Supporting Information

Microplastic release to water, via laundering, and to air, via everyday use: a comparison between polyester clothing with differing textile parameters

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Number of Pages: 11

Number of Figures: 3

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Materials: detergent composition

The detergent had the following composition: 5-15% of anionic and non ionic surfactants; < 5% of soap and phosphonates; optical whitening agents; enzymes and perfume.

QA/QC

- **Pre-washing of the garments:** Cross-contamination of microfibres between washes was prevented by running two consecutive empty washing cycles, the first at 60 °C, 1400 rpm for 1h 10 min, the second at 40° C, 1400 rpm and 45 min. The wastewater coming from these cleaning cycles was filtered through sieves of decreasing porosity (500, 63, 25 µm), placed at the end of the draining pipe. No fibres were found in the sieves, so the cleaning cycles were considered efficient.
- **Release of microfibres from synthetic clothes during washing**: Cross-contamination of microfibres between washes was prevented by running, after the end of each washing test, two consecutive empty washing cycles, both at 40 °C, 1200 rpm for 30 min each. Blanks were obtained by filtering the wastewater from 5 of these empty washing cycles, using the same procedure, to check for the presence of contaminants. Results showed a change in the filter weight < 1 mg so the cross-contamination was considered negligible and no correction of the data was performed. Furthermore, to avoid cross contamination of microfibres among the different filtrations, tygon tubes, filter holders and tanks were cleaned with distilled water and with a jet of compressed air. Cotton lab coats and nitrile gloves were worn during all the experimental work.
- **Release of microfibres from synthetic clothes to air:** The filters used were always kept closed in Petri dishes and the observations under the microscope were carried out very carefully to avoid sources of contamination. In any case, the morphology and colour (white) of the cotton fibres of the lab coats and leggings made them very recognisable in the microscopy observations, avoiding mistakes in fibre counting. The peculiar morphology of cotton fibres characterized by convolutions with a typical twisted ribbon form, make them recognizable from synthetic fibres whose shape is mainy a long thin cylinder (Houck, 2009). Also considering the cotton fibres released by the garment composed by a blend of 50%:50% polyester/cotton, they were easily discernible from those of the lab coats and leggings since they were pink whereas the latter were white.

Materials: Textile characteristics of tested garments

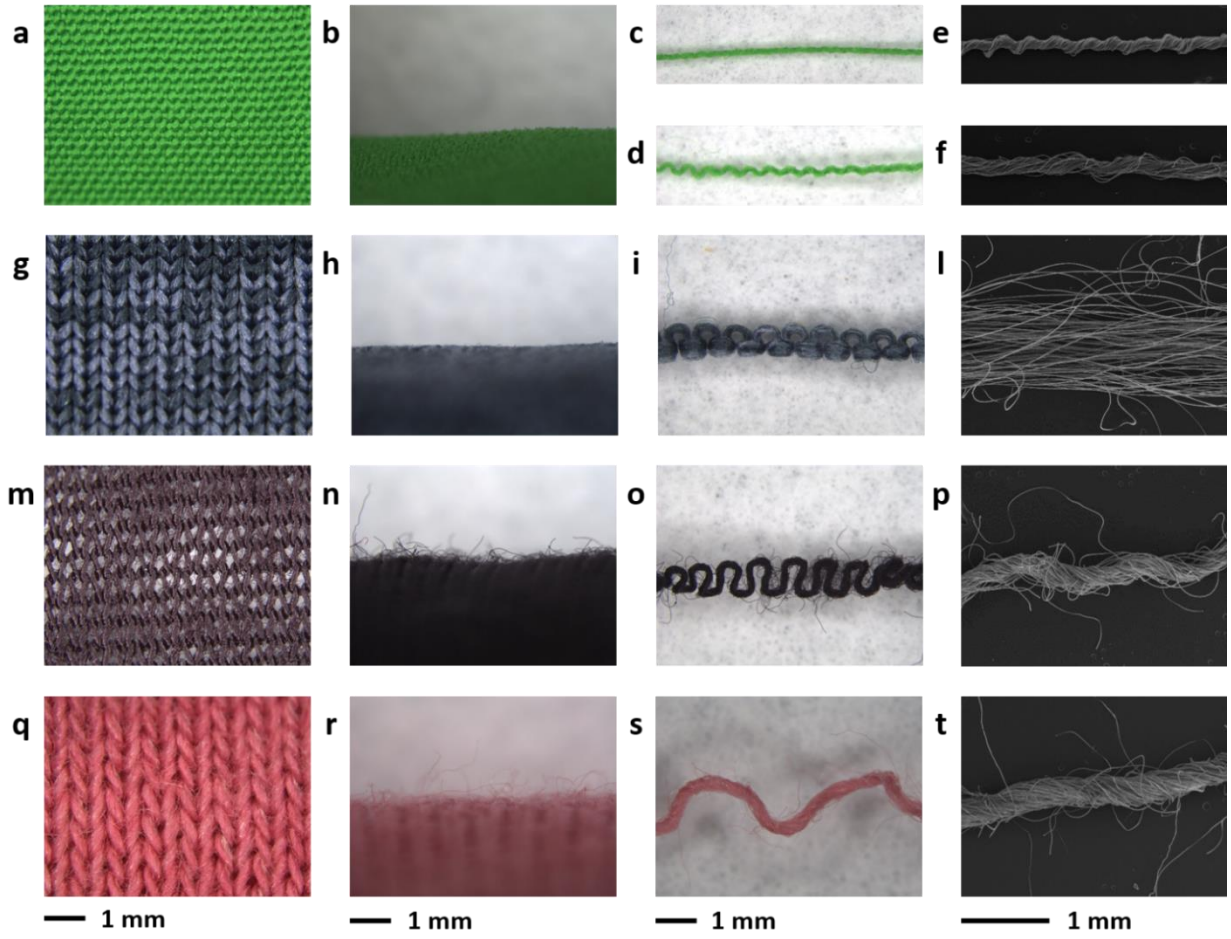

Figure S1. Optical micrographs (OM) and SEM micrographs (SM) of selected garments. 100% polyester blouse: a) OM of the plane surface, b) OM of the cross section, c-d) OMs of the warp and weft yarns, and e-f) SMs of the warp and weft yarns; 100% polyester t-shirt: g) OM of the plane surface, h) OM of the cross section, i) OM and l) SM of the yarn; 100% polyester dress: m) OM of plane surface, n) OM of the cross section, o-p) OM and SM of the yarn; 50%:50% polyester/cotton sweatshirt: q) OM of the plane surface, r) MO of the cross section, s-t) OM and SM of the yarn.

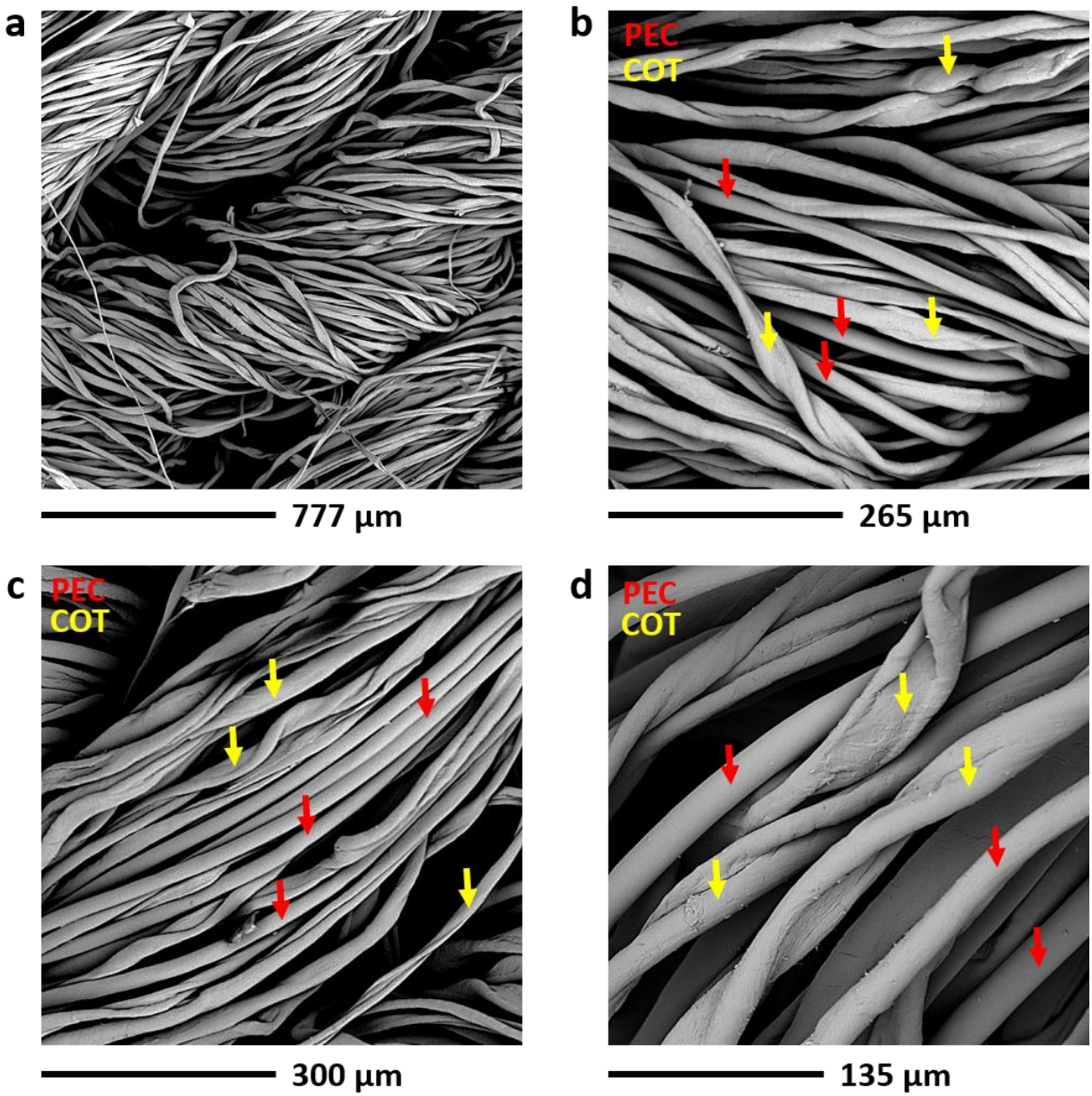

Figure S2. SEM micrographs of 50%:50% polyester/cotton sweatshirt fabric at different magnifications. Colored arrows indicate polyester (PEC) or cotton (COT) fibres.

Materials: washing program

The tested garments were mainly composed by polyester, the washing program "synthetic" of the Bosch washing machine was applied, whose default washing time, set by the producer, was of 1h 47 min. This choice is in line with previous works which used similar washing duration (Napper et al. 2016: 1h15min; Sillanpää et al. 2017: 1h15min; Kelly et al 2019, full scale testing: 1h25 min).

Release of microfibres from synthetic clothes during washing: multistep filtration procedure

The wastewater, coming directly from the drainpipe of the washing machine, was firstly filtered through a 400 µm pore size mesh. Wastewater was recovered into tanks and filtered by means of a peristaltic pump (SP 311/60 Velp Scientifica) connected with Tygon tubes, throughout a nylon net filter with a 60 µm pore size (Merck) and then through a nylon net filter with a 20 µm pore size (Merck). Finally, 300 ml of the filtered wastewater was filtered on a Durapore PVDF membrane with a 5 µm pore size (Merck). The filters were placed inside closed filter holders, avoiding exposure to air and possible contaminations. At the end of each tank, 1L of distilled water was poured into, the tank and shaken, and the water filtered. Such procedure was carried out twice for each tank to collect any microfibres that remained on the sides of the tanks. Finally, 1L of distilled water, heated at 70 °C in a covered glass becker, was fluxed in the closed filtration system to clean the filters from excess of detergent. After this rinsing step, the filter was removed from the filter holder under a fumehood and placed inside a closed glass Petri dish. All the filters, inside closed glass Petri dishes, were dried in an oven at 105 °C for 1 h and then weighed. Comparison of weight before and after the filtration was used to quantify the amount of microfibre released. The weight of microfibre recovered on 400, 60 and 20 µm pore size filters, was normalized for the washing load, obtaining W in mg/kg. The amounts obtained from 5 µm pore size filters were normalized for the amount in liter of filtered wastewater, obtaining the concentration C of mg of microfibres per L. The mean C_a and the SD were calculated considering the C values of the 4 washed samples for each type of garment.

Release of microfibres from synthetic clothes during washing: microfibre dimensions

The dimensions of the collected microfibers were examined using ImageJ (release 1.43u) on optical micrographs acquired on a fraction of the microfibres recovered on the 400 µm mesh, and on SEM micrographs collected on those recovered from 60, 20 and 5 µm pore size filters. The average values of length, L, and diameter, D, along with the SD, were calculated on the basis of the measurements of 25 microfibres per filter (for a total of 100 microfibres per garment).

Release of microfibres from synthetic clothes to air: sequence of movements for the tests

The sequence of movements performed by the volunteers during the tests, was the following:

- 2 min and 30 sec: lateral opening and closing of arms and legs;
- 1 min: steady oscillation near the desk, gently shaking the garment;

- 1 min: steady with hands leaning against the desk;
- 1 min: walking, gently shaking the garment;
- 1 min 30 sec: walking opening and closing the arms laterally.
- 1 min: steady oscillation near the desk, gently shaking the garment;
- 1 min: steady with hands leaning against the desk;

This sequence, with a duration of 10 min, was performed twice to reach an overall testing time of 20 min.

Release of microfibres from synthetic clothes to air: microfibre identification

Colour and morphology of the microfibres were mainly taken into account. Colour was used as a guide to identify the microfibres coming form the four type of garments tested (since they all had different colours). Then, synthetic and cotton microfibres were discriminated considering that: synthetic fibres have to be equally thick through their entire length and should not be entirely straight, neither cellular nor organic structures should be visible (Dris et al., 2017) and their shape is mainly a long thin cylinder (Houck, 2009); cotton fibres are characterized by convolutions, with the typical twisted ribbon form (Houck, 2009).

To confirm the nature of the counted microfibres, FTIR spectroscopy was applied to analyse the microfibres collected in the Petri dishes after testing of each garment, by using the equipment and the procedure described in the Materials and Methods section. 16 microfibres randomly selected were analysed for both PES-Knit-Filament and PES-Knit-Staple, and only 4 microfibres for PES-Woven-Filament due to the low overall amount of microfibres released by this garment. Since for PES/Cot-Knit-Staple tests microfibres could be of double nature, cotton or polyester, 32 microfibres randomly selected among the 8 Petri dishes were analysed.

Statistics

Statistical analysis of the amount and number of microfibres released to water and air, respectively, was carried out by using IBM® SPSS® Statistics software. The data were tested for normality using a Shapiro-Wilk test and for homogeneity of variance by using Levene's test. One-way Analysis of Variance (ANOVA) with a Student–Newman–Keuls (SNK) post hoc test was performed to assess any significant differences among the types of garments. When data did not comply with the assumption of homogeneity of variances, Welch ANOVA with a Games-Howell post hoc test was

performed. Two-sample t-test and Mann-Whitney U (MWU) were also applied for analysis of two groups of data, depending if the data were normally distributed or not. A 5% significance level was used for all statistical tests; p values <0.05 indicate significant difference among the data.

Microplastic release to water: additional information

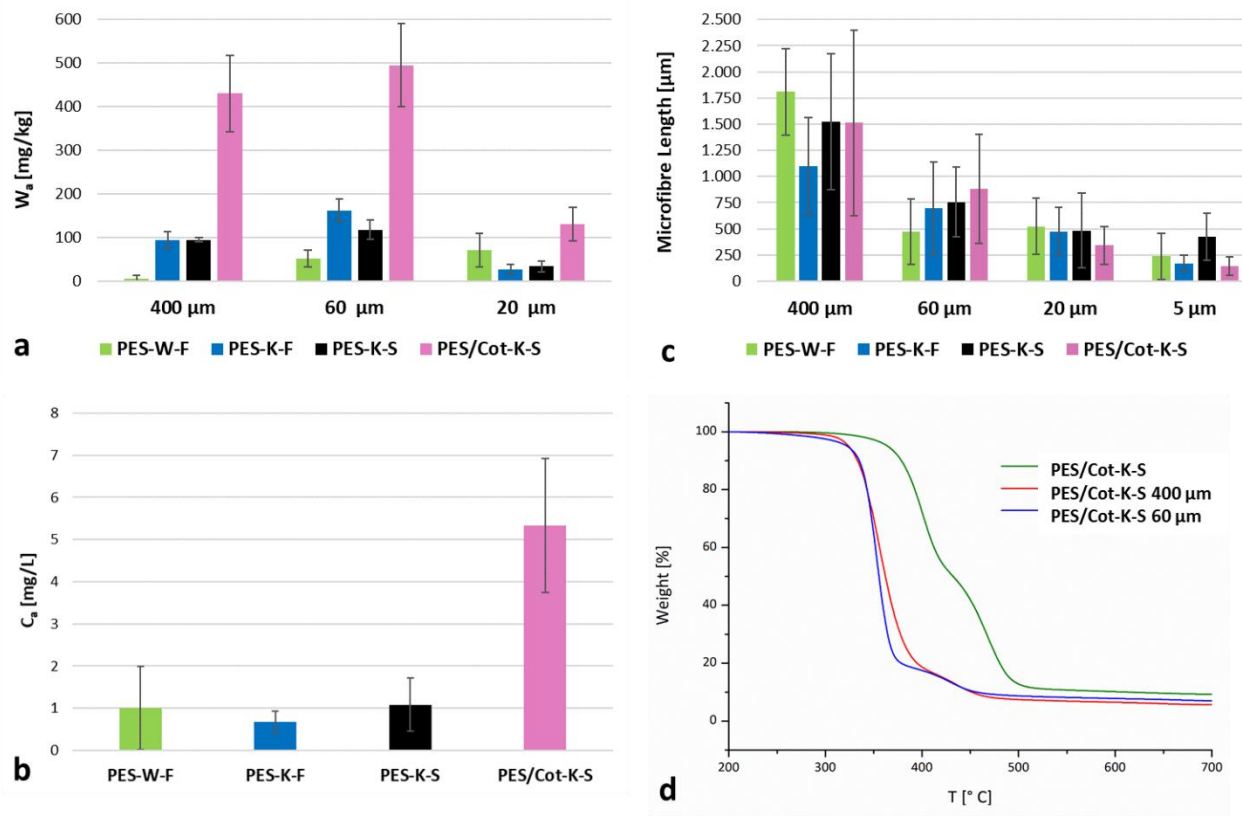

Figure S3. Amount of microfibres recovered on a) 400, 60 and 20 µm pores size filters ($W_a \pm SD$) and b) on 5 µm pore size filters ($C_a \pm SD$), released during washing from PES-Woven-Filament (PES-W-F), PES-Knit-Filament (PES-K-F), PES-Knit-Staple (PES-K-S) and PES/COT-Knit-Staple (PES/Cot-K-S); c) Length of microfibres released from PES-W-F, PES-K-F, PES-K-S, PES/Cot-K-S during washing and recovered on 400, 60, 20 and µm pore size filters; d) Thermogravimetric curves of PES/Cot-K-S fabric, PES/Cot-K-S microfibers released during washing of PES/Cot-K-S recovered from 400 and 60 µm pore size filters.

Figure S2a shows that the largest quantity of microfibers was recovered on the filter with a 60 µm pore size for all garments except PES-Woven-Filament, for which the greatest aliquot was recovered on the 20 µm filter. Statistical analysis on the different concentrations of microfibres released from the tested garments, reported in Figure S2b, confirmed that PES/Cot-Knit-Staple, the garment made

with polyester/cotton blend, was characterized by a significantly higher C_a value than the other garments (Table S2), but since the SD was quite high for some values, C_a was only considerate as indicative of the presence of smaller microfibers in the wastewater and was not used to calculate microfiber presence in the entire effluent.

The thermogravimetric curves reported in Figure S2d, show that all three samples present a twostep thermal degradation, starting from 200° C. PES/Cot-Knit-Staple fabric had a weight loss of around 51% during the first step, corresponding to a temperature of max weight loss (T_{max}) of 402° C; the weight loss in the second step is of around 41% at a T_{max} of 468° C. These values are perfectly in line with the composition of the fabric that is 50% polyester and 50% cotton. In fact, the first step correspond to the degradation of the cotton part, which usually involves the decomposition of the glycosyl units to char at lower temperatures and the depolymerization of such units to volatile products containing levoglucosan at higher temperatures (Alongi et al., 2011). The second step corresponds to the further degradation of polyester, due to the decomposition of the main chain (Alongi et al., 2012). The aliquots of microfibres recovered from both 400 and 60 µm pore size filters, presented similar behaviors, with a weight loss of 84% and 80% respectively during the first step (400 µm: T_{max}=346° C; 60 µm T_{max} =353° C), and of 10 % for both of them in the second step (400 µm: T_{max}=435° C; 60 µm T_{max} =429°).

Table S. ANOVA with SNK post hoc test on the concentrations C [mg/L] of microfibres released from PES-Woven-Filament (PES-W-F), PES-Knit-Filament (PES-K-F), PES-Knit-Staple (PES-K-S) and PES/Cot-Knit-Staple (PES/Cot-K-S)
recovered on 5 µm pore size filters filtering an aliquot of 300 ml of wastewater.

ANOVA

C

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	56,509	3	18,836	19,058	,000
Within Groups	11,861	12	,988		
Total	68,369	15			

Student-Newman-Keuls[a]

Garment	N	Subset for alpha = 0.05	
		1	2
PES-K-F	4	,6667	
PES-K-S	4	1,0842	
PES-W-F	4	1,3342	
PES/Cot-K-S	4		5,3333
Sig.		,621	1,000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 4,000.

Table S1. Welch ANOVA with Games-Howell post hoc test on the number of microfibres per gram of washed fabric released to water from PES-Woven-Filament (PES-W-F), PES-Knit-Filament (PES-K-F), PES-Knit-Staple (PES-K-S) and PES/Cot-Knit-Staple (PES/Cot-K-S).

N

	Statistic[a]	df1	df2	Sig.
Welch	45,198	3	5,989	,000

a. Asymptotically F distributed.

Dependent Variable: N

Games-Howell

(I) Garment	(J) Garment	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
PES-W-F	PES-K-F	-1037,79375*	196,84969	,014	-1781,1538	-294,4337
	PES-K-S	-418,61112	180,41414	,248	-1192,7907	355,5685
	PES/COT-K-S	-3189,00077*	289,98720	,000	-4221,7619	-2156,2396
PES-K-F	PES-W-F	1037,79375*	196,84969	,014	294,4337	1781,1538
	PES-K-S	619,18263*	111,17408	,011	202,0460	1036,3192
	PES/COT-K-S	-2151,20702*	252,79040	,004	-3181,4683	-1120,9457
PES-K-S	PES-W-F	418,61112	180,41414	,248	-355,5685	1192,7907
	PES-K-F	-619,18263*	111,17408	,011	-1036,3192	-202,0460
	PES/COT-K-S	-2770,38965*	240,21334	,003	-3851,5636	-1689,2157
PES/COT-K-S	PES-W-F	3189,00077*	289,98720	,000	2156,2396	4221,7619
	PES-K-F	2151,20702*	252,79040	,004	1120,9457	3181,4683
	PES-K-S	2770,38965*	240,21334	,003	1689,2157	3851,5636

*. The mean difference is significant at the 0.05 level.

Table S2. ANOVA with SNK post hoc test on the number of microfibres per gram of worn fabric released to air from PES-Woven-Filament (PES-W-F), PES-Knit-Filament (PES-K-F), PES-Knit-Staple (PES-K-S) and PES/Cot-Knit-Staple (PES/Cot-K-S).

ANOVA

N

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	439136,384	3	146378,795	35,450	,000
Within Groups	49549,324	12	4129,110		
Total	488685,709	15			

N

			Subset for alpha = 0.05		
	Garment	N	1	2	3
Student-Newman-Keuls[a]	PES-W-F	4	1,2942		
	PES-K-F	4		108,1607	
	PES-K-S	4			346,7786
	PES/COT-K-S	4			402,9730
	Sig.		1,000	1,000	,240

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 4,000.