Tanja Pušić[1], Ana Šaravanja[1], Tea Bušac[1], Kristina Šimić[1], Nino Dimitrov[2], Mirjana Čurlin[3]

Assessment of polyester knitted fabrics and effluents from standard and innovative washing processes

[1]University of Zagreb Faculty of Textile Technology
[2]Croatian Institute for Public Health
[3]University of Zagreb Faculty of Food Technology and Biotechnology

Abstract

The purpose of this research was to establish the link between the surface of pristine polyester knitted fabrics before and after alkali hydrolysis modification, as well as the composition of effluents following standard and innovative washing processes. Using the streaming potential method, substantial variations in the surface of the pristine knitted fabrics after ten washes in the standard and innovative washing processes were discovered in comparison to the alkali-hydrolysed knitted fabrics. The results of the tests carried out with the selected processes establish a relation between the properties of pristine and the alkaline hydrolysed knitted fabrics and the composition of the effluents and filter cakes. The results revealed that the innovative washing process has the potential for further investigations into the release of microfibers from knitted polyester fabrics.

***Keywords**: polyester knit, alkali hydrolysis, washing, microfibers*

1. Introduction

The global issue of the past few decades has been the presence of a variety of microscopic particles, such as spheres, fragments, and debris, or fibres, in the environment from different sources, including wastewater, air, soil, and sediments. Microplastic (MP) is a collective name for small plastic fragments with an approximate length of 1 to 5 mm or for fibres with a length of 3 to 15 mm and a length to diameter ratio > 3 [1, 2]. Microfibres (MFs) are similar in size to microplastics, but their composition is not exclusively limited to plastics. Microfibres of natural origin are biodegradable, but the functionalisation of textiles made from natural fibres can slow down or prevent biodegradation, which can be harmful to aquatic organisms [3]. The fragments from synthetic materials such as polyamide (PA), polyester (PES), polypropylene (PP), polyacrylonitrile (PAN), polyethylene (PE) and polyurethane (PU) in the form of flexible foams pose a significant environmental threat to internal organs due to their accumulation [4, 5]. Studies indicate that synthetic textiles are a source of secondary MPs, i.e. microplastics produced by fragmentation, weathering/ageing or maintenance of larger items [6]. Among these sources, household clothes washing has the greatest potential for the formation of microplastics and microfibers [7-9]. Many structural factors influence the formation and release of microfibres when washing synthetic textiles, including type, geometry, yarn type, processing, etc. [4]. The extent of the changes depends on the factors of the washing process, which are determined by a Sinner cycle (chemicals, temperature, time, mechanical agitation) [10].

Accordingly, intensive research has been conducted on the washing process of synthetic textiles over the last decade, with some of the research devoted to factors in the washing process [11, 12], prevention of particle release [13, 14], installation of filters in household washing machines [15, 16] and methodology of analysis of released particles [17, 18]. For a long time obtaining comparable and accurate results was difficult due to the lack of standardised methods, protocols and approaches. The American Association of Textile Chemists and Colorists, AATCC, released the first technique for evaluating particle/fibre fragment release from home washing in 2021, AATCC TM212-2021 [19] which goes some way to resolving the inconsistencies in the protocol for conducting the washing process and the method for analysing the released particles that are dubious when assessing the extent of pollution in the effluent from the washing process. The procedure defines key terms that are often the subject of discussion in scientific and professional communities when it comes to defining released particles, namely: Fibre, Fibre Fragment and Microfibre [20].

Further progress followed in 2023 with the announcement of three parts of the international standard ISO 4484 dedicated to microplastics from textile sources [21-23]. Standard ISO 4484-1 describes a protocol and gravimetric method for material loss of all types from fabrics under washing test conditions [21]. Detergent has been left out of the test method due to clogging of filters during the filtration procedure, stick to fibers and filters, which can interfere with fiber fragment release by adding mass and lead to misinterpretation of results. Despite these facts, the washing process in water, without detergent, has a different effect on fiber fragment release than washing with detergent. ISO 4484-2 [22] specifies a method for determining microplastics (from the textile industry) collected in various matrices (such as textile process wastewater,

clothes washing water, textile process air emissions, and textile process solid waste). ISO 4484-3 [23] specifies a method for measuring the collected material mass released from the outlet hose of a standard washing machine, during the washing operation, as specified in EN ISO 6330 [24].

The research contribution to this interdisciplinary topic from 2020 is provided by the project HRZZ-IP-02-2020, 7575 "Assessment of microplastic shedding from polyester textiles in washing process" [25]. The research area of this project focuses on the assessment of microplastics of textile origin released into the environment using innovative washing process and chitosan treatment of polyester fabrics in an eco-friendly manner.

The standard method, EN ISO 6330, is adjusted in the innovative washing process to adapt the washing parameters for synthetic textiles, polyester and polyester/cotton fabrics, and knitted fabrics. Surface modification using the biopolymer chitosan is used in the environmentally friendly treatment of polyester and polyester/cotton fabrics and knitted fabrics to minimize MP shedding from synthetic textiles into the environment. Alkaline hydrolysis of polyester textiles is one of existing methods for modifying the surface of polyester to optimize its interaction with chitosan. This topochemical reaction in sodium hydroxide solution is saponification, which occurs through the hydrolysis of ions attached to the carbonyl group in the polyester chain [26-28].

This research focuses on the investigation of pristine and alkaline hydrolysed polyester knitted fabric as a donors of textile origin particles in standard and innovative washing processes with reference detergent at 60 °C. The effects of these processes were investigated using the fabric surface properties before and after ten washing cycles, the composition of the effluents collected after ten cycles, and the filter cake.

2. Experimental

Material

Double faced interlock standard polyester knit, MRF-0008, Wfk, Germany with a mass per unit area of 139 g/m^2, Dh= 16 stitches/cm and Dv = 21 stitches/cm was used in the research.

Alkali hydrolysis

The fabric was alkaline hydrolysed in a Mathis laboratory apparatus for 30 minutes in a 2 % sodium hydroxide solution at 98 °C. This was followed by two hot rinse water cycles and two cold rinse water cycles.

Washing process

The washing of pristine and alkali-hydrolysed PES knitted fabric was performed in SDL Atlas Rotawash equipment using the 2A protocol of standard EN ISO 6330: 2021[24] in a solution of 1.25 g/L ECE A phosphate-free reference detergent [29] at 60 °C with a bath ratio of 1:7. The fabric was rinsed four times with cold water at a temperature of 20 °C and a bath ratio of 1:8 after each cycle. The innovative process follows the 2A protocol for washing, with modifications made for gradual cooling throughout the four times rinsing. The first cycle is done at 50 °C, the second at 40°C, the third at 30°C, and the fourth at 20 °C. After each individual wash and rinse cycle, the effluents were collected as composite samples from 10 washings. Table 1 lists the characteristics of PES knitted fabrics, both before and after ten washes.

Table 1. Designation and description of samples

Labels	Description of standard polyester knit
P_PES	Pristine
P_PES-St_10	Pristine 10 times washed by standard process
P_PES-In_10	Pristine 10 times washed by innovative process
P_PES_AH	Alkali hydrolysed
P_PES_AH-St_10	Alkali hydrolysed 10 times washed by standard process
P_PES_AH-In_10	Alkali hydrolysed 10 times washed by innovative process

Characterisation of the polyester knitted fabrics

The fabric surface before and after alkaline hydrolysis and 10 washing cycles was characterised by determining the zeta potential as a function of pH 1 mmol/L KCl using the streaming potential method in the SurPASS electrokinetic analyser, A. Paar, Austria. Polyester knitted fabrics with a higher percentage of breaks and wrinkles are a potential donor of particles in the wash and rinse cycles, so their surface and appearance [30] before and after 10 wash cycles of the standard and innovative processes was evaluated by a panel of four examiners and presented as an average value. The surface appearance of the pristine and 10 times washed samples after cyclic rubbing of 125, 600, 1000, 2000, 5000 and 7000 cycles before and after washing in dry condition was evaluated according to standard method [31]. The surface of the polyester knitted fabrics previously coated with gold and palladium for a period of 90 s was examined with a scanning electron microscope (SEM) tt. Tescan, MIRA /LMU, Czech Republic using magnifications of 1,000x.

Characterization of washing effluents

The effluent was separated into a filtrate and a filter cake using membrane filtration; a glass fibre membrane with a pore size of 0.7 μm was selected for this purpose.

Since microplastic emissions are not directly monitored, according to the Best Available Techniques (BAT) reference document for the textile industry should be considered as total suspended solids (TSS) [32, 33]. Accordingly, the analysis of the composite effluent samples was performed by determining TSS [34], turbidity (NTU) according to [35] and pH [36]. In addition, the particle size distribution (PSD) was determined by the laser diffraction method using the particle size analyser PSA 1090 LD, A.

Paar, Graz, Austria [37]. All measurements were taken in triplicate and the results were reported as mean values.

To facilitate sampling of the filter cake sample using a digital microscope, the fibrous formations on the surface were counted and labelled as a preliminary measure for the examination of the filter cake sample by pyrolysis-gas chromatography-mass spectrometry (Py-GC/MS). Measurements were carried out using a micro-furnace pyrolyzer (EGA/Py-3030D, Frontier Laboratories, Ltd.) equipped with an auto-shot sampler (AS-1020E, Frontier Laboratories, Ltd.). The pyrolyzer was interfaced directly to a split/splitless injection port of a GC/MS instrument (GCMS Shimadzu QP2010 Plus). The GC injection port was connected to a quadrupole mass detector through a separation column (Ultra ALLOY+-5, 30 m×0.25 mm i.d., coated with 0.5 µm film thickness of 5% diphenyl 95 % dimethylpolysiloxane, Frontier Laboratories, Ltd.) and a vent-free GC/MS adapter (Frontier Laboratories, Ltd.). The detailed analytical conditions are listed in Table 2.

Table 2. Analytical conditions for Py-GC/MS

Instrument	Parameters	Settings
Pyrolyzer	Furnace temperature	600°C
	Interface temperature	300°C
GC	Injection port temperature	300°C
	Column oven temperature	40°C (2 min hold) - 320°C (20°C min^{-1}, 16 min hold
	Flow Control mode	Pressure
	GC/MS interface temperature	300°C
	Injection mode	Split (split ratio: 1:16)
	Carrier gas	Helium (column flow rate: 0.87 mL min^{-1})
MS	Ion source temperature	250°C
	Ionization method	Electron ionization (EI), 70 eV
	Scan range	*m/z 29– 350*

The pyrolysis temperature was preheated at 600 °C and the resulting pyrolyzates of the sample of 0.2 mg, placed into the deactivated stainless steel sample cup.

The qualifications and identifications of peaks in the chromatograms were confirmed by comparing the mass spectrum of each peak in the chromatogram with those in data search libraries of F Search all in one ver. 3.8 (Frontier Laboratories Ltd., Japan) and NIST/EPA/NIH (NIST 17).

3. Results and discussion

The effects of standard and innovative washing processes on the surface properties of polyester knitted fabrics were analysed by streaming potential, wrinkle appearance, resistance to surface fuzzing and pilling and scanning electron microscopy. Zeta potential values of pristine and alkali-hydrolysed PES knitted fabric before and after 10 washing cycles according to standard and innovative procedures in variation of the pH of 1 mmol/L KCl are shown in Fig. 1.

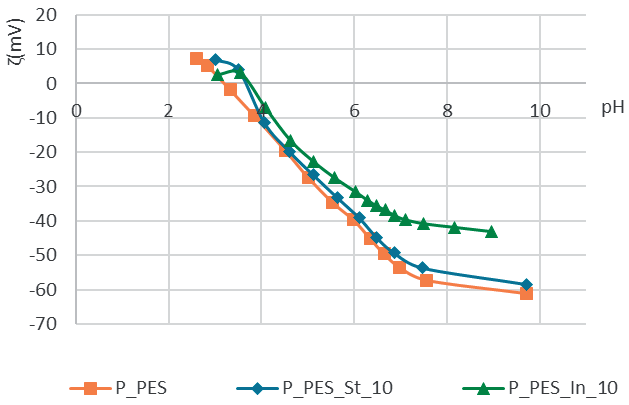

Fig. 1a. Zeta potential of pristine knitted fabrics before and after 10 washing cycles according to standard and innovative process in variation of the pH of 1 mmol/L KCl

Figure 1a shows the zeta potential of pristine (P_PES) knitted fabric sample as shown by a typical titration curve [38]. The zeta potential values of polyester knitted fabrics washed according to standard process (P_PES-St_10) are nearly equal to the values pristine samples. Polyester knitted fabrics washed 10 times using an innovative process (P_PES-In_10) had a lower negative impact than the original sample (P_PES). When compared to the standard wash, the knitted fabric that went through the new method exhibited a lower negative charge, which might imply that its hydrophilicity was improved.

Zeta potential of alkali-hydrolysed PES knitted fabric before and after 10 washing cycles according to standard and innovative process in variation of the pH of 1 mmol/L KCl is shown in Fig. 1b.

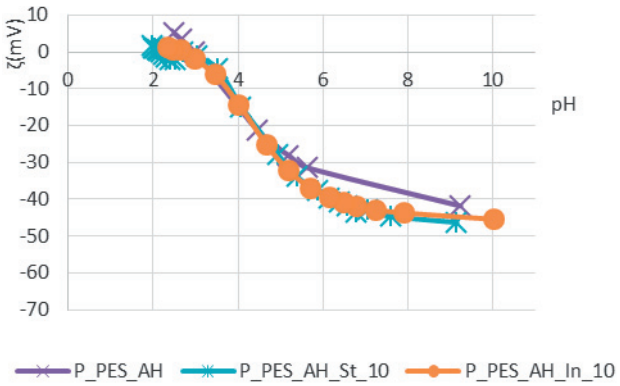

Fig. 1b. Zeta potential of alkali-hydrolysed knitted fabrics before and after 10 washing cycles according to standard and innovative process in variation of the pH of 1 mmol/L KCl

The influence of the alkaline hydrolysis process (AH) on the surface of the fabric is demonstrated by decreasing the

negative value of the zeta potential compared to the original PES fabric in the observed pH range, and the value of the isoelectric point (IEP) of the alkaline hydrolysed fabric confirms the modification of the surface.

Titration curves for alkaline hydrolysed polyester fabric were identical when washed 10 times using the standard procedure (P_PES_AH-St_10) and the innovative method (P_PES_AH-In_10). The differences between the two alkaline processes, hydrolysis and standard washing process, are confirmed by the obtained ratios of the zeta potential titration curves. The degree of modification of the polyester fabric during alkaline hydrolysis is more reflected in the change of surface properties [26, 39, 40], and both the standard and innovative alkali washing processes have no influence on the surface of the alkaline hydrolysed samples.

SEM micrograph of knitted fabrics before and after standard and innovative washing processes are shown in Fig. 2.

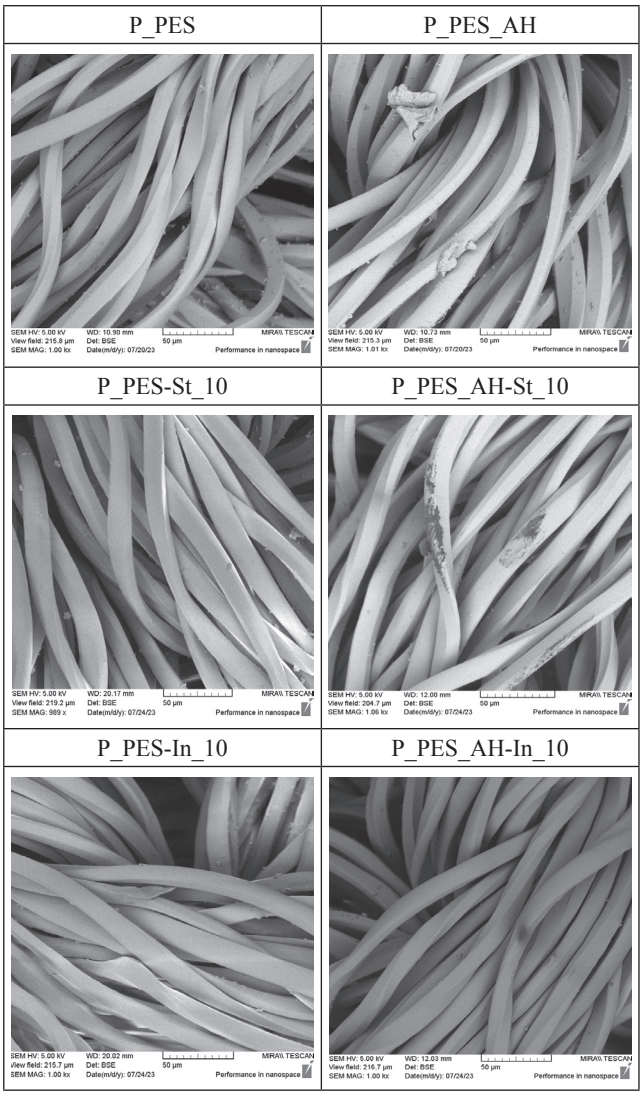

Fig. 2. SEM micrographs of knitted fabrics under magnification of 1,000x

Micrographs of PES knitted fabrics before and after 10 cycles of the standard and innovative washing processes reveal no modifications, showing that the washing procedures have no prominent effect on the surface of PES knitted fabrics (Fig. 2). The micrographs of the alkaline hydrolysed knitted fabric after the standard and innovative washing processes differ. The surface of this sample is partially peeled after the standard washing process, in contrast to the sample washed using the innovative process, where no surface changes are visible.

Despite this, all knitted fabrics score a 5 for resistance to surface fuzzing and pilling, which is evaluated both before and after 10 washing cycles using standard and innovative methods. Grades for wrinkle appearance of assessed after ten cycles washings of PES knitted fabrics are summarised in Table 3.

Table 3. Surface appearance grades

P_PES-St_10	P_PES-In_10	P_PES_AH-St_10	P_PES_AH-In_10
2-3	3	1-2	1-2

Grade: 1- surface with many wrinkles; 5 – smooth surface

The results (Table 3) demonstrate that alkaline hydrolysed PES knitted fabrics washed using standard and innovative processes have a greater level of wrinkling than PES knitted fabrics washed using these processes.

Characterisation of the washing effluents

Composite effluents of 10 cycles washing were evaluated by TSS, NTU, pH listed in Table 4, and particle size distribution as shown in Fig. 3 and Fig. 4.

Table 4. Characteristics of 10 cycles washing effluents

Effluent	TSS (mg/L)	NTU	pH
P_PES-St_10	128.3	70.2	7.8
P_PES-In_10	150.5	68.4	8.4
P_PES_AH-St_10	113.3	48.7	7.8
P_PES_AH-In_10	171.5	42.2	8.1

When the results of the characterization of the composite effluents from 10 cycles washing PES knits (P_PES) and alkaline hydrolysed (P_PES_AH) according to the standard and innovative processes are compared (Table 4), a higher content of total suspended solids, TSS, can be observed in the effluent of the innovative process. The surfactant components in the detergent are primarily responsible for the turbidity of the effluent. The obtained results show that the turbidity value of alkaline hydrolysed knitted fabrics is lowered. The surface is modified and the structure is opened up by alkaline hydrolysis. This modification improves the surface ability to interact with detergent ingredients that affect turbidity. The pH of the composite phase effluents from the innovative washing process (pH ~ 8) is higher than that from standard washing.

Figure 3 shows the PSA analysis results as a particle size distribution curve in effluents from 10 cycles of washing PES knitted fabrics using standard and innovative methods, whereas Figure 4 shows effluent after washing alkaline hydrolysed PES knitted fabrics using the same processes.

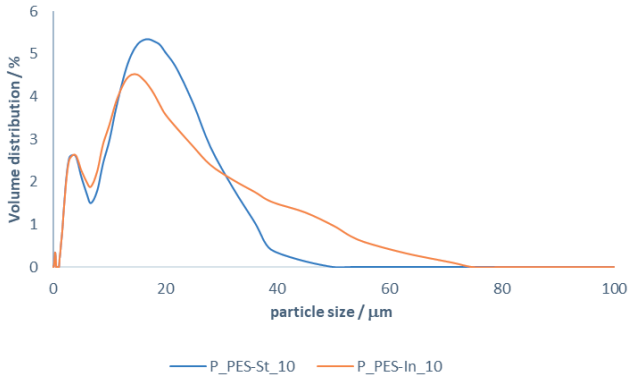

Fig. 3. Volume distribution of particle sizes in the effluent from 10 cycles washing PES knitted fabric (P_PES) using the standard and innovative processes

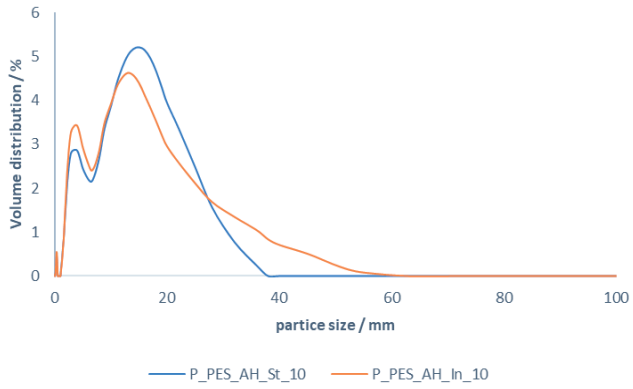

Fig. 4. Volume distribution of particle sizes in the effluent from 10 cycles washing alkaline hydrolysed PES knitted fabric (P_AH_PES) using the standard and innovative processes

The particle size distribution curves for effluents from the standard and innovative washing processes of PES knitted fabrics (P_PES and P_PES_AH) differ in shape, with the innovative process exhibiting a greater range of particle sizes and the lowest volume distribution.

The characteristic diameters D_{10}, D_{50}, D_{90}, average diameter (A) and their ratio as Span value and k parameters of the curve computed from the acquired distribution curves using the computer support of the PSA instrument are presented in Table 5.

Table 5. Characteristic parameters for PSD curves

Parameter	P_PES-St_10	P_PES-In_10	P_PES_AH-St_10	P_PES_AH-In_10
D_{10} (µm)	2.014	2.029	1.866	1.794
D_{50} (µm)	10.776	10.130	8.641	7.473
D_{90} (µm)	23.748	28.836	19.924	21.446
Average (µm)	12.246	13.544	10.216	10.345
k	1.117	1.091	1.122	1.073
Span	2.017	2.646	2.090	2.630

The mean particle diameter in the effluent from washing alkali hydrolysed knitted fabric has reduced, and equivalent values are seen in the effluent from standard and innovative processes. The alkaline hydrolysis affects the particles of smaller diameter in comparison to pristine polyester knitted fabric. According to the reported ratios of characteristic diameters, indicated as span value, the wastewater from the innovative washing process has a higher value. The lowest value of the shape factor k is typical for the effluent from the innovative process involving both PES knitted fabrics.

Filter cakes produced by filtering effluents from 10 cycles of standard and innovative washing processes (PES -St_10; PES -In_10; AH -St_10; AH -In_10) were analysed using Py - GC/MS. The extracted ion chromatographs (EIC), with particular mass values of the selected samples were compared. When comparing the EICs, no significant difference was found between the analysed samples of the cake filter sample after 10 cycles of the standard and innovative washing processes. For the comparison EIC with filter cake samples (PES-St_10; PES-In_10; AH-St_10; AH-In_10) the polyester (AATCC & ISO) multifibre test fabric # 10A (DW) textile standard was used. Pyrolytic decomposition of the textile standard revealed that benzoic acid is one of its main components. The characteristic mass spectrum and EIC of benzoic acid with the intensities of the main m/z values of the polyester (Terylene) textile standard is shown in Fig. 5.

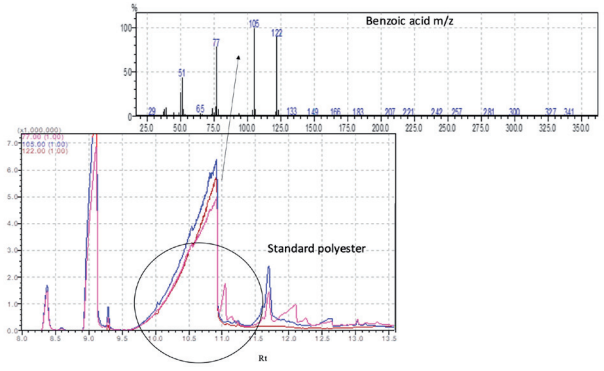

Fig. 5. EIC of standard polyester Multifiber test fabric # 10A (DW) with characteristic mass fragments m/z of benzoic acid

A match may be detected after a specified retention period by monitoring and comparing the EICs, with specific m/z values typical for benzoic acid, of the standard samples with the filter cake samples, Figs. 6-9.

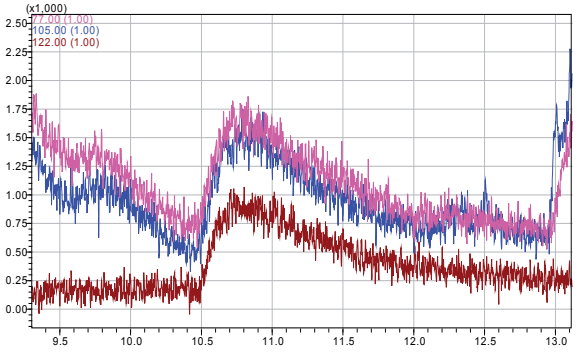

Fig. 6. EIC of filter cake P_PES-St_10

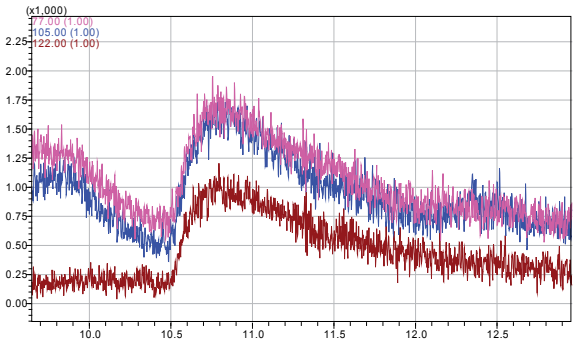

Fig. 7. EIC of filter cake P_PES-In_10

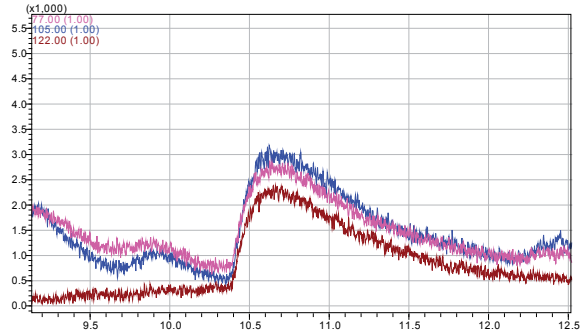

Fig. 8. EIC of filter cake P_PES-AH-St_10

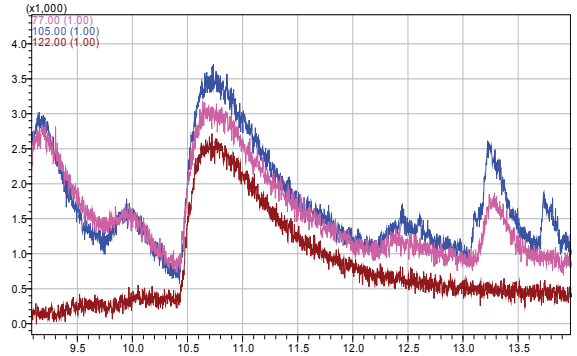

Fig. 9. EIC of filter cake P_PES-AH-In_10

All of these results might point to polyester as the source of the benzoic acid intensities that were seen in the filter cake samples (Figs. 6–9).

4. Conclusions

The surface of pristine PES knitted fabric has been affected by alkaline hydrolysis, as evidenced by the zeta potential curves.

In comparison to the innovative one, washing did not significantly modify the surface of the PES knitted fabric, resulting in a smaller negative surface charge.

The value of the zeta potential does not indicate the difference between the standard and innovative washing processes of alkaline hydrolysed PES knitted fabrics.

Grade 5 for all knitted fabrics showed no tendency to surface fuzz and pilling before and after 10 washing cycles.

The average particle diameter in the effluents from washing of alkali-hydrolysed polyester knitted fabric is smaller than in the effluents from washing of pristine polyester knitted fabric by standard and innovative washing process.

The intensities of benzoic acid originating from the polyester may be seen in the obtained programs of all filter cakes.

The results of the research carried out using the selected methods connect the properties of pristine and alkaline hydrolysis modified knitted fabrics with the composition of the effluent and the filter cake.

Funding

This research was funded by the Croatian Science Foundation, grant number HRZZ-IP-2020-02-7575.

Acknowledgments

The research was performed on equipment purchased by K.K.01.1.1.02.0024 project “Modernization of Textile Science Research Centre Infrastructure” (MI-TSRC).

5. References

[1] ECHA, Information note on restriction report for intentionally added microplastic particles, 2019. https://echa.europa.eu/documents/10162/5a730193-cb17-2972-b595-93084c4f39c8 (15.9.2023.)

[2] Choobar, B. G., Shahmirzadi, M. A. A., Kargari, A., Manouchehri, M. Fouling mechanism identification and analysis in microfiltration of laundry wastewater, Journal of Environmental Chemical Engineering 7 (2019) 103030; doi:10.1016/j.jece.2019.103030

[3] Periyasamy, A.P. Microfiber Emissions from Functionalized Textiles: Potential Threat for Human Health and Environmental Risks. Toxics (2023) 11, 406

[4] Periyasamy, A.P. Environmentally Friendly Approach to the Reduction of Microplastics during Domestic Washing: Prospects for Machine Vision in Microplastics Reduction. Toxics (2023) 11, 575. https://doi.org/10.3390/toxics11070575

[5] Bhavsar, P., Bhave, M., Webb, H.K. Solving the plastic dilemma: the fungal and bacterial biodegradability of polyurethanes. World J Microbiol Biotechnol 39, 122 (2023). https://doi.org/10.1007/s11274-023-03558-8

[6] Šaravanja, A., Pušić, T., Dekanić, T. Microplastics in Wastewater by Washing Polyester Fabrics. Materials (2022) 15, 2683. https://doi.org/10.3390/ma15072683

[7] Pušić, T., Vojnović, B., Flinčec Grgac, S., Čurlin, M., Malinar, R. Particle Shedding from Cotton and Cotton-Polyester Fabrics in the Dry State and in Washes. Polymers (2023) 15, 3201. https://doi.org/10.3390/polym15153201

[8] Schöpel, B., Stamminger, R. A Comprehensive Literature Study on Microfibres from Washing Machine. Tenside Surfactants Detergents 56 (2019) 94-101

[9] Henry, B., Laitala, K., Klepp, I. G. Microfibres from apparel and home textiles: Prospects for including microplastics in environmental sustainability assessment. Science of the Total Environment. 20 (2019) 2, 483-494. doi: 10.1016/j.scitotenv.2018.10.166. Epub 2018 Oct 12. PMID: 30368178.

[10] Sinner, H. Über das Waschen mit Haushaltswaschmaschinen, 2nd ed.; Auflage Haus und Heim Verlag, Hamburg, Germany, 1960, 9–10.

[11] Hernandez, E., Nowack, B., Mitrano, D. M. Synthetic Textiles as a Source of Microplastic from Households: A Mechanistic Study to Understand Microfiber Release During Washing. Environmental Science & Technology, 51 (2017) 7036–7046.

[12] Dris, R., Gasperi, J., Mirande, C., Mandin, C., Guerrouache, M., Langlois, V., Tassin, B. A first overview of textile fibres, including microplastics in indoor and outdoor environments. Environmental Pollution 221 (2017) 453–458

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

[14] McIlwraith, H. K., Lin, J., Erdle, L.M., Mallos, N., Diamond, M. L., Rochman, C. M. Capturing microfibres-marketed technologies reduce microfiber emissions from washing machines. Marine Pollution Bulletin 139 (2019) 40–45.

[15] Pribauer, B., Laminger, T., Ipsmiller, W., Koch, D., Bartl, A. Assessment of microplastics in the Environment—Fibres: The disgregarded twin? Detritus (2019) 9, 1–12

[16] Volgare, M., Avolio, R., Castaldo, R., Errico, M.E., El Khiar, H., Gentile, G., Sinjur, A., Susnik, D., Znidarsic, A., Cocca, M. Microfiber Contamination in Potable Water: Detection and Mitigation Using a Filtering Device. Microplastics (2022), 1, 322-333 https://doi.org/10.3390/microplastics1030024

[17] Tiffin, L., Hazlehurst, A., Sumner, M., Taylor, M. Reliable quantification of microplastic release from the domestic laundry of textile fabrics. Journal of Textile Institute 113 (2021) 558–566

[18] Mariano, S., Tacconi, S., Fidaleo, M., Rossi, M., Dini, L. Micro and Nanoplastics Identification: Classic Methods and Innovative Detection Techniques. Frontiers in Toxicology, 26 (2021) https://doi.org/10.3389/ftox.2021.636640

[19] AATCC TM212-2021: Test Method for Fiber Test Method for Fiber Fragment Release During Home Laundering

[20] Kaurin, T., Šaravanja, A. Protokoli za analizu otpuštenih čestica u procesu kućanskog pranja. Zbornik radova 15. Znanstveno-stručnog savjetovanja Tekstilna znanost i gospodarstvo. Vujasinović, E., Dekanić, T. (ur), Tekstilno-tehnološki fakultet, Zagreb, 26.1.2023., 2023, 126-130

[21] ISO 4484-1:2023: Textiles and textile products - Microplastics from textile sources - Part 1: Determination of material loss from fabrics during washing

[22] ISO 4484-2:2023: Textiles and textile products -Microplastics from textile sources, Part 2: Qualitative and quantitative analysis of microplastics;

[23] ISO 4484-3:2023: Textiles and textile products - Microplastics from textile sources, Part 3: Measurement of collected material mass released from textile end products by domestic washing method

[24] EN ISO 6330: 2021: Textiles - Domestic washing and drying procedures for textile testing

[25] https://inwashed.wixsite.com/unizg-ttf, 1.9.2023.

[26] Grancarić, A. M., Pušić, T., Kallay, N. Modifikacija poliesterskog vlakna alkalnom hidrolizom. Polimeri 12 (1991) 9-12, 141-146.

[27] Hilal, N. M., Gomaa, S., ELsisi, A. Improving Dyeing Parameters of Polyester/Cotton Blended Fabrics by Caustic Soda, Chitosan, and Their Hybrid', Egyptian Journal of Chemistry 63(2020) 6, 2379-2393. doi: 10.21608/ejchem.2020.25571.2498

[28] Čorak, I., Tarbuk, A., Đorđević, D., Višić, K., Botteri, L. Sustainable alkaline hydrolysis of polyester fabric at low temperature. Materials (2022) 15(4), 1−17, doi: 1.21608/ejchem.2020.25571.2498

[29] Kaurin, T.; Čurlin, M.; Šaravanja, A.; Vojnović, B.; Pušić, T. Design of Chitosan-Polyester Composites to Reduce Particulate Contamination of Washing Wastewater. Water (2023), 15, 2418. ttps://doi.org/10.3390/w15132418

[30] AATCC Test Method No. 124, Test Method for Smoothness Appearance of Fabrics after Home Laundering

[31] EN ISO 12945-2:2020, Textiles: Determination of fabric propensity to surface fuzzing and to pilling, Part 2: Modified Martindale method

[32] Roth, J., Zerger, B., De Geeter, D., Gómez Benavides, J., Roudier, S. Best Available Techniques (BAT) Reference Document for the Textiles Industry, Publications Office of the European Union, Luxembourg, 2023, doi:10.2760/355887, JRC 131874, p. 68.

[33] Se Jeong, L., Young-Kwon, P., Hyein, K., Jihye, K., Hye, M.M., Yeonsook, L., Atsushi, W., Norio, T., Hajime, O., Young-Min, K. Selective solvent extraction and quantification of synthetic microfibers in textile laundry wastewater using pyrolysis-gas chromatography/mass spectrometry. Chemical Engineering Journal 434 (2022), 134653, https://doi.org/10.1016/j.cej.2022.134653.

[34] EN 872:2005: Water quality - Determination of suspended solids -- Method by filtration through glass fibre filters

[35] ISO 7027-1:2016 Water quality - Determination of turbidity - Part 1: Quantitative methods

[36] EN ISO 10523:2012, Water quality - Determination of pH

[37] ISO 9276-5:2020, Representation of results of particle size analysis — Part 5: Methods of calculation relating to particle size analyses using logarithmic normal probability distribution

[38] Grancarić, A. M., Tarbuk, A., Pušić, T. Electrokinetic properties of textile fabrics. Coloration Technology 121 (2005) 221-227

[39] Pušić, T., Kaurin, T., Liplin, M., Budimir, A., Čurlin, M., Grgić, K., Sutlović, A., & Volmajer Valh, J. The Stability of the Chitosan Coating on Polyester Fabric in the Washing Process. Tekstilec 66 (2023) 85–104. https://doi.org/10.14502/tekstilec.66.2023010

[40] Kaurin, T., Pušić, T., Čurlin, M. Biopolymer textile structure of chitosan with polyester. Polymers (2022) 14(15), 1−15, doi: 10.3390/polym14153088