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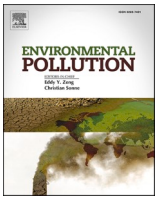
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Mitigation of laundering-induced microplastic fiber release from polyester fabric via plasma-assisted chemical surface modification

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ABSTRACT

Microplastic fiber (MPF) release from polyester textiles during laundering is a recognized pathway contributing to aquatic contamination and potential human exposure. In this study, atmospheric-pressure plasma activation followed by chemical surface modification was evaluated as an at-source strategy to mitigate MPF shedding from a 100% polyester (PET) fabric. Fabrics were plasma activated and subsequently treated with sodium hydroxide (NaOH) or chitosan at two concentrations (0.6% and 1.2% w/v). MPF release was quantified using an EN ISO 4484-1:2023-based laboratory washing protocol (Gyrowash) over five sequential wash cycles with procedural blanks used to correct background contamination. Released MPFs were assessed using complementary metrics, including filter mass gain (gravimetry), image-based MPF counting, MPF length distribution, and scanning electron microscopy (SEM) of fabric surfaces and collected MPFs. All surface-treated samples exhibited lower MPF counts than the untreated fabric. The strongest mitigation was observed for 0.6% chitosan ($-63.0 \pm 4.2\%$), followed by 1.2% chitosan ($-40.1 \pm 11.0\%$), while NaOH treatments showed intermediate reductions ($-32.4 \pm 5.4\%$ to $-34.8 \pm 7.1\%$). Length distribution analysis showed treatment-dependent shifts in emitted MPF size, with 1.2% NaOH increasing the relative contribution of short MPFs (<1.0 mm), whereas chitosan treatments increased the proportion of MPFs ≥1.0 mm. SEM observations supported these trends by revealing differences in surface disruption after washing and abrasion, as well as corresponding differences in MPFs retained on filters. Overall, plasma-assisted chitosan finishing—particularly at 0.6%—showed the strongest mitigation among the tested conditions, indicating that combined plasma–chemical modification is a promising at-source strategy for reducing laundering-induced MPF shedding from polyester.

1. Introduction

Polyester (polyethylene terephthalate, PET) is the most widely used synthetic textile fiber worldwide due to its low cost, durability, and versatile performance in apparel and technical textiles. In 2024, polyester accounted for ~59% of global fiber production (~77.7 million tonnes) (Textile Exchange, 2025). Despite its widespread use, polyester presents environmental concerns because it can release fibrous microplastics throughout its life cycle. Synthetic fibrous particles released from polyester textiles are commonly referred to as microplastic fibers (MPFs) (Yang et al., 2023). In the context of environmental pollution, the term microfiber is often used broadly to denote any fibrous particle released from textile materials, regardless of composition (e.g., natural, regenerated, or synthetic). This broad category should be distinguished from microplastic fibers or fibrous microplastics, which specifically refer to fibers composed of synthetic polymers or other environmentally persistent modified materials. Within this framework, synthetic microfibers or microplastic fibers released during laundering are more appropriately classified as secondary microplastics, because they originate from a larger textile product and are generated through fiber detachment and fragmentation during wear, laundering, and end-of-life handling (Akyıldız et al., 2025b; Hossain et al., 2025; Luzi et al., 2025). Polyester-based textiles are considered major contributors to MPF pollution during domestic laundering. For instance, De Falco et al. (2018) estimated that a 5 kg laundry load consisting solely of polyester fabrics could release more than 6 million MPFs into wastewater.

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Across the textile life cycle, the highest emissions have been reported during manufacturing (49%), followed by washing (28%) and wearing (23%) (Akyildiz et al., 2024; Lim et al., 2022). Domestic laundering is recognized as a key pathway for MPF emissions because mechanical agitation, fabric–fabric abrasion, and hydro-mechanical forces detach fibers from yarn surfaces and fabric structures. Comprehensive reviews describe how textile construction, yarn hairiness, finishing, and washing conditions (e.g., temperature, duration, liquor ratio, chemistry, and mechanical action) govern the magnitude of MPF shedding (Cesa et al., 2020; De Falco et al., 2019; Henry et al., 2019; Periyasamy and Tehrani-Bagha, 2022; Tedesco et al., 2024; Akyıldız et al., 2025b; Luzi et al., 2025; Himu et al., 2026; Theeratampitak & Charusiri, 2026). However, domestic laundering practices are globally heterogeneous, and hand-washing remains common in many regions. Hand-washing and machine washing involve different hydro-mechanical forces and can produce different MPF release patterns (Wang et al., 2023). Beyond laboratory observations, global modeling has estimated that laundry can generate emissions on the order of millions of tons of MPFs annually, highlighting the potential significance of this source at environmental scales (Wang et al., 2024).

Once released, MPFs may be partially captured in wastewater treatment systems; however, removal is incomplete, and MPFs can reach aquatic environments via treated effluent and sludge-related pathways, supporting the need for upstream mitigation at the textile level (Liu et al., 2021; Sun et al., 2019; Avilés et al., 2025). Following their release into aquatic systems, microplastic fibers have been widely detected across various environmental matrices, including surface waters, wastewater, sediments, rivers, and marine systems (Suaria et al., 2020; Ziajahromi et al., 2016; Athey and Erdle, 2022; Balestra et al., 2024). In textile wastewater treatment plants, microfiber concentrations of up to 825 MF L^{-1} in influent and 520 MF L^{-1} in effluent have been reported, with cellulose-based and polyester fibers as the dominant types (Akyıldız et al., 2025a). At broader environmental scales, ocean surface waters contain 0.02–25.8 microfibers L^{-1}, with a median concentration of 1.7 microfibers L^{-1}, further demonstrating their widespread distribution (Suaria et al., 2020). MPFs exhibit distinct pollution characteristics due to their high aspect ratio, persistence, and large surface area, which enhance their ability to adsorb and transport co-contaminants such as heavy metals, hydrophobic organic compounds, and microorganisms (Mishra et al., 2019; Frost et al., 2022; Aslam et al., 2025; Chouchene et al., 2026). These properties also influence their environmental fate by promoting transport, prolonged residence, and interactions with surrounding biota, thereby increasing the likelihood of biological exposure in aquatic systems (Wang et al., 2021). Due to their small size and fibrous morphology, MPFs are readily ingested by aquatic organisms, and may also cause physical abrasion, gut blockage, tissue injury, and trophic transfer, meaning that the fibers themselves can exert ecological harm beyond their role as contaminant carriers (Wang et al., 2021; Singh et al., 2020; Rakib et al., 2023). Recent studies have shown that MPF exposure can induce oxidative stress, inflammation, and intestinal damage, highlighting their ecotoxicological significance (O'Connor et al., 2025; Singh et al., 2020; Rakib et al., 2023; Chouchene et al., 2026).

In parallel with environmental concerns, potential human health implications of MPs have gained increasing attention. Fibrous particles are of particular interest due to their aerodynamic behavior and potential relevance to respiratory exposure (Song et al., 2024). In addition, MPFs may contain and release chemical additives (e.g., plasticizers and flame retardants), and these substances may leach into aquatic environments. Because additives such as phthalates and bisphenols (including BPA) are endocrine-active and have been associated with reproductive and genotoxic endpoints, additive leaching represents an additional ecotoxicological concern, alongside the physical and biological risks posed by the fibers themselves, for aquatic organisms and ecosystems (Chen et al., 2022; Hermabessiere et al., 2017; Jeng, 2014; Li et al., 2024; Meeker et al., 2009).

A continuing challenge in the MPF field is ensuring robust and comparable quantification across studies, as results can vary markedly with washing protocols, sampling/filtration steps, and analytical approaches (Palacios-Marín et al., 2026; Lupato et al., 2025). To improve reproducibility, standardized test methods have been developed. EN ISO 4484-1:2023 provides a harmonized laboratory method to collect and quantify material loss from fabrics during controlled laundering, enabling more consistent evaluation of shedding performance across textile types and mitigation strategies. In response to MPFs pollution, mitigation approaches include machine-level filtration, consumer behavior changes, textile design optimization, and chemical or physical surface treatments aimed at stabilizing fabric surfaces and reducing fiber detachment (Akyıldız et al., 2025b).

Surface treatments are especially promising because they can directly target fiber-shedding mechanisms—by stabilizing fiber ends, minimizing protruding fibrils, and/or creating protective films that reduce mechanical abrasion. Among bio-based options, chitosan has attracted attention as a potential finish due to its film-forming ability and capacity to bind fibers at the fabric surface. In controlled laundering studies, chitosan pretreatment markedly decreased MPFs release from polyester garments (up to ~95% under optimized conditions), highlighting its potential as an at-source mitigation strategy (Kang et al., 2021; Periyasamy and Tehrani-Bagha, 2022; Ramasamy & Subramanian, 2023). Among chemical approaches, alkali treatment (NaOH) is widely used in polyester processing and has been reported to reduce MPF shedding during laundering by modifying the fiber surface through hydrolysis-driven changes in wettability and morphology, thereby influencing fiber detachment and fragmentation behavior. Chemically modified polyester fabrics have shown reduced MPFs release and shorter average emitted MPF lengths under more severe alkali conditions, indicating that NaOH concentration and treatment intensity can critically affect shedding outcomes (Rathinamoorthy & Raja Balasaraswathi, 2022; Tavanai, 2009; Rathinamoorthy & Raja Balasaraswathi, 2023a; Čorak et al., 2022).

Plasma-based surface modification has also emerged as a clean and scalable strategy to improve textile surface functionality without altering bulk properties. By increasing surface energy and introducing polar functional groups, plasma activation can enhance the adhesion and durability of subsequent coatings or finishes. Importantly, plasma treatment has recently been reported to reduce the shedding mass of microplastic fibers from polyester textiles (e.g., ~43% reduction reported for oxygen plasma treatment), highlighting its potential as a mitigation tool either alone or in combination with chemical finishes (Jabbar et al., 2024; Leroux et al., 2009; McCoustra & Mather, 2019; Primc & Mozetič, 2024). However, comparative evidence remains limited on how plasma activation combined with specific chemical finishes alters not only the magnitude of MPF release but also emitted MPF size distributions under standardized ISO laundering conditions.

Previous studies have investigated plasma treatment and chemical modifications separately as strategies to reduce microplastic fiber release from synthetic textiles (Kang et al., 2021; Rathinamoorthy & Raja Balasaraswathi, 2023a; Rathinamoorthy & Raja Balasaraswathi, 2022; Ramasamy & Subramanian, 2023; De Falco et al., 2019). Plasma treatment can act as a surface activation step prior to chemical finishing, introducing polar functional groups and increasing the surface energy of polyester fibers, thereby enhancing the adhesion and effectiveness of subsequent chemical treatments such as chitosan or NaOH. In the present work, the focus is to evaluate the combined plasma-assisted treatment route rather than isolating the independent effects of plasma activation and chemical treatment.

Within this context, the present study evaluates plasma-assisted chemical surface modification as a strategy to reduce microplastic fiber (MPF) release from a 100% polyester knitted fabric during repeated washing. Atmospheric-pressure plasma activation was applied prior to chemical finishing using NaOH and chitosan at two concentrations (0.6% and 1.2%, w/v). By integrating mass-, count-, and

morphology-based metrics under standardized conditions, this study aims to clarify how plasma-assisted alkali and bio-based coatings influence MPF shedding mechanisms and to provide evidence for effective at-source mitigation of microplastic fibers. Because plasma-only and chemical-only controls were not included in the experimental design, the individual contributions of each treatment step cannot be separated, and the results should therefore be interpreted as the performance of the combined treatment route.

2. Materials and methods

2.1. Sample materials and preparation

A 100% polyester (polyethylene terephthalate, PET) interlock knitted fabric was used in this study. The fabric was supplied by a commercial textile manufacturer in Türkiye. Its structural characteristics included an areal weight of 200 g/m^2, a stitch density of 16 wales cm^{-1} and 20 courses cm^{-1}, an average fiber diameter of 11.6 μm, a yarn count of Nm 65 (metric number), an indirect yarn count system expressing yarn length per unit mass. Sample specimens (10 × 25 cm) were prepared in accordance with EN ISO 4484-1:2023, which standardizes methodologies for quantifying microplastic release from textile materials during washing procedures.

2.2. Plasma surface activation and optical emission spectroscopy

Surface activation was performed using atmospheric-pressure plasma glow discharge (APP-GDBD) at the Environmental Park facility (Turin, Italy). The system (Grinp S.r.l., Turin, Italy) operated with a gas mixture of He–O_2–H_2O under 1050 W power, maintaining an electrode temperature of 50 °C. Both sides of each fabric specimen were exposed to plasma for 30 s. This process was designed to enhance hydrophilicity and introduce polar functional groups (e.g., hydroxyl and carboxyl), thereby improving subsequent chemical treatment efficiency (Jabbar et al., 2024).

The plasma discharge was characterized using optical emission spectroscopy (OES) with an Ocean Optics LIBS2500 2plus spectrometer equipped with a QP600-2-SR/BX optical probe and an integration time of 100 ms. The recorded emission spectrum indicated the presence of excited helium species (501, 587, 667, and 706 nm) together with reactive species such as OH (309 nm), oxygen lines at 776 and 844 nm, and nitrogen-related bands (N_2 and N_2^+), which are commonly associated with plasma-induced surface activation processes in polymer substrates (Plăcinta et al., 1997). The OES analysis was used to qualitatively characterize the plasma discharge and confirm the presence of reactive species generated under the selected treatment conditions.

2.3. Chemical treatments

Four chemical solutions were prepared: sodium hydroxide (NaOH, Sigma-Aldrich) at 0.6% and 1.2% (w/v) and chitosan (Sigma-Aldrich) at 0.6% and 1.2% (w/v). NaOH solutions were prepared by dilution from a 0.1 M stock solution using deionized water. Chitosan solutions were prepared using distilled water containing citric acid, as chitosan is not soluble in water alone. For the 0.6% solution, 1.2 g of chitosan and 1.2 g of citric acid (Sigma-Aldrich) were added to 200 mL of distilled water. For the 1.2% solution, 2.4 g of chitosan and 2.4 g of citric acid were added to 200 mL of distilled water. The citric acid was first dissolved in water, after which chitosan powder was slowly added under constant magnetic stirring at 50 °C for 4 h to ensure complete dissolution and homogenization. Immediately following plasma activation, fabric specimens were immersed in the respective solutions. For each treatment condition, three fabric specimens were processed. After immersion, samples were padded and dried overnight at 40 °C. The experimental design included one untreated control (Raw) and four combined plasma-assisted chemical treatment groups. The untreated control received no plasma or chemical treatment, whereas all modified groups were first plasma treated and then chemically treated with either NaOH or chitosan at concentrations of 0.6% or 1.2%.

2.4. Washing protocol and filtration

Microplastic fiber release was quantified following EN ISO 4484-1:2023. All washing, filtration, and gravimetric analyses were conducted at STIIMA-CNR (Biella, Italy). Fabric specimens were sealed in stainless-steel containers with 360 mL of grade 3 demineralized water and 50 stainless-steel balls (6 mm diameter). Washing was carried out using a James Heal Gyrowash apparatus at 40 °C for 45 min. Each treatment was tested in triplicate (n = 3), and each replicate underwent five sequential wash cycles. Effluents from cycles 1, 3, and 5 were retained for analysis, while cycles 2 and 4 were not analyzed individually. This interval-based sampling strategy was selected to capture early-, intermediate-, and later-stage release behavior across repeated washing while maintaining analytical feasibility across all treatment groups and replicates. Similar interval-based approaches have been reported in previous studies on textile MPF release during laundering (Jabbar & Tausif, 2023; Jabbar et al., 2024).

To assess potential contamination, one blank control was included in each washing cycle. Blank samples consisted of containers filled with water and stainless-steel balls but no fabric. Identical washing and filtration procedures were applied. For count-based results, blank counts were subtracted from sample counts prior to averaging.

After each wash cycle, the wash liquor was collected. To recover

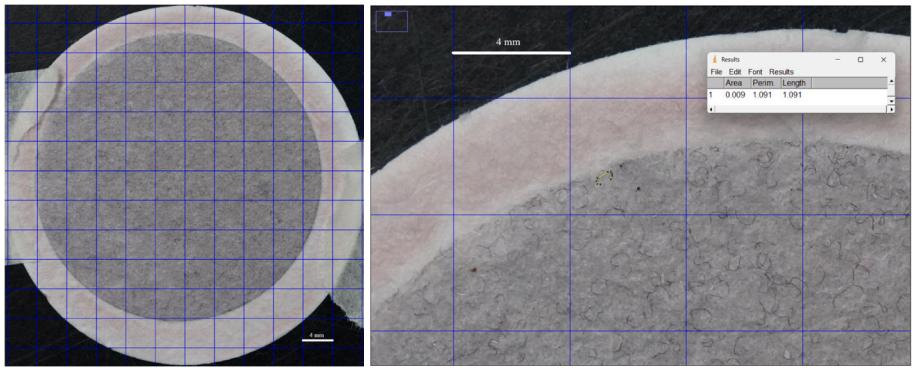


Fig. 1. Image analysis of MPFs released from raw polyester fabric after the 5th washing cycle. **(a)** Filter image with a grid overlay used to evaluate the distribution of MPF counts. **(b)** Magnified region showing MPF length measurement performed using ImageJ.

microplastic fibers remaining in the container, an additional rinse-only step was performed using the same container and stainless-steel balls but without fabric. The container and balls were rinsed with distilled water, followed by an ethanol rinse, and all rinses were combined with the corresponding wash liquor prior to vacuum filtration. This step was included to minimize MPF losses due to adhesion to container walls and stainless-steel balls. The combined effluent was vacuum-filtered through 47-mm binder-free glass fiber filters (1.2 μm pore size). Filters were transferred to aluminum foil, dried overnight, and stored in a desiccator until analysis. Containers and stainless-steel balls were cleaned with grade 3 demineralized water and ethanol between cycles to prevent cross-contamination. The same procedure was applied to all samples and procedural blanks.

2.5. Microplastic fiber analysis: mass, count, and length

Microplastic fiber release was assessed via gravimetric, count-based, and length-based methods. Filters and fabric specimens were dried overnight and equilibrated in a desiccator prior to weighing with an analytical balance of ±0.1 mg readability. Filters were weighed before and after filtration, while fabric specimens were weighed before and after washing. Desiccator equilibration was used to minimize weighing variability associated with residual moisture, and the gravimetric measurement capability of the method was governed by the balance readability (±0.1 mg precision).

For count analysis, filters were photographed under standardized lighting using a Sony ILCE-7RM3 camera with a Zeiss 100-mm macro lens. All filter images were acquired under identical lighting and optical conditions. Because MPF counting and length measurements were based on visible fibers in digital images, the practical lower detection threshold of the method was determined by image resolution and visual distinguishability from the filter background; accordingly, only clearly resolved fibers were included in the analysis. Image analysis was performed in ImageJ (Fig. 1). To estimate the total microplastic fiber count, 20 predefined grid zones, each with an area of 15 mm^2, were selected and analyzed on each filter. The total number of MPFs counted in these 20 grid zones was then multiplied by a correction factor of 4.63, corresponding to the ratio between the analyzed grid area and the total grid area covering the full filter surface. In addition, because the filtration device had a smaller diameter than the filter paper, a second correction factor of 1.2664 was applied to account for the ratio between the full filter area and the effective filtration area. Blank filters were fully analyzed across their entire surface, and no extrapolation was applied. The average MPF count from blank filters was directly subtracted to isolate MPF release attributable exclusively to the fabric treatments and exclude potential environmental or procedural contamination.

Statistical analyses were performed using replicate-level data (n = 3 per treatment group). Given the limited sample size, differences among treatment groups were assessed using the non-parametric Kruskal–Wallis test. When a significant overall effect was detected, post hoc pairwise comparisons were performed using multiple comparisons of ranked data. Statistical significance was accepted at $p < 0.05$. Results are presented as mean ± standard deviation.

For length-distribution analysis, a grid-based image-analysis approach was applied using ImageJ's calibrated line tool. All predefined squares along the filter diagonal were evaluated following the counting strategy reported by De Falco et al. (2018), in which all diagonal squares are included in the analysis (De Falco et al., 2018; Rathinamoorthy and Raja Balasaraswathi, 2023b). Fibers detected within these selected grid areas were measured and classified into four length intervals: <0.5 mm, 0.5–1.0 mm, 1.0–1.5 mm, and >1.5 mm. Results are presented as percentage distributions within each size class (mean ± SD, n = 3).

2.6. Abrasion testing

Surface durability and mechanical resistance were evaluated using a Martindale abrasion tester in accordance with ISO 12947. Abraded specimens were subsequently examined to compare surface damage and fiber integrity with untreated controls.

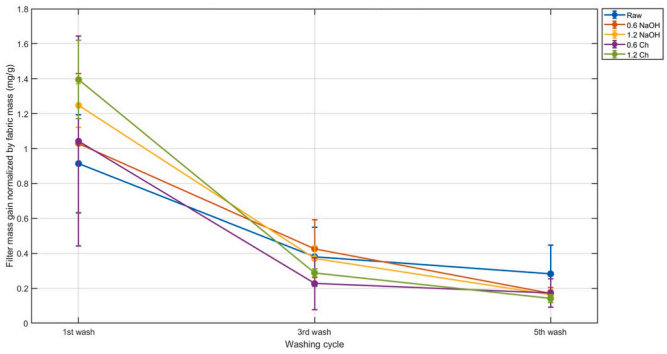


Fig. 2. Filter mass gain normalized by fabric mass (mg g^{-1} fabric) after the 1st, 3rd, and 5th washing cycles for untreated and treated samples. Values represent mean ± SD (n = 3).

2.7. FTIR, microscopic, and SEM analysis

Chemical characterization of raw and treated fabric surfaces was further investigated by Fourier transform infrared spectroscopy (FTIR) equipped with an attenuated total reflectance (ATR) accessory (Shimadzu AIM-9000). Microscopic images were obtained using a digital microscope (RH-2000, Hirox, HRS-3DT). In addition, the surface morphology, fiber degradation, and characteristics of released microplastic fibers were analyzed using scanning electron microscopy (SEM) (FEI Inspect). Images were acquired from: (i) fabrics before washing, (ii) fabrics after washing (after cycle 5), (iii) after Martindale abrasion of washed specimens, and (iv) MPFs retained on filters. SEM was used for qualitative morphological assessment of fabric surfaces and released fibers rather than as a primary quantitative method for MPF enumeration.

3. Results and discussion

3.1. Gravimetric analysis

Gravimetric results indicated a general decline in filter mass gain with repeated laundering, with substantially lower values observed in the third and fifth washing cycles compared with the first wash (Fig. 2). The error bars shown in Fig. 2 represent the standard deviation among replicates, indicating moderate variability between samples but consistent trends across treatments.

This decreasing trend is consistent with systematic observations for polyester textiles, where early washes remove loosely bound surface fibers and processing-derived debris, followed by reduced release as the readily detachable fraction becomes depleted. Here, filter mass gain is interpreted as retained solids representing total material loss because gravimetry is not polymer-specific and may include detached finishing residues. Statistical analysis of filter mass gain normalized by fabric mass showed no significant differences among treatment groups at any of the evaluated washing stages. The Kruskal–Wallis test gave $p = 0.2457$ for the 1st wash, $p = 0.5861$ for the 3rd wash, and $p = 0.4290$ for the 5th wash. Although differences in mean normalized filter mass gain were observed among treatments, these variations were not statistically significant under the present experimental conditions (n = 3) (Fig. 2).

The elevated filter mass observed in the first wash for the 1.2% chitosan-treated samples may not exclusively represent polyester MPFs, because gravimetric measurements capture total retained solids, including potential contributions from detached finishing residues. Chitosan coatings—particularly when not covalently fixed or

Table 1
MPF counts after the fifth wash cycle (mean ± SD, n = 3). Blank counts (31.20 ± 12.9) were subtracted from sample counts to obtain blank-corrected values.

Sample	Average MPFs Count (mean ± SD)
Raw	1622.1 ± 292.8
0.6% NaOH	1086.5 ± 219.8
1.2% NaOH	1033.8 ± 90.7
0.6% Chitosan	575.8 ± 85.9
1.2% Chitosan	937.5 ± 33.0

sufficiently crosslinked—can exhibit limited wash durability and may undergo partial removal during early laundering cycles (Pušić et al., 2023; Kalazić et al., 2024). Under such conditions, loosely bound chitosan residues may partially detach during early laundering, contributing to apparent filter mass gain without necessarily reflecting MPF emission. (Abu Bakar et al., 2025). The elevated first-wash filter mass observed for the 1.2% chitosan treatment may partly reflect detached finishing residues rather than polyester MPFs alone, since gravimetric analysis captures total retained solids. Although chitosan is a bio-based polymer and would not represent secondary microplastic pollution, its release may still increase the organic load of the wash effluent. However, this potential trade-off could not be directly evaluated in the present study because COD and BOD were not determined.

A similarly elevated first-wash mass was observed for the 1.2% NaOH-treated samples. One possible explanation is that the combined effects of plasma activation and subsequent alkaline exposure increased the susceptibility of the near-surface region to damage, thereby promoting fragmentation during the initial wash. Although NaOH treatments have been reported to reduce MPFs shedding from polyester by inducing surface hydrolysis and modifying surface morphology/wettability (Rathinamoorthy & Raja Balasaraswathi, 2023a), more aggressive conditions (e.g., higher concentration and/or prolonged exposure), particularly following plasma activation, may also lead to surface etching and localized weakening, which can increase early-cycle material loss.

By the third wash, treated samples exhibited lower mass release than untreated controls, consistent with the combined effects of (i) depletion of loosely attached fibers after the first cycles and (ii) improved surface stability due to the applied modifications. This interpretation aligns with Ramasamy & Subramanian (2023), who reported sustained reductions in MPFs shedding from polyester fabrics treated with chitosan- and alkali-based finishes over extended laundering, whereas untreated fabrics continued to release higher quantities of MPFs.

While gravimetric analysis provides an estimate of the total retained solids released during washing, it does not distinguish between microplastic fibers and other detached materials. Therefore, a complementary count-based analysis was performed to quantify the number of released microplastic fibers, as discussed in Section 3.2.

3.2. Count-based analysis

The quantity of microplastic fibers released after the fifth washing cycle was quantified by count-based analysis. To control for background contamination, results were blank corrected by subtracting the mean MPFs count measured in procedural blanks (31.2 ± 12.9 MPFs/filter) from each replicate prior to calculating treatment means and standard deviations. The resulting blank-corrected MPF counts per filter are summarized in Table 1, where the untreated fabric (Raw) exhibited the highest release and all treated fabrics showed reduced MPF counts. These results demonstrate the efficacy of chemical and plasma-assisted surface modifications in mitigating MPF shedding during laundering (Akyildiz et al., 2025). Compared with the untreated raw PET fabric (1622.1 ± 292.8 MPFs), all treated fabrics exhibited lower mean MPF counts. The lowest count was observed for the 0.6% chitosan treatment

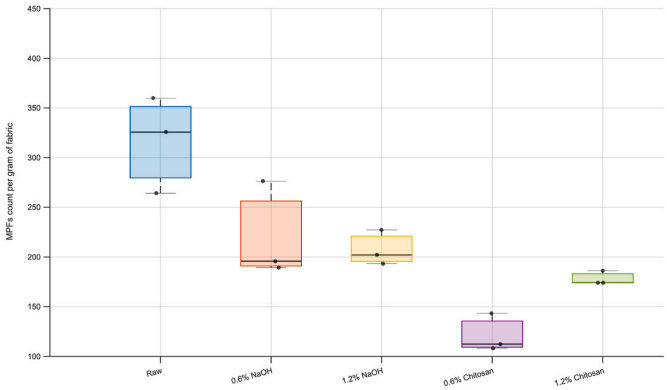


Fig. 3. Microplastic fiber counts normalized by fabric mass after the fifth wash cycle (MPFs g^{-1} fabric; n = 3 per treatment).

(575.8 ± 85.9 MPFs), followed by 1.2% chitosan (937.5 ± 33.0 MPFs), 1.2% NaOH (1033.8 ± 90.7 MPFs), and 0.6% NaOH (1086.5 ± 219.8 MPFs). For graphical presentation and statistical comparison, the blank-corrected count data summarized in Table 1 were normalized by fabric mass and expressed as MPFs g^{-1} fabric (Fig. 3). Kruskal–Wallis analysis of the normalized data showed a significant overall treatment effect ($p = 0.0146$), and post hoc pairwise comparison indicated a significant difference only between the untreated control and the 0.6% chitosan treatment ($p = 0.0122$) (Table 1, Fig. 3). No other pairwise comparisons were statistically significant ($p > 0.05$).

Among the NaOH-treated groups, MPF counts were reduced by 32.4 ± 5.4% (0.6% NaOH) and 34.8 ± 7.1% (1.2% NaOH) relative to untreated controls. Chitosan treatments resulted in even greater mitigation, with reductions of 63.0 ± 4.2% (0.6% Chitosan) and 40.1 ± 11.0% (1.2% Chitosan). Chitosan pretreatment, particularly at 0.6%, demonstrated the strongest effect in reducing release. This trend is consistent with prior studies reporting that chitosan coatings on synthetic textiles can significantly reduce MPF emissions during washing—by up to ~95% in some cases—owing to their film-forming capacity and surface-binding potential (Kang et al., 2021). Such coatings may act as physical barriers, encapsulating surface fibers and inhibiting detachment during mechanical agitation. The superior performance of the 0.6% chitosan treatment compared with the 1.2% concentration may be related to differences in coating morphology and stability. Lower chitosan concentrations can form relatively thin and uniform films on the fiber surface, enhancing fiber cohesion and reducing detachment during laundering. In contrast, higher concentrations may produce thicker or less homogeneous coatings that are more prone to cracking or partial removal under mechanical stress, thereby reducing their mitigation efficiency. Similar concentration-dependent behavior has been reported in textile finishing studies using chitosan-based coatings (Kang et al., 2021).

NaOH treatments also resulted in reductions in MPF shedding. This outcome aligns with previous work demonstrating that alkali treatment of polyester fibers alters surface morphology and improves hydrophilicity, thereby reducing the extent of fiber fragmentation during laundering (Rathinamoorthy and Raja Balasaraswathi, 2023a).

For cross-treatment comparability, the dataset was additionally normalized to specimen mass and expressed as MPFs per gram of fabric (MPFs g^{-1}) (Fig. 3). Mass normalization is important because small differences in specimen weight among replicates can influence absolute counts when reported on a per-filter basis. The normalized results indicate that MPF release count per gram of fabric increased in the following order: 0.6% chitosan <1.2% chitosan <0.6% NaOH $\lesssim$ 1.2% NaOH < Raw, indicating that the 0.6% chitosan treatment resulted in the lowest MPF release. This pattern confirms that plasma-assisted chemical surface modification, particularly chitosan-based finishing, was the most effective among the tested conditions for reducing MPFs

Table 2
Microplastic fiber counts and filter mass gain after the fifth wash cycle (mean ± SD, n = 3). Mass gain reflects total retained solids.

Sample	MPFs Count (mean ± SD)	Filter Mass Gain (mg)
Raw	1622.1 ± 292.8	1.5 ± 0.8
0.6% NaOH	1086.5 ± 219.8	0.87 ± 0.2
1.2% NaOH	1033.8 ± 90.7	0.83 ± 0.2
0.6% Chitosan	575.8 ± 85.9	0.77 ± 0.4
1.2% Chitosan	937.5 ± 33.0	0.87 ± 0.1

release.

3.3. Mass–count comparison

Microplastic fiber release after the fifth wash was evaluated using two complementary approaches: MPFs count and filter mass gain (Table 2). Because gravimetry quantifies total retained solids rather than polymer-specific MPFs mass, finishes (especially chitosan) may contribute detached residues that increase filter mass without increasing MPFs counts; therefore, gravimetry is interpreted here alongside MPF counts, length distributions, and SEM observations. As expected, the untreated fabric (Raw) showed the highest release, with an average of 1622.1 ± 292.8 MPFs per filter and a filter mass gain of 1.5 ± 0.8 mg. These results are consistent with previous findings indicating that untreated polyester textiles release both higher MPFs counts and higher mass than surface-treated fabrics (Periyasamy & Tehrani-Bagha, 2022).

Notably, the relationship between mass and count was not strictly proportional across treatments. The 0.6% chitosan group displayed the lowest count but a mass gain comparable to 0.6% NaOH, suggesting that either (i) longer and/or thicker MPF contributed disproportionately to mass and/or (ii) a fraction of non-fibrous or weakly attached finishing residues contributed to gravimetric gain without increasing the number of counted MPFs. This highlights a recognized limitation of relying on gravimetry alone when coatings or finishes may partially detach; therefore, pairing mass measurements with imaging and morphology-based analyses strengthens interpretation. Similar effects have been reported for polymeric coatings such as chitosan, which may shed non-fibrous fragments when not strongly fixed to the fabric (Periyasamy & Tehrani-Bagha, 2022).

In contrast, NaOH-treated fabrics exhibited a more parallel decrease in both mass and count, suggesting a different shedding/fragmentation behavior. Alkali treatment is known to modify polyester surface properties via hydrolysis and morphological changes, which can influence fiber detachment and the size distribution of released MPFs (Rathinamoorthy & Raja Balasaraswathi, 2023a).

Overall, these results underline the importance of combining count- and mass-based metrics: counts reflect the number of discrete released MPFs, whereas gravimetry reflects total retained material, potentially including detached finishes or non-fibrous fragments. This distinction is particularly relevant when comparing synthetic substrates with bio-derived surface finishes, which may contribute additional non-microplastic residues. All treated groups exhibited lower mass release than raw. Notably, 0.6% chitosan minimized MPFs counts, whereas 1.2% NaOH yielded the lowest filter mass gain, indicating that both finishing chemistry and concentration influence shedding dynamics. Collectively, the mass–count comparison supports plasma-assisted chemical surface modification as an effective strategy to mitigate MPFs release from polyester textiles during repeated laundering.

3.4. Length distribution

The length distribution of microplastic fibers released after the fifth washing cycle was assessed to examine whether plasma-assisted chemical treatments influenced MPFs fragmentation and detachment behavior. Microplastic fiber lengths were classified into four intervals: <0.5 mm, 0.5–1.0 mm, 1.0–1.5 mm, and >1.5 mm (Fig. 4).

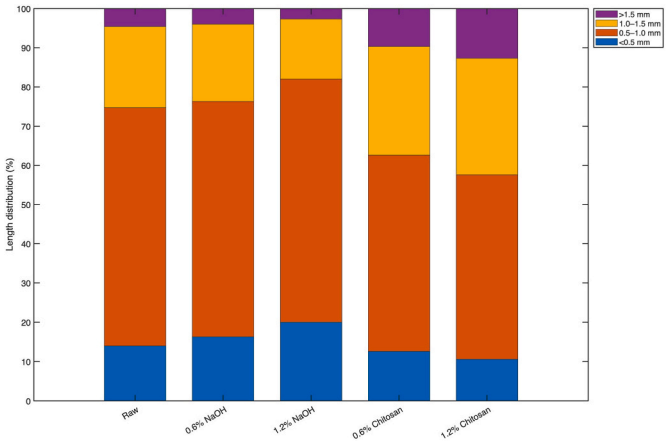


Fig. 4. Length distribution (%) of released microplastic fibers collected after the fifth wash cycle for each treatment.

Across all sample groups, the dominant fraction of released MPFs was within the 0.5–1.0 mm interval, accounting for 47.0–62.0%. However, the treatments induced noticeable shifts in the relative contribution of longer MPFs. Compared with the raw fabric, in which fibers ≥1.0 mm accounted for 25.3% of the measured fraction, the 0.6% chitosan and 1.2% chitosan treatments exhibited substantially higher proportions of MPFs ≥1.0 mm, reaching 37.4% and 42.4%, respectively. This increase was driven primarily by a larger 1.0–1.5 mm fraction and, particularly for chitosan-treated samples, by a higher contribution of the >1.5 mm class. In the case of chitosan, this shift toward a greater long-fiber fraction is consistent with a surface-stabilization mechanism: chitosan is known to form films and promote fiber–fiber binding, which can increase surface cohesion and reduce mechanically induced micro-damage during laundering (Kang et al., 2021). Accordingly, the reduced count-based release observed for the chitosan-treated fabrics, especially 0.6% chitosan (Section 3.2), may reflect both fewer detachment events and a shift in the size distribution of the emitted MPFs.

In contrast, the 1.2% NaOH treatment produced the highest share of short MPFs, with 82.0% of measured fibers below 1.0 mm, and the lowest >1.5 mm fraction (2.7%). This pattern suggests enhanced fragmentation into shorter pieces under laundering stress. The 0.6% NaOH treatment showed a distribution closer to that of the raw sample, with the 0.5–1.0 mm class remaining dominant and only a modest contribution of longer fibers (≥1.0 mm = 23.7%). The stronger shift toward shorter fibers observed for 1.2% NaOH is consistent with previous reports that alkali treatment of polyester can alter surface morphology and influence shedding behavior through hydrolysis-driven surface etching, with the outcome depending on treatment severity (Rathinamoorthy & Raja Balasaraswathi, 2023a).

Taken together, the length-based results complement the count-based findings by showing that surface treatments influenced not only the amount of MPF release but also the size characteristics of the emitted fraction. In particular, chitosan-treated fabrics, especially at 1.2%, showed a relatively greater contribution of longer fibers, whereas the 1.2% NaOH treatment was associated with a stronger shift toward shorter length classes. These treatment-dependent differences in emitted fiber size are environmentally relevant because fiber length may influence transport, persistence, and biological interactions in aquatic systems.

3.5. FTIR, microscopic, and SEM analysis

ATR-FTIR spectra collected before washing retained the characteristic absorption bands of PET in all samples, indicating that the main polyester backbone remained preserved after treatment. Only moderate

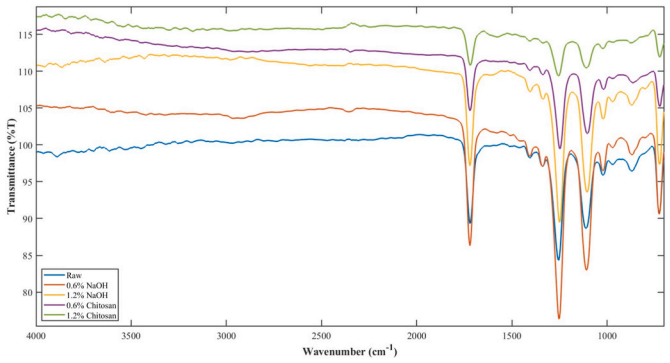


Fig. 5. ATR-FTIR spectra of raw and plasma-assisted chemically treated PET fabrics (0.6 and 1.2% NaOH; 0.6 and 1.2% chitosan) collected before washing.

differences in intensity and spectral profile were observed between raw and treated fabrics, particularly in the fingerprint region. The relatively subtle changes may be related to the low concentration of the applied chemical treatments and to the fact that the ATR-FTIR response remained dominated by the underlying PET substrate. Thus, the FTIR results are consistent with limited surface modification rather than major bulk structural alteration (Fig. 5).

Microscopic images revealed the presence of microplastic fibers retained on the filter surface after washing. Observations were performed at magnifications corresponding to scale bars of 500 μm and 50 μm (Fig. 6). The images show numerous MPFs distributed across the filter surface. The fibers exhibited elongated shapes, smooth surfaces, and relatively uniform diameters characteristic of synthetic polyester fibers, facilitating their visual identification as textile-derived MPFs.

Scanning electron microscopy (SEM) was used to qualitatively evaluate surface morphology and damage features of the polyester fabric before washing, after washing, after abrasion, and to examine the morphology of released MPFs retained on the filters (Fig. 7). It should be noted that the plasma treatment applied in this study was relatively short and primarily intended for surface activation rather than extensive etching or structural modification of the polyester fibers. Consequently, plasma exposure alone did not induce pronounced morphological changes on the fiber surfaces but rather facilitated improved interaction between the polyester substrate and the subsequent chemical treatments. Consistent with observations reported by Morent et al. (2008), mild atmospheric plasma treatments primarily modify surface chemistry rather than inducing significant topographical changes on polymer fibers. In the raw fabric before washing, fibers exhibited relatively smooth surfaces and compact yarn structures typical of knitted PET. After washing, visible signs of mechanical disturbance were observed, including local fiber displacement and surface fibrillation. Following abrasion, raw samples showed pronounced damage, with clear fiber breakage and protruding fibrils, indicating reduced resistance to repeated mechanical stress.

NaOH-treated samples exhibited surface modifications that were consistent with treatment-driven changes in polyester morphology. After abrasion, both 0.6% and 1.2% NaOH samples displayed evidence of increased surface disruption, including fractured fiber ends and localized splitting, suggesting that alkali exposure may have weakened near-surface regions and promoted fragmentation under mechanical loading.

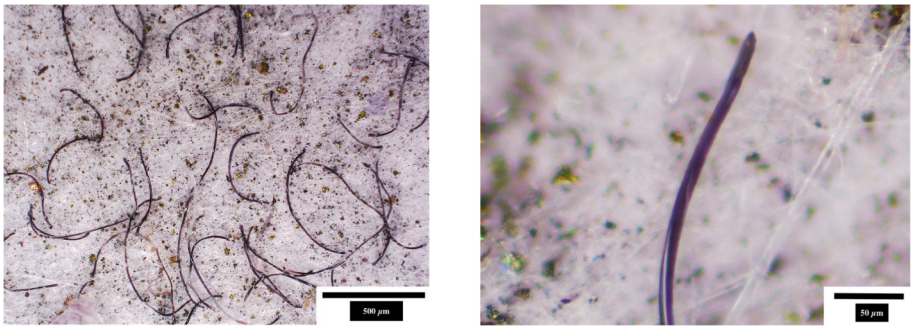
In contrast, chitosan-treated fabrics showed a comparatively more cohesive fiber arrangement after washing, suggesting improved stabilization of the fiber/yarn surface. After abrasion, although chitosan-treated samples still exhibited damage features (e.g., broken protruding fibers), the overall surface structure appeared less disrupted than NaOH-treated fabrics. Overall, SEM observations confirm that plasma-assisted chemical surface modification influences both fabric surface integrity and microplastic fiber detachment mechanisms.

4. Conclusion

This study investigated plasma-assisted chemical surface modifications as an at-source mitigation strategy for microplastic fiber release from a 100% PET knitted fabric during repeated washing under EN ISO 4484-1:2023 conditions. By integrating gravimetric measurements, image-based MPF counting, length distribution analysis, and SEM observations, the work provides a comprehensive evaluation of how surface modification influences both the quantity and characteristics of released MPFs.

All treatments reduced MPF release relative to the untreated fabric, demonstrating that modifying the fiber surface can effectively mitigate laundering-induced MPF detachment. Among the evaluated conditions, chitosan-based finishing delivered the strongest mitigation in count-based results, with 0.6% chitosan achieving the lowest MPF emission in the fifth wash. NaOH treatments also reduced release, but with lower effectiveness than chitosan under the tested conditions. Gravimetric outcomes generally supported these trends by showing lower mean filter mass gains for treated samples than untreated controls, although these differences were not statistically significant under the present experimental conditions.

The length distribution results demonstrated that treatments influenced not only the amount of released MPFs but also their size characteristics. Chitosan-treated groups exhibited increased proportions of MPFs $\geq$1.0 mm compared with untreated fabric, whereas 1.2% NaOH yielded the highest proportion of short MPFs (<1.0 mm), suggesting a treatment-severity-dependent shift in fragmentation behavior. SEM observations qualitatively supported these findings by revealing treatment-dependent differences in surface disruption after washing and abrasion, and corresponding differences in the presence of MPFs on filters.



(a) (b)

Fig. 6. Optical microscopy images of microplastic fibers (MPFs) retained on the filter after washing of polyester fabric. (a) Overview of MPFs distributed on the filter surface (scale bar = 500 μm). (b) Magnified view of an individual MPF showing typical polyester fiber morphology (scale bar = 50 μm).

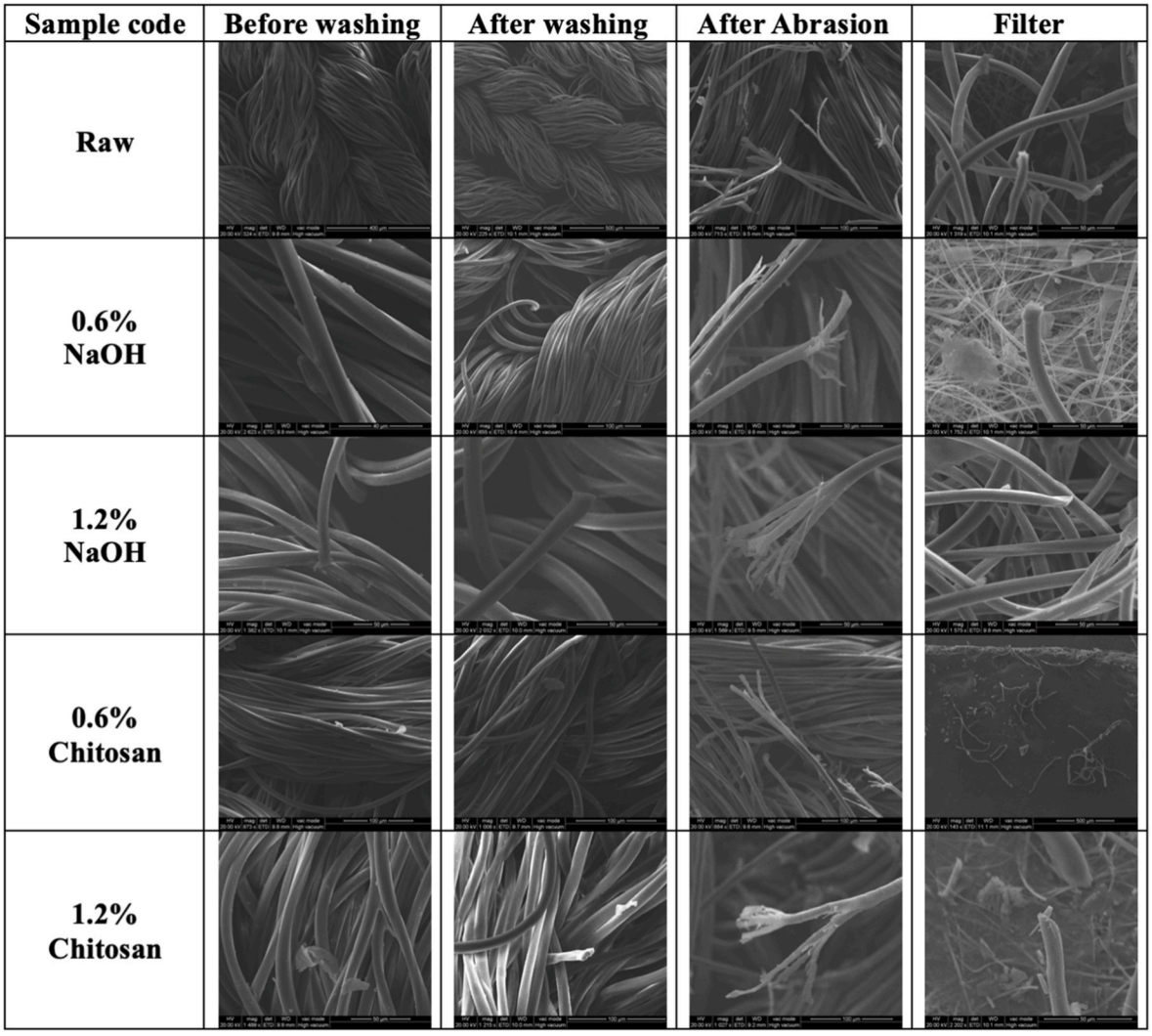


Fig. 7. Representative SEM photos of PET knit fabric surfaces and released MPFs for untreated and surface-treated samples. Columns show (i) before washing, (ii) after washing (after cycle 5), (iii) after Martindale abrasion of washed specimens, and (iv) MPFs retained on filters from the corresponding wash effluent.

Overall, the results demonstrate that plasma-assisted chemical surface modification—particularly low-concentration chitosan finishing—represents a promising and potentially scalable strategy to mitigate microplastic fiber release from polyester textiles during laundering.

A limitation of the present study is that plasma-only and chemical-only control groups were not included. Therefore, although the results demonstrate the performance of the combined plasma-assisted chemical treatments, the independent contributions of plasma activation and subsequent chemical finishing cannot be quantitatively separated in the current experimental design. Future work should include these controls to distinguish independent, additive, and potentially synergistic effects.

CRediT authorship contribution statement

Sinem Hazal Akyıldız: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Conceptualization. **Barış Alp Sevgi:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Raffaella Mossotti:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Domenico D'Angelo:** Writing – review & editing, Methodology, Conceptualization. **Rossana Bellopede:** Writing – review & editing, Supervision.

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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Data availability

Data will be made available on request.

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