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Oxygen plasma treatment to mitigate the shedding of fragmented fibres (microplastics) from polyester textiles

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ABSTRACT

The release of fragmented fibres (FFs) during the manufacture and service life of textiles is one of the key sources of microplastic pollution. Polyester (polyethylene terephthalate (PET)) is the most widely used fibre with production volume of 63 million tonnes - represents 55% of all textile fibres and 80% of synthetic fibres. The impact of textile material and structural variables as well as chemical modification of textiles to mitigate the release of FFs has previously been reported. For the first time, the current research brings together disciplines of textile technology and tribology to understand the impact of inter-fibre friction on the release of FFs. The oxygen plasma, a sustainable alternative approach to chemical treatment methods, was employed to alter the surface morphology and consequently the frictional behaviour of fibre surfaces and eventually its impact on the release of FFs. Using commercially relevant methods, bespoke polyester fabric was manufactured and treated with low pressure oxygen plasma. The treated and untreated samples were characterised for surface roughness (fibre surface profilometry), coefficient of friction (nanotribometer), FFs shedding, shear properties and wettability. An increase in surface roughness of polyester fibres was observed after the treatment. The oxygen plasma treatment significantly increased the coefficient of friction, shear hysteresis, wettability and reduced the FFs shedding by 43 % in mass (accumulative of prewash and 5 accelerated washes) and 73 % in count (number) compared to untreated sample, without impacting the fabric handle and imparting hydrophilic property. Strong negative correlations between shed FFs and fabric coefficient of friction ($r = -0.907$ for FFs mass and $r = -0.918$ for FFs count) were observed. The outcomes of this study confirm that the fibre tribological properties can play a role to modulate the release of FFs from polyester textiles, using a sustainable method of plasma treatment technology.

1. Introduction

Textiles have been reported as one of the major sources of fibrous microplastic pollution in our ecosystem (Hernandez et al., 2017; Cai et al., 2020a). Textiles manufactured from both natural and synthetic fibres shed fragmented fibres (FFs), including fibrous microplastics from synthetic textiles, during their whole life cycle starting from production (Cai et al., 2020b), everyday use (De Falco et al., 2020), laundry (Cai et al., 2020b; De Falco et al., 2019a), drying (O'Brien et al., 2020; Kärkkäinen et al., 2021) to end of life disposal (Sun et al., 2021). The FFs are described as the fibrous mass released from any textile material and majority of FFs released from textiles are reported to be < 5 mm in length. Textiles are estimated to contribute 35% of total microplastics present in world oceans (Henry et al., 2019). This figure highlights that the textiles are one of the largest sources of microplastic pollution in the oceans. An estimated 0.5 million tonnes of fibrous microplastics are leaked to the oceans from textile washings annually, and it's expected that their accumulated quantity in oceans would exceed 22 million tonnes between 2015 and 2050 (Foundation, 2017). The global fibre production has been nearly quintupled since the last five decades reaching almost 114 million tonnes in 2022 and is expected to increase up to 149 million tonnes by 2030 (Global production volume of textile, 2023). Amongst textile fibres, polyester is the dominant synthetic fibre used in the textile manufacturing globally. In 2022, its production was 63 million tonnes with a market share of approximately 55% (Zhang et al., 2021) and is expected to go above 92 million tonnes in the

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following next 10 years (Gu and Qiu, 2022). The fast fashion and increasing population of the world are the dominant factors responsible for the rise in global economic activities of fibres and textiles (Periyasamy et al., 2022).

The adverse effects of synthetic FFs on our ecosystem have been reported by various researchers. The earthworms exposed to polyester FFs demonstrated transcriptional responses related to general stress and change in casting behaviour which highlights potential implications of microplastics for soil ecosystem (Prendergast-Miller et al., 2019). The negative impact of polyester FFs on the reproduction of soil invertebrates has also been reported (Selonen et al., 2020). The presence of polyester FFs in soil influenced the microbial communities by modifying soil physical properties (Guo et al., 2021). Several studies highlighted the toxic effect of fibrous microplastics on aquatic organisms which include reduced growth, mortality, body condition and tissue damage (Rebelein et al., 2021). The structural alteration to fish gills was observed when exposed to polyester FFs in water column (Hu et al., 2020). The microplastics were observed in 54% of fish stomachs and higher rate of occurrence of microplastics was noted in fish captured near the urban area of water body (Chan et al., 2019). The studies have also highlighted that microplastics may disturb fish metabolism, interfere growth rates, reproduction, and immune function (Thacharodi et al., 2024). The risk of direct exposure to humans has been increased due to the presence of microplastics in the indoor environments, which are likely to be inhaled (Dris et al., 2017), and in the path of food chain such as sea food, sugar, salt, honey, drinking water, beer, fruits & vegetables, milk, rice, meat and other processed food items (Al Mamun et al., 2023). It is pertinent to mention that the studies reporting the direct effect of textile origin synthetic FFs (microplastics) on human health are very limited.

The widespread presence of microplastics (including textile origin FFs) in our ecosystem calls for immediate measures to mitigate the pollution and their likely toxic impact on life. Textiles are one of the dominant sources of the release FFs during manufacturing, service life and end of life disposal. Suitable mitigation strategies at each stage of a textile life cycle can help to mitigate the release and consequently negative impact to the environment. The generation of FFs during textile manufacturing cannot be underestimated as it adds to 49 % of total released fibre mass followed by laundry (28%) and wearing (23%) (Lim et al., 2022). At the manufacturing stage, textiles from tough and fine longer length fibres assembled into less hairy compact yarn structure with finer yarn counts (linear density), highly twisted continuous filament yarns, compact fabric structure with higher yarn interlacement and yarn densities result in reduced FFs emissions (De Falco et al., 2020; Periyasamy et al., 2022; Berruezo et al., 2021). It is reported that rotor, air jet (Jabbar et al., 2023) and modified ring spun yarns (Jabbar et al., 2022) result in relatively less release of FFs fibres from textiles. The low temperature dyeing with light coloured shade has been reported to lower emission of FFs from polyester spun yarns (Mondal et al., 2023). End-of-pipe solutions at textile manufacturing (Chan et al., 2021) or service stage (McIlwraith et al., 2019) includes the filtration of effluent to capture FFs. However, filtration of effluent still presents the challenge of disposal of collected FFs and an at-source approach to mitigate the release of FFs is desirable. Several researchers attempted to reduce the release of FFs by modifying textile surfaces with different chemical agents/finishes. The synthetic textiles were modified with chitosan (Kang et al., 2021), poly butylene succinate-co-butylene adipate (De Falco et al., 2019b), pectin modified with glycidyl methacrylate (De Falco et al., 2018), poly lactic acid (De Falco et al., 2019b), polydimethylsiloxane (Lahiri et al., 2023), sericin and poly vinyl alcohol (Ramasamy et al., 2023), lipase enzyme (Ramasamy et al., 2022) and reduction in FFs release were reported from surface modified textiles. The chemical modification of polyester fabrics with alkali (Rathinamoorthy et al., 2023), 1% trichloroacetic acid and methylene chloride solvent system (Rathinamoorthy et al., 2024) were also reported in a reduced shedding of FFs from textiles during laundry. It is important to note that conventional chemical finishing treatments result in changing the handle of the fabric and are also typically known to results in loss of efficacy over time.

The fibre surface tribology (fibre-to-fibre friction) is not only important in textile manufacturing processes but it also affects the physical (i.e., smoothness, handle) and mechanical (i.e., shear, stiffness, strength, elastic recovery) properties of textiles (Gupta, 2008). The inter-fibre friction in textiles affects textile fibre interactions in yarn/fabric structures, ease of fuzz formation and release from a textile structure (Jabbar et al., 2022). Zambrano et al. modified the surface of cotton knitted fabrics with different finishing agents and pointed out that fabrics treated with silicon softener released more FFs during laundry as compared to untreated fabrics. The result was attributed to coefficient of friction between fibres which was decreased after treatment making the fabric surface smooth, resulting in easy loosening of fibres from textile structure, increase in fuzz formation and more emission of FFs (Zambrano et al., 2021). Inter-fibre friction in textiles is anticipated to be a key property affecting the FFs release from textiles.

Textile fibre surfaces undergo significant transformations through gas plasma treatment, modifying their chemistry and morphology. The treatment process involves exposing textile substrates to various gas plasmas, which ionise and dissociate into a complex mixture of reactive species. These include ions, electrons, photons, free radicals and metastables etc. As these energetic species interact with the fibre surfaces, they trigger chemical reactions that alter the surface properties (McCoustra et al., 2018). During plasma treatment, the gas plasma's composition and energy determine the type and extent of surface modifications. The reactive species can remove hydrogen atoms, break chemical bonds, and introduce functional groups, leading to surface etching and roughening, introduction of polar groups, crosslinking, and grafting of new molecules. The versatility of gas plasma treatment allows for tailored surface modifications, depending on the type of chosen gas, treatment conditions, and textile substrate. The plasma treatment has a superficial impact, modifying only the outermost surface layer of the substrate (Jelil, 2015).

Oxygen plasma treatment is a highly effective method for enhancing the surface properties of polyester fabrics, particularly in terms of increase in surface energy, roughness, and wettability. By exposing the textile substrate to oxygen plasma, functional groups such as carboxyl, carbonyl, and/or hydroxyl etc. are introduced onto the polymer surface. These polar groups significantly increase the surface energy of the material, rendering it more hydrophilic and receptive to wetting agents (Wei et al., 2007). The increase in surface roughness lead to an increase in total surface area, thereby further enhancing hydrophilic capacity (Azeem et al., 2021). Moreover, the enhanced surface roughness by oxygen plasma may alter the inter-fibre friction within the textile structure.

In the current research, bespoke polyester fabric was exposed to oxygen plasma to alter the frictional properties of textiles and to quantify its impact on the FFs release from textiles during laundry. The extracted FFs, after laundry, were characterised for FFs mass, length profile and count. The fabrics were also characterised for surface frictional coefficient, shear property, wettability and wicking to elucidate the oxygen plasma impact on these properties and shedding of FFs.

2. Materials and methods

2.1. Bespoke textile sample development

The 100% polyethylene terephthalate (PET, 38 mm and 1.2 dtex) staple fibre yarn, with 29.53 Tex (20's Ne) nominal linear density and 7.04 turns.cm^{-1} (17.88 turns. inch^{-1}) nominal twist level, was spun using ring spinning technology, a predominant method to produce staple yarns. The virgin raw PET staple fibres were processed through a short staple spinning setup which includes Rieter blowroom line (mixing bale opener B34, condenser A21, unistore A79), Rieter C75 card, Toyoda

DYH 500C draw frame, Rieter RSB D40 draw frame, Toyota FL16 roving frame, and Toyota RY5 ring frame to manufacture ring spun yarn. The properties of PET staple fibres and the developed ring yarn are given in Tables S1 and S2 respectively. A rapier weaving loom by CCI Taiwan was used to convert the developed yarn into woven plain fabric with 24 threads cm^{-1} in warp and 22 threads.cm^{-1} in weft directions. The warp yarns were treated with polyvinyl alcohol (PVA) to improve their weavability.

A 5 g.L^{-1} of reference detergent by AATCC was used to prewash the fabric in a lab scale Jet machine maintaining 10: liquor ratio at 70 °C for 45 min. The washed fabric was subsequently dyed in a dye bath with 5% omf Foron® Navy Blue disperse dye (Archroma, Singapore) and 0.5 g. L^{-1} dispersing agent maintaining pH 4–4.5 and liquor ratio 20:1 at 130 °C for 1 h. Finally, the hot washing of fabric was done after the completion of dyeing process at 70 °C for 15 min. The fabric was washed, neutralised and dried at room temperature after the completion of dyeing process.

2.2. Oxygen plasma treatment

The PET fabric was exposed to oxygen plasma at a pressure ranging from 0.4 to 0.8 mbar using a low-pressure plasma (PICO Diener electronic Germany) coupled with a vacuum pump and an oxygen tank. Before the treatment, the plasma reacting chamber was cleaned with oxygen plasma at 10 mL min^{-1} gas flow rate and 150W electric power. After that, each side of the fabric specimens of dimensions 200 × 340 mm^2 was exposed to pure oxygen plasma in the reacting chamber for 30 min at 10 mL min^{-1} gas flow rate and 250 W electric power.

2.3. Gyrowash and FFs mass measurement

The untreated (U), and oxygen plasma (OP) treated polyester (PET) fabric specimens of dimensions 200 × 340 mm^2 were under folded 50 mm to back from each edge and lockstitched (2.5 mm per stitch, stitch type 301) near the edge using 27 Tex 100% staple polyester light green colour thread sourced from Coats Astra (13ANT, Tkt 120). The colours of sewing thread and textile samples were kept different from each other to identify easily any FFs release from the sewing thread. The finished size of specimens was 100 × 240 mm^2 approximately. The samples were subjected to laboratory washing using GyroWash machine (James Heal) to measure the FFs released mass according to standard test method, AATCC TM212-2021. The full detail of the washing and FFs mass measurement process is reported elsewhere (Jabbar et al., 2023). Briefly, prewashing of four fabric specimens from each sample was carried out without adding any steel balls in stainless-steel canisters. The specimens were put again in stainless-steel canisters along with 50 steel balls (6 mm diameter) and 360 mL of detergent solution per canister and washed up to 5 accelerated washing cycles at 40 °C and 40 rpm for 45 min. Before the start of each washing cycle, a thorough washing of canisters with distilled water was insured. Four replicates of each sample were washed to compute average values.

The rinsing of each specimen after each cycle with distilled water was carried out three times to recover the effluent for filtration subsequently. The excessive water from the specimens was removed using a pair of tweezers. The rinsing of beakers, mesh, tweezers and canister was done three times to recover all the effluent from each specimen in one beaker. A binder-free glass fibre filter (47 mm diameter, 1.6 μm pore size, Merck Millipore Ltd. Ireland) was used to filter all the recovered effluent. A sensitive precision weighing balance with a resolution of 0.00001 g (Mettler Toledo AE160) was used to weigh the filters before filtration. After filtration. the wet filters were allowed to completely dry at 50 °C by putting them overnight in a fan oven followed by in a desiccator for 1 h. The filters were re-weighed again to calculate the filter mass increase. The difference of filters' mass before and after filtration equals to the FFs mass released. The textile specimens were dried in a fan oven at 50 °C overnight before using them for next washing cycle.

2.4. Count and length profile of FFs

The Diamlength instrument from Fibremetrics Australia, was used to measure the number of FFs released from untreated and oxygen plasma treated polyester textiles after prewash, 1st, 3rd' and 5th washes and their length distribution. This instrument captures live images of water immersed FFs and uses image processing technique to compute FFs. The glass filter, containing mass of FFs over its surface was immersed in 1 L of distilled water for 5 min and stirred for a few seconds with the help of a glass rod to loosen the FFs from the filter surface and disperse in water. The FFs were successfully released from filters. However, many glass fibres were found in water which were probably detached and dispersed from glass filter during stirring. The opacity parameter of the instrument was employed to reject them from calculation because of their different refractive index and material density from FFs.

2.5. Fibre surface profilometry

The fibre surfaces from untreated and treated fabric specimens were scanned using a Bruker NPFLEX white light vertical scanning interferometer (VSI) under an optical magnification level of 100 × to produce topographic heightmaps and to calculate their relevant roughness parameters. The specimens were sputter coated with gold before measurement. A white light source was used to maximise the signal-to-noise ratio during interference pattern localisation, owing to its low coherence. Rectangular 62 × 46 μm^2 scans of each fibre surface were initially produced. These scans were then further masked to isolate individual fibres for topographical measurement. The resulting height data from each fibre were fitted to a toroidal model surface. The curvature terms for each relevant toroid were then subtracted from their respective height data to remove the effects of fibre geometry on the measurement of surface roughness.

The roughness parameter most encountered in profilometry is the centre-plane mean arithmetic roughness, normally referred to as R_a. This parameter is defined as the mean deviation of surface height ($z(x, y)$) from the mean height (z_0). of the surface being measured – as defined by the equations below.

$$R_a = \frac{1}{\Delta_x\ \Delta_y} \iint |z(x,y) - z_0| dx\ dy = \frac{1}{N_x\ N_y} \sum_{n_x=1}^{N_x} \sum_{n_y=1}^{N_y} \left| z(n_x, n_y) - z_0 \right|$$

$$z_0 = \frac{1}{\Delta_x\ \Delta_y} \iint z(x,y) dx\ dy = \frac{1}{N_x\ N_y} \sum_{n_x=1}^{N_x} \sum_{n_y=1}^{N_y} z(n_x, n_y)$$

Here, Δ_x and Δ_y are the dimension lengths of the area being measured in the x and y directions respectively, making their product the nominal surface area. N_x and N_y are the total number of heights datums (n) in the x and y directions respectively, making their product the total number of datums within the measurement area. It is important to note that R_a neglects other surface properties – such as skewness and kurtosis – although such parameters are beyond the scope of this study.

2.6. SEM analysis

The untreated and oxygen plasma treated textiles was introduced to Jeol JSM-6610 scanning electron microscope, after sputter-coating with a thin gold layer using sputter coater Q150RS (Quorum Technologies), to visualise the topology of polyester fibres on the fabric surface.

2.7. Fabric surface tribology

The friction experiments of untreated, and oxygen plasma treated samples were performed on NTR3 nanotribometer (Anton-Paar GmbH

Switzerland). The specially designed upper and lower sample holders were used to mount the fabric specimens as shown in Fig. S1. To find the surface coefficient of friction (COF), the top and bottom fabric specimens were cut along the warp direction to the width of 5 mm and 8 mm respectively as highlighted in Fig. S1 and mounted on specimen holders with a double-sided adhesive tape. The lower fabric specimen was rubbed in back-and-forth motion against the upper one for 20 rubbing cycles by applying 100 mN normal load at 0.2 Hz sliding frequency and 2 mm sliding amplitude.

2.8. Kawabata shear

The both oxygen plasma and untreated textile samples were characterised for shear properties including shear rigidity (G), shear hysteresis at shear angle of 0.5° (2HG), and shear hysteresis at shear angle of 5°(2HG5)) on Kawabata tensile and shear module (KES-FB1A).

2.9. Wettability and wicking

The wettability of PET fabrics was determined by measuring water contact angle (WCA) at the fabric surface and water absorption time to understand the influence of oxygen plasma treatment on their hydrophilic character. The WCA was measured by putting a water drop of 10 μL size on the fabric surface by employing sessile drop measurement method and using drop shape analyser instrument (model DSA 30S, Kruss GmbH Germany). The water absorption time of fabrics was measured by falling a water drop of 25 μL volume from a burette fixed at a height of 10 mm from the fabric surface. The wicking ability was evaluated using WickView instrument from James Heal by releasing 0.2 mL volume of water on each test specimen of 150 mm diameter approximately to measure the dynamic increase in wicking area for 120 s. The test specimens were kept horizontal during the test (with fabric side facing up assumed as skin side by the instrument) and water released on skin side was transported through the thickness of fabric and spread on both skin and face sides. Deionised water was used as a test solution and five replicates were taken for every sample in all three tests to compute average values.

2.10. Statistical analysis

The effect of oxygen plasma treatment on the released mass and number of FFs, frictional coefficient, Kawabata shear, and wettability of fabric samples was determined statistically using Tukey's method within one-way ANOVA in Minitab® 22.1 software. The correlation between measured fabric properties and FFs released mass was measured using Pearson correlation coefficient (r).

3. Results

3.1. Surface roughness

Example surface profiles of the untreated and plasma treated PET fibres are presented in Fig. 1 in the format of 3D heightmaps. The oxygen plasma treated fibres exhibit a notably more uneven surface in contrast to the untreated fibres upon examination. The calculated R_a values of fibres treated with oxygen plasma were also noted to be higher than that of the untreated fibres on average; increasing from 0.192 ± 0.027 for the untreated fibres, to 0.226 ± 0.034 for the oxygen plasma treated fibres.

Fig. 2 shows scanning electron microscopic images of the untreated fibres and oxygen plasma treated fibres under a magnification level of 4000 × . The observed increase in surface roughness of the oxygen plasma treated fibres in the VSI measurements is also reflected in these images. This outcome may be attributed to etching phenomenon of oxygen plasma treatment on the surface of fibre, contributing to an increase in surface roughness (Wei et al., 2007). This was further witnessed by the increased effectiveness of oxygen plasma over nitrogen (N_2), argon (Ar) and air plasma in the surface etching of polyester fabrics (Kamel et al., 2011).

3.2. Fabric coefficient of friction

The mean coefficient of friction (COF, μ) curves with standard error of 5 measurements up to 20 cycles for oxygen plasma treated and untreated and specimens are plotted in Fig. 3. It shows that the surface COF of fabric after oxygen plasma treatment is increased. The box plot given in Fig. S2 reveals the spread of 20 cycles mean COF data. The statistical analysis shows a significant difference of COF of oxygen plasma sample from the untreated one at 95% confidence interval (p-value = 0.000).

3.3. The gravimetric mass and length profile of FFs

The FFs mass released from untreated and oxygen plasma treated samples during each washing cycle up to 5 washes is plotted in Fig. 4 and presented in Table S3. A higher release of FFs mass was noted after prewash irrespective of fabric sample which was decreased gradually during subsequent washes. The decrease over repeated cycles is well known and also reported in previous studies (Jabbar et al., 2022, 2023). When compared with untreated sample, oxygen plasma (OP) treated sample has shown a significantly less release of FFs mass during prewash step where no steel balls are added during washing. However, this

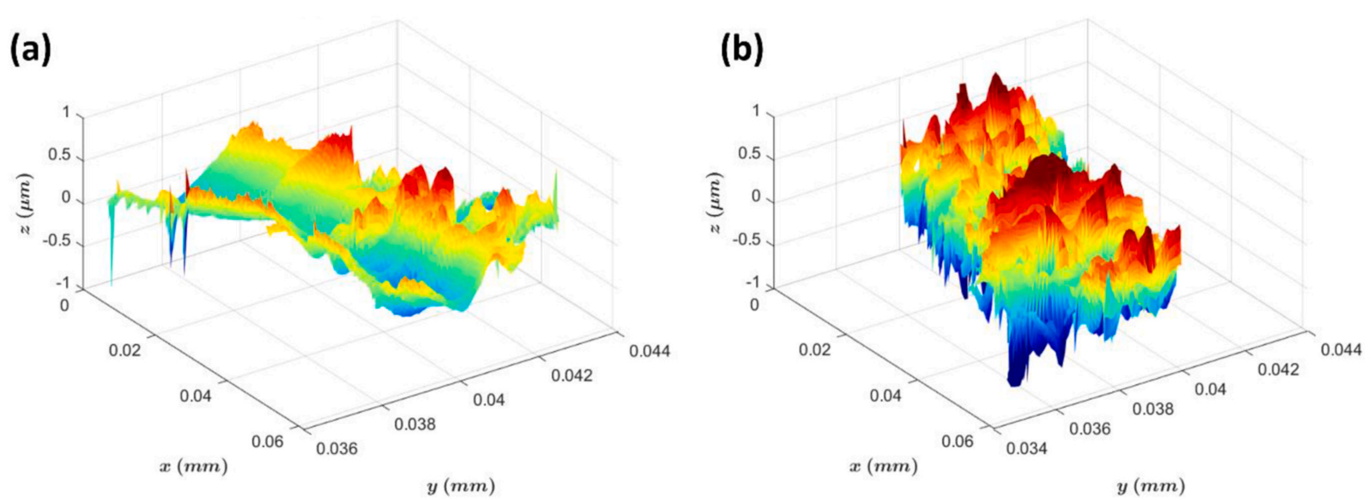

Fig. 1. Vertical scanning interferometry 3D images showing surface topology of polyester fibres (a) untreated and (b) oxygen plasma treated.

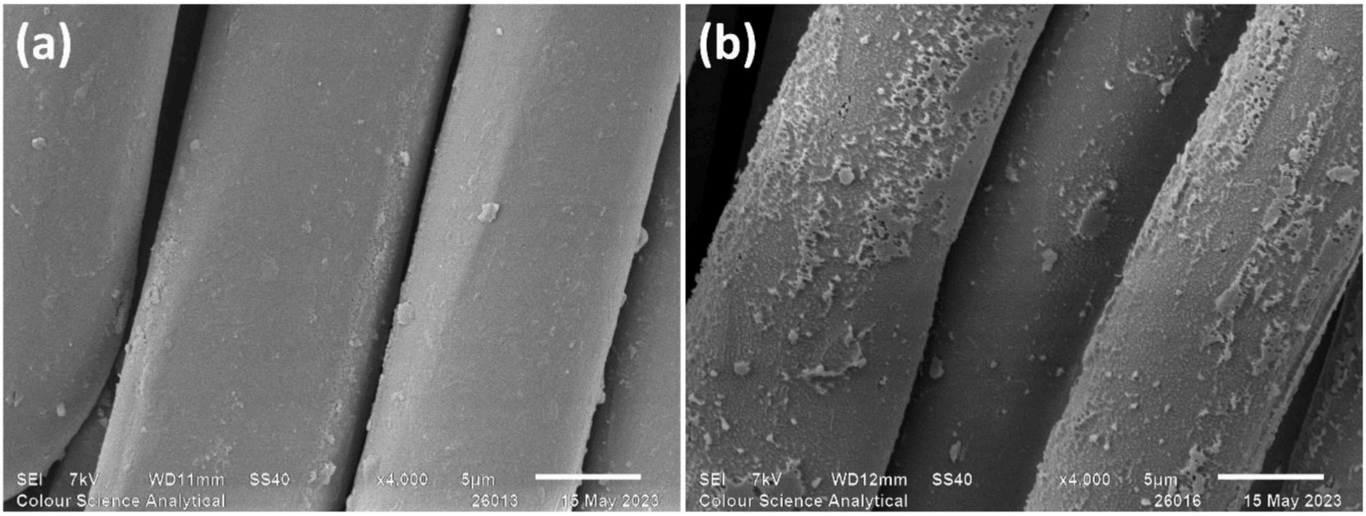

Fig. 2. Scanning electron microscopy of polyester fibres (a) untreated, (b) oxygen plasma treated.

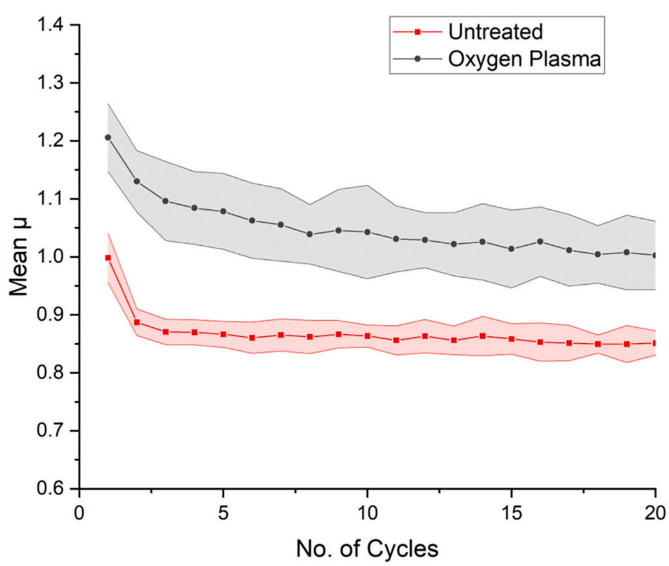

Fig. 3. Mean coefficient of friction (μ) curves with standard error of 5 measurements each for oxygen plasma treated and untreated polyester fabrics.

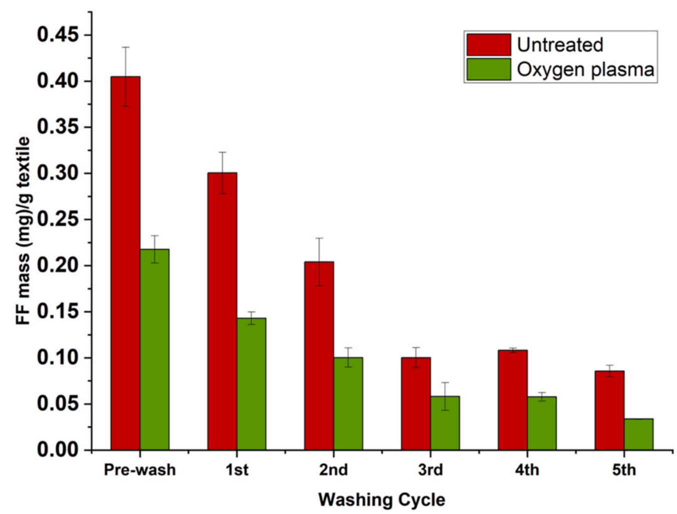

Fig. 4. FFs mass released from untreated and oxygen plasma treated samples after each wash cycle up to 5 washes.

difference of shed FFs mass was reduced from 3rd wash cycle onwards. During prewash, OP sample released 0.22 mg/g of FFs mass as compared to 0.404 mg/g for untreated sample resulting in 45.6 % decrease. This emission of FFs during prewash highlights the significance of textile manufacturing processes in fibre damage and generation of FFs. The boxplot in Fig. S3 highlights the FFs mass accumulated from all washing cycles with OP sample releasing 0.68 mg/g of FFs mass as compared to 1.2 mg/g for untreated sample resulting in 43.05% decrease. The Tukey's method showed that this difference in shedding of FFs was also statistically significant (*p*-value = 0.000) at 95% confidence level as compared to untreated one. A strong negative correlation (r = −0.907) between shed FFs mass and fabric COF is revealed by the correlation analysis i.e., a higher COF resulted in less shedding of FFs mass.

The FFs count showed a declining trend like the gravimetric FFs mass during repeated washing cycles. As evident in Fig. 5 and Table S3, the number of FFs were greater during pre-wash for both types of samples, which were decreased gradually in the following washing cycles. By calculating the total amount of FFs, the oxygen plasma treated sample released on average 73% a smaller number of FFs per gram of textile (1785 FFs/g textile) as compared to untreated sample (6638 FFs/g textile) which was also statistically significant (*p-value* = 0.000) at 95% confidence level according to Tukey's comparison method.

The length data of FFs was presented on a log scale as shown in Fig. 6. Like previous studies, the untreated and treated samples showed a relatively small variation in FFs length distribution. The median length of FFs is increased over cycles as shown in Fig. 6, which indicates that the shorter length FFs are released during pre-wash in contrast to subsequent wash cycles. Moreover, the untreated sample showed a considerable increase in median length of FFs in the following washing cycles. An increase in median length from 0.41 mm for pre-wash to 0.49 mm for 5th wash was observed. However, oxygen plasma sample disclosed a relatively less increase in this parameter over cycles where the increase in median length from 0.34 mm for pre-wash to 0.37 mm for 5th wash was noted. These results may be reasoned by the probability of shorter length FFs to come out easily from the internal structure and release the textile surface during initial wash cycle. Whereas more time is needed for comparatively longer and tangled FFs to release from textile structure during following washing cycles. Furthermore, over 90% of released FFs were observed to be less than 1 mm for both samples similar to reported previous studies (Hernandez et al., 2017; Jabbar et al., 2023).

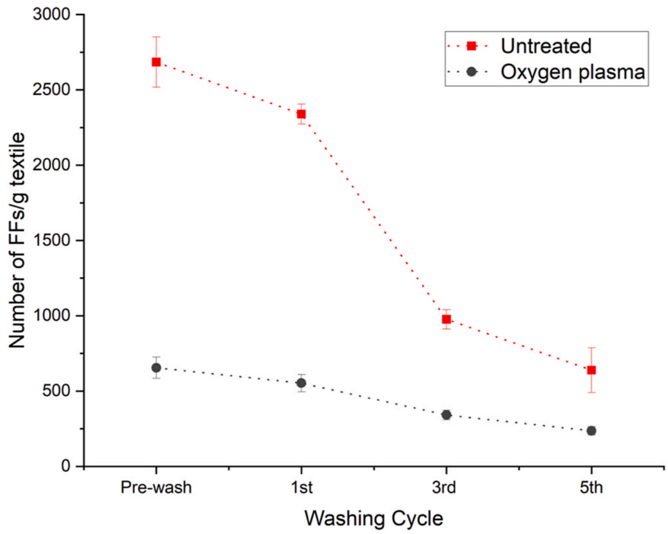

Fig. 5. The number of FFs from oxygen plasma treated and untreated samples during washing cycles. The data error is based on triplicate experiments.

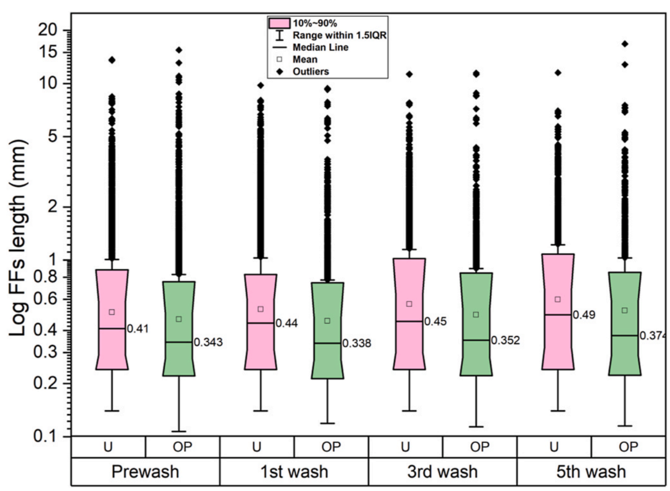

Fig. 6. The plot of FFs length distribution after the prewash, 1st, 3rd and 5th washing cycles. The plotted data is the sum of triplicate experiments.

3.4. Kawabata shear properties

The shear rigidity (G) describes the pliability of fabrics and indicates the freedom with which the fibres/yarns in the fabric structure slide against each other. The shear hysteresis both at 0.5° and 5° shear angles give an indication of energy loss caused by yarn-to-yarn friction in the fabric structure. The greater hysteresis values means that large recovery forces are needed to overcome fabric internal friction and vice versa. The results of G, 2HG and 2HG5 in weft and warp directions are plotted and shown in Fig. 7 and presented in Table S4. It can be noted that shear rigidity, G of oxygen plasma treated sample is relatively greater than untreated sample. However, there was no significant difference of mean G for untreated and oxygen plasma samples in both warp and weft directions at 95% confidence level as revealed by Tukey's comparison method shown in Table S5. Similarly, the increase in shear hysteresis 2HG for oxygen plasma sample was also statistically insignificant at 95% confidence level as compared to untreated sample (Table S5). A weak negative correlation of the release of FFs mass with shear properties G ($r = -0.371$), 2HG ($r = -0.672$) was exhibited by correlation analysis. The effect of oxygen plasma treatment on shear hysteresis 2HG5 was statistically significant at 95% confidence level as compared to untreated one. A strong negative correlation of the release of FFs mass with shear hysteresis 2HG5 ($r = -0.895$) was observed. The analysis of results showed that the effect of oxygen plasma was more significant on shear hysteresis rather than that of shear rigidity. This phenomenon may be explained by the distribution of shear stresses in the fabric plane during shear deformation and oxygen plasma treatment is a surface treatment with no major influence on the bulk properties of the fabric. The treatment predominantly modified the surface of fibres located on the fabric surface which may affect the yarn displacement at crossover points during shear deformation. Overall, the shear results verify that oxygen plasma has enhanced the shear hysteresis, especially 2HG5 which might be related to increase in inter-yarn friction at crossover points resulting in more energy loss during shear deformation. This is a favourable result that the treatment has little impact on the handle of the fabric which is critical to achieve the desired aesthetic and functional properties from a fabric.

3.5. Fabric wettability and wicking

The way a solid surface interacts with a water drop can be assessed by measuring WCA of a solid surface. It is also considered a useful technique to judge the hydrophilic or hydrophobic character of textiles. A material surface with a WCA greater than or equal to 90° is considered a hydrophobic otherwise hydrophilic if WCA is less than 90° (Marmur et al., 2017). Table 1 presents the measured WCAs of untreated and treated polyester fabrics. It is evident that oxygen plasma treatment has significantly lowered the water contact angles by changing the fabric surface from hydrophobic to hydrophilic. The mean WCA is reduced from 128.84° for untreated sample to 59.86° for oxygen plasma treated sample. Fig. 8 provides the details of water drop shape and WCAs of representative oxygen plasma treated and untreated specimens.

The Tukey's comparison method, as shown in Table S5, highlights statistical significance of decrease in WCA of oxygen plasma sample at 95% confidence level as compared to untreated sample. The boxplot in Fig. S4 further discloses the disperse of 5 measurements of WCA data. The decrease in WCA confirmed the change in surface properties of fabric such as the introduction of polar groups and rough surface of fibres after oxygen plasma treatment (Wei et al., 2007). The correlation analysis showed a strong positive relationship of WCA with the release of FFs ($r = 0.984$). The hydrophilicity of textiles can also be estimated by observing the absorption time of a water drop on the textile surface. The results of absorbency time, presented in Table 1, show that hydrophilicity is significantly increased for oxygen plasma treated sample. It has been reported in a recent study (Rathinamoorthy et al., 2023) that the increase in hydrophilicity of polyester fabrics (i.e., decrease in WCA and increase in absorbency) is strongly correlated with the decrease in shed FFs. However, the change in hydrophobic character of polyester textiles may not be the only single metric responsible for reduced shedding of FFs but some other important parameters such as surface roughness and fibre-to-fibre friction may have an important impact in controlling the freedom of movement and escape of already embedded FFs in textile structures. Fig. 9 shows the average curves of wicking area on face side of the fabric with standard error of 5 measurements for oxygen plasma treated and untreated specimens. It is evident that wicking area rises rapidly right after releasing the water on skin side of fabric for oxygen plasma treated sample and reaches to approximately 182 mm^2 within 15 s whereas it is only 2 mm^2 approximately for untreated sample for the same time. This phenomenon may be explained by the increase in hydrophilicity (Table 1 and Fig. 8) and nano roughness (Fig. 2b) of oxygen plasma treated fabric which facilitate the water transport through the fabric thickness and spreading along the fabric surface.

4. Discussion

Most of the previous studies have mainly investigated the effect of various textile manufacturing, materials, structural and washing

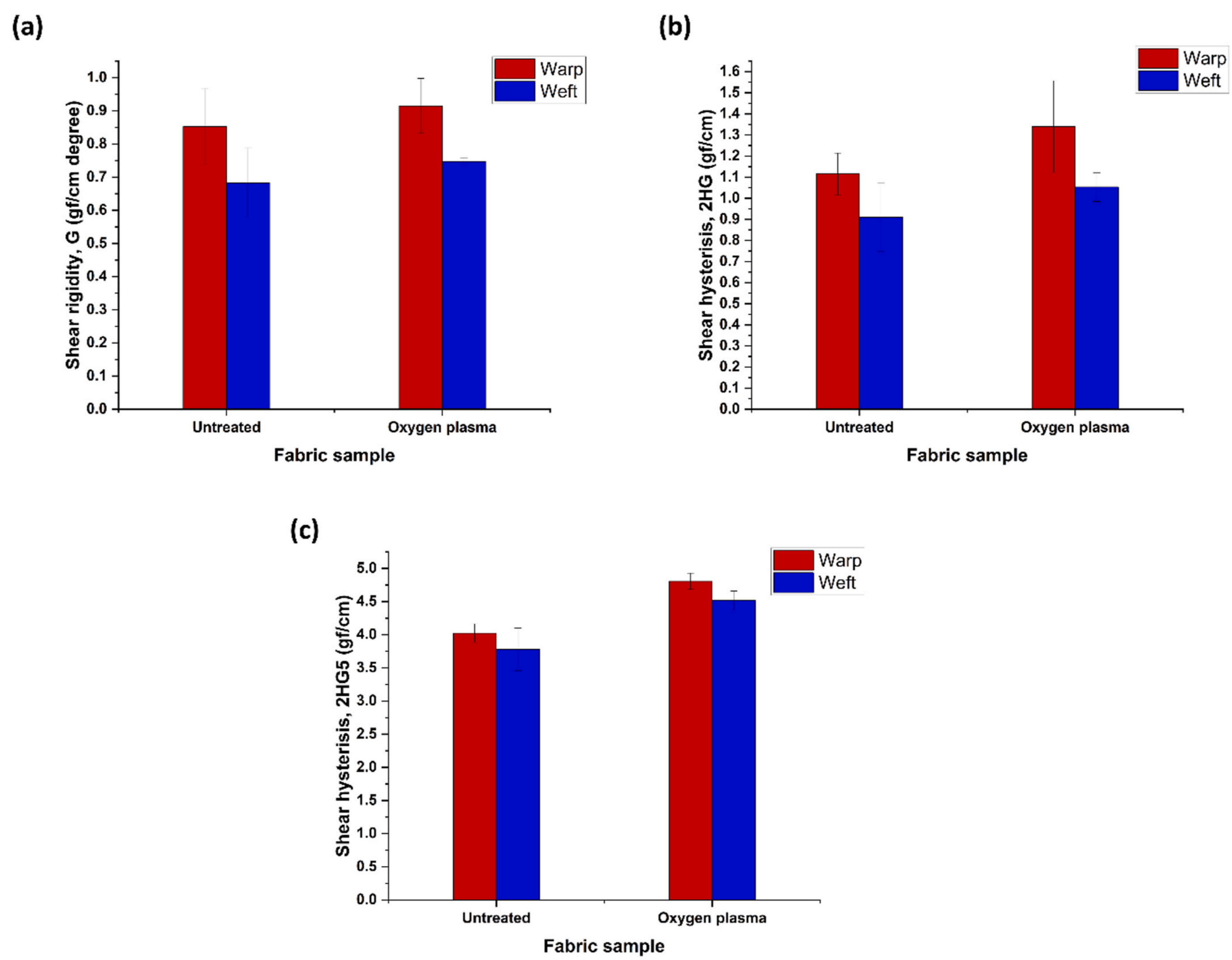

Fig. 7. Kawabata shear properties of in warp and weft direction for untreated and oxygen plasma treated PET fabrics.

Table 1
WCA and absorbency of polyester textile samples under study.

Sr. No.	PET fabric sample	Water contact angle (°)	Absorption time (sec)
1	Untreated	128.84 ± 5.20	71.8 ± 6.97
2	oxygen plasma	59.86 ± 8.34	<1

parameters on the release of FFs as reviewed in a study (Periyasamy et al., 2022). It is recognised that fibre damage and generation during textile production is the prominent reason (among others) of FFs release during washing of textiles (Cai et al., 2020b; Pinlova et al., 2022). The researchers have reported various chemical finishing/coating techniques to mitigate shedding of FFs from synthetic textiles as summarised in Table 2. The restriction of free movement of fibres by penetration of finishing agent into the textile structure has been reported to reduce the shedding of FFs in the most of published work. The conventional chemical finishing treatments introduce a layer on the fabric surface which results in changing the handle of the fabric and are also typically known to results in loss of efficacy over time when exposed to different conditions during use and service. Furthermore, limited knowledge exists on the mechanism of release of already present FFs in textile structure during laundry. In addition, the environmental impact of chemicals used in previous mitigation studies cannot be underestimated. The role of oxygen plasma to change the surface structure (Wei et al., 2007), wettability (Kamel et al., 2011; Wrobel et al., 1978) and dyeability (Kamel et al., 2011) of polyester fabrics has been reported but the impact of change in surface tribology to limit the release of FFs from polyester textiles is not established. This study therefore adopted a sustainable approach to get insight into the role of surface modification by oxygen plasma treatment on the FFs shedding from polyester textiles without altering the bulk properties and any undesired changes in fabric handle. Furthermore, the change in moisture wicking can offer additional functionality for the wearer's comfort. The advantage of the present work over previous mitigation studies, summarised in Table 2, can be explained by its simple, clean, and sustainable approach to mitigate the release of FFs from textiles thus overcoming a barrier to sustainable textile manufacturing. It also brings the additional benefits of no significant impact on fabric handle and improved moisture management for standard round polyester fibres.

The increase in surface roughness of polyester fibres has been reported after oxygen plasma treatment (Wei et al., 2007). A strong positive correlation has also been reported between the coefficient of friction and surface roughness of woven fabrics (Sülar et al., 2013). The increase in COF may be related to the increased surface roughness of fibres after oxygen plasma treatment. The sliding behaviour of textile fibres over each other may be related to their surface roughness and COF. A higher COF may restrict the freedom with which the fibres slide over each other and escape the textile structure. The published work witnesses the presence of FFs in textile structures possibly generated

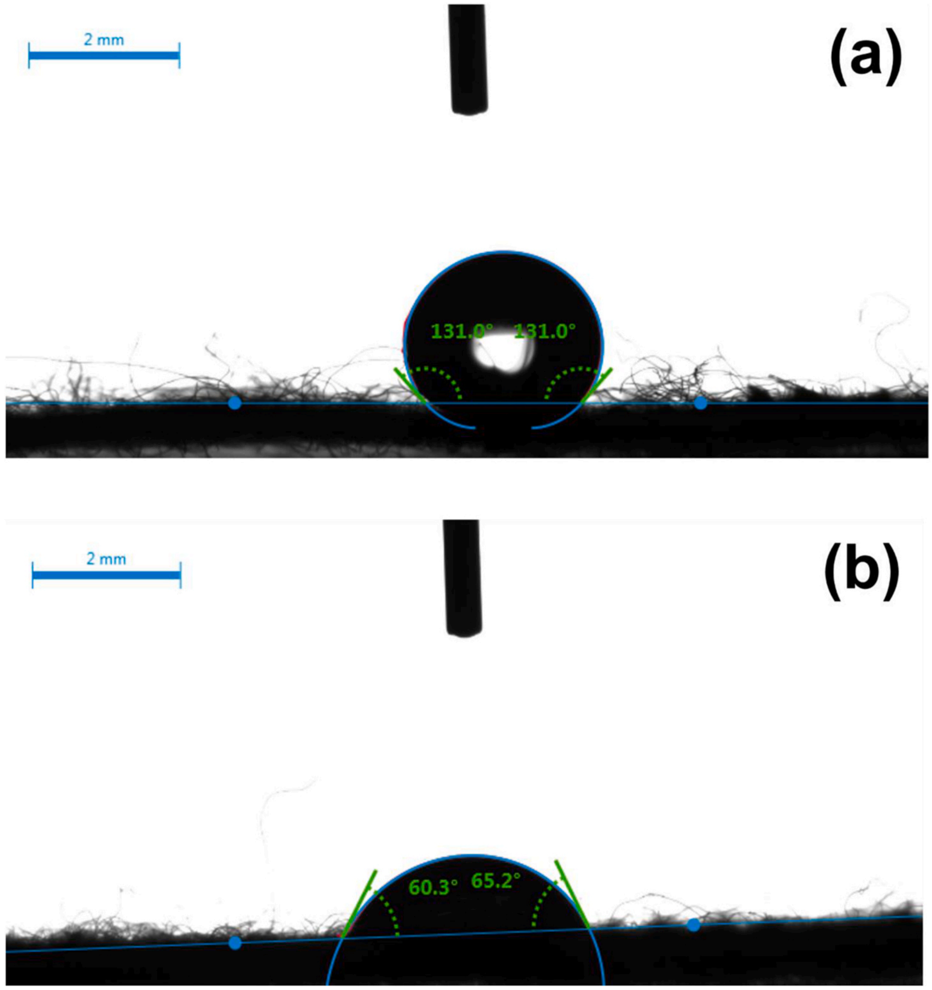

Fig. 8. Shapes of water drops and WCAs of representative (a) untreated, (b) oxygen plasma treated specimens.

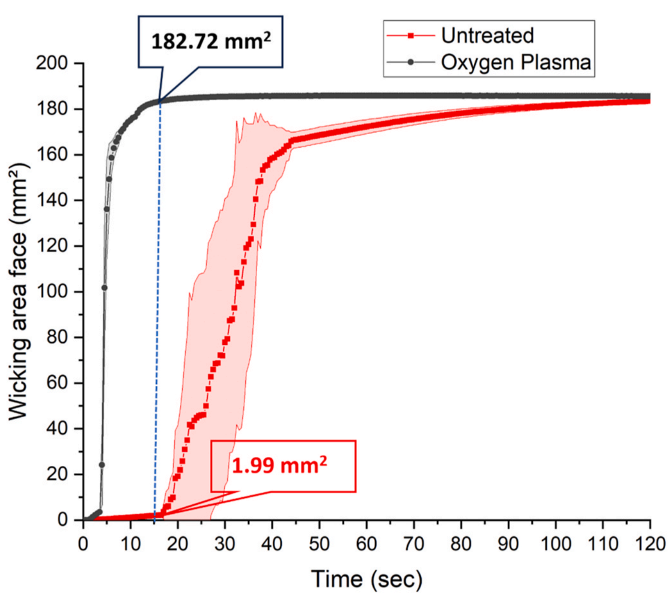

Fig. 9. Average wicking area on the face side of fabric with standard error of 5 measurements each for oxygen plasma treated and untreated polyester fabrics.

during textile manufacturing. (Cai et al., 2020b; Jabbar et al., 2023; Pinlova et al., 2022). The reduced shedding of FFs after oxygen plasma treatment may be explained by the fact that the freedom of movement and escape of already present FFs from textile structure are restricted due to increase in fibre surface roughness and COF of polyester fabrics. The higher surface roughness and COF imparts resistance to fibres sliding over each other and restricting their movement to escape the textile structure.

The inter-yarn friction in woven structure has a greater influence on fabric shear properties. In woven fabrics, the in-plane shear properties are mainly influenced by inter-yarn friction at warp and weft yarns crossover points which move relative to each other by changing cross-over angle between weft and warp yarns. Apart from improving the wettability of polyester textiles, the surface roughness of PET fibres is also increased due to oxygen plasma treatment (Sun et al., 2005) as discussed before and shown in Fig. 1. This increase in surface roughness may also contribute to increase in inter-yarn friction in a textile structure. This phenomenon may restrict the freedom of yarn movement and offer more resistance to yarns to return to their original place during shear deformation thus directing to increase in shear hysteresis as presented in Fig. 7 and Table S4.

The wettability of polymer materials is known to be depended on their surface roughness and polarity (Wrobel et al., 1978). The increase in wettability of polyester fibres, by oxygen plasma treatment, has been reported in literature through induction of hydrophilic functional groups on polyester fibre surface (Wei et al., 2007). The higher surface roughness of fibres by oxygen plasma is also witnessed in the current and

Table 2
Summary of published literature for mitigation of FFs release from synthetic textiles.

Mitigation technique	Chemical/Finishing agent used	Textile substrate	Outcome in percentage reduction of FFs	Reference
Surface coating/ Chemical modification	Poly (lactic acid)	Polyamide-6,6 fabric	More than 80% reduction of the number of FFs released.	De Falco et al. (2019b)
Grafting/chemical modification	Pectin modified with glycidyl methacrylate	Polyamide-6,6 fabric	90% reduction of the number of FFs released.	De Falco et al. (2018)
Chemical modification	Chitosan	Polyester, polyamide, and acrylic garments	Polyamide fabric 48% reduction in number; acrylic fabric, 49% reduction in number; polyester fabric 95% reduction in number.	Kang et al. (2021)
Surface coating/ Chemical modification	Poly butylene succinate-co-butylene adipate	Polyamide-6,6 fabric	More than 80% reduction in the number of FFs released.	De Falco et al. (2019b)
Surface coating/ Chemical modification	Polydimethylsiloxane (PDMS)	Polyamide fabric	93% reduction in number of FFs released.	Lahiri et al. (2023)
Chemical finishing	Chitosan with alkali pretreatment	Polyester fabric	87.61% reduction of FFs mass released.	Ramasamy et al. (2023)
Chemical modification	Sericin with alkali pretreatment	Polyester fabric	47.94% reduction of FFs mass released.	Ramasamy et al. (2023)
Chemical modification	Polyvinyl alcohol with alkali pretreatment	Polyester fabric	40.19% reduction of FFs mass released.	Ramasamy et al. (2023)
Surface/chemical modification	Lipase enzyme	Polyester fabric	79.11% reduction of FFs mass for knitted polyester.	Ramasamy et al. (2022)
Chemical modification	Alkali	Polyester fabric	89.6% reduction of FFs mass for woven polyester fabric and 68% of FFs mass for knitted polyester fabric.	Rathinamoorthy et al. (2023)
Chemical modification	Methylene chloride (TCA–MC) and trichloroacetic acid solvent system	Polyester fabric	64.72% reduction in number for woven polyester fabric and 93.79% reduction in number for knitted polyester	Rathinamoorthy et al. (2024)
Physical modification	oxygen plasma	Polyester fabric	43% reduction of FFs released mass and 73% reduction in FFs count.	Current study

previous (Wei et al., 2007) studies. The wettability of polyester fabric (as witnessed by decreased WCA and water absorption time) can be conferred by the rise in surface roughness and introduction of polar functional groups over fibre surface after oxygen plasma treatment.

5. Conclusion

In the present study, an effort was made to mitigate the FFs shedding, through oxygen plasma treatment, by modifying the surface characteristics of polyester textiles without affecting fabric handle and imparting moisture management properties. The increase in surface roughness, coefficient of friction and wettability were strongly correlated with reduced FFs shedding from polyester fabrics after the treatment. It was disclosed that oxygen plasma has significantly increased the coefficient of friction, shear hysteresis, wettability and reduced the FFs shedding from polyester textiles during laundry. A cumulative 43 % decrease in shed FFs mass (accumulative of pre-wash and 5 accelerated wash cycles) and 73 % in count (number) were noted for oxygen plasma treated sample in contrast to untreated one. It can be proposed from the outcomes of this study that the treatment with oxygen plasma can be used as a clean alternative finishing step during textile production to mitigate shedding of FFs from polyester clothing without affecting bulk properties and offering additional functionality. It further signifies the direction for future research to explore the role of other commercially well-known gases plasmas to create bespoke surface profiles and mitigate FFs release from textiles.

CRediT authorship contribution statement

Abdul Jabbar: Writing – review & editing, Writing – original draft, Project administration, Methodology, Conceptualization. **Michael Bryant:** Methodology, Investigation. **Josh Armitage:** Writing – review & editing, Visualization, Software. **Muhammad Tausif:** Writing – review & editing, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.clet.2024.100851.

Data availability

Data will be made available on request.

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