in clean environments, and analysts should wear lab coats to avoid contamination.

In conclusion, the ISO 4484 standard series provide a guideline to evaluate microplastic fibre shedding from textiles during washing. The use of standardized methodologies is essential since the microplastic fibre pollution is rapidly growing. ISO 4484 is a useful guide for analysing synthetic textiles and producing mitigation strategies in the textile industry.

1.3. Techniques for Preventing Microplastic Fiber Release in Textiles

Increasing microplastic pollution created a serious concern. The research and policy work on this matter are trending due to the environmental hazard of these materials. One of the most effective interventions can be made during the design and production phases. The modifications made during these phases can minimize the microplastic fibre emissions before

it occurs. In this chapter, details and methods of minimizing the microplastic fibre emissions are presented.

1.3.1 Prevention Methods

One of the most direct ways of minimization is designing a textile that sheds fewer microplastic fibres. Fabrics composed of filaments are continuous and smooth. They tend to shed significantly less than the ones produced with staple fibres. They are known to consist short and loose segments which are more prone to mechanical breakage and detachment (De Falco et al., 2018). Similarly, fabrics produced by using high-twist yarns show less fibre shedding (S. Choi et al., 2021)

Fabric density is another factor which affects the shedding. Finishing techniques such as sanding, sueding, or brushing improve softness and increase the exposure of fibre ends on the fabric surface. The balance between environmental friendliness and comfort is a challenge in textile production (Shen et al., 2020). Although there is no global standard for shedding, some textile producers have begun to experiment low-shedding fabrics. The ISO 4484 standard guides and supports these initiatives.

In addition to improved fabric design, microplastic fibre capturing systems have been developed. They are able to prevent the release of microplastic fibres into wastewater streams. External filter units can be connected to household washing machines. These filters can capture 80–90% of fibres depending on flow rate, maintenance, and installation quality (McIlwraith et al., 2019). On the other hand, in-drum devices reduce mechanical agitation and collect loose fibres in the washing chamber. The effectiveness of these solutions is arguable however they provide and important solution for the commercial machines.

A few numbers of washing machine manufacturers have been working on built-in microplastic fibre filtration systems. Increase in this trend is caused in by legislation in France (AGEC Law, 2020). Which indicates that all new washing machines sold after January 1, 2025, must include a built-in microplastic fibre filter. This is the first national-level policy targeting microplastic fibre emissions at the household level. It is expected to influence the European Union to work on these types of policies in the future.

While technical solutions are essential, consumer behaviour also plays a critical role in microplastic fibre mitigation. Studies have shown that washing garments at lower temperatures with shorter cycles and low agitation can lead to a significant reduction in fibre release. Even

switching from 60 °C to 30 °C washing may reduce shedding by 30% (Belzagui et al., 2021). Similarly, fully loaded washed reduce the friction between the garments and the drum. This situation can limit fibre fragmentation. Drying machines can also prevent airborne fibre emissions.

Consumers can be educated through product labelling and better washing instructions. However, the industries should collaborate and try to decrease the shedding risk before the product reaches to the final customer.

In summary, effective prevention of microplastic fibre release is complicated and multi-layered strategy depending on various individuals and entities. While the creation of new strategies in textile design and filtration technology is in progress, the integration of these practices into consumer habits will be a long-term job.

1.3.2 Surface Treatments

Surface treatments have become more popular research focus in the recent years to mitigate microplastic fibre shedding from synthetic textiles. These techniques are useful when they are applied during the postproduction process. The aim of these techniques is reinforcing the integrity of the fabric and prevent shedding during washing processes. Surface treatment is a good option for materials such as polyester and polyamide due to their tendency of high shedding (Ali et al., 2024).

Conventional synthetic textiles contain loose fibre ends, and they can easily break under the mechanical stress. Surface treatments try to prevent this situation by either forming a physical barrier or strengthening chemical bonds within the fibre. This type of treatments can seriously reduce the number of shedding fibre without altering the quality of the textile (Rathinamoorthy & Balasaraswathi, 2024).

One of the most common surface coating techniques is chemical coatings. Done by applying polymeric or resin-based solutions onto the fabric surface via dipping, spraying or padding. Polymers such as polyurethanes, silicones, and polyacrylics are being used to enhance the abrasion resistance and form protective films which bind surface fibres in place (Belzagui et al., 2021). These coatings may be hydrophobic or hydrophilic depending on intention of the application. For microplastic fibre control, generally hydrophobic coatings are preferred due to their lower surface friction and reduced water penetration.

The polymer coating is effective in early wash cycles; however, the durability of coatings is a critical issue. Consecutive laundering leads to film erosion or micro-cracking in coating layers. For example, in commercial finishing silicone-based polymers are being used and they have shown promising results in the first three to five laundering cycles. After a certain point, a plateau phase or decline in yield follows (Rathinamoorthy & Balasaraswathi, 2024). This has led the investigations into hybrid coatings which combine mechanical adhesion and chemical anchoring mechanisms.

More recently, bio-based coating techniques gained attention due to their environmental side and versatility. Chitosan can be given as a sample which is a biopolymer derived from chitin, a natural polysaccharide abundant in crustacean shells and fungal cell walls. It is partially deacetylated, giving it a linear structure with positively charged amine groups under physiological conditions. These properties make chitosan a biodegradable, biocompatible material with strong potential for use in surface modification and other industrial applications (Queiroz et al., 2014). It is one of the most studied biopolymers in this sector. When chitosan is applied to polyester or polyamide fabrics, it forms a cationic film that adheres strongly to the negatively charged fibre surface. Especially if the fabric had another activation treatment such as alkaline hydrolysis or plasma discharge. Studies have shown that, polyester fabrics treated with chitosan, reduced microplastic fibre release by approximately 95% (Kang et al., 2021). Chitosan enhances textile performance by forming a thin, hydrophilic film on fibre surfaces that physically binds loose fibres and smooths the structure. Its amine and hydroxyl groups also promote inter-fibre adhesion through ionic and hydrogen bonding, reducing felting, shrinkage, and fibre shedding while improving dye affinity and antimicrobial properties (Tonin et al., 2007, Wong et al., 2003)

Pectin is another popularly used biopolymer, a plant-derived anionic polysaccharide. It can form cohesive films on synthetic surfaces. Pectin-treated polyamide fabrics released up to 90% less microplastic fibres compared to untreated samples (De Falco et al. 2019). This effect is due to the film's flexibility and fibre penetration ability. Both chitosan and pectin are biodegradable and non-toxic materials. That makes them suitable for environmentally sensitive textile applications.

Physical finishing techniques are also useful for surface stabilization. Calendaring, which compresses the fabric between heated rollers, can flatten loose fibres and reduce surface roughness. Similarly, singeing, which passes the fabric over a controlled flame or hot plate,

removes loose fibre ends by thermally degrading them. These methods are commonly used in cotton and polyester finishing processes and may provide minor reductions in shedding. However, the impact is generally less than chemical or polymer-based treatments and may be temporary if not followed by additional reinforcement layers (Periyasamy, A. P., & Tehrani-Bagha, A., 2022b).

Among the technologies, plasma surface treatment offers solvent-free and low-waste solutions. This treatment is made by exposing the fabric surface to ionized gas under vacuum or atmospheric pressure. Resulting with surface activation of the fabric. Plasma treatment can be standalone treatment or used as primary supporting process to the coating applications (Muthu et al., 2021). It can modify hydrophobic synthetic fibres without altering bulk properties. Plasma can enhance the binding efficiency of future coatings by bringing polar functional groups (–OH, –COOH) onto the textile surface. Studies have shown that plasma and chitosan treatment combinations provide better results compared to standalone versions (Rathinamoorthy & Balasaraswathi, 2024).

Surface treatment efficiency is typically assessed using mass loss measurements, fibre counting, and microscopic analysis with the use of ISO 4484 standards. Additional tools such as scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR) can be used to analyse the surface coating.

These treatments show promising results; however, their longevity is an issue. Coatings perform well in early washing cycles but they either wash-off slowly from the surface or lose their abilities. Another issue is cost and scalability, since these applications requires specializing and additional machines to apply.

The environmental impact of these mentioned treatments should be evaluated as well. They may reduce the microplastic fibre shedding but also introduce chemicals to the environment. Biodegradable solutions like chitosan and pectin offer more environmentally sensitive approaches (Ali et al., 2024).

In conclusion, surface treatments provide important strategies to textile industry on reducing microplastic fibre release. Even single solution can offer good results, while combined treatments can provide better solutions in reducing the microplastic fibre release while washing (Ramasamy & Subramanian, 2023). More research needs to be done to optimize these treatment methods and apply to the industry in the full scale.

1.4. Plasma Pre-Treatment Technology

1.4.1 Plasma Technology Basics

Plasma technology became a widely used in the surface modification methods due to its efficiency. Also not changing the bulk structure of the material while enhancing the physical and chemical properties. When the plasma interacts with textile it creates surface level reactions. These interactions occur only in the outer layer of the fabric, this makes plasma an effective modification method without altering the integrity (Labay et al., 2012).

In textile applications, plasma is generated through energy, usually in from of radiofrequency or microwave. Plasma treatment can reduce impurities and increase surface energy by introducing polar groups (hydroxyl, carboxyl, amine). This process enhances the chemical reactivity of the fibre. Plasma can also provide crosslinking between polymer chains at the surface and enhance coating application performance (De Falco et al., 2019). These effects support the reduction of microplastic fibre shedding through reducing weakly bound fibres, and by modifying the fibre surface. Plasma can enhance binding of biopolymer coatings like chitosan or pectin (Rathinamoorthy & Balasaraswathi, 2024).

Plasma treatments can be classified according to their operational pressure. Low-pressure or vacuum plasma treatments allow high control and more uniform but slower process. Atmospheric pressure treatments are being done in ambient conditions and well-suited for industrial processes. They don’t require vacuum chambers and can be directly installed at the end of production lines. Both systems are efficient in terms of surface optimization (Labay et al., 2012; De Falco et al., 2019).

The effectiveness of the plasma treatment depends on several factors such as type of gas, power level, exposure duration, distance between textile and source. Commonly, oxygen plasma is applied to polyester due to its capability of increasing wettability and reactivity. These parameters should be optimized in order to get the most efficient performance and reduce the undesirable side effects (De Falco et al., 2019).

In the environmental perspective, plasma offers several advantages compared to traditional chemical treatments. Such as no water or solvent use since it is a dry process. By that, the waste generation is eliminated, and chemical risks are reduced. This makes plasma processes more environmentally sustainable (Ramasamy & Subramanian, 2023).

In conclusion, plasma technology provides an efficient and versatile method for surface modification. It improves adhesion, increases surface energy and enhances durability of coatings while preserving the backbone of the material.

1.4.2 Plasma Application on Synthetic Textiles

Synthetic textiles, especially polyester (PES) and polyamide (PA) are the most produced and used fibre types due to their strength, durability, elasticity and cost effectiveness (Labay et al., 2012). In recent years, plasma treatment is one of the favourite technologies used for surface level modification due to its ability of not altering the bulk properties of the fabric.

Plasma treatment modifies the synthetic textile by exposing the fabric to a plasma field consisting of electrons, ions, and reactive substances. When applied to synthetic textiles, physiochemical effects appear including surface etching, removal of weak boundaries, and introduction of polar functional groups (such as hydroxyl (–OH), carboxyl (–COOH), and amino ($–NH_2$) groups). These groups significantly increase surface energy and hydrophilicity. They also enhance the effectiveness of applied hydrophilic finishes and biopolymer coatings,

Oxygen plasma treatment is more efficient on polyester fabrics. Through polymer surface oxidation, the wettability and adhesion characteristics can be improved. This can increase the performance of biopolymer coating processes due to high presence of carboxylic and hydroxyl sites (Ramasamy & Subramanian, 2023). Without surface activation, chitosan would have less adhesion, leading to weak coating and rapid wash-off during the laundering.

Studies have shown that polyester fabrics treated with oxygen plasma and chitosan coating showed up to 80–90% reductions in microplastic fibre shedding compared to untreated fabrics (De Falco et al., 2019). Similarly, polyamide fabrics' adherence of coatings enhanced when underwent plasma pretreatment. This resulted in significant reductions in microplastic release (Labay et al., 2012).

Plasma causes subtle surface morphology beyond the chemical activation. Microscopic analysis revealed that plasma treated fabrics demonstrate increased surface roughness. This situation can cause interlocking between fabric surface and applied coating (Labay et al., 2012). It can be said that plasma treatment does not only provide chemical activation; it also

Plasma treatments shown positive effects also with alkaline pre-treatment combinations. By using sodium hydroxide (NaOH), alkaline hydrolysis can occur and increase hydrophilicity. However, it has been observed that alkali chemicals can also cause damage to the microfibre

structure (Akyildiz, Sezgin, et al., 2022). When applied with plasma activation, alkaline pre-treatments amplify surface polarity and provide more efficient bonding (Rathinamoorthy & Balasaraswathi, 2024). According to a study, the substrates cannot bind chitosan, and they need to be pretreated, and a widely used technique is the application of nonequilibrium gaseous plasma. Usually, oxygen, argon, and air plasmas were used. (Vesel, 2023)

From the sustainability perspective, there are many environmental benefits of plasma treatment. Since it's a dry process, neither water is required nor effluent is produced, and it requires minimal chemical input. This make is suitable for circular economy and waste reduction systems. Plasma treated fabrics keep their mechanical strength, flexibility, and dye retention ability (Ramasamy & Subramanian, 2023).

In summary, plasma treatment offers an effective method for modifying surface properties of synthetic textiles. When combined with alkaline modifications or bio-polymer coating, plasma can enhance the adhesion and reduce the release of microplastic fibres during washing. Despite the advantages of these techniques, this technology is still in its early stages. Further research and developments must be done in order to optimize this technology.

1.4.3 Plasma Treatment Effectiveness in Reducing Microplastic Release

Repeated washing, drying and wear contribute to detachment and release of microplastic fibre into the air and wastewater system. Several studies identified surface finishings (especially when combine with plasma treatment) are effective to stabilize or reduce this detachment activity of synthetic textiles (De Falco et al., 2019; Rathinamoorthy & Balasaraswathi, 2024).

In a study, plasma treated, and chitosan coated polyester demonstrated microplastic fibre release reduction of 84.29% in fibre count and 87.61% in mass during washing cycles (Rathinamoorthy & Balasaraswathi, 2024). Similar results are present also for alkaline treatments, especially with sodium hydroxide. When NaOH and plasma treatments are combined, the physical activation and chemical modification effects are amplified (Rathinamoorthy & Balasaraswathi, 2023). This enhances the coating durability and adhesion (Ramasamy & Subramanian, 2023).

Despite these promising results, there are many factors that affect the efficiency of treatments such as fabric type, washing conditions, coating formulations, and plasma parameters. A study comparing plasma-chitosan-treated and untreated polyester fabrics reported an 84% reduction

in microplastic fibre count, however the effect diminished slightly after ten laundering cycles (De Falco et al., 2019). Similarly, polyester knits treated with trichloroacetic acid and methylene chloride showed microplastic fibre release reductions as high as 93.79%, the woven fabrics exhibiting lower reductions (Rathinamoorthy & Balasaraswathi, 2024). This shows that high reductions are possible, but the effectiveness is not universal among all conditions.

In this context, the current study aims to investigate the effectiveness of plasma-assisted surface treatments on polyester fabrics using two concentrations (0.6 % and 1.2 %) of chitosan and sodium hydroxide. These treatment types have been selected based on their documented ability. This study seeks to confirm the reliability and environmental impact of these treatments towards microplastic fibre release control.

In conclusion, surface treatment strategies, particularly plasma treatment followed by chemical or biopolymer finishing, show strong potential for reducing microplastic fibre release from synthetic textiles.

2. Materials and Methods

This study aimed to assess the microplastic fibre release behaviour of polyester fabrics that underwent plasma treatments. Polyester fabrics were subjected to separate chemical treatments using chitosan and sodium hydroxide (NaOH) prior to plasma exposure to assess their individual effects on surface modification and microplastic fibre release. The impact of each treatment was evaluated by measuring microplastic fibre mass and release count during washing and characterized by using Fourier-transform infrared (FTIR) spectroscopy to detect surface chemical changes. The washing experiments were conducted in full accordance with relevant ISO standards, specifically ISO 4484-1:2023 for quantifying microplastic release from textiles, which incorporates procedures from ISO 105-C06 for standardized laundering.

2.1 Sample Materials and Preparation

The fabric used in the study was a knitted 100% polyester textile, representative of commonly used synthetic clothing materials. The samples were cut into uniform pieces of 10 cm × 25 cm based on ISO 4484-1:2023 to simulate the real-world washing conditions. Samples fabrics have no pilling, 0.0116 mm average fibre diameter and 65 nm yarn count.

2.2 Plasma Surface Treatment

All fabric samples were subjected to a low-pressure plasma treatment prior to the application of chemical agents. This process was conducted at the Environmental Park in Turin, using a low-pressure plasma system under controlled conditions of power, gas flow, and exposure time.

The plasma treatment was applied to both sides of the mesh samples using an open-air atmospheric pressure plasma glow discharge system (APP-GDBD, Grinp S.r.l., Torino, Italy). The setup consisting of two stainless-steel plates (80 × 23 × 3.5 cm) and five electrodes (80 × 1 cm) to obtain stable glow discharge. The electrode spacing and dielectric composition determined whether the discharge was filamentary or glow. The maximum power was 1500 W (3.75 W/cm^2), with approximately 40% energy loss. Water vapor was provided by a rotary pump and heating box, with the electrode temperature set to 50 °C. A He-O_2-H_2O gas mixture (1050 W nominal power) was used to generate plasma, which was applied to both sides of the fabric for 30 seconds. The method, which is a lab-scale roll-to-roll counterpart of an industrial setup, ensures both continuity and industry application.

The aim of this process is activating the polyester fibre surface. The plasma environment consists of reactive species such as positive ions, free radicals, electrons, and UV photons. These species modify the surface chemistry and increase surface energy. Also, they enhance surface porosity and improve wettability by introducing polar functional groups (hydroxyl and carboxyl). Improved wettability supports more uniform spreading and stronger adhesion. This enhances the effect of sodium hydroxide (NaOH) and chitosan in subsequent treatment stages.

2.3 Chemical Treatment Procedure

Sodium hydroxide (NaOH) solutions at concentrations of 0.6% and 1.2% were prepared by diluting 0.1 M NaOH with deionized water. Specifically, 12 mL and 24 mL of 0.1 M NaOH were mixed with 188 mL and 176 mL of water. These concentrations are based on a previous study of Rathinamoorthy et al., 2024.

Citric acid is used to dissolve chitosan powder in order to prepare the solutions. 1.2 g and 2.4 g of chitosan powder were slowly added in to 200 mL of distilled water containing 1.2 g and 2.4 g of citric acid to prepare 0.6% and 1.2% chitosan solutions. Then, solutions were stirred with magnetic stirrer at 50 °C for 4 hours to ensure full dissolution and homogeneity.

All fabric samples were subjected to low-pressure plasma treatment under controlled conditions. Right after plasma activation, the fabrics were immersed in NaOH or chitosan solutions. Excess liquid was removed with a laboratory padder to ensure uniform chemical distribution. The treated samples were then dried at 40 °C overnight to stabilize the chemical adhesion and support film formation.

Control (RAW) samples received no plasma or chemical treatment and were used for comparison in the experimental analysis.

2.4 Washing Protocol and Microplastic Fibre Collection

Microplastic fibre release tests were conducted following the standard method mentioned in the ISO 4484-1:2023. All laundering, filtration, and weighing processes were performed at the CNR Biella Campus (Italy), using certified laboratory equipment to ensure the reliability and repeatability of results.

Each polyester fabric sample was placed in a separate sealed stainless-steel container filled with 360 mL of purified water and 50 stainless steel balls (6 mm in diameter) to simulate mechanical agitation. Washing was carried out using a James Heal Gyrowash machine, with each cycle lasting 45 minutes at 40 °C. Each sample underwent five consecutive washing cycles. For analysis, wastewater was collected from the first, third, and fifth cycles. The second and fourth cycles were discarded, and containers have been cleaned to prevent cross-contamination.

After each cycle, the contents of the container (fabric, steel balls, and residual water) were carefully removed and rinsed with distilled water and ethanol to ensure complete recovery of released microplastic fibres. The collected wastewater was then filtered through glass microplastic fibre filters (47 mm diameter, 1.2 μm pore size) using glass filtration holders connected to a vacuum pump with regulated pressure.

To prevent environmental contamination, the filters were immediately transferred into aluminium foil covers after filtration. They were then dried overnight and stored in a desiccator prior to weighing and further analysis.

2.5 Microplastic Fibre Analysis: Mass, Count, and Length Based Methods

To quantify microplastic fibre release and characterize fibre morphology; gravimetric, image-based count, and length-based analyses was performed.

2.5.1 Mass-Based Analysis

After filtration, each glass microplastic fibre filter was dried overnight in an oven and stored in a desiccator to eliminate residual moisture. Filters were weighed before and after washing using an analytical microbalance (±0.1 mg resolution). The filter mass gain (post- mass minus pre-filtration mass) was normalized to the initial dry weight of the fabric and reported as grams of microplastic fibre released per gram of fabric (g/g). This provided a quantitative estimate of total fibre loss (De Falco et al., 2019).

2.5.2 Count-Based Analysis

Each filter paper was places on a black matte background to enhance visual contrast. Using a calibrated high-definition SL.R camera (Sony ILCE-7RM3 v.1.01) equipped with a Zeiss 100 mm macro 1:2, photographs of each filter were captured under standardized magnification

settings. These conditions were kept the same for all samples to ensure uniform conditions and comparisons.

All the captured images were imported into ImageJ software, a widely used open source image processing software designed for biological and materials analysis. Images were calibrated to convert pixel measurements into millimetres. To estimate the total microplastic fibre count, 20 pre-defined grid zones, each measuring 15 mm^2 area, were selected and analysed on each filter. The sum of fibres from the 20 grid zones was then multiplied by a correction factor of 4.63, representing the ratio between the counted grids and the total number of grids covering the full surface of the filter. In addition, because the used filtration device had a smaller diameter than the filter paper, another correction factor of 1.2664 was applied to calculate for the ratio between the full filter area and the effective filtration area. This ensured the acquiring accurate fibre count released during washing. For filters with particularly low fibre densities, such as blanks or control samples, manual counts were conducted to ensure precision and avoid underestimation.

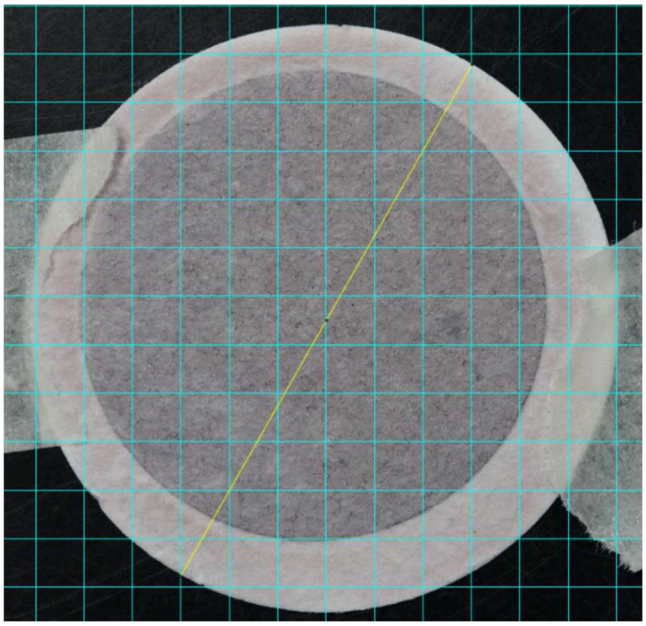

Figure 1. Image of filter paper of Raw sample after 5th wash on ImageJ software

2.5.3 Length-Based Analysis

To evaluate the fibre fragmentation, the length measurement is made for 50 randomly selected microplastic fibres from each sample, from the same high-resolution images. This process used ImageJ's calibrated line segment tool, allowing for precise dimensional data in millimetres. Length distributions were categorized into defined bins: <0.5 mm, 0.5–1.0 mm, 1.0–1.5 mm,

and >1.5 mm, to analyse distribution trends. Shorter average lengths were interpreted as signs of increased fibre breakage due to either chemical weakening or mechanical abrasion.

By combining mass, count, and length-based analyses, this methodology provided a multidimensional understanding of the effect of surface treatments on microplastic fibre shedding and fibre integrity during domestic washing.

2.6 Abrasion Test

To assess the durability and surface resistance of the treated and untreated fabric samples, abrasion tests were conducted using a Martindale abrasion tester in accordance with the ISO 12947 protocol. This standardized method is widely recognized for simulating wear under controlled mechanical conditions and allows the interpretation of structural integrity and surface damage due to friction. The testing process imitates mechanical stress that textiles experience during laundry, wear or usage over time.
Each fabric sample, cut into circular discs, was firmly installed in the Martindale device and subjected to a specified cyclic rubbing motion under constant pressure. An abradant fabric was placed in contact with the sample, and the device was programmed to perform a certain number of rub cycles (according to ISO criteria), assuring consistency across all samples. The abrasion followed a Lissajous pattern that provides uniform wear over the test surface.
After the abrasion test, samples were visually evaluated under SEM to assess surface degradation, fibre breakage, and detachment.

2.7 FTIR Spectroscopy

To investigate chemical changes on the fabric surface, FTIR (Fourier Transform Infrared Spectroscopy) performed with the use of ATR accessory (Shimadzu AIM-9000 – Figure 1). Raw and treated samples were analysed using spectra ranging between 700-4000 cm^{-1}. Two versions of each spectrum were captured: raw (unprocessed) and processed (smoothed and baseline-corrected). Multi-point baseline correction and spectral smoothing were used during processing to improve clarity and interpretation without affecting the core data. A reference image of the FTIR device used in the research can be seen below alongside the spectrum graphs for more detail and clarity. In the Figure 2 an example of FTIR spectra on RAW sample is shown.

Figure 2. Shimadzu AIM-9000 Micro-FTIR

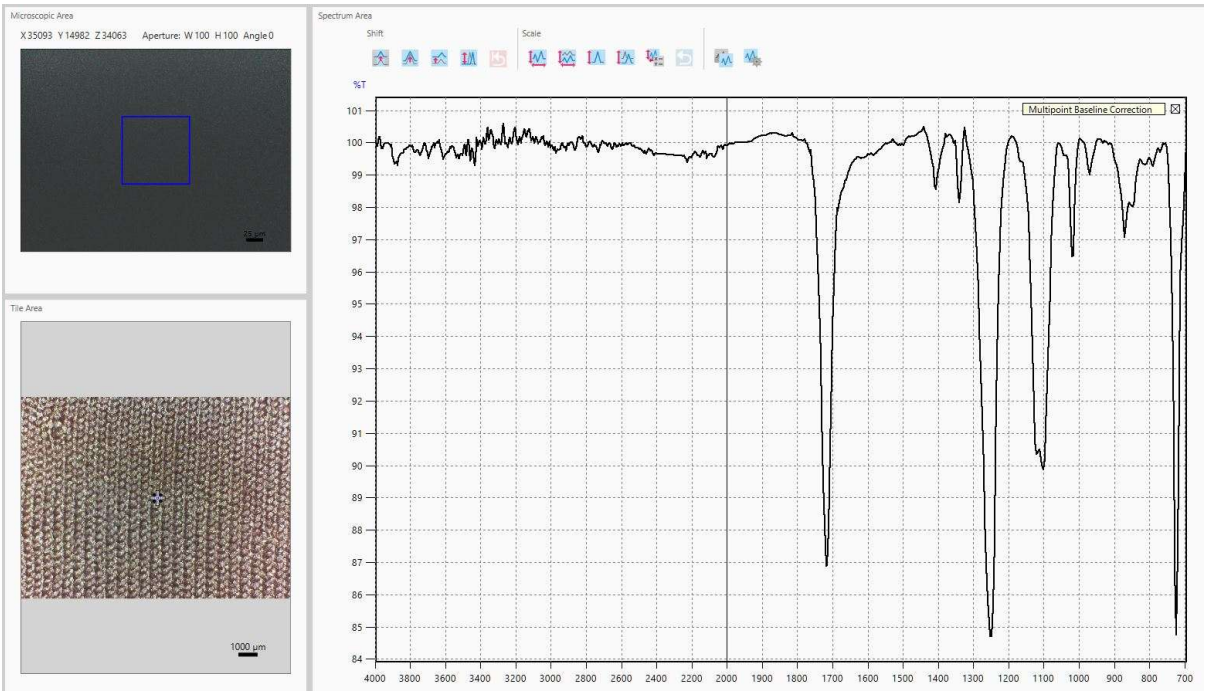

Figure 3. Screen image from the FTIR analysis software for Raw sample

2.8 Scanning Electron Microscopy (SEM) Analysis

SEM provides high-resolution imaging, enabling direct viewing of surface topology, fibre integrity, fibrillation, and damage morphology at the micro scale. This method is useful in detecting signs of degradation such as surface fractures, fibril development, and fibre breaking, which are generally undetectable by macroscopic or gravimetric examination alone.

SEM (Fei Inspect Eds Edax, Genesis) was used in this research to analyse different phases of the fabric's life cycle. Images were gathered from (i) dry fabric samples before experimental washing, (ii) washed and dried samples to assess the effect of washing on fibre morphology, (iii) samples that underwent abrasion after washing to examine mechanical durability under stress, and (iv) glass fibre filter papers used for capturing shed fibres, which were analysed to investigate the morphology of the released microplastic fibres. This method allows an advanced comprehension of how fabric structure changes during each phase of treatment and mechanical wear.

Previous studies have shown that fibre surface properties such as microcracks, fibrils, or surface coating detachment are signs of mechanical and chemical stress-related degradation (Cai et al., 2021).

SEM enables a visual comparison of the protective effects of plasma-assisted chemical modifications, such as alkaline hydrolysis with sodium hydroxide (NaOH) and chitosan. These treatments are hypothesized to enhance surface bonding and reduce fragmentation by improving inter-fibre adhesion or altering the fibre's susceptibility to mechanical failure (Periyasamy & Tehrani-Bagha, 2022). Reviewing both treated and raw fabrics, the SEM data provide important visual data to support these hypotheses.
Finally, SEM analysis helps assess quantitative results, count, and FTIR analysis. Fibres with smooth, unbroken surfaces, for example, are expected to release fewer microplastic fibres after washing, while highly fibrillated or broken surfaces may explain higher counts or filter mass gains. These connections improve the overall reliability.

3. Results

3.1 Microplastic Fibre Release Counts

As seen Table 1, the average microplastic fibre release count for the untreated (Raw) samples was 1653 fibres per filter. Among the treated samples, 0.6% NaOH resulted in an average of 1117.7 fibres, and 1.2% NaOH with 1065.0 fibres. The chitosan-treated groups demonstrated even lower counts: 0.6% Chitosan gave an average result of 607.0, and 1.2% Chitosan with 968.7 fibres on average. These counts, as seen below on the table, represent the replicated average values.

Sample Type	Average Fibre Count
Raw (Untreated)	1653.0
0.6% NaOH	1117.7
1.2% NaOH	1065.0
0.6% Chitosan	607.0
1.2% Chitosan	968.7

Table 1. Average Fiber Count per Sample Type

When compared to the raw sample, all chemical treatments demonstrated a reduction in microplastic fibre release. The 0.6% NaOH treatment reduced fibre count by 32.4%, while the 1.2% NaOH shown a reductio of 35.6%. The 0.6% Chitosan group reduced microplastic fibre shedding by 63.3%, and the 1.2% Chitosan group by 41.4%. These findings suggest that while both NaOH and chitosan treatments mitigate fibre release, the effectiveness is influenced by both the chemical agent and its concentration.

The findings of this study indicate that 0.6% Chitosan, exhibited greater effectiveness in reducing microplastic fibre release when compared to NaOH. While both chemical agents reduced the amount of fibre shedding, the level of reduction differed depending on the concentration and type of agent, indicating different modes of interaction with the fabric structure.

Figure 4 shows the average microplastic fibre release count per sample on 5th wash, calculated from three replicate samples for each treatment condition. The untreated (raw) samples displayed the highest microplastic fibre release and thus served as a baseline for the assessment of the impact of chemical treatments. The findings indicate that both NaOH treatments (0.6% and 1.2%) resulted in a significant decrease in microplastic fibre release, suggesting that these alkaline treatments may break down or weaken fibre structures, leading to a reduction in recoverable material. However, chitosan-treated samples, particularly at 0.6%, exhibited relatively lower release counts, suggesting that chitosan treatments may be less destructive to the fibre structure.

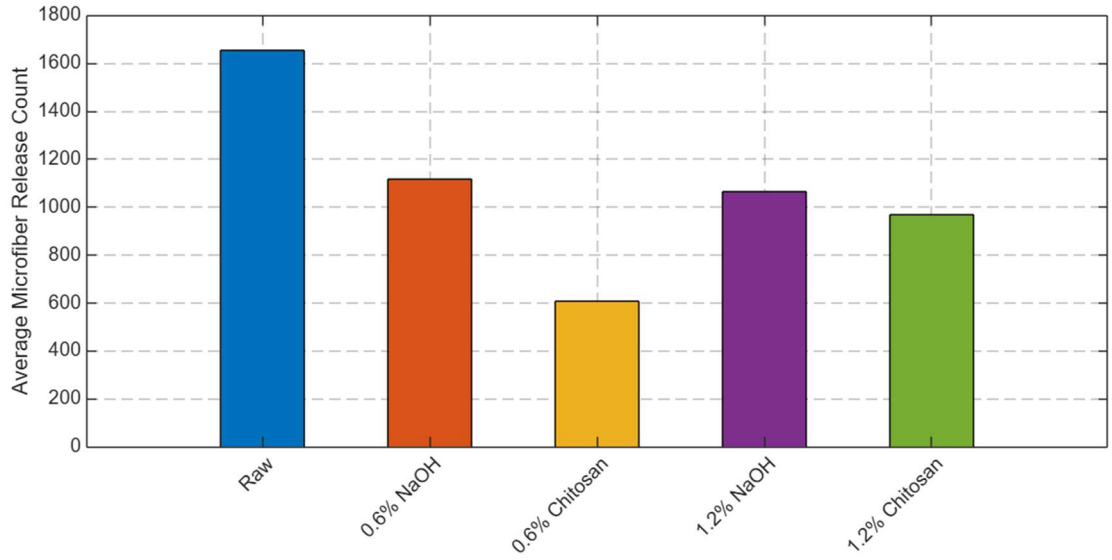

Figure 4. Average Microplastic Fibre Count per Sample Type on 5th wash

These replicate-based averages provide information about the influence of different surface treatments on fibre release behaviour. The findings suggest that NaOH contributes to structural degradation or enhanced fibre removal, while chitosan preserves fibre integrity, resulting in improved recovery during analysis. This evaluation is critical for determining the effectiveness of chemical agents in applications.

3.2 Mass-Count Comparison

The microplastic fibre release after the fifth wash was evaluated using both fibre count and filter mass gain. As shown in the table below, the Raw samples exhibited the highest microplastic fibre count (1653.0) and the highest filter mass gain (0.0015 g) after the fifth wash.

Both NaOH and chitosan treatments demonstrated a clear reduction in both count and mass compared to the raw fabric.

Sample	Avg. Microplastic Fibre Count	Filter Mass Gain (g) – 5th Wash
Raw	1653.0	0.0015
0.6% NaOH	1117.7	0.0009
1.2% NaOH	0	0.0008
0.6% Chitosan	607.0	0.0009
1.2% Chitosan	968.7	0.0008

Table 2. Filter mass gain (5^{th} wash) - average microplastic fibre count comparison

Between treatments, chitosan showed a more visible drop in microplastic fibre count. However, the filter mass gains were nearly the same with NaOH treated samples. The reason may be the NaOH samples release less number but potentially denser or more fragmented microplastic fibres.

The difference between count and mass may be due to fibre lengths sizes. Shorter fibres can be heavier fibres and increase the total mass even if there are fewer fibres. Also, dyes and chemicals on the fabric surface, especially in untreated samples, might dissolve or detach during washing and increase the filter mass.

Chitosan treatments preserved fibre count better than NaOH, especially at 0.6%, while keeping similar filter mass gain. This may indicate that chitosan treatment effectively decreased microplastic fibre release without serious fibre degradation.

Overall, both count and mass data support that both treatments reduced microplastic fibre shedding. The low filter mass gains of treated samples, compared to raw, show the positive effect of these treatments in limiting the fibre release. But the differences between mass and count proves the effect of other factors such as fibre size or extra substances on the.

3.3 Microplastic Fibre Mass Calculation

By utilizing a volumetric technique based on fibre count and size, the mass of released microplastic fibres was determined. The average fibre diameter and length values derived from microscopic analysis were coupled with the total number of fibres detected on the filter for each sample. The following formula was utilized to get the microplastic fibre mass (m):

$$m = T \times d \times \pi \times (D / 2)^2 \times L$$

Total fibre count (T), polyester density (d) (1.38 g/cm³), average fibre diameter (D in mm), and average fibre length (L in mm) are all accounted for in this equation. It was then converted to grams. Instead of using gravimetric measurements, this method provides a standardized estimation of fibre mass based on physical features, assuming cylindrical fibre geometry (Rathinamoorthy & Balasaraswathi, 2023). Microplastic fibre masses calculated using data from the fifth washing cycle for each sample can be seen in the table below.

Sample	Microplastic Fibre Mass (g)
RAW-1	0.000179
RAW-2	0.000154
RAW-3	0.000146
0.6 % NaOH-1	0.000144
0.6 % NaOH-2	0.000095
0.6 % NaOH-3	0.000093
1.2 % NaOH-1	0.000097
1.2 % NaOH-2	0.000054
1.2 % NaOH-3	0.000059
0.6 % Ch-1	0.000153
0.6 % Ch-2	0.000131
0.6 % Ch-3	0.000152
1.2 % Ch-1	0.000130
1.2 % Ch-2	0.000130
1.2 % Ch-3	0.000128

Table 3. *Calculated microplastic fibre masses*

The computed microplastic fibre mass and the filter mass gain after washing are two independent techniques. The computed mass is based on the number, length, and diameter of microplastic fibres with ideal cylindrical shape and homogenous density. On the contrary, the filter mass gain indicates the filter's overall gravimetric increase after washing and drying, capturing not only microplastic fibres but also additional material that remain on the surface.

One main cause of difference is the existence of non-fibrous residues on the sample fabric, including surface coatings, dye particles, or finishing chemicals which are released during washing. These chemicals contribute to filter weight, resulting in potential inaccurate estimation in mass-based assessments. Another cause can be the shorter lengths of NaOH treated samples compared to chitosan treated ones.

On the other hand, the fibre count-based calculation may underestimate the total microplastic fibre mass due to variables such as unnoticed or overlapping fibres, or fibre shape used in volumetric-to-mass calculations. These methodological variations point out the difficulties in directly comparing gravimetric and visual methods in microplastic fibre analysis.

In combination, these elements explain why filter mass gain numbers tend to be greater than calculated microplastic fibre mass, and why the correlation between the two methods might be weak.

3.4 Statistical Comparison

A Pearson correlation analysis was performed to assess the connection among microplastic fibre mass obtained using geometric fibre properties and gravimetric mass gain measured on filter paper after the fifth washing cycle.

The results showed a Pearson correlation coefficient of 0.30, indicating a weak positive correlation between calculated microplastic fibre mass and gravimetric filter mass gain. Nevertheless, the corresponding p-value was 0.280, which exceeded the commonly acknowledged limit for statistical significance. This indicates that the correlation is not strong, and the observed correlation could be related to random variation instead of a solid, anticipated link between the two data.

The variations between the two methodologies can explain the absence of significant correlation. The microplastic fibre mass is determined using visual-based analysis, which only takes into account microplastic fibres that have been discovered. The gravimetric approach, on the contrary, determines total retained substance on the filter, which includes non-fibrous

residues such as finishing chemicals and dye particles. These external factors influence the filter mass gain, decreasing the correlation between the two values found from two different methods.

A one-way ANOVA was used to see if there was a significant difference in microplastic fibre mass between treatment groups. The obtained p-value of 0.0017 indicates a statistically significant difference in group averages. This indicates that chemical treatments, especially those applied after plasma activation, have a significant impact on the amount of fibre released after washing.

In conclusion, while the relationship between computed microplastic fibre mass and weighed filter gain is weak and statistically insignificant, the ANOVA results precisely support the theory that surface treatments substantially minimize microplastic fibre release. The difference between the two measurements is most likely due to the complexities of real-world fibre capture and weighing. Together, these methodologies provide a comprehensive assessment of microplastic fibre shedding behaviour and treatment effectiveness.

3.5 Result Interpretation

In this section, the results were discussed, and comparisons were made with combining analysis data.

3.5.1 Average Filter Mass Gain per Gram of Fabric

Figure 5 displays the average filter mass gain per gram of fabric among three washing cycles for each treatment group.

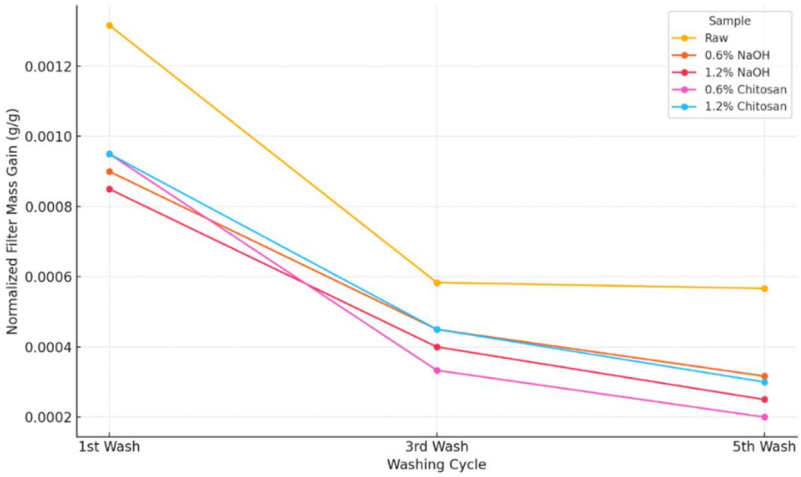

Figure 5. Average Filter Mass Gain per Gram of Fabric Across Washing Cycles

All samples demonstrate a clear decrease in microplastic fibre release from the first to the fifth wash, showing that the majority of fibre detachment occurs during the initial phases of laundering. This is corresponding to previously observed release behaviour in synthetic fabrics, where surface-bound and loosely attached fibres are released during initial cycles.

The raw (untreated) samples began with the largest normalized release around 0.00132 g/g in the first wash, which decreased to 0.00057 g/g by the fifth. All treated fabrics had lower beginning and end release values compared to raw sample, revealing that the treatment was effective.

Between treatments, 1.2% NaOH and 0.6% chitosan exhibited the lowest mass gains in the fifth wash (0.00025 g/g and 0.00020 g/g), showing significant reduction of microplastic fibre release throughout multiple cycles. While both NaOH and chitosan successfully reduced release, NaOH performed slightly more consistently across all washes, whereas chitosan had a steeper drop, implying that it may stabilize the fibre surface over time. The NaOH treatment is a surface modification, which reinforces fibre bonds and slows down release. In contrast, chitosan forms a surface coating that may detach more particles during early wash cycles.

These results validate that plasma-assisted treatments, especially NaOH and chitosan, are effective techniques for reducing microplastic fibre shedding. The observed reductions over time suggest that these treatments assist in longer-term release stabilization in repetitive laundering conditions.

3.5.2 Microplastic Fibre Count per Gram of Total Fabric Mass

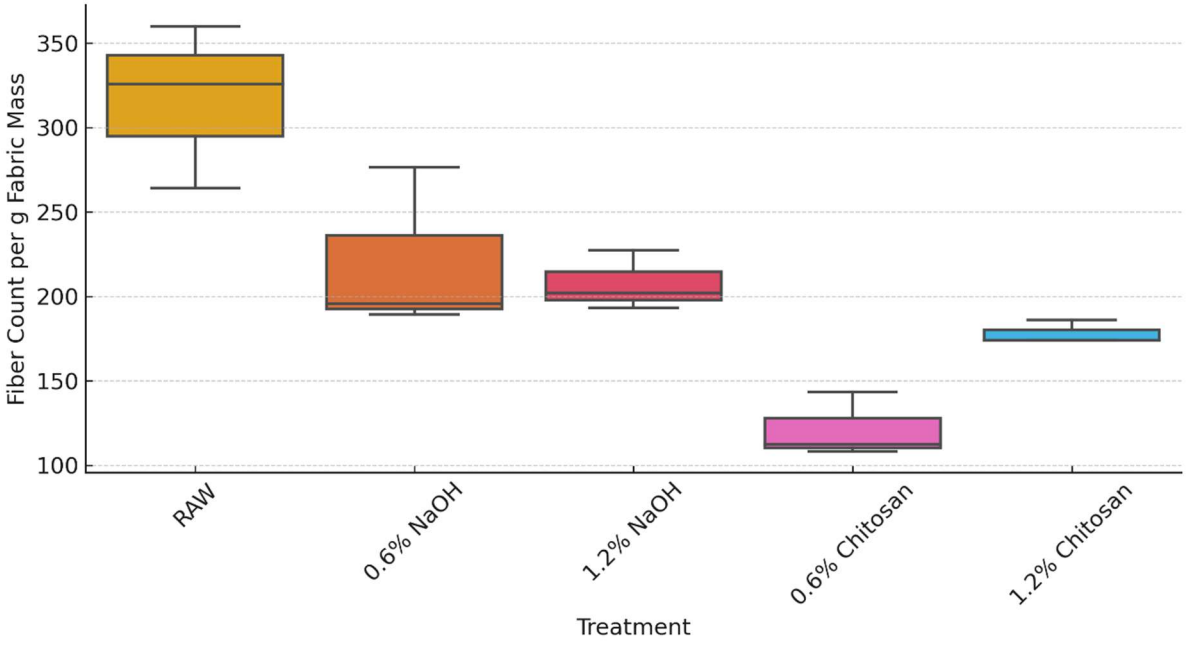

Figure 6. Microplastic fibre count per gram of total fabric mass

The number of microplastic fibres released during washing relative to the total initial mass of the fabric is exhibited in Figure 6. It provides an intensity-based view of fibre detachment, normalized by fabric size, and is particularly useful for comparing shedding rates across different treatment types while accounting for fabric weight.

Raw samples show the highest microplastic fibre count per gram of fabric, averaging around 300-350 fibres per gram. On the other hand, treated fabrics gave lower results. The average for the 1.2% NaOH samples is approximately 208 fibres/g, indicating a 34.4% reduction compared to raw samples. The 0.6% NaOH-treatment samples follow with 30.4% reduction. These results show that the NaOH pre-treatment significantly restricts fibre release by improving surface integrity or altering the morphology of polyester surfaces, resulting in more cohesive fibre bonding.

Chitosan-treated samples also demonstrate a reduction in fibre release. The 1.2 % chitosan group shows a reduction nearly 44%, while the 0.6 % chitosan group shows approximately 62% reduction compared to the RAW samples. However, chitosan treatments have slightly higher variability, and average fibre counts than NaOH treatments at the same dosage, reflecting a less significant effect on microplastic fibre detachment under the examined conditions.

Overall, the data show that surface treatments with NaOH and chitosan, particularly when plasma-assisted, are effective at lowering the amount of microplastic fibre shedding in polyester fabrics. NaOH is slightly better in minimizing fibre release relative to fabric mass, especially at higher concentrations, indicating that it is suitable for applications that require improved fibre preservation performance.

3.5.3 Filter mass gain per gram of fabric

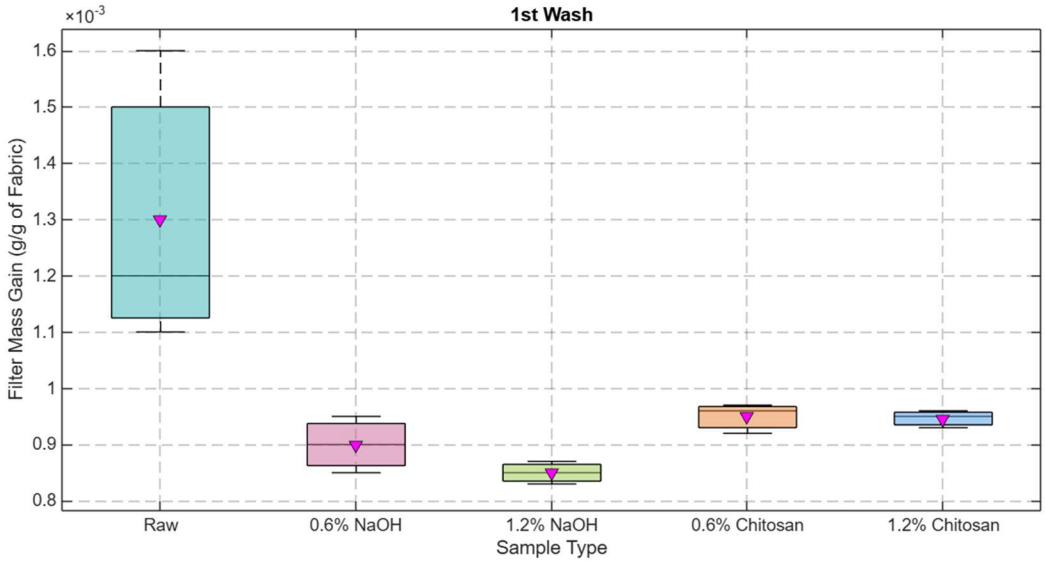

Figure 7. Filter mass gain per gram of fabric 1st wash

In the initial wash, all samples released the greatest amount of material (Figure 7). The raw group having the largest mass gain per gram of fabric. This is expected due to the detachment of loosely attached surface fibres and treatment residues. Both NaOH and chitosan treatments significantly decreased filter mass gain compared to raw, confirming their effectiveness of reducing early microplastic fibre release. NaOH-treated samples, particularly the 1.2%, had slightly lower values than chitosan-treated samples, showing a higher control of surface fibre detachment. The release of chitosan itself from the fabric during initial stages may be the reason of high filter mass of chitosan treated samples.

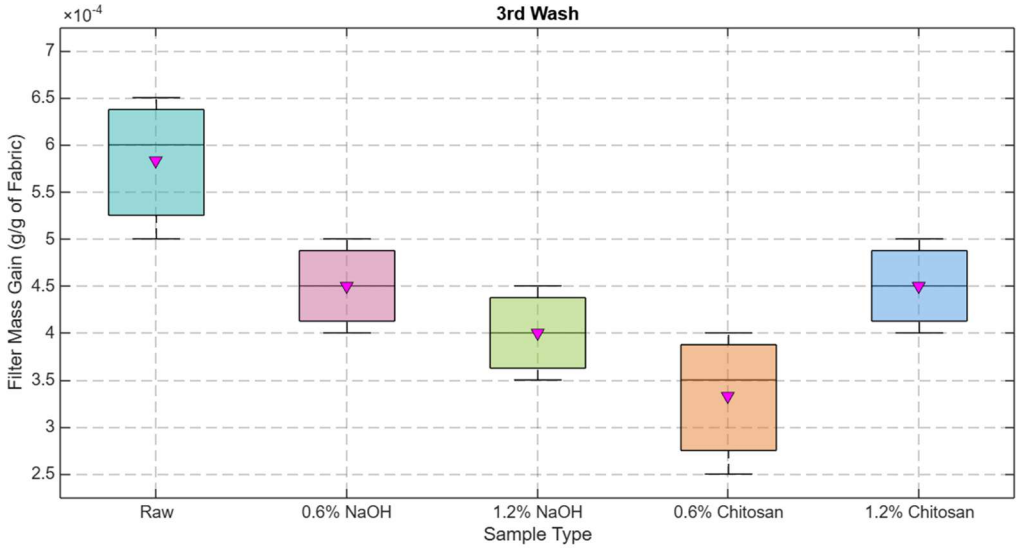

Figure 8. Filter mass gain per gram of fabric 3rd wash

By the third wash, filter mass gain values had decreased significantly among all samples, indicating a decrease in microplastic fibre release. The raw group maintained the highest average value, but all treated fabrics went through significant reductions. Notably, 0.6% chitosan displayed the greatest decline from the first wash, with a 64.9% reduction, followed by 1.2% NaOH with a 52.9% reduction. The 0.6% NaOH, 1.2% chitosan, and raw groups followed closely with reductions of 50.0%, 52.5%, and 55.1%, respectively.

These results show that surface treatments, specifically chitosan at 0.6% concentration, effectively decrease the amount of fibre shed in the washing. A commonality of results also indicates that the majority of weakly bound or fragile fibres and excess chitosan had been released by this stage, and treated samples achieved a more stable release pattern.

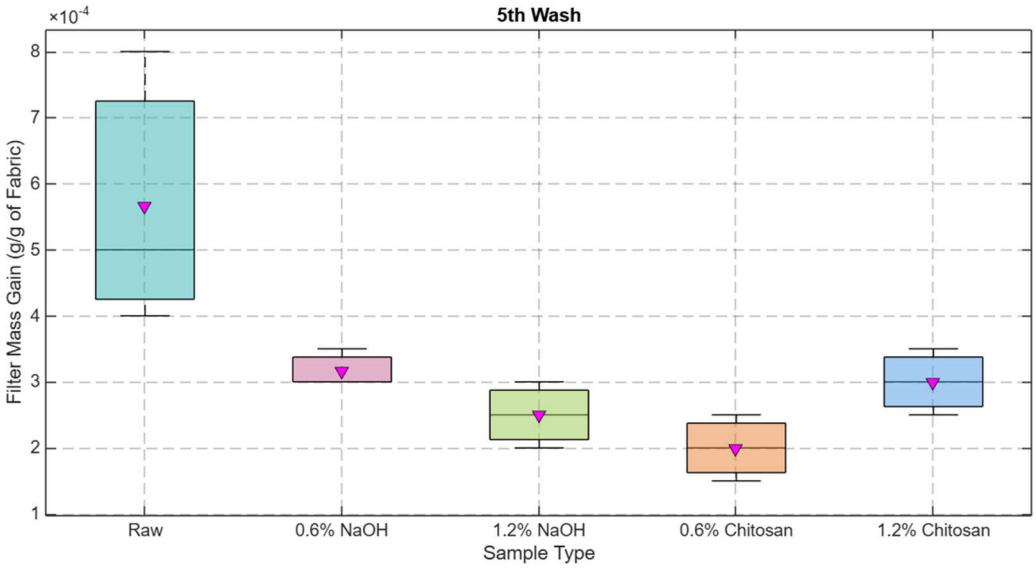

Figure 9. *Filter mass gain per gram of fabric 5th wash*

In the fifth wash, mass gain values reached their lowest levels across all groups, affirming that the majority of microplastic fibre release occurs early in the washing lifecycle. Compared to the first wash, raw samples saw a 56.4% reduction, while treated samples showed even more pronounced declines: 64.8% for 0.6% NaOH, 70.6% for 1.2% NaOH, 78.9% for 0.6% chitosan, and 68.3% for 1.2% chitosan.

Reductions from the third to fifth wash were also substantial in treated samples—0.6% chitosan dropped an additional 40.0%, 1.2% NaOH by 37.5%, and 1.2% Chitosan by 33.3%. In contrast, the raw fabric showed minimal change between these washes (2.9%), reinforcing that untreated fabrics release most fibres early, with sustained shedding afterward.

These trends confirm that surface treatments not only reduce initial fibre release but also help maintain lower, more stable shedding levels across repeated laundering cycles, even after the first wash removes any excess chitosan.

3.6 Length Distribution

The average fibre length measured in the untreated (Raw) samples was 0.82 mm. Following treatment with 0.6% NaOH, the average length was slightly higher at 0.86 mm. In contrast, the 1.2% NaOH group showed a lower average of 0.76 mm, indicating a significant reduction in fibre length compared to the raw sample. This indicates that the higher concentration of NaOH may have a degrading effect on fibre structure. The results can be seen in Figure 10.

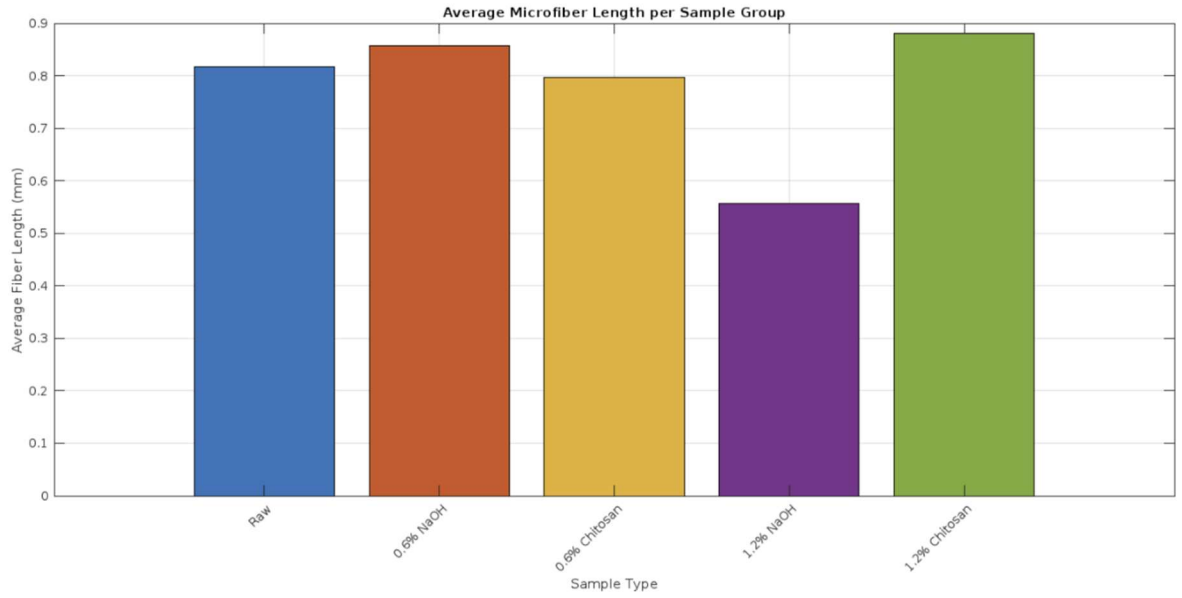

Figure 10. Average fibre length for each sample type

The 0.6% Chitosan treatment resulted in an average fibre length of 0.80 mm, which is close to the raw average, while the 1.2% Chitosan had a slightly higher average of 0.88 mm. Unlike the 1.2% NaOH, chitosan treatments did not lead to a decrease in fibre length, indicating better preservation of fibre structure during processing.

These results show that NaOH (especially at higher concentration) can shorten the microplastic fibres, chitosan treatments are more effective at preserving fibre length and decreasing structural damage.

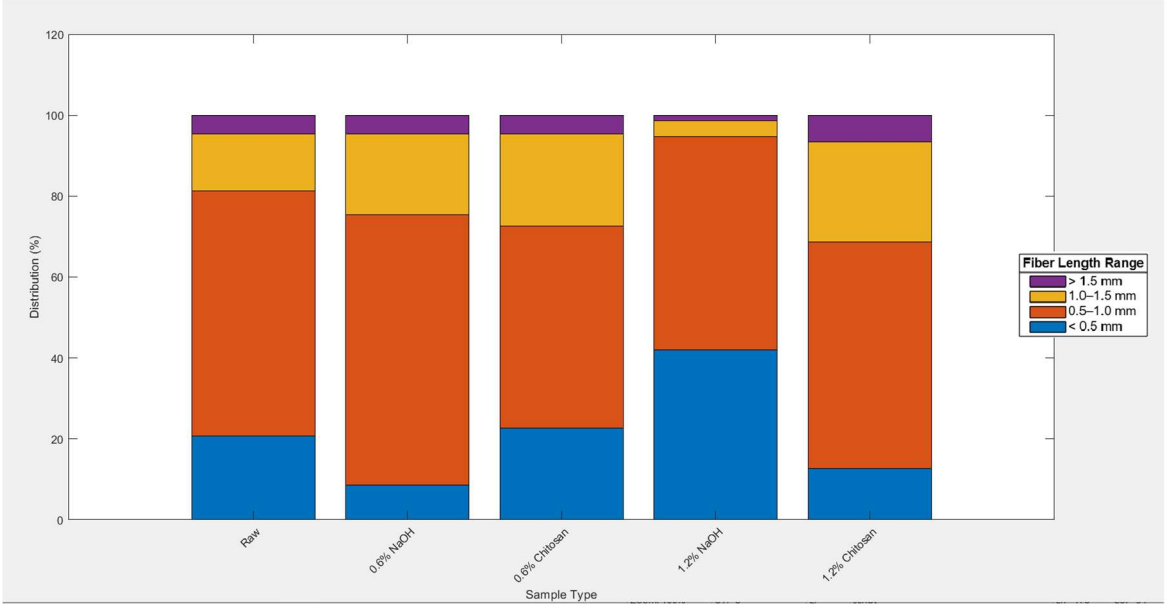

Figure 11. Fibre Length Distribution by Treatment Group

Figure 11 presents the percentage distribution of microplastic fibre lengths for each treatment group and raw sample. The outcomes show how different treatments affect fibre fragmentation and preservation during processing.

Raw samples presented a slightly balanced distribution, with a higher percentage of longer fibres, indicating minor structural changes. Whereas NaOH-treated groups, especially 1.2% treatment, showed shorter fibre lengths, indicating a higher rate of fibre breakage. Chitosan-treated samples, maintained a greater share of longer fibres, pointing to better preservation of fibre structure compared to NaOH.

These trends demonstrate how chemical treatments affect microplastic fibre morphology and support the evaluation of treatment methods based on their ability to maintain or alter fibre integrity.

3.7 FTIR Analysis

All sample groups went through Fourier Transform Infrared Spectroscopy (FTIR) analysis to determine the chemical structure of polyester fabric samples and evaluate any potential changes caused by surface treatments.

The spectra were captured between 700-4000 cm^{-1}, and both raw and processed data (baseline-corrected and smoothed) were analysed. FTIR scans were carried out with an ATR (Attenuated Total Reflectance) accessory, which allowed for precise surface characterization without requiring any further sample preparation.

Each sample was analysed separately. The corresponding FTIR spectra are displayed in different plots to differentiate the unprocessed and processed signal data. NaOH and Chitosan treated samples were compared to Raw sample for detecting changes and differences. The results presented show similar polyester fingerprints in all groups, with specific features such as the C=O stretching vibration near 1715 cm^{-1}, C-H bending around 800-700 cm^{-1}, and strong C-O-C ester bonds near 1270-1100 cm^{-1} (Paz & Sousa, 2024).

NaOH-treated textiles had minor shifts and intensity reductions near the ester and carbonyl peaks, especially at 1.2% concentration suggesting surface hydrolysis. Chitosan-treated fabrics showed minimal chemical changes supporting a physical coating effect rather than structural change.

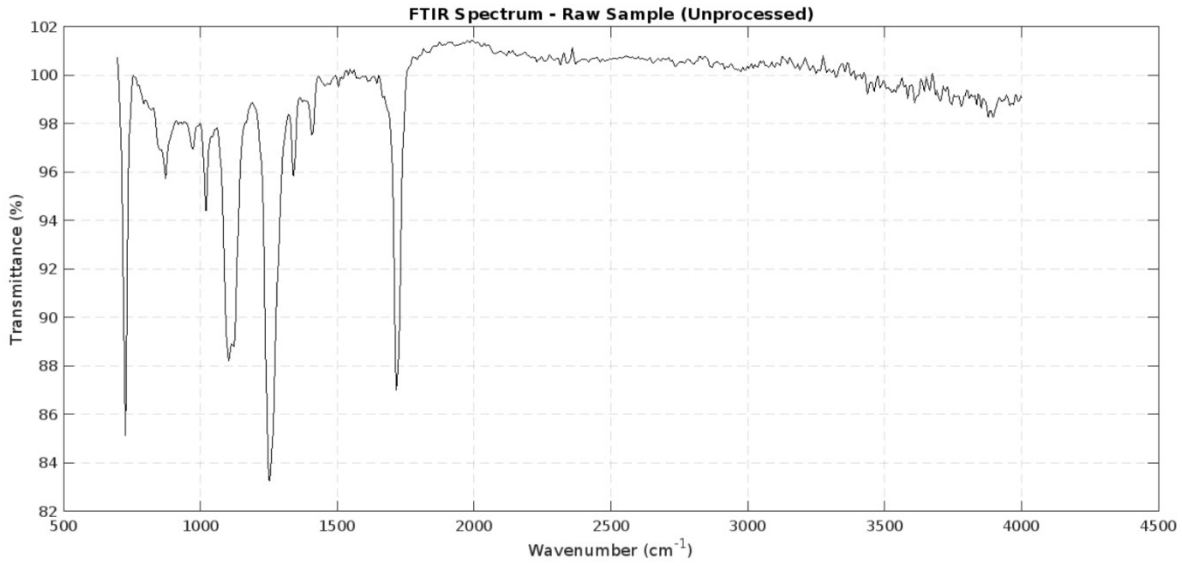

Figure 12. FTIR spectrum of Raw polyester sample

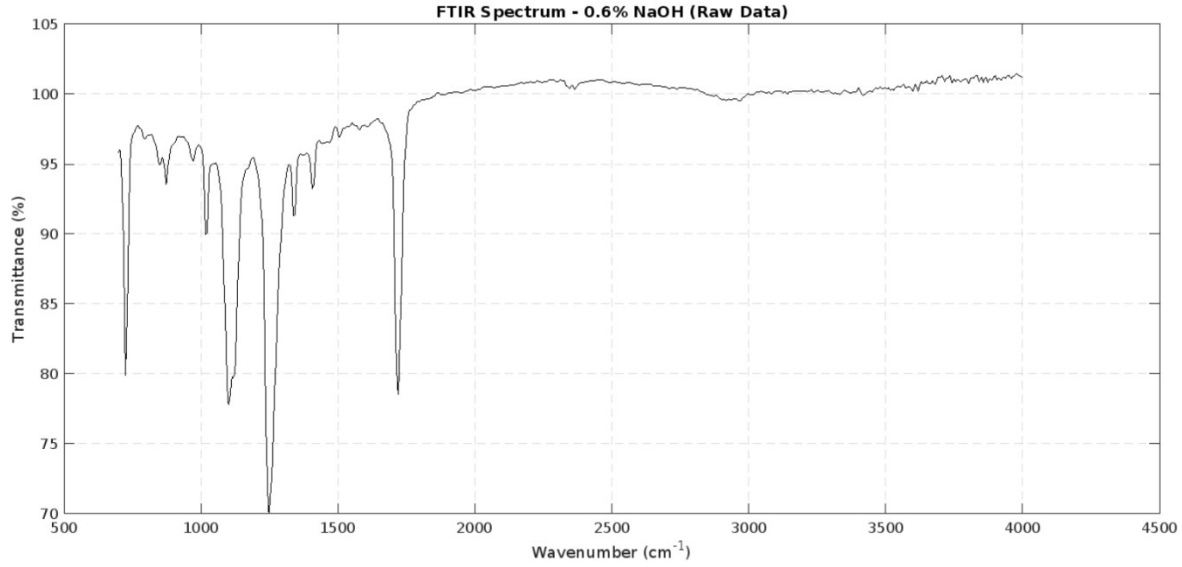

Figure 13. FTIR spectrum of 0.6% NaOH treated sample

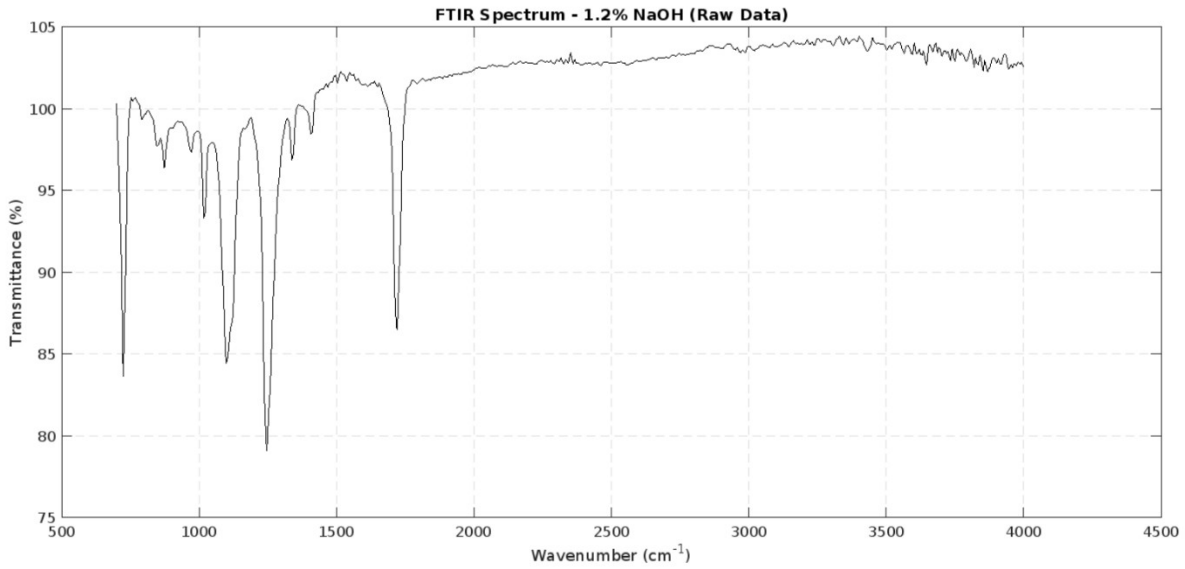

Figure 14. FTIR spectrum of 1.2% NaOH treated sample

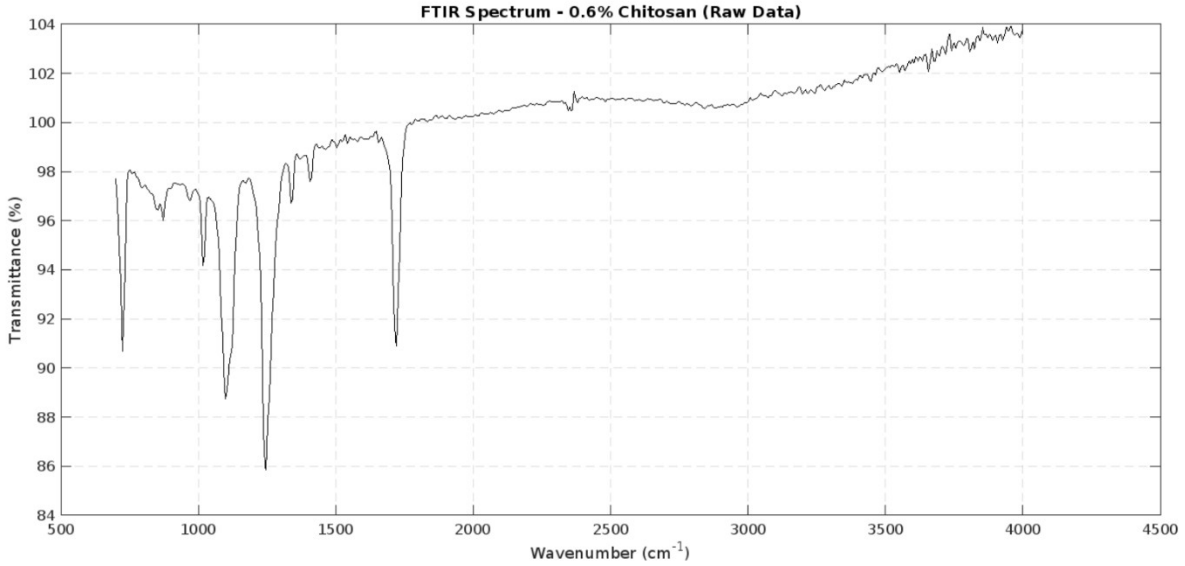

Figure 15. FTIR spectrum of 0.6% Chitosan treated sample

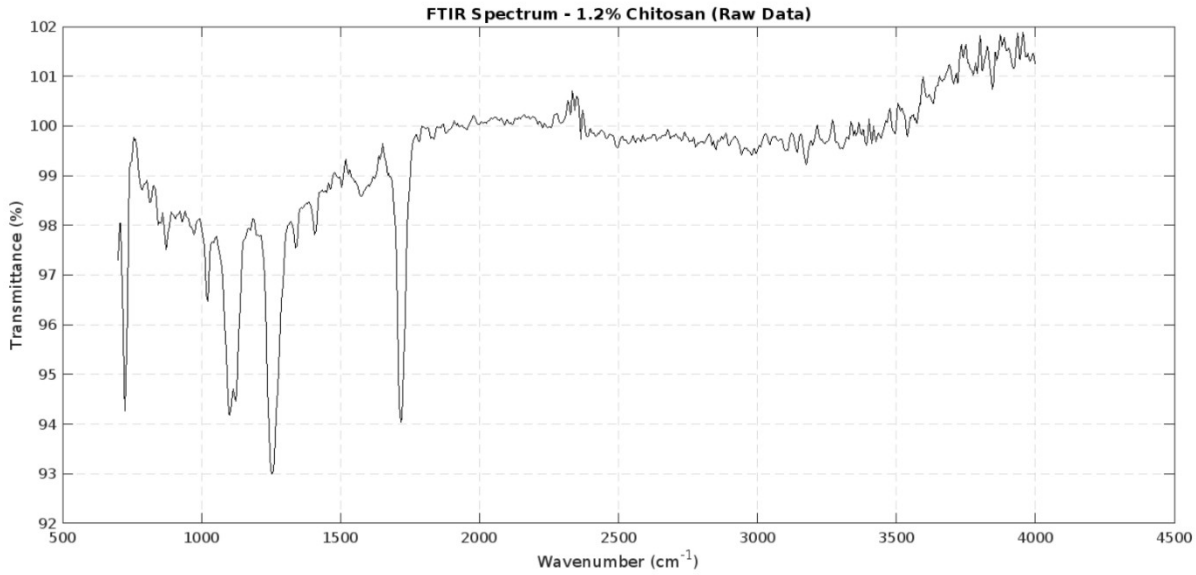

Figure 16. FTIR spectrum of 1.2% Chitosan treated sample

The RAW and treated samples show minor but significant differences (Figure 12-16). NaOH-treated fabrics display a small stretching or decrease in intensity around the ester (C-O-C) and carbonyl (C=O) bands, indicating partial surface hydrolysis or molecular rearrangement of ester groups. At higher NaOH concentrations (1.2%), the C=O peak near 1715 cm^{-1} shifts and changes shape (Cammarano et. al., 2013).

In contrast, chitosan-treated fabrics (0.6% and 1.2%) maintain the core polyester peaks with higher fidelity, though small variations can be seen in the lower-wavenumber region (<1200 cm^{-1}). This may result from weak hydrogen bonding or surface deposition of chitosan interacting with the polymer's surface groups. Chitosan-treated polyester fabrics also display additional peaks after 3500 cm^{-1} (−OH and −NH stretching) (Grgac et al., 2020) and presence of residual N-acetyl groups around 1645 cm−1 (C=O stretching of amide I) (Queiroz et al., 2014). Unlike NaOH treatment, chitosan does not appear to degrade or break the polyester structure but may introduce additional functional layers. Differences between the 0.6% and 1.2% chitosan-treated spectra are minimal, suggesting a threshold of chemical interaction or saturation at the fibre surface.

The absence of new peaks or significant spectrum changes in the core polyester region indicates that the treatments did not change the polyester's foundational chemical structure. This suggests that both NaOH and chitosan modifications affect the fibres' surface rather than penetrating or changing the molecular backbone.

Overall, FTIR analysis confirms the polyester content of all samples and indicates that NaOH treatment causes noticeable changes to surface chemistry, specifically at higher doses. Chitosan treatment, on the other hand, displays more surface-adherent and less chemically disruptive, indicating that it functions as a biopolymer coating rather than a reactive modifier.

3.8 SEM Analysis

SEM images analysed from (i) dry fabric samples before experimental washing, (ii) washed and dried samples to assess the effect of washing on fibre morphology, (iii) abraded samples after washing to examine mechanical durability under stress, and (iv) glass fibre filter papers used for capturing shed fibres. This analysis is made to investigate the morphology of the released microplastic fibres.

NaOH and chitosan treated samples, showed significantly improved resistance to surface damage, with fewer broken filaments and smoother zones. These results were correlated with microplastic fibre release and mass gain data for a thorough assessment of the treatment's effectiveness in reducing mechanical wear-caused fibre shedding.

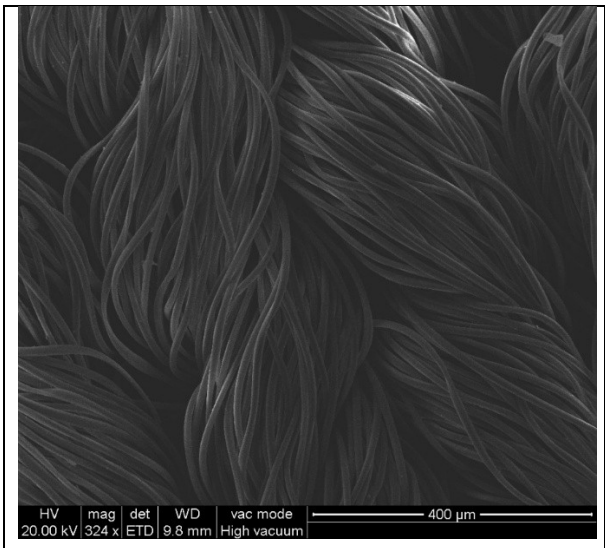

Figure 17. Untreated raw polyester fabric, before washing trials SEM image

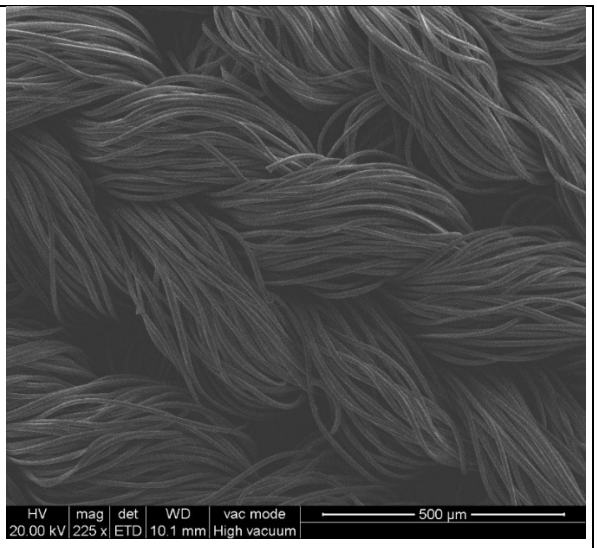

Figure 18. Untreated raw polyester fabric, after washing trials SEM image

Pre-wash sample show smooth, continuous fibres with minimal surface damage. Boundaries are well-defined and compact, indicating the structural integrity before going through mechanical or chemical processes. Post-wash fibres show early signs of surface disruption.

Minimal fibrillation and decrease of adhesion between fibres can be observed, indicating microstructural weakening under repeated mechanical stress (Figure 17-18).

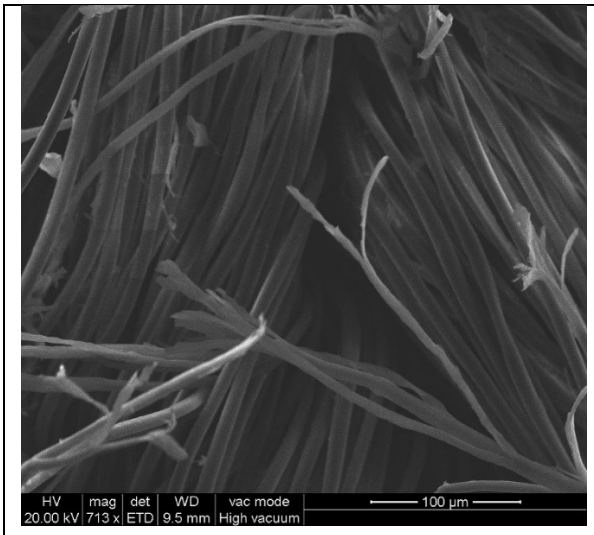

Figure 19. Untreated raw polyester fabric, after washing trials and abrasion test SEM image

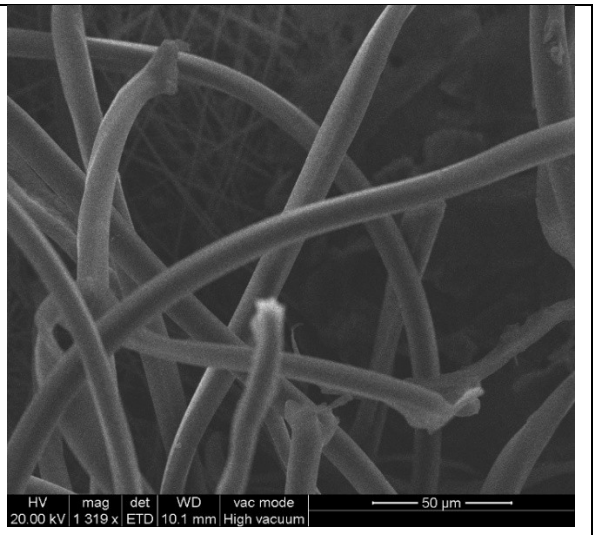

Figure 20. Glass fibre filter SEM image after filtering wash water from raw polyester sample

Significant surface damage is visible on the abraded sample as seen in figure 19. Multiple fibres show fibrils and surface cracking indicating fragmentation. The signs of wear show the lack of treatment on the raw fabric. On the filter paper, presence of short fragments and thin fibrils leads to intensive fibre breakage and shedding, consistent with mechanical degradation in untreated fabrics.

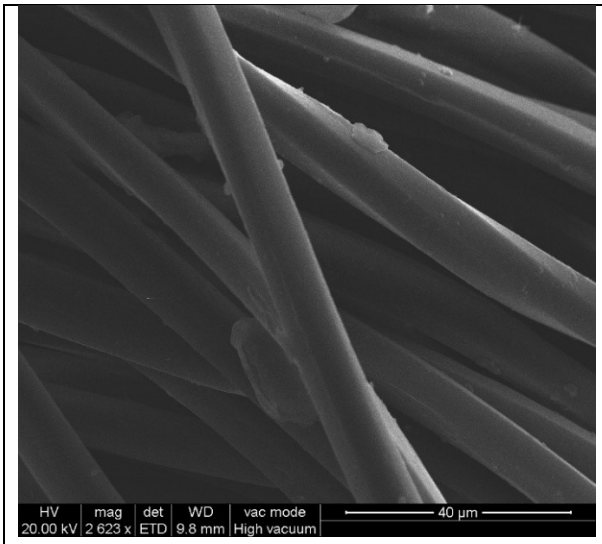

Figure 21. Plasma-treated 0.6% NaOH polyester fabric, before washing trials SEM image

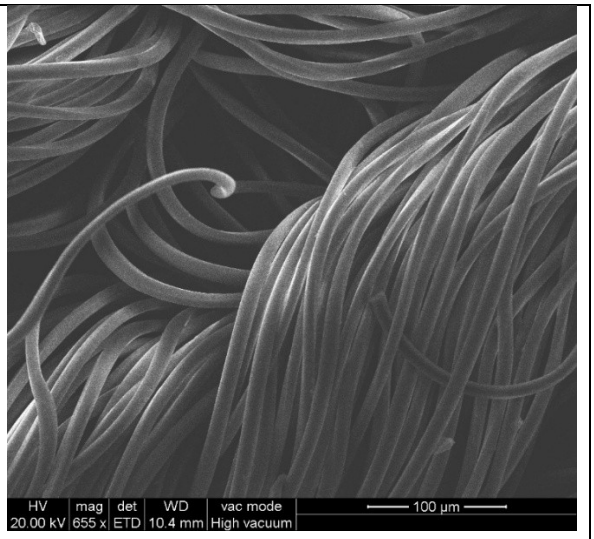

Figure 22. Plasma-treated 0.6% NaOH polyester fabric, after washing trials SEM image

The fibre surface on pre-wash sample seems slightly disrupted due to alkaline pretreatment but keep continuity and structural cohesion. Indicating controlled chemical modification (Figure 21). On the post-wash sample, fibres show minimal fibrillation and maintain smoother profiles compared to raw samples. The filament compactness appears decent, suggesting that the NaOH treatment provides resistance against washing caused degradation (Figure 22).

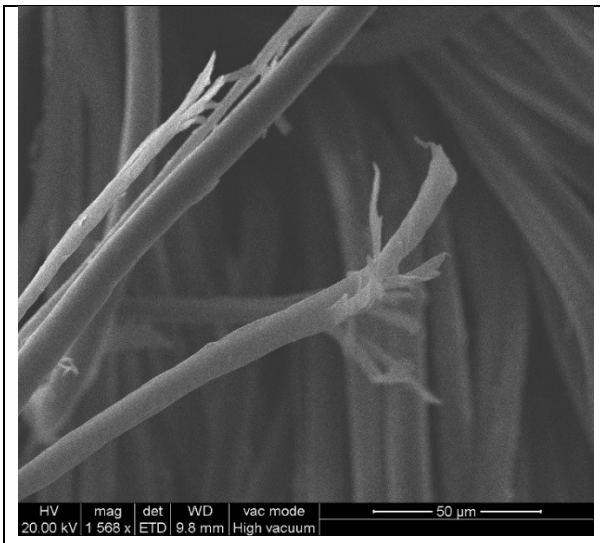

Figure 23. Plasma-treated 0.6% NaOH polyester fabric, after washing trials and abrasion test SEM image

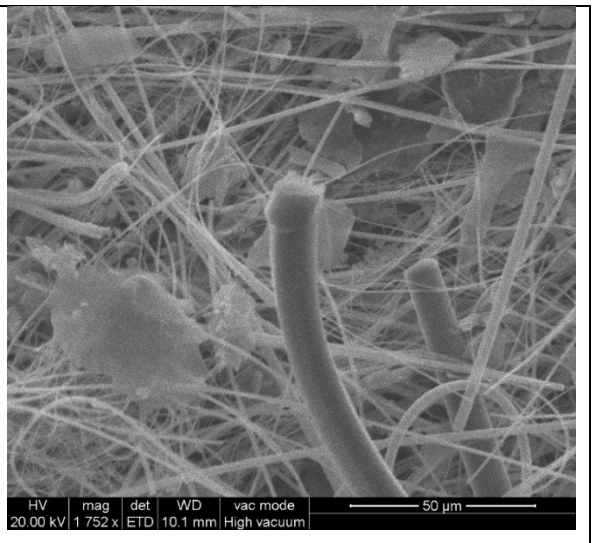

Figure 24. Glass fibre filter SEM image after filtering wash water from Plasma-treated 0.6% NaOH polyester fabric

Even though the exposure to abrasive forces, fibres demonstrate better defined edges than the raw samples (Figure 23). Fibrillation is marginally reduced, indicating the protective effect of the alkaline treatment under mechanical stress. Compared to the raw sample filter, Figure 24 shows fewer short particles. The fibres appear more intact, supporting reduced fragmentation.

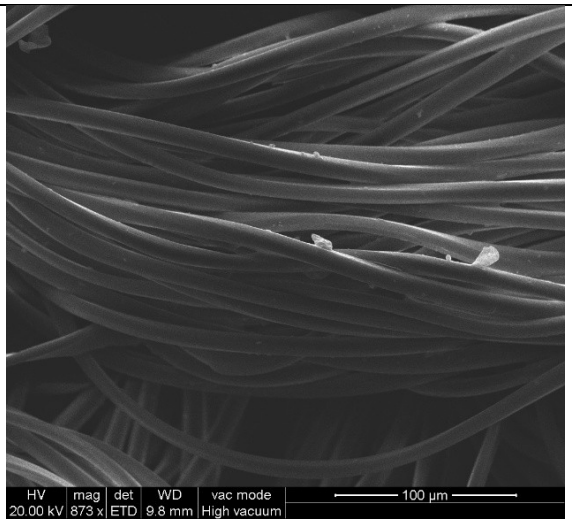

Figure 25. Plasma-treated 0.6% Chitosan polyester fabric, before washing trials SEM image

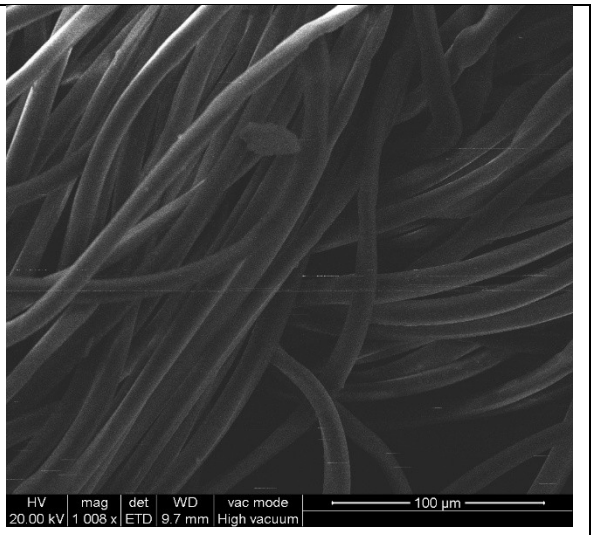

Figure 26. Plasma-treated 0.6% Chitosan polyester fabric, after washing trials SEM image

The pre-wash fibres show a relatively uniform and smooth surface (Figure 25). The treatment appears gentler than NaOH, preserving fibre continuity. Fibre surfaces of post-wash samples look smooth, showing minimal surface wear or fibrillation (Figure 26). When compared to 0.6% NaOH, 0.6% chitosan sample shows slightly smoother surfaces with low levels of damage.

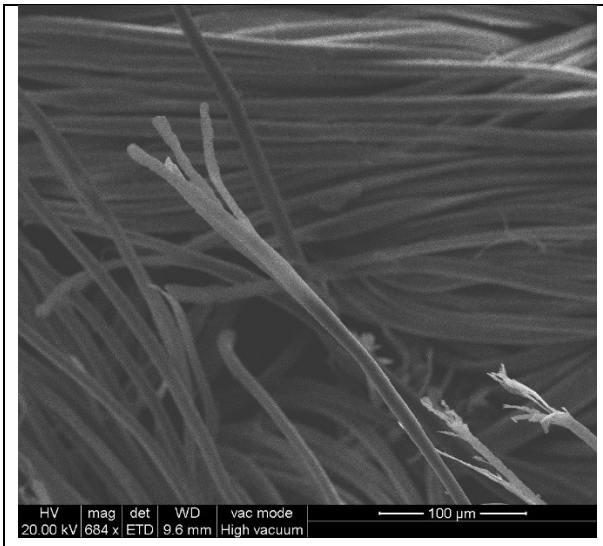

Figure 27. Plasma-treated 0.6% Chitosan polyester fabric, after washing trials and abrasion test SEM image

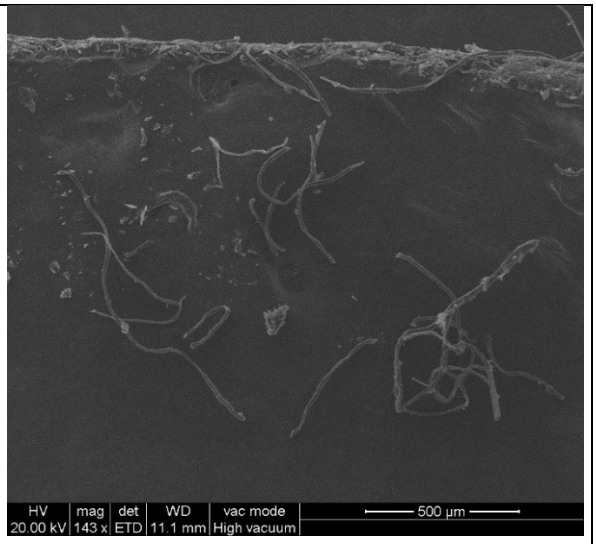

Figure 28. Glass fibre filter SEM image after filtering wash water from Plasma-treated 0.6% Chitosan polyester fabric

Even under mechanical stress and abrasion, most of the fibres maintain structural integrity. There is slightly better surface prevention than the 0.6% NaOH-treated sample, suggesting significant resistance to wear-caused fragmentation. Microplastic fibres on the filter appear less fragmented than raw fabric microplastic fibres. Compared to the 0.6% NaOH filter, fibre edges may be slightly more curled or irregular, but the average particle size distribution remains controlled.

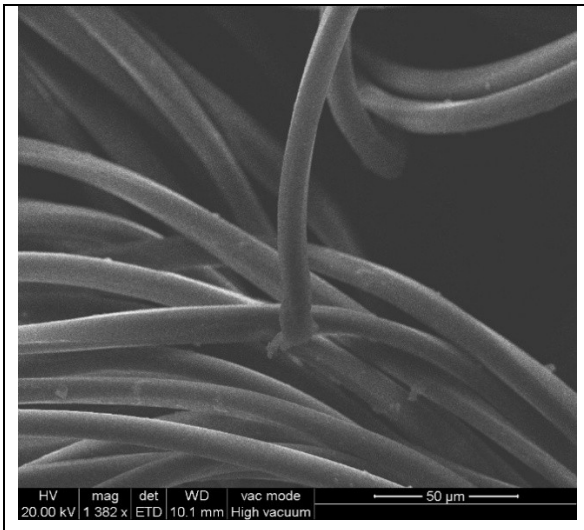

Figure 29. Plasma-treated 1.2% NaOH polyester fabric, before washing trials SEM image

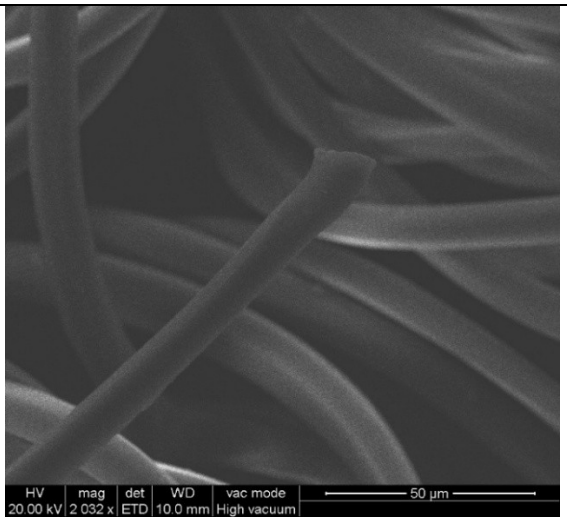

Figure 30. Plasma-treated 1.2% NaOH polyester fabric, after washing trials SEM image

Fibre surfaces on the pre-wash fabric display more visible damage compared to the 0.6% NaOH version (Figure 29). While the structural integrity remains intact, the alkaline treatment appears to have slightly roughened the surface. On the post-wash fabric, fibres maintain continuity (Figure 30). Compared to raw fabric, there is reduced fibrillation; compared to 0.6% NaOH, the effects are similar.

Figure 31. Plasma-treated 1.2% NaOH polyester fabric, after washing trials and abrasion test SEM image

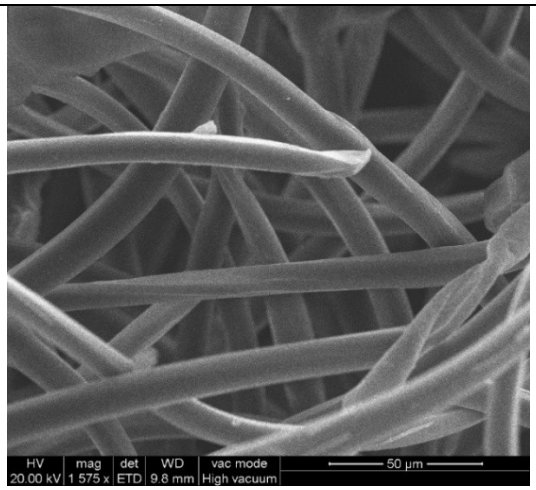

Figure 32. Glass fibre filter SEM image after filtering wash water from Plasma-treated 1.2% NaOH polyester fabric

Fibril formation is evident in abraded fabrics (Figure 31). Fibres appear more worn than 0.6% NaOH-treated samples, suggesting that the higher NaOH concentration may marginally affect durability under friction. The fibre fragments on filter paper appear controlled in length and density, showing a relatively uniform morphology. There is a modest reduction in fragmentation compared to the raw sample, and the appearance is similar to or slightly improved from the 0.6% NaOH case (Figure 32).

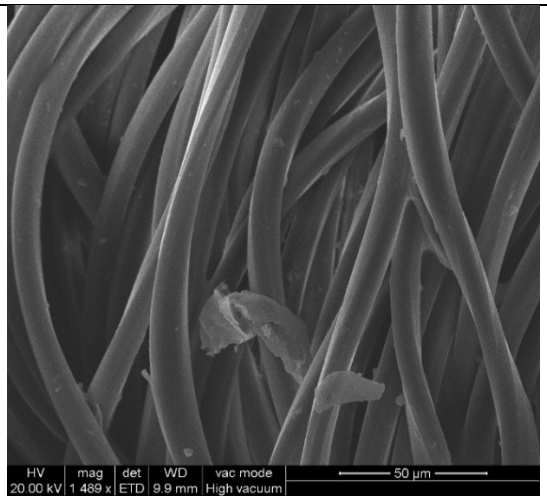

Figure 33. Plasma-treated 1.2% Chitosan polyester fabric, before washing trials SEM image

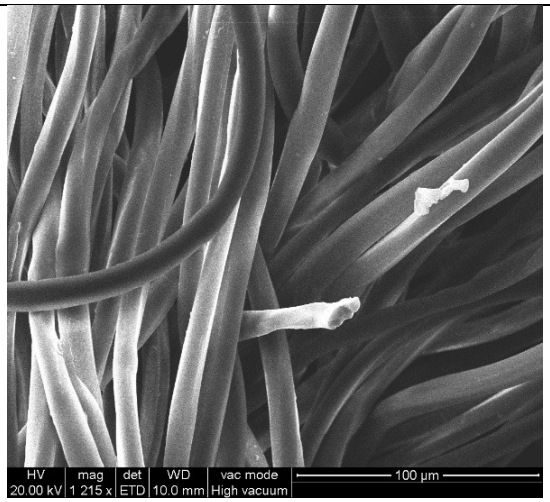

Figure 34. Plasma-treated 1.2% Chitosan polyester fabric, after washing trials SEM image

Fibres on pre-wash sample seem smoother compared to ones treated with NaOH, potentially due to surface film development. The coating is noticeable compared to 0.6% chitosan, although it has no visible impact on the fibre structure (Figure 33). On the post wash fabric, the fibres remain intact and smooth, with barely noticeable fibrillation. Surface damage is less noticeable than in raw and NaOH-treated fabrics (Figure 34). The results are similar to the 0.6% chitosan-treated sample, indicating minimal extra benefit from the higher concentration.

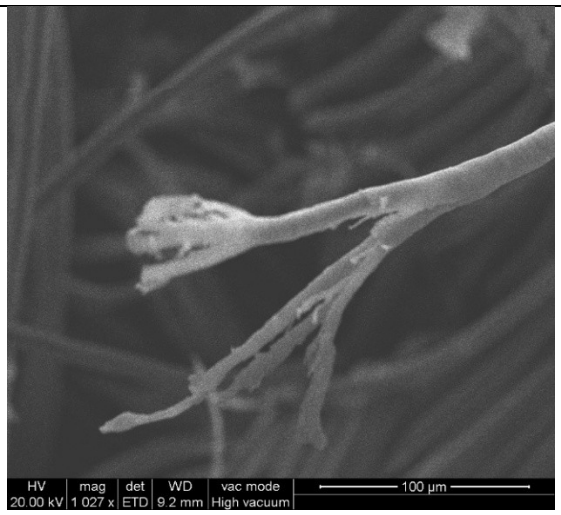

Figure 35. Plasma-treated 1.2% Chitosan polyester fabric, after washing trials and abrasion test SEM image

Figure 36. Glass fibre filter SEM image after filtering wash water from Plasma-treated 1.2% Chitosan polyester fabric

On the abraded fabric, some fibre edges show signs of wear, but overall integrity is conserved. The chitosan-treated sample appeared slightly less abraded than the 1.2% NaOH-treated fabric, although the difference is fairly low (Figure 35). The captured fibres on filter are largely intact with fewer visible fragments (Figure 36). The results align closely with 0.6% chitosan in terms of fragment size and count. Compared to NaOH filters, the morphology may suggest slightly less mechanical breakup.

SEM analysis revealed that untreated polyester fabrics experienced notable surface damage and fibre fragmentation after washing and abrasion. On the contrary, NaOH and chitosan-treated samples showed smoother surfaces and better fibre integrity. Both treatments decreased visible wear. SEM observations indicate that no visible surface damage occurred on NaOH-treated samples. This is likely due to the low concentration of NaOH used, which may have been sufficient to modify the PET surface without causing etching. The results obtained correspond with fibre count and mass data, suggesting that surface treatments can reduce microplastic fibre release while enhancing structural durability.
The relatively low surface roughening found in chitosan-treated fibres might be the result of cortification, a densification and hardening activity that improves the outer structure's resistance to mechanical stress.

4. Conclusion

This study deeply examined the efficiency of plasma-assisted surface treatments in reducing microplastic fibre release from polyester-based knitted fabrics during domestic washing. A variety of analysis were carried out to compare the structural, physical, and morphological responses of treated and untreated samples.

The combination of gravimetric measurements, microplastic fibre counting and length analysis, spectroscopic profiling (FTIR), and morphological inspection (SEM) provided a multidimensional understanding of the fibre shedding mechanisms and the influence of different chemical treatments.

Overall, the analyses confirmed the reliability of the surface treatments, significantly reduce microplastic fibre release during washing cycles. The gravimetric data, although vulnerable to uncertainties from non-fibrous elements and environmental factors, presented a reliable foundation for comparing fibre release between treatments. Microplastic fibre count and length data collected by high-resolution imaging and ImageJ analysis provided more detailed data on fibre fragmentation. FTIR spectra validated the used fabrics' polyester composition and the presence of chemical compounds after treatment, while SEM images provided important qualitative evidence of surface damage, fibre integrity, and treatment effects at the microstructural level. These procedures were supportive and essential in determining the intensity and nature of microplastic fibre shedding.

The results support the hypothesis that surface treatments involving plasma surface activation followed by NaOH or chitosan application reduce microplastic fibre release during washing. The combination of chemical agents with plasma appears to promote better surface adhesion and enhanced surface cohesion of fibres, leading to lower fibre fragmentation. This was evident in both the gravimetric filter mass gain and the count of fibres released. Furthermore, FTIR analysis showed no evidence of alterations in the polymer structure, suggesting that these treatments affect only the outer surfaces of the fibres, enhancing hydrophilicity and potentially creating surface effects, as supported by previous literature findings.

Both treatment methods performed well in decreasing microplastic fibre shedding. Lower fibre counts in treated samples, especially in chitosan samples, indicate a significant improvement in reducing microplastic shedding. Mass-based analysis also confirms reduced microfiber release from pretreated samples.

SEM pictures revealed minor changes in surface roughness and abrasion effects; NaOH-treated fabrics displaying smoother textures and fewer fragmentation. Chitosan treatments resulted in rougher surface morphology, possibly due to surface-level cortification effect. Although, both treatments enhanced the fabric's durability compared to untreated samples.

However, this study does not certainly favour one treatment over another. The variation in shedding behaviour and the effects of different concentrations suggest that further investigation is required. Future research could investigate alternative natural treatments, agent combinations, or new surface-modifying technologies. Long-term wear simulations, water quality studies, and industrial-scale tests would help to improve the implementation of these methods in sustainable textile manufacturing.

In conclusion, while this study confirms the effectiveness of plasma-assisted NaOH and chitosan treatments in reducing microplastic fibre release, further research to optimize these surface modification techniques is needed. The results obtained during this research contribute to the ongoing development of textile solutions aimed at mitigating microplastic pollution and promoting more sustainable fabric production.

5. References

[1]. Akyildiz, S. H., Sezgin, H., Yalcin, B., & Yalcin-Enis, I. (2022). Optimization of the textile wastewater pretreatment process in terms of organics removal and microplastic detection. Journal of Cleaner Production, 384, 135637. https://doi.org/10.1016/j.jclepro.2022.135637

[2]. Akyildiz, S. H., Bellopede, R., Sezgin, H., Yalcin-Enis, I., Yalcin, B., & Fiore, S. (2022). Detection and Analysis of Microfibers and Microplastics in Wastewater from a Textile Company. Microplastics, 1(4), 572–586. https://doi.org/10.3390/microplastics1040040

[3]. Akyildiz, S. H., Fiore, S., Bruno, M., Sezgin, H., Yalcin-Enis, I., Yalcin, B., & Bellopede, R. (2024). Release of microplastic fibers from synthetic textiles during household washing. Environmental Pollution, 357, 124455. https://doi.org/10.1016/j.envpol.2024.124455

[4]. Ali, A., Qamer, S., Shahid, M., Tomkova, B., Khan, M. Z., Militky, J., Wiener, J., & Venkataraman, M. (2024). Micro- and Nanoplastics Produced from Textile Finishes: A Review. Langmuir. https://doi.org/10.1021/acs.langmuir.4c00552

[5]. Allen, E., Henninger, C. E., Wood, J., Garforth, A., & Asuquo, E. (2024). Designing Out Microplastic Pollution Released from Textiles and Apparel During Laundering. Cambridge Prisms Plastics, 2. https://doi.org/10.1017/plc.2024.20

[6]. Belzagui, F., Buscio, V., Gutiérrez-Bouzán, C., & Vilaseca, M. (2020b). Cigarette butts as a microfiber source with a microplastic level of concern. The Science of the Total Environment, 762, 144165. https://doi.org/10.1016/j.scitotenv.2020.144165

[7]. Boucher, J., & Friot, D. (2017). Primary microplastics in the oceans: A global evaluation of sources. https://doi.org/10.2305/iucn.ch.2017.01.en

[8]. Cai, Y., Mitrano, D. M., Hufenus, R., & Nowack, B. (2021). Formation of Fiber Fragments during Abrasion of Polyester Textiles. Environmental Science & Technology, 55(12), 8001–8009. https://doi.org/10.1021/acs.est.1c00650

[9]. Cammarano, A., Luca, G., & Amendola, E. (2012). Surface modification and adhesion improvement of polyester films. Open Chemistry, 11(1), 35–45. https://doi.org/10.2478/s11532-012-0135-x

[10]. Choi, J., Jeong, Y., Kim, S., Lee, B., Ariyasiri, K., Kim, H., Jung, S., Hwang, K., Choi, T., Park, C. O., Huh, W., Carl, M., Rosenfeld, J. A., Raskin, S., Ma, A., Gecz, J., Kim, H., Kim, J., Shin, H., . . . Kim, C. (2018). Targeted knockout of a chemokine-like gene increases anxiety and fear responses. Proceedings of the National Academy of Sciences, 115(5). https://doi.org/10.1073/pnas.1707663115

[11]. Choi, S., Kwon, M., Park, M., & Kim, J. (2021). Analysis of Microplastics Released from Plain Woven Classified by Yarn Types during Washing and Drying. Polymers, 13(17), 2988. https://doi.org/10.3390/polym13172988

[12]. Cox, K. D., Covernton, G. A., Davies, H. L., Dower, J. F., Juanes, F., & Dudas, S. E. (2019). Human consumption of microplastics. Environmental Science & Technology, 53(12), 7068–7074. https://doi.org/10.1021/acs.est.9b01517

[13]. De Falco, F., Di Pace, E., Cocca, M., & Avella, M. (2019). The contribution of washing processes of synthetic clothes to microplastic pollution. Scientific Reports, 9(1). https://doi.org/10.1038/s41598-019-43023-x

[14]. Dris, R., Gasperi, J., Mirande, C., Mandin, C., Guerrouache, M., Langlois, V., & Tassin, B. (2016). A first overview of textile fibers, including microplastics, in indoor and outdoor environments. Environmental Pollution, 221, 453–458. https://doi.org/10.1016/j.envpol.2016.12.013

[15]. El-Kheir, A. A., El-Ghany, N. A., Fahmy, M., Aboras, S., & El-Gabry, L. (2019). Functional finishing of polyester fabric using bentonite Nano-Particles. Egyptian Journal of Chemistry, 63(1), 85–99. https://doi.org/10.21608/ejchem.2019.20404.2223

[16]. European Environment Agency. (2019). Plastic in textiles: towards a circular economy for synthetic textiles in Europe.

[17]. Frias, J., & Nash, R. (2018b). Microplastics: Finding a consensus on the definition. Marine Pollution Bulletin, 138, 145–147. https://doi.org/10.1016/j.marpolbul.2018.11.022

[18]. Geiser, M., Rothen-Rutishauser, B., Kapp, N., Schürch, S., Kreyling, W., Schulz, H., Semmler, M., Hof, V. I., Heyder, J., & Gehr, P. (2005). Ultrafine particles cross cellular membranes by nonphagocytic mechanisms in lungs and in cultured cells. Environmental Health Perspectives, 113(11), 1555–1560. https://doi.org/10.1289/ehp.8006

[19]. GESAMP. (2015). Sources, fate and effects of microplastics in the marine environment: Part one of a global assessment (IMO/FAO/UNESCO-IOC/UNIDO/WMO/IAEA/UN/UNEP/UNDP Joint Group of Experts on the Scientific Aspects of Marine Environmental Protection, Reports and Studies No. 90). International Maritime Organization. https://www.gesamp.org/publications/microplastics-in-the-marine-environment-part-1

[20]. Grgac, S. F., Tarbuk, A., Dekanić, T., Sujka, W., & Draczyński, Z. (2020). The Chitosan Implementation into Cotton and Polyester/Cotton Blend Fabrics. Materials, 13(7), 1616. https://doi.org/10.3390/ma13071616

[21]. Hartline, N. L., Bruce, N. J., Karba, S. N., Ruff, E. O., Sonar, S. U., & Holden, P. A. (2016). Microfiber Masses Recovered from Conventional Machine Washing of New or Aged Garments. Environmental Science & Technology, 50(21), 11532–11538. https://doi.org/10.1021/acs.est.6b03045

[22]. Henry, B., Laitala, K., & Klepp, I. G. (2018). Microfibres from apparel and home textiles: Prospects for including microplastics in environmental sustainability assessment. The Science of the Total Environment, 652, 483–494. https://doi.org/10.1016/j.scitotenv.2018.10.166

[23]. Kang, H., Park, S., Lee, B., Ahn, J., & Kim, S. (2021). Impact of Chitosan Pretreatment to Reduce Microfibers Released from Synthetic Garments during Laundering. Water, 13(18), 2480. https://doi.org/10.3390/w13182480

[24]. Koelmans, A. A., Nor, N. H. M., Hermsen, E., Kooi, M., Mintenig, S. M., & De France, J. (2019). Microplastics in freshwaters and drinking water: Critical review and assessment of data quality. Water Research, 155, 410–422. https://doi.org/10.1016/j.watres.2019.02.054

[25]. Labay, C., Canal, J. M., & Canal, C. (2011b). Relevance of surface modification of polyamide 6.6 fibers by air plasma treatment on the release of caffeine. Plasma Processes and Polymers, 9(2), 165–173. https://doi.org/10.1002/ppap.201100077

[26]. McIlwraith, H. K., Lin, J., Erdle, L. M., Mallos, N., Diamond, M. L., & Rochman, C. M. (2018). Capturing microfibers – marketed technologies reduce microfiber emissions from washing machines. Marine Pollution Bulletin, 139, 40–45. https://doi.org/10.1016/j.marpolbul.2018.12.012

[27]. Monira, S., Roychand, R., Bhuiyan, M. A., & Pramanik, B. K. (2023). Role of water shear force for microplastics fragmentation into nanoplastics. Environmental Research, 237, 116916. https://doi.org/10.1016/j.envres.2023.116916

[28]. Muthu, M., Gopal, J., Kim, D., & Sivanesan, I. (2021). Reviewing the Impact of Vehicular Pollution on Road-Side Plants—Future Perspectives. Sustainability, 13(9), 5114. https://doi.org/10.3390/su13095114

[29]. Napper, I. E., & Thompson, R. C. (2016). Release of synthetic microplastic plastic fibres from domestic washing machines: Effects of fabric type and washing conditions. Marine Pollution Bulletin, 112(1–2), 39–45. https://doi.org/10.1016/j.marpolbul.2016.09.025

[30]. Pauly, D., Christensen, V., Dalsgaard, J., Froese, R., & Torres, F. (1998). Fishing down marine food webs. Science, 279(5352), 860–863. https://doi.org/10.1126/science.279.5352.860

[31]. Paz, M. L., & Sousa, C. (2024). Discrimination and quantification of cotton and polyester textile samples using Near-Infrared and Mid-Infrared spectroscopies. *Molecules*, *29*(15), 3667. https://doi.org/10.3390/molecules29153667

[32]. Periyasamy, A. P., & Tehrani-Bagha, A. (2022). A review on microplastic emission from textile materials and its reduction techniques. Polymer Degradation and Stability, 199, 109901. https://doi.org/10.1016/j.polymdegradstab.2022.109901

[33]. Periyasamy, A. P., & Tehrani-Bagha, A. (2022b). A review on microplastic emission from textile materials and its reduction techniques. Polymer Degradation and Stability, 199, 109901. https://doi.org/10.1016/j.polymdegradstab.2022.109901

[34]. Pirc, U., Vidmar, M., Mozer, A., & Kržan, A. (2016). Emissions of microplastic fibers from microfiber fleece during domestic washing. Environmental Science and Pollution Research, 23(21), 22206–22211. https://doi.org/10.1007/s11356-016-7703-0

[35]. Ramasamy, R., & Subramanian, R. B. (2023). Microfiber mitigation from synthetic textiles — impact of combined surface modification and finishing process. Environmental Science and Pollution Research, 30(17), 49136–49149. https://doi.org/10.1007/s11356-023-25611-7

[36]. Queiroz, M. F., Melo, K., Sabry, D., Sassaki, G., & Rocha, H. (2014). Does the use of chitosan contribute to oxalate kidney stone formation? Marine Drugs, 13(1), 141–158. https://doi.org/10.3390/md13010141

[37]. Rathinamoorthy, R., & Balasaraswathi, S. R. (2023). Impact of quantification method on microfiber assessment–A comparative analysis between mass and count based methods. Journal of Environmental Management, 347, 119040.

[38]. Rathinamoorthy, R., & Balasaraswathi, S. R. (2024). Microfibre Pollution from Textiles. In CRC Press eBooks. https://doi.org/10.1201/9781003331995

[39]. Rathinamoorthy, R., Nivruthi, K., Puvisha, R., Suganthini, S., & Balasaraswathi, S. R. (2024). A Novel Approach to Combat Microfiber Release from Polyester Textiles Through Surface Treatment. Fibers and Polymers, 25(3), 961–976. https://doi.org/10.1007/s12221-024-00489-5

[40]. République Française. (2020). Loi n° 2020-105 du 10 février 2020 relative à la lutte contre le gaspillage et à l'économie circulaire (AGEC). Journal officiel de la République française. Retrieved from https://bcome.biz/regulation-radar/anti-waste-law-for-a-circular-economy-agec-law-loi-n-2020-105-article-13/

[41]. Rosa, R. P., Rosace, G., & Trovato, V. (2024). Recent Advancements in Acrylic Fabric Applications: A Comprehensive review and Future Trends. Polymers, 16(15), 2111. https://doi.org/10.3390/polym16152111

[42]. Shen, L., Worrell, E., & Patel, M. K. (2010). Environmental impact assessment of man-made cellulose fibres. Resources Conservation and Recycling, 55(2), 260–274. https://doi.org/10.1016/j.resconrec.2010.10.001

[43]. Shen, M., Song, B., Zeng, G., Zhang, Y., Huang, W., Wen, X., & Tang, W. (2020). Are biodegradable plastics a promising solution to solve the global plastic pollution? Environmental Pollution, 263, 114469. https://doi.org/10.1016/j.envpol.2020.114469

[44]. Shirvanimoghaddam, K., Motamed, B., Ramakrishna, S., & Naebe, M. (2020). Death by waste: Fashion and textile circular economy case. The Science of the Total Environment, 718, 137317. https://doi.org/10.1016/j.scitotenv.2020.137317

[45]. Tatsii, D., Bucci, S., Bhowmick, T., Guettler, J., Bakels, L., Bagheri, G., & Stohl, A. (2023). Shape matters: Long-Range transport of microplastic fibers in the atmosphere. Environmental Science & Technology, 58(1), 671–682. https://doi.org/10.1021/acs.est.3c08209

[46]. Vesel, A. (2023). Deposition of Chitosan on Plasma-Treated Polymers—A Review. Polymers, 15(5), 1109. https://doi.org/10.3390/polym15051109

[47]. Wong, Y., Szeto, Y., Cheung, W., & McKay, G. (2003). Adsorption of acid dyes on chitosan—equilibrium isotherm analyses. Process Biochemistry, 39(6), 695–704. https://doi.org/10.1016/s0032-9592(03)00152-3

[48]. Xu, J., Thomas, K. V., Luo, Z., & Gowen, A. A. (2019). FTIR and Raman imaging for microplastics analysis: State of the art, challenges and prospects. TrAC Trends in Analytical Chemistry, 119, 115629. https://doi.org/10.1016/j.trac.2019.115629

[49]. Zubris, K. a. V., & Richards, B. K. (2005). Synthetic fibers as an indicator of land application of sludge. Environmental Pollution, 138(2), 201–211. https://doi.org/10.1016/j.envpol.2005.04.013