

Article

# Assessment of Polyester Fabrics, Effluents and Filtrates after Standard and Innovative Washing Processes

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**Abstract:** Textile materials from polyester fibres are sensitive to washing, especially at higher temperatures, due to their thermoplastic properties, hydrophobic nature and sensitivity to the alkaline medium. The issue of microplastic fibres' (MFs) release from polyester textiles is a topic that attracts the attention of researchers from different scientific fields, since microplastics are now among the serious environmental risks. In this study, two washing protocols, a standard and an innovative procedure, were presented, aiming to preserve the properties of polyester fabrics and reduce the pollution of washing effluents. The standard procedure followed HRN EN ISO 6330, while the innovative procedure was a modification of the standard that involved gradually cooling the bath before rinsing. The effects of these washing protocols were studied based on the physicochemical properties of the fabrics compared to the unwashed material, the composition of the effluents, and the filtrates after 10 cycles. The characterisation parameters of the fabrics, effluents and filtrates according to the standard and the innovative washing protocols showed differences in the observed parameters during the 10 washing cycles. The obtained results show the usefulness of the proposed concept of cooling the bath before rinsing in order to preserve the properties of polyester fabrics and reduce the load of washing effluents. Hierarchical cluster analysis (HCA) also confirmed differences in pH, conductivity and turbidity for effluents and filtrates from standard and innovative washing protocols.

**Keywords:** fabric; polyester; washing; effluents; characterisation

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## 1. Introduction

Within the synthetic fibre group, polyester is the most widely used in the world, with an estimated growth rate of 34% over the next decade [1,2].

Textiles made of polyester have certain advantages compared to other textiles, but also disadvantages that have been highlighted recently, especially due to their negative impact on the environment, which may be related in part to the effects of wearing them in dry and wet conditions, as well as alkalinity, mechanical stress and increased washing temperatures [3,4]. Alkaline hydrolysis of polyester by strong alkalis and high processing temperatures leads to irreversible changes in the polyester material and increases its reactivity and hydrophilic character [5–7].

A global problem in recent decades is the presence of microplastic particles in the environment (wastewater, air, soil and rocks). Microplastics are all plastic particles whose size/diameter is less than 5 mm and which can appear in various forms, such as spheres, fragments or fibres, and can be divided according to their origin into primary and secondary plastics [5,8,9].

Microplastic fibres (MFs) are released when synthetic fabrics are treated in horizontal and vertical household washing machines. According to the results of some studies, it is

estimated that about 30% of these particles are released into the environment through the washing process of polyester textiles [10–13].

According to some estimates,  $162 \pm 52$  MFs/g of clothing is released in one wash cycle, so about 6 million MFs are released annually from 5 kg of polyester clothing [9,13].

The release rate of these particles depends on the properties of the textiles and the stages of the technology and life cycle [14]. Quantification of released MF has been extensively studied, but the large number and variety of methods make it difficult to draw conclusions about the magnitude of the impact [9]. The estimation of MF loading during a washing cycle depends on the parameters of the washing process and on the structural parameters and properties of polyester textiles as a source of MF [15–17].

Preventive measures are needed to reduce the rate of released fibres, and one of the possible solutions to reducing the release of MFs from synthetic textiles is their structural properties' functionalisation, e.g., with chitosan [18,19], and optimal conditions in the washing process according to the principles of Sinner's cycle [20,21]. It is also possible to prevent MF emissions by implementing mitigation strategies in washing machines [22].

Shedding of MFs may be caused by temperature differences during washing and rinsing, which can sometimes exceed 30 °C, i.e., washing at 60 °C and rinsing with water at  $14 \pm 3$  °C.

Thus, the study addresses the effects of two washing protocols for polyester fabrics, the standard and the innovative protocols. The standard washing protocol was performed according to HRN EN ISO 6330 [23], while the innovative washing protocol was the standard protocol modified by a cool-down concept.

The effects of these washing protocols were studied based on the physicochemical properties of the fabrics after 10 cycles compared to the unwashed material, the composition of the effluents and the filtrates after the first, second, third, fourth, fifth and sixth to tenth washing cycles.

Polyester fabrics before and after standard and innovative washing protocols were analysed based on surface characterisation (electrokinetic properties, SEM images, tendency of pilling and wrinkles) and tensile properties.

The physicochemical properties of the effluents and filtrates were monitored based on pH, conductivity, turbidity, total solids (TS), total dissolved solids (TDS) and total suspended solids (TSS) using standard analytical methods [24]. All effluent and filtrate characterisation data were used for hierarchical cluster analysis (HCA). After membrane filtration of the wastewater, the filter cake was examined by digital microscopy.

## 2. Materials and Methods

The study was performed using standard white polyester staple fibre fabric supplied by the Centre for Tesmaterials. B.V., The Netherlands, with a surface mass of 156 g/m<sup>2</sup>, density 27.7 threads/cm in the direction of the warp, and 20 thread/cm in the weft direction, with the fineness of the warp thread being 30.4 tex and the warp threads 31.9 tex.

The standard washing process was carried out with the reference detergent ECE A (non-phosphate detergent) at a concentration of 2.0 g/L at 60 °C and a bath ratio of 1:8 according to the standard method of HRN EN ISO 6330 in a laboratory washing equipment Linitest, Original Hanau. The washing protocol consists of 1 main wash and 4 rinses with water at 30 °C, followed by extraction and air drying.

The innovative washing protocol, designed as a standard protocol, was modified by cooling; fresh water was added at the end of the main wash cycle at 60 °C until the bath temperature was reduced to 30 °C by a temperature gradient of 4 °C/min. The effluents from the 1st, 2nd, 3rd, 4th and 5th wash cycles according to the standard and innovative protocols were collected separately, while the effluents from the 6th to 10th were pooled for further analysis.

Polyester fabrics were designated as non-washed (N), polyester fabric washed 10 times with the standard protocol (N-St\_10) and polyester fabric washed 10 times with the innovative protocol (N-In\_10).

Surface characterisation of fabrics marked in this way was carried out using methods of streaming potential, scanning electron microscopy, propensity to pilling and smoothness appearance.

The streaming potential of polyester fabrics was measured using electrokinetic analyser SurPASS, A. Paar, Graz, Austria, depending on the pH 1 mmol/L KCl. Next, the equations H-S a zeta potential values were calculated [25].

The surface of the polyester fabrics was observed by means of a scanning electron microscope tt. Tescan, MIRA/LMU, Brno, Czech Republic, at a magnification of 3000 $\times$ . Prepared samples for microscopy were previously coated with gold (Au) and palladium (Pd) over a period of 90 s.

The smoothness appearance of polyester fabrics after being subjected to repeated standard and innovative protocols was also assessed [26].

The propensity of polyester fabrics to surface fuzzing and to pilling was assessed by grades after 125, 500, 1000, 2000, 5000 and 7000 rubs [27].

Tensile properties of polyester fabrics through maximum force and elongation at a maximum force were determined using a Tensolab 3000 dynamometer, Mesdan s.p.A., Puegnago del Garda, Italy [28]. The change in breaking strength ( $\Delta F$ ) was calculated as a percentage of the difference between breaking force of fabrics before and after 10 washing cycles according to standard and innovative protocols.

For a complete evaluation of the innovative protocol in relation to the standard protocol of washing polyester fabrics, it is of particular importance to determine the physico-chemical parameters of the washing effluents using standard methods.

Physico-chemical characteristics of processing waters were monitored by determining the pH value, conductivity, turbidity, total solids (TS), total dissolved solids (TDS) and total suspended solids (TSS) using standard analytical methods. Characteristics TS, TSS and TDS were determined by implementation of in-house methods. Conductivity was measured using the conductivity meter CG 853 and pH using the pH meter CG 842, both supplied by Schott, Mainz, Germany [29,30]. Turbidity of effluents was determined by Turbidimeter, Hach TL2350, Manchester, UK [31].

After membrane filtration of effluents using a filter with 0.45 mm diameter and pore size of 0.7  $\mu\text{m}$ , Ahlstrom-Munksjö, Aalen, Germany, filtrates (f) of the 1st, 2nd, 3rd, 4th and 5th and collected baths from the 6th to the 10th were analysed by measuring pH, conductivity and turbidity. All effluent and filtrate characterisation data were used for hierarchical cluster analysis. HCA is a set of methods for multidimensional classification with the aim of forming groups of similar objects, taking into account all grouping features simultaneously [32].

Filter cakes obtained from the 1st, 2nd, 3rd, 4th and 5th and from the 6th to the 10th standard and innovative protocols were observed by digital microscopy, Dinolite AM7013MZT, type Premier, The Netherlands, and characterised by counting of MFs.

### 3. Results and Discussion

Studies on MF release during washing that examined fibre release as a function of material type, washing/drying method and variables tested [33] revealed the complexity, research priorities for improving MF monitoring and mitigation strategies [34], difficulties in comparison and the need for global standardisation protocols and methods.

This study focuses on standard and innovative washing polyester fabrics without impurities with reference detergents at 60 °C in 10 cycles. Results are presented for each observed system: polyester fabric, wash effluent, filtrate and filter cake.

#### 3.1. Characterisation of Polyester Fabric

The effects of standard and innovative washing protocols on polyester fabric modification were evaluated by surface characterisation.

The analysis of the surface area of unwashed and washed standard fabrics based on zeta potential as a function of pH at 1 mmol/L KCl is shown in Table 1.

**Table 1.** Zeta potential of fabrics in a variation of pH 1 mmol/L KCl.

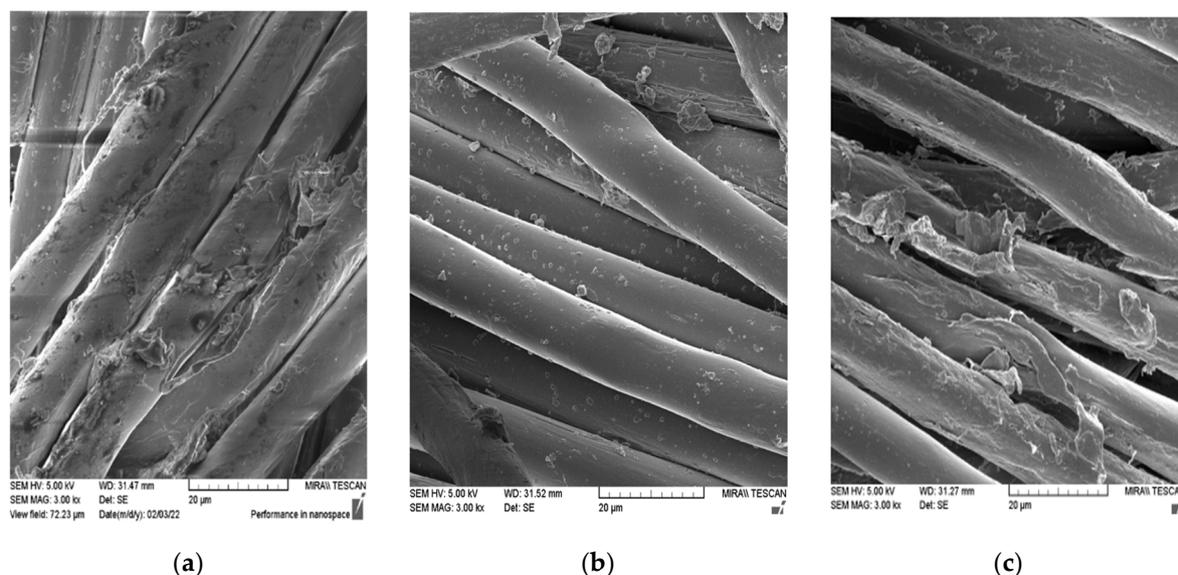
N		N-St_10		N-In_10	
pH	$\zeta$ (mV)	pH	$\zeta$ (mV)	pH	$\zeta$ (mV)
9.95	−17.80	9.54	−32.70	9.96	−35.0
6.39	−13.30	6.82	−30.30	6.55	−29.0
5.62	−10.90	6.19	−28.10	5.94	−25.0
4.98	−8.22	5.85	−25.70	5.44	−21.0
4.45	−5.25	5.34	−21.60	4.84	−15.0
3.75	−1.58	4.83	−15.30	3.97	−7.0
3.2	0.454	3.97	−6.59	3.39	0.97
2.71	1.594	3.47	2.17	2.9	9.95
2.45	1.786	2.97	13.60	2.55	8.30

The zeta potential of standard polyester fabric before and after standard and innovative washing protocols has positive and negative values. Unwashed fabric has the lowest negative zeta potential in the whole pH range, and the magnitude is similar to that of cellulose fabrics [35,36]. Such low negative values of zeta potential of standard polyester fabrics indicate a deviation in comparison with polyester textiles, which usually have a much more negative zeta potential [35]. This state of the surface may indicate the presence of preparations covering the surface and preventing the complete dissociation of the reactive groups of this polymer in an alkaline medium and adsorption in an acidic medium. Polyester fabrics washed according to standard and innovative protocols are more negative than unwashed ones in the whole pH range. The magnitude of the zeta potential values for these two samples shows that the effects of both protocols are similar. The difference in the size of the zeta potential of the washed samples according to the standard and innovative protocols is specific only for pH values below 3.5. The washed standard sample (N-St\_10) has more positive values than the washed innovative sample (N-In\_10). According to the results obtained, this is not due to the conductivity of an electrolyte solution in the titration process. Since the chemical structure of the polyester fabric is not changed during washing according to both protocols, this behaviour could be due to the release of the specific compounds from the surface. Similar isoelectric points of all fabrics prove that no chemical change occurred during the 10 cycles of washing.

The characterisation of the fabric surface before (N) and after 10 cycles of standard (N-St\_10) and 10 cycles of innovative (N-In\_10) washing protocols was carried out using the scanning electron microscopy method with a magnification of 3000 $\times$ ; see Figure 1.

The alkali content of the detergent, temperature and mechanical agitation during several washing cycles resulted in slight surface changes in the polyester fabrics studied. The morphologies of samples N and N-In\_10 are similar, while the surface of sample N-St\_10 is smoother.

The panels evaluated the appearance of the washed fabrics primarily based on the presence of wrinkles over 10 wash cycles. The average test score is 5 for unwashed polyester fabric, 1 for fabric washed according to a standard protocol and 2 for fabric washed according to an innovative protocol. The results indicate that a standard washed fabric has more wrinkles than a fabric washed with an innovative protocol, which can be justified by a higher stabilisation rate due to a cooling concept of the innovative washing protocol.



**Figure 1.** SEM images of fabrics before and after 10 washing cycles. (a) N. (b) N-St\_10. (c) N-In\_10.

Assessments of the appearance of the surface of unwashed and washed polyester fabrics after different numbers of cyclic rubbings are given in Table 2.

**Table 2.** Grades of fabric propensity to surface pilling.

	Number of Rubs					
	125	500	1000	2000	5000	7000
N	5	5	5	5	4–5	3–4
N-St_10	5	5	5	5	4	3–4
N-In_10	5	5	5	5	5	4

The evaluations presented in Table 2 show that cyclic rubbing has no effect on the surface change up to 2000 cycles. After 5000 cycles of cyclic rubbing, the surface appearance of the unwashed and washed fabric samples is unchanged. Increasing the cyclic rubbing to 7000 cycles further affected the appearance of all samples. Both the unwashed fabrics and the fabrics washed according to the standard protocol are rated 3–4, which corresponds to slight to moderate pill formation on a large part of the sample surface. The best behaviour is shown by the samples washed according to the innovative protocol, which were rated 4, corresponding to slight surface fluffing and/or partially formed pills. According to the obtained scores, the innovative protocol is more suitable for surface preservation when a tendency of pilling is observed. The results for polyester fabrics washed according to the standard protocol are consistent with previous results after 10 washing cycles [15], although there are differences in the selection of polyester fabrics and washing conditions.

The results of the reduction in the tensile strength of the fabric after 10 washing cycles with the standard (N-St\_10) and innovative (N-In\_10) protocols compared to the unwashed condition are shown in Table 3.

**Table 3.** The change in the reduction of the breaking force,  $\Delta F$  and the breaking elongation,  $\epsilon$ .

Sample	$\Delta F(\%)$	$\epsilon$ (%)
N	-	20.03
N-St_10	17.8	22.18
N-In_10	4.2	21.25

Polyester fabrics washed according to standard and innovative protocols have lower strength than unwashed standard polyester fabrics. The loss of strength properties or the reduction in breaking force is greater for fabrics washed according to the standard protocol. The integrity of a fabric washed according to the innovative protocol is better compared to the standard protocol. The obtained results in terms of surface are consistent with the morphological characteristics (SEM), which show that the fabric washed according to the standard protocol was more altered compared to the unwashed fabric and the fabric washed according to the innovative protocol. Additionally, the scores for the fabric's tendency of surface pilling agree well with  $\Delta F$  and SEM. This proves that a cooling concept or gradual temperature stabilisation in the innovative protocol preserves the surface and integrity properties of the polyester fabric. It can be elaborated as an effect of less shock.

### 3.2. Characterisation of the Effluent and Filtrate

To assess the release of MFs, it is of particular importance to determine the physico-chemical parameters of the wash effluent. The results for the individual wash cycles according to standard and innovative procedures (1st, 2nd, 3rd, 4th and 5th and cumulative cycles from 6th to 10th) are presented in Tables 4 and 5.

**Table 4.** Physico-chemical parameters of effluents from washing of polyester fabrics by the standard protocol.

Effluent	TSS (mg/L)	TS (mg/L)	TDS (mg/L)	pH	$\kappa$ ( $\mu\text{s}/\text{cm}^2$ )	Turbidity (NTU)
St_1	114.75	735.67	561.9	8.20	825.00	52.63
St_2	118.17	716.33	590.95	8.06	797.67	62.7
St_3	126.17	701.33	580.68	8.14	792.00	66.3
St_4	121.67	677.0	535.24	8.08	754.67	64.47
St_5	121.17	634.67	552.86	7.97	811.67	70.2
St_6-10	124.50	663.00	527.86	8.06	740.00	56.8

**Table 5.** Physico-chemical parameters of effluents from washing of polyester fabrics by the innovative protocol.

Effluent	TSS (mg/L)	TS (mg/L)	TDS (mg/L)	pH	$\kappa$ ( $\mu\text{s}/\text{cm}^2$ )	Turbidity (NTU)
In_1	127.0	680.7	560.95	8.39	732.0	72.7
In_2	125.0	654.0	556.43	8.42	710.0	70.8
In_3	137.0	634.7	508.57	8.40	680.0	76.9
In_4	179.0	628.0	481.91	8.58	624.7	88.5
In_5	210.0	733.3	508.57	8.53	670.0	102.1
In_6-10	156.3	645.5	507.86	8.26	702.5	56.4

The issue of MFs required control of parameters relating to solid and suspended substances (TS, TSS), turbidity and conductivity. The results obtained indicate that the effluent does not place a significant burden on the environment [37].

Comparing the results of the parameters of the effluents from the standard and innovative washing protocols, the turbidity values for the effluents from the standard washing are lower than those from the innovative washing protocol. The results for conductivity are lower for the standard wastewater than for the innovative wash protocol. No clear trends for solids and suspended solids were observed for the analysed wash cycles, confirming the dispersion of the system and the importance of sampling and analysis.

The wastewater characterisation parameters were used to verify the applicability of a particular method and its relevance to the characterisation of wastewater from washing processes according to standard and innovative protocols.

Membrane filtration was used to separate the effluents into a filtrate and a cake according to previously described conditions [37]. Physico-chemical parameters for filtrates' characterisation were pH, conductivity ( $\kappa$ ) and turbidity; see Table 6.

**Table 6.** Physico-chemical parameters of filtrates (f) from washing of polyester fabrics by the standard protocol.

Filtrate	pH	$\kappa$ ( $\mu\text{S}/\text{cm}^2$ )	Turbidity (NTU)
St_1f	8.45	883.33	1.53
St_2f	8.55	796.00	1.4
St_3f	8.47	774.67	3.43
St_4f	8.44	714.00	3.08
St_5f	8.56	738.00	4.19
St_6-10f	8.37	666.00	3.99

The results obtained from these parameters for the individual washing cycles by the standard and innovative protocols are shown in Tables 6 and 7.

**Table 7.** Physico-chemical parameters of filtrates (f) from washing of polyester fabrics by the innovative protocol.

Filtrate	pH	$\kappa$ ( $\mu\text{S}/\text{cm}^2$ )	Turbidity (NTU)
In_1f	8.54	731.0	2.42
In_2f	8.57	704.0	6.19
In_3f	8.53	680.0	5.47
In_4f	8.51	638.0	5.48
In_5f	8.49	655.0	4.60
In_6-10f	8.48	692.5	2.91

The conductivity of the innovative filtrate lowers the values compared to the standard filtrate, as previously observed on the effluent. Filtrates' turbidities are significantly lower than those of the effluents, confirming the efficiency of filtration as well as retention of MFs from polyester fabrics.

The characterisation of the filter cake was analysed with a digital microscope with magnification of  $50\times$  by MFs number on the glass-fibre filter, Table 8.

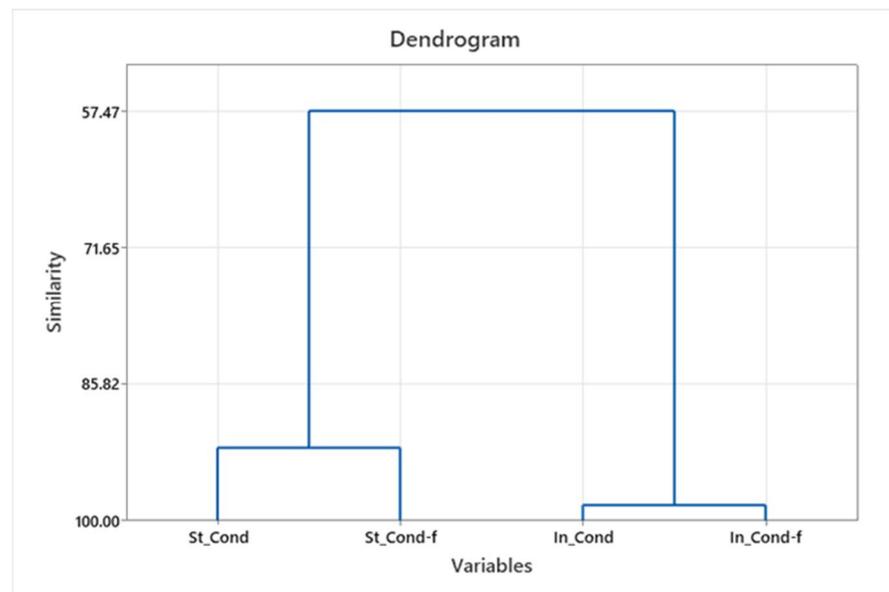
**Table 8.** Counted MFs on the filter.

Protocol	Washing Cycles						$\Sigma$
	1st	2nd	3th	4th	5th	6–10th	
	Counted MFs						
St	103	97	114	37	34	35	420
In	143	164	105	88	70	23	593

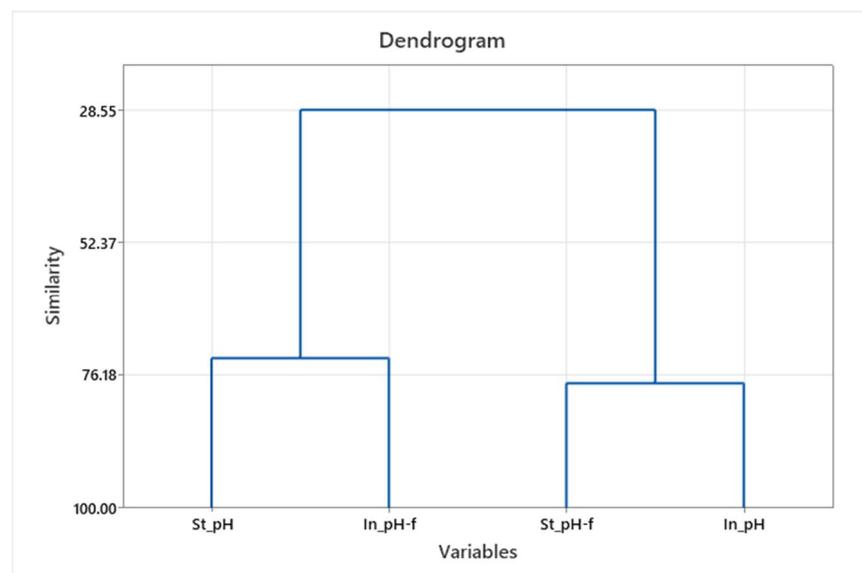
The number of MFs on the filters decreased with an increasing number of cycles, which is consistent with the results of a previous study despite different washing protocols [38].

It was also found that the amount and length of released fibres were influenced by the structural polymer parameters but also by the number of washing cycles [39]. The number of MFs on the filter from the innovative protocol is higher than from the standard protocol. This can be associated with a decrease in tensile properties and smoothness of fabrics washed according to the standard protocol.

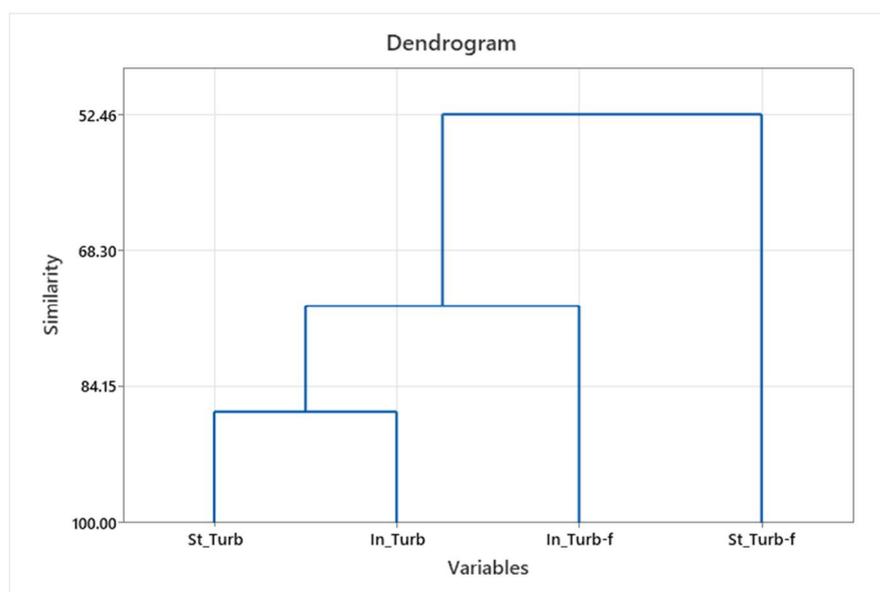
The similarity of individual parameters in effluents and filtrates across all washing cycles and the significance in the standard and innovative process were obtained by hierarchical cluster analysis (HCA). The results are shown with dendrograms showing the similarities of the parameters considered, as well as grouping them at the hierarchical level; see Figures 2–4.



**Figure 2.** Conductivity grouping dendrogram—effluent and filtrate for standard and innovative protocols.



**Figure 3.** Dendrogram of pH grouping—effluent and filtrate for standard and innovative protocols.



**Figure 4.** Turbidity grouping dendrogram—effluent and filtrate for standard and innovative protocols.

Obtained groups for individual parameters (conductivity) were formed separately for the standard and innovative protocol, and for individual parameters (turbidity and pH), the grouping was obtained separately for effluent and filtrate.

#### 4. Conclusions

Research has pointed out the usefulness of the proposed concept of cooling the bath before rinsing to preserve the properties of polyester fabrics and to reduce the load of washing wastewater.

Based on the results of the characterisation of fabrics, effluents and filtrates according to the standard and innovative washing protocols, differences in parameters were found.

The effects of the innovative process are more favourable for the tensile properties and appearance of the surface evaluated by both methods used. The SEM image of the surface favours the innovative protocol. The zeta potential results showed no differences between the fabrics washed with the standard process and the innovative process.

Wastewater analysis showed differences between the individual characterisation parameters. The turbidity of the effluent from the innovative process is higher than that from the standard process. TS, TDS and TSS are different, which did not harmonise the changes in the individual cycles (1–5) and the other cycles (6–10). The grouping of the parameters obtained by the hierarchical cluster analysis shows the differences between the standard protocol and the innovative protocol (conductivity) and the differences between effluent and filtrate (pH and turbidity). The characterisation of the filter cake by the number of MFs showed a higher number of MFs released in the innovative washing process.

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