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► **To cite this version:**

Matthieu Dreillard, Caroline de Faria Barros, Virgile Rouchon, Coralie Emonnot, Veronique Lefebvre, et al.. Quantification and Morphological Characterization of Microfibers Emitted from Textile Washing. *Science of the Total Environment*, 2022, 832, pp.154973. 10.1016/j.scitotenv.2022.154973 . hal-03641143

HAL Id: hal-03641143

<https://ifp.hal.science/hal-03641143v1>

Submitted on 14 Apr 2022

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1 **Quantification and morphological characterization of microfibers emitted from textile**
2 **washing**

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11 **Abstract**

12 Microplastics are a subject of growing interest as they are a potential threat for living organisms.
13 Textile microfibers (MFs) are an important microplastics sub-group that have been reported as
14 a major source of microplastics release into the environment. This pollution occurs mainly
15 during the washing of synthetic garments. However, standardized methods to quantify and
16 characterize these MFs are scarce. This study proposes a new analytical protocol to characterize
17 these MFs in number and size by means of filtration techniques, optical and electronic
18 microscopy and automatic image post-processing. This approach was developed and validated
19 on effluents from washing machines produced in different conditions (5 different garments,
20 sequential cycles, and presence or not of detergent). Among the analyzed effluents, it was found
21 that 40 to 75% of microfibers have a length comprised between 50 and 200 μm , with average
22 microfiber diameters ranging from 8 to 17 μm depending on the type of textile. The emission
23 range of microfibers was estimated to be between 220 000 to 2 820 000 microfibers per kg of
24 textile depending on the type of garment and the washing conditions. The counting method
25 developed is adapted to a certain range of textiles, such as 100% polyester fleece jackets (PET-
26 1), 100% smooth polyester T-shirt (PET-2) and 100% acrylic sweater (PAN), and is not affected

27 by the presence of detergent. The proposed method of characterization of these MFs lengths
28 can also be extrapolated to the counting of other objects that have a similar morphology to the
29 analyzed fibers. Hence, it can be helpful to develop new testing capture technologies and, thus,
30 contribute to the enhancement of filtering techniques of several pollutants.

31

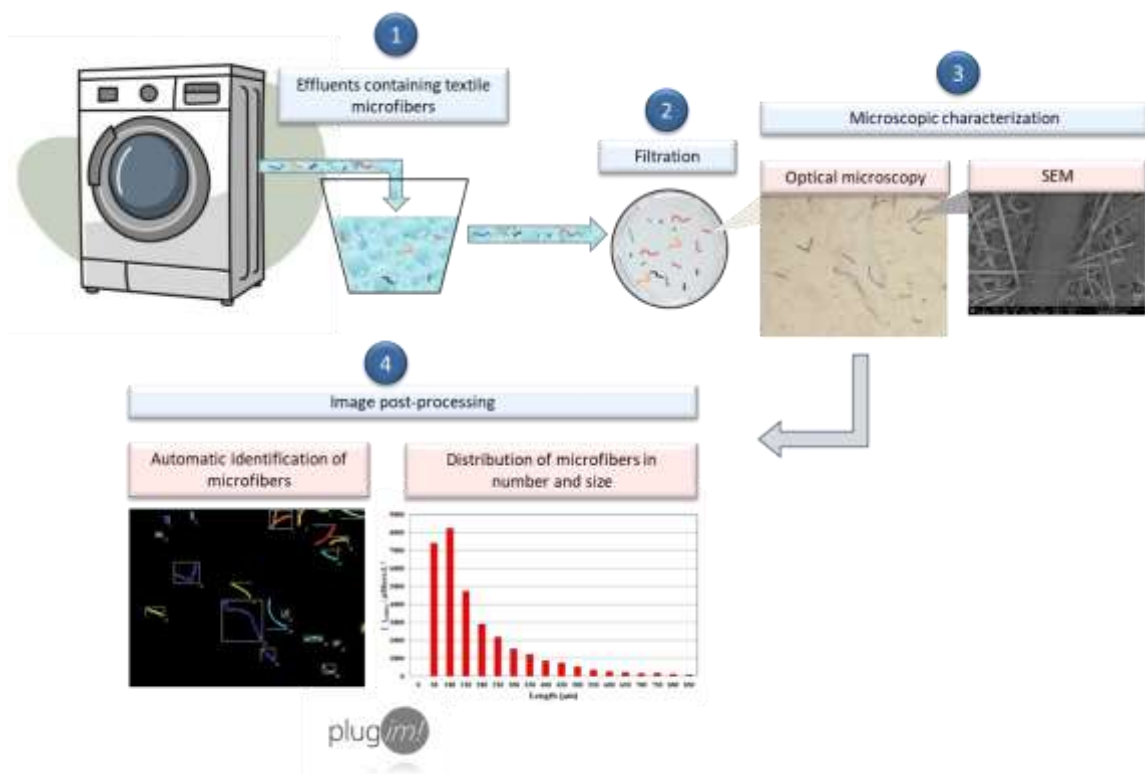
32 **Highlights**

- 33 • New method for quantitative determination of textile microfibers release.
- 34 • Automatic counting and acquisition of detailed length distributions of microfibers.
- 35 • Most microfibers (40 to 75%) are between 50 and 200 μm long.
- 36 • Average microfiber diameters ranging from 8 to 17 μm .

37

38

Graphical abstract



39

40 **Keywords: Microplastic; Filtration; Microscopy; Counting method; Length distribution.**

41

42 **1. Introduction**

43 Due to attractive properties such as high durability, high versatility and low cost, plastics are
44 widely used in day-to-day life and their production has increased exponentially since the mid-
45 20th century, exceeding 300 million tons produced per year [1], [2]. The textile industry
46 accounts for an important part of this share, with more than 80 million tons of synthetic fibers
47 produced in 2020 – a value largely driven by the production of polyester [3],[4].

48 The abrasion of synthetic textiles during washing leads to shedding and releasing of plastic
49 fragments due to the mechanical stress of the textile fibers [5],[6]: over 700 000 synthetic
50 microfibers (lengths < 5 mm) can be generated during a simple domestic wash [7], [8]. Despite
51 a relatively high retention efficiency of microplastics in conventional wastewater treatment
52 plants (WWTP), microfibers are still the most abundant item remaining in discharged effluents
53 (up to 83%) [9]. Moreover, most of the plastic microfibers retained in WWTP end up in sludges
54 during the first steps of the treatment [10], which are in many countries spread on agricultural
55 lands for fertilization purposes [11], [12].

56 The ecotoxicology of microplastics has been demonstrated in many areas of environmental
57 sciences [13], which rises an urgent necessity of developing regulatory measurement analysis
58 and technologies for microfibers capturing [14]. Since the distribution of the microfibers
59 lengths is a fundamental parameter for designing and testing these devices, accurate microfibers
60 characterization method is essential to evaluate their performances.

61 Filtration is still a conventional method to measure the weight and estimate the amount of
62 microfibers released by washing machines [7], [8], [15]–[22]. It can be coupled to additional
63 characterization techniques to improve the quantification of these microfibers such as optical
64 microscopy [18], [19], [23]–[28], scanning electron microscopy [23], [27], [29], Fourier
65 transform infrared spectroscopy [19], [30]–[32], and Raman spectroscopy [31]–[35]. However,
66 a comparison between the results of the literature is not straightforward due to the absence of a

67 standard protocol. Moreover, the length distribution profiles are seldom estimated [30], [36],
68 [23].

69 This work aims at contributing to the progress in microfiber quantification by presenting a new
70 protocol of characterization based on microscopy and automatic image post-processing
71 techniques, which can provide a distribution of the microfibers in number and length. The
72 results obtained in this study can also be helpful for the design of future devices to capture these
73 microfibers and for evaluating environmental impact.

74

75 **2. Material and methods**

76 **2.1. Textiles and washing protocol**

77 For this study, five different types of brand-new commercial garments were selected: 100%
78 polyester fleece jacket (PET-1), 100% smooth polyester T-shirt (PET-2), 88% polyester/12%
79 polyamide (PET/PA), 100% cotton (CO) towels, and 100% acrylic sweater (PAN). These
80 garments were all black or navy-blue to ensure an easier characterization of the microfibers by
81 optical microscopy. Other characteristics related to manufacturing stages (i.e. fabric type, fabric
82 weight per unit of area, fabric thickness and yarn type) were also evaluated (Table A1).

83 The washing machine used in this study was a Miele W5794 (2010) with a nominal capacity of
84 7 kg of dry laundry. This washing machine was selected for its high level of reproducibility.

85 All washing experiments were carried out using 3 kg of dry garment, a temperature of 30°C, a
86 spin cycle of 1200 rpm and a washing cycle adapted to the type of fabric (synthetic or natural).

87 For the trial with detergent, 57.8 g of a commercial detergent (commercial liquid Ariel Power
88 Original from Procter & Gamble France) was added. Prior to each test with a new garment type,
89 a rinsing cycle was performed on the washing machine to minimize contamination. For
90 successive washings, garments were dried between each washing on a hanger in air for a
91 minimum of 4 hours. For each experiment, the entire water effluent (around 70 L) was collected

92 in a stainless-steel vessel. This tank was homogenized to hold a representative water sample of
93 around 1 L (glass bottle) for analyses and was rinsed between each test.

94

95 **2.2. Filtration**

96 Filtration aims at quantifying particles mass and deposit them on a surface that is best adapted
97 to be characterized by microscopy. The water samples obtained from the wash tests were
98 filtered through a 0.7 μm glass fiber filter (VWR I516-0345) at room temperature. The filtration
99 system used was a Setaclean Total Sediment Tester. All filters were dried at 60°C before and
100 after filtration during 1 hour to avoid humidity. All masses were measured after this drying
101 procedure with a Mettler Toledo AG204 scale (± 0.1 mg) and were normalized to the mass of
102 garment employed for each wash.

103 The volume to be filtered was adapted to the microfiber concentration of the washing effluents:
104 200 mL for all tests performed with 100% polyester fleece jacket (PET-1) without detergent
105 (including successive washes), and 20 mL for the remaining tests. It must be large enough to
106 allow measuring the deposited mass (limit of quantification of 0.1 mg on the scale) and small
107 enough to limit the entanglement of microfibers that complicates the counting method. To avoid
108 cross contamination, the filtration system was rinsed with demineralized water after each test.
109 Water samples were manually agitated before filtration to ensure a good homogeneity of the
110 solution. Two filtrations were performed for each wash test and both filters were then
111 characterized by microscopy to evaluate the repeatability of the sampling and analytical
112 method. All results presented in this study are the average of both analyses.

113

114 **2.3. Microscopic characterization**

115 Scanning electron microscopy (SEM) analysis were performed as complementary
116 characterization to verify the detailed morphology of initial garment fibers (before washing)
117 and microfibers released in water effluents and further deposited on glass fiber filters. This

118 technique allowed to precisely measure fiber diameters and identify possible by-products (other
119 than microfibers) that could have been released through the washing process and retained by
120 the filtration method. For this study, the analyses were performed with a Nova NanoSEM 450
121 FEI scanning electron microscope using a GAD (Gaseous Analytical Detector) detector adapted
122 for low vacuum analysis. The characterization parameters were defined as an acceleration
123 voltage of 5 kV, spot size of 3, diaphragm of 30 μm , working distance of 6 mm and chamber
124 pressure between 30 and 50 Pa.

125 Glass fiber filters from the filtration method were also characterized via an optical microscope
126 (OM) Olympus BX51 equipped with 6 objective lenses (5 polychromatic and 1 immersive).
127 The images were taken with a $\times 2.5$ objective lens in reflective mode with polarizing filter. The
128 chosen exposure time was 16 ms with a gain of 16 dB. Images of size 2080×1544 were taken
129 at a resolution of $2.73 \mu\text{m}/\text{pixel}$. The protocol for image acquisition consists in randomly taking
130 15 images of the area of the filter covered by the microfibers (about 11.34 cm^2). These 15
131 images correspond to about 32% of the total surface of the filter. The values obtained can then
132 be extrapolated to the entire surface of the filter and the volume of the filtered solution. Fig. 1
133 shows an example of a surface analyzed using optical microscopy. It is important that no blurred
134 areas are shown in the analysis since it could impact the post-processing steps.

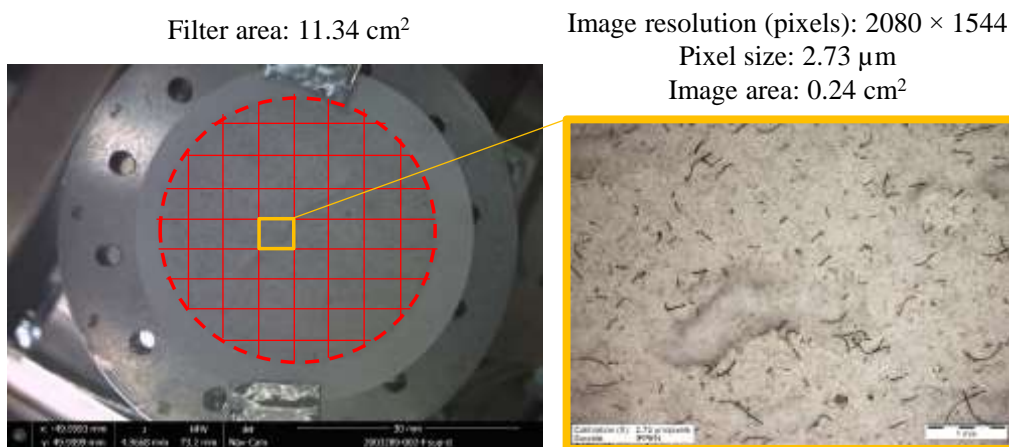


Fig. 1. Filter surface area analyzed by optical microscopy.

136 **2.4. Counting Method**

137 In order to extract the number and size of the filtered microfibers, a post-processing protocol
138 was applied on the acquired images using the plug im! platform, which is an image processing
139 platform in open access for the scientific community that is capable of providing advanced
140 functionalities [37].

141 A set of classical post-processing image techniques was used to segment the OM images. At
142 first, a set of nonlinear transformations was used since the contrast between the microfibers and
143 the filters was clearly visible. First, a grey level segmentation operation was performed to
144 binarize the image and extract the pixels corresponding solely to the microfibers. This
145 segmentation is based on Otsu's method [38] that calculates the optimal threshold separating
146 two classes on the histogram of pixel intensities by minimizing their intra-class variance.
147 Finally, the negative of these segmented images was calculated.

148 A length criteria opening [39] allows to clean the binary images of all connected components
149 below a predefined length threshold. The segmentation step may generate connected
150 components related to residual noise or to particles not considered as textile microfibers with
151 length smaller than 50 μm , i.e., 18 pixels considering image resolution. This value was
152 determined after the identification and sizing of non-fiber particles (amorphous deposit,
153 particles) by SEM that were dominantly characterized by sizes below 50 μm . Hence, the
154 microfibers concerned by this method have a minimum size of 50 μm .

155 A binary morphological operation was performed to keep only connected components
156 completely contained in the image and remove all those in contact with the border of the image,
157 since in this case the full length of the microfibers is impossible to quantify. Then, a
158 morphological analysis algorithm [40] was used to determine the geodesic length of each
159 connected component in the resulting binary image, which renders the length of each fiber in
160 pixel. This value can then be converted to metric units (pixel size of 2.73 $\mu\text{m}/\text{pixel}$).

161 All these image processing steps are automated and can be performed in batch mode. Fig. 2
162 shows an example of the image processing used to quantify microfibers. This set of data
163 processing allows quantifying the total number of microfibers, as well as their distribution in
164 number and size.

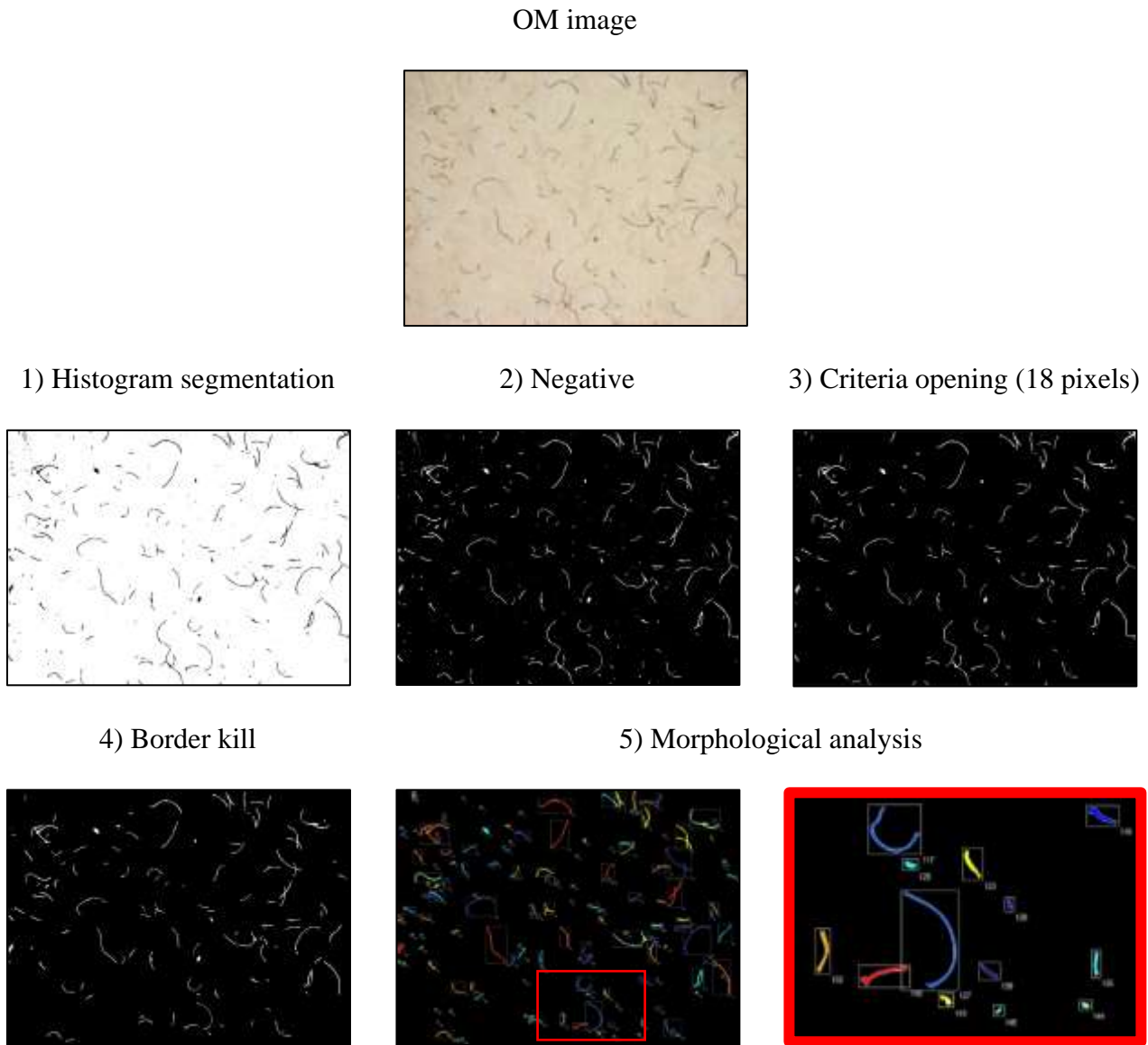


Fig. 2. The several steps of the counting method processed within the plug im! platform.

165

166 2.5. Microfiber weight estimation

167 The results obtained by the quantification method can be used to calculate the microfibers mass.

168 These values can then be compared to the results obtained by filtration. The SEM images were

169 analyzed with ImageJ [41] to measure the diameter of the fibers before washing and after being
 170 retained by the filter. Thirty measurements per fabric type were performed to determine the
 171 average diameter D of the fibers, as well as their deviation. Results of average diameter are
 172 presented in Table A2. The diameter was assumed to be homogeneous along the fibers. The
 173 mass of the released microfibers was then calculated using the Eq. 1:

174

$$175 \quad M_f = \rho \sum_{i=50\mu m}^{5mm} N_i \cdot \left(\frac{\pi \cdot D^2}{4}\right) \cdot L_i \quad \text{Eq. (1)}$$

176

177 where ρ : density of the material;

178 M_f : total mass of released microfibers;

179 N_i : total number of microfibers released in class i of length L_i counted by plug im!;

180 D: average diameter of microfibers measured by ImageJ;

181 L_i : length of microfibers considered in class i and measured by plug im!.

182 For this equation, the length distributions of microfibers obtained by the counting method were
 183 divided into 101 classes of 50 μm width. Polymer densities were taken from Cesa et al.[17]:
 184 1.365 g.cm⁻³ for polyester, 1.150 g.cm⁻³ for acrylic.

185

186 **2.6. Microfiber average length**

187 The average microfiber length (in number) can be derived from microfibers length distributions
 188 using the Eq. 2:

189

$$190 \quad L_{avg} = \frac{\sum_{i=50\mu m}^{5mm} N_i \cdot L_i}{N} \quad \text{Eq. (2)}$$

191

192 where N is the total number of released microfibers counted by plug im!.

193

194 **3. Results and Discussion**

195 **3.1. Preliminary characterization before washing**

196 The five different garments were analyzed by SEM prior to washing. The SEM micrographs
197 (Fig. A.1) show different shape and configuration of fibers depending on the type of fabric. The
198 organization of the macroscopic fibers is not well defined except for those from the 100%
199 smooth polyester T-shirt (PET-2). PET fibers from fleece jacket (PET-1) or T-shirt (PET-2)
200 show a smooth surface and a constant cylindrical section. The same morphology is found for
201 acrylic fibers (PAN). In the case of PET/PA textile, 2 fibers morphologies can be distinguished
202 corresponding to PET and PA, with the latter having a flatter morphology (ribbon shape).
203 Acrylic fibers (PAN) have the largest diameter (16.99 μm) among the tested textiles (Table
204 A.2). The cotton fibers are morphologically different from the synthetic ones presenting a
205 twisted ribbon shape well-known for this fabric [42].

206
207 **3.2. Results after washing**

208 In order to test the applicability of the developed analytical procedure, a total of 10 washing
209 experiments were performed. The first washing of PET-1 textile was repeated twice (PET-1 C1
210 and PET-1 C1-R) to verify the repeatability of the washing protocol. It should be mentioned
211 that these tests were made on two different PET-1 garment lots. The impact of the number of
212 washing cycles was investigated on the PET-1 garment up to 4 washes (PET-1 C1, C2, C3 and
213 C4). The effect of the detergent was also tested during the first washing of PET -1 (PET-1 C1-
214 D). Finally, the five different textiles types were washed once (PET-1 C1, PET/PA C1, PET-2
215 C1, PAN C1, CO C1).

216 Table 1 and Fig. 3 and Fig. 4 show the results obtained by the developed analytical protocol.
217 The counted filtered microfibers are expressed with respect to the mass of the initial dry
218 garment. One should note that all results presented in this study are average values obtained
219 from the filtration duplicates performed for each experiment. Moreover, it was decided to set a

220 minimum length of 50 μm for the identification of microfibers by the counting method (optical
 221 microscopy + post-processing image techniques) since the SEM observations did not reveal the
 222 presence of microfibers with a length below this value. One possible explanation is that an
 223 elongated shape object such as a fiber would not easily undergo a mechanical rupture at a low
 224 length to diameter ratio [43].

225 Table 1. Main Results.

Test Tag	Type of fabric		N° washing cycles	Filtration	plug im!	Eq. (2)	Eq. (1)
	Detergent			[] mg.kg ⁻¹	[] $\mu\text{fibers.kg}^{-1}$	L _{avg} μm	[] mg.kg ⁻¹
PET-1 C1	PET-1	-	1	485	690 000	194	87
PET/PA C1	PET/PA	-	1	1520	ND*	ND*	ND*
PET-2 C1	PET-2	-	1	428	1 570 000	241	149
PAN C1	PAN	-	1	339	1 390 000	181	273
CO C1	CO	-	1	305	ND*	ND*	ND*
PET-1 C1-D	PET-1	57.8 g	1	1751	2 820 000	160	294
PET-1 C1-R	PET-1	-	1	289	670 000	229	100
PET-1 C2	PET-1	-	2	72	530 000	237	82
PET-1 C3	PET-1	-	3	59	330 000	285	61
PET-1 C4	PET-1	-	4	53	220 000	301	43

226 *ND: Not determined due to high level of entanglement of microfibers.

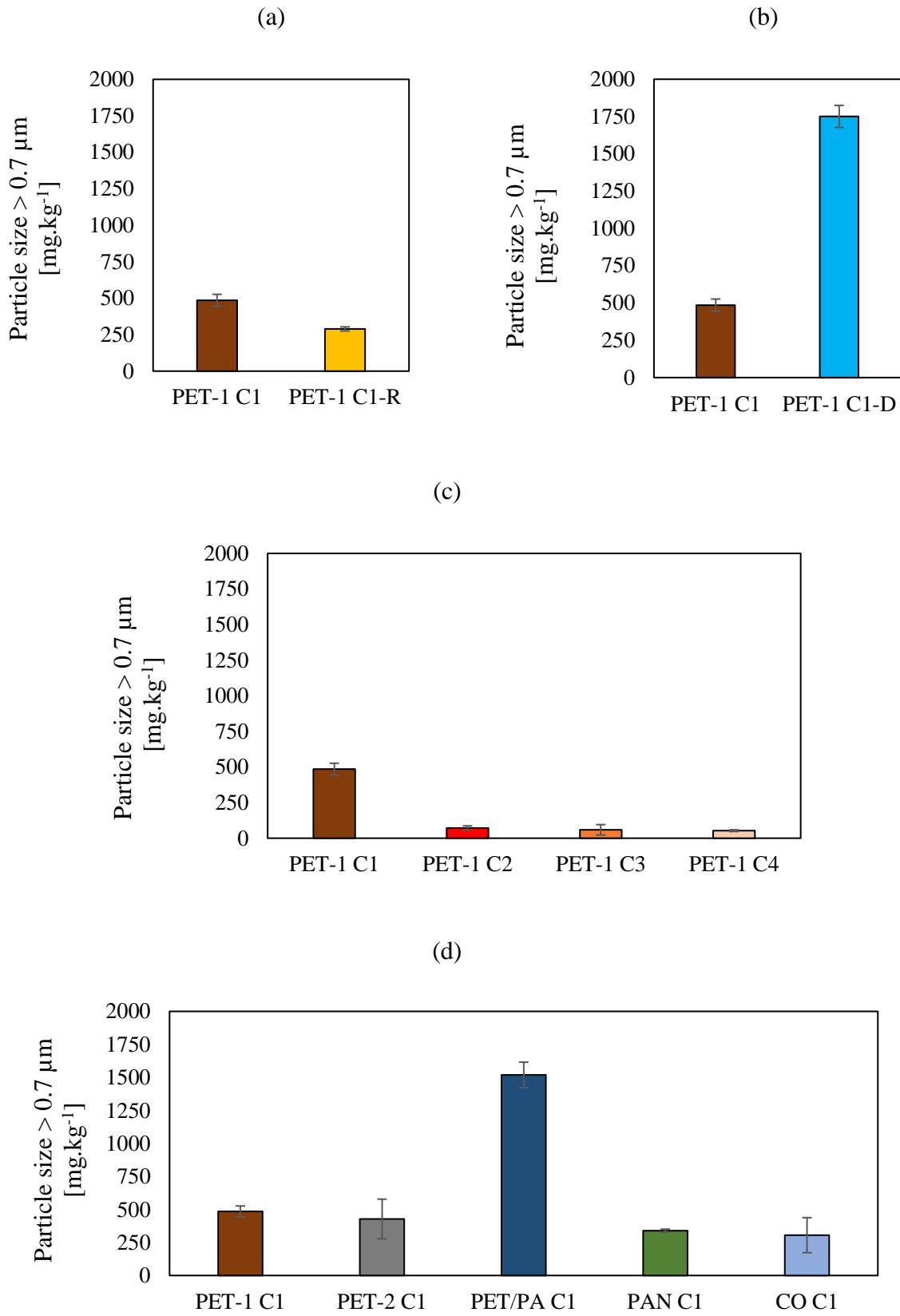
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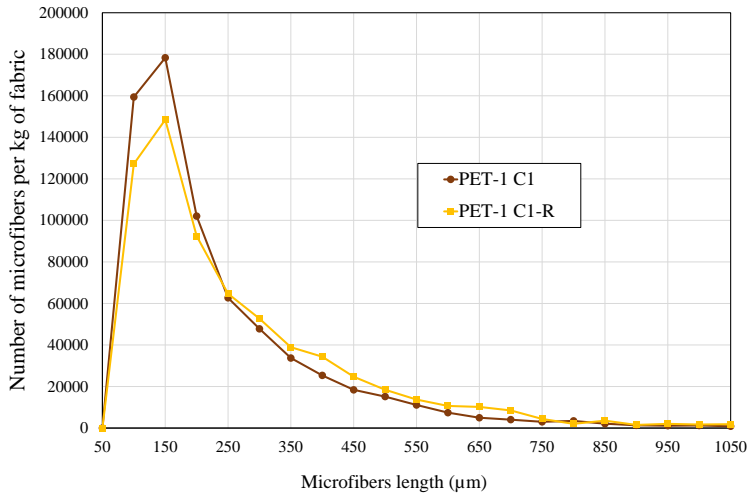
233 Fig. 3. Filtration results – a) repeatability test, b) effect of detergent, c) effect of number of
 234 washing cycles, d) effect of fabric type – PET-1: 100% polyester fleece jacket, PET-2: 100%

235 smooth polyester T-shirt, PET/PA: 88% polyester/12% polyamide towel, CO: 100% cotton

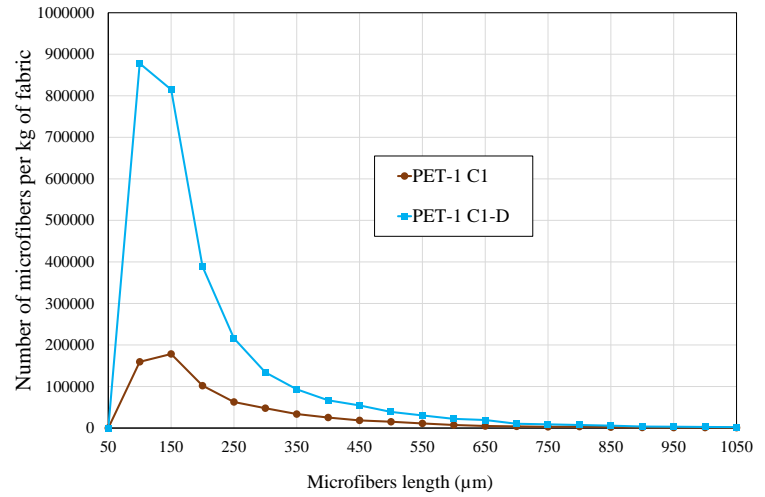
236 towel, PAN: 100% acrylic sweater.

237

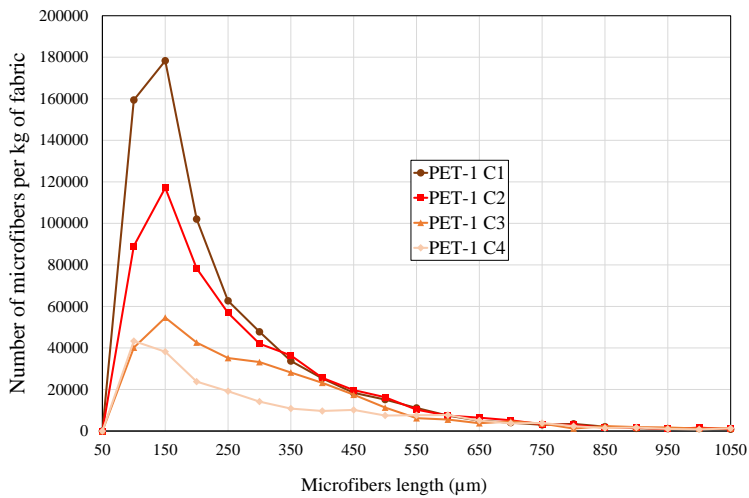
(a)



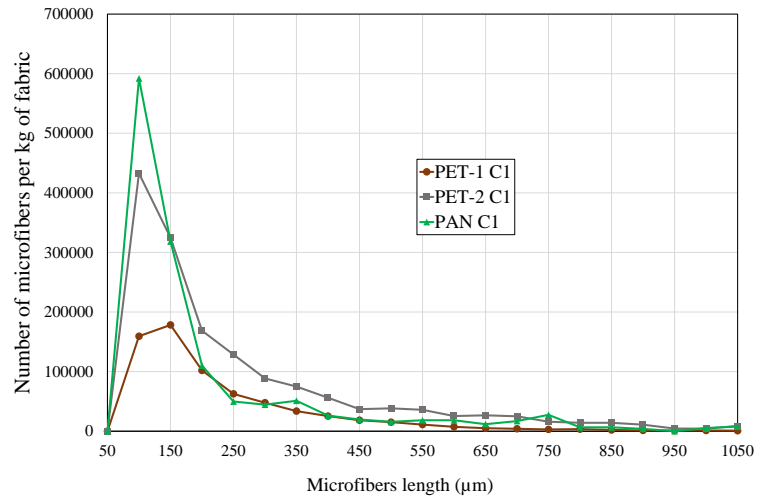
(b)



(c)



(d)



238 Fig. 4. Microfibers length distributions – a) repeatability test, b) effect of detergent, c) effect

239 of the number of washing cycles, d) effect of fabric type – PET-1: 100% polyester fleece

240 jacket, PET-2: 100% smooth polyester T-shirt, PAN: 100% acrylic sweater– horizontal axis

241 was cut at 1050 µm for visualization purposes.

242

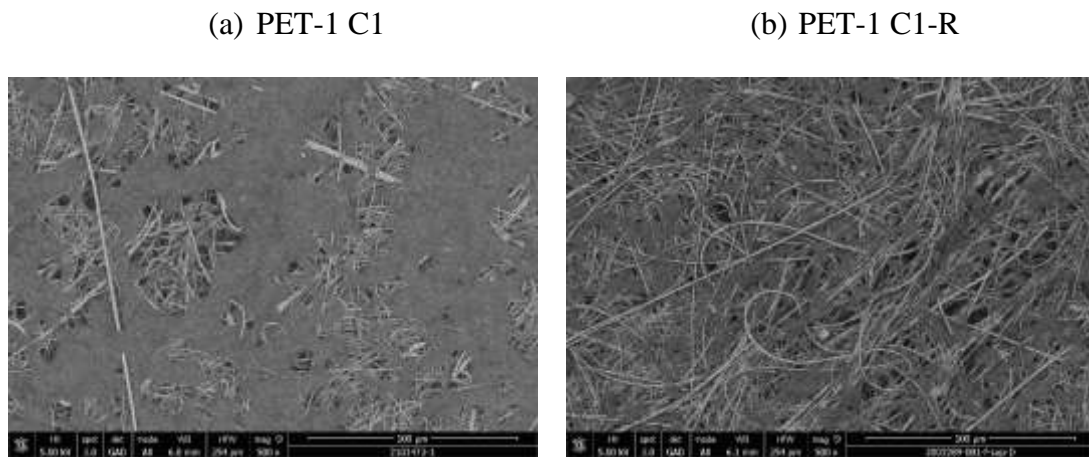
243 **3.2.1. Repeatability tests (PET-1 C1 and PET-1 C1-R)**

244 From Table 1, it can be seen that the number of microfibers released is near those of the
245 reference (PET-1 C1) and the repeatability tests (PET-1 C1-R): respectively 690 000 and 670
246 000 $\mu\text{fibers.kg}^{-1}$ of garment. Length distributions are also relatively similar (Fig. 4a),
247 confirming the repeatability of these washing and sampling protocols with respect to
248 microfibers release.

249 Around 65 to 70% of the microfibers are smaller than 200 μm long and only 5% are longer than
250 600 μm for both tests (Fig. 4a), while the length of the longest microfiber was 3.25 mm for the
251 reference test (PET-1 C1) and 3.4 mm for the repeatability test (PET-1 C1-R). From these two
252 distributions, one can calculate average microfiber lengths of respectively 194 and 229 μm .
253 These values correspond to 16 to 19 times the diameter of PET-1 microfibers determined by
254 SEM (12.3 μm). This is a much smaller average length than what has been reported by Napper
255 and Thompson [7] but more consistent with recent works that used dedicated techniques to
256 access information on microfibers length [30],[36],[23]. For instance, Zambrano et al. [30]
257 analyzed PET microfibers length distributions in washing machine effluents with a commercial
258 instrument (HiRes Fiber Quality Analyzer). They determined that the most abundant length
259 population was 200 μm which was the detection limit of their analytical equipment. Very few
260 microfibers above 2.5 mm were detected by these authors.

261 Despite the good repeatability in terms of microfibers release, a significant difference in mass
262 was found by the filtration method (Table 1) between the PET-1 C1 and PET-1 C1-R
263 experiments – a particle concentration ($>0.7 \mu\text{m}$) of 485 mg.kg^{-1} for the first was larger than
264 the 289 mg.kg^{-1} for the latter. This difference is significant with respect to the uncertainty of
265 the filtration method (Fig. 2a). In addition, one can notice from Table 1 that the calculated
266 microfibers mass obtained from the length distributions using Eq. 1 is lower than the mass
267 obtained by filtration: the reference test microfibers seem to account for less than 20wt% of the
268 total mass obtained by filtration, which suggests the presence of non-fiber material release. This

269 was confirmed by SEM imaging (Fig. 5) that shows an amorphous deposit on the surface of the
270 filters. This deposition is more present on PET-1 C1 which is likely related to the higher mass
271 obtained by filtration. This additional material release could be related to textile treatment such
272 as dyeing or finishing – these processes use various chemicals such as dyes, biocides or stain
273 repellents which could release residual quantities during garment washing. [44]. The
274 repeatability test was carried out with another garment lot and, since residual quantities of
275 chemicals may differ from one lot to another, it could explain the large difference in the mass
276 filtration results obtained.



277 Fig. 5. SEM observations of the amorphous deposit present at the surface of glass fibers filters
278 (0.7 μm porosity) after the filtration of water samples from (a) PET-1 C1 and (b) PET-1 C1-R
279 experiments.

280 3.2.2. Effect of the number of washing cycles (PET-1 C1, C2, C3 and C4)

281 Table 1 shows a large decrease in mass recovered by filtration between the first (PET-1 C1,
282 485 mg.kg^{-1}) and the second cycle (PET-1 C2, 72 mg.kg^{-1}). This phenomenon is mainly due to
283 the reduction of materials other than microfibers since their calculated mass from length
284 distributions (Eq. 1) is only reduced by 5 mg.kg^{-1} . This hypothesis is confirmed by SEM
285 observation (Fig. A.2) that shows the absence of the amorphous deposit after the second cycle.
286 Moreover, calculated microfibers masses are very close to those obtained by filtration for cycles
287 2, 3 and 4 (Table 1). This shows that textile emissions are mainly composed of microfibers after

288 the first cycle. Even if filtration results are always to be taken with caution for the quantification
289 of microfibers, latter observation tends to validate (for washing cycle > 1) the common literature
290 approach that directly assimilates filtration mass to microfiber mass [16]-[20].

291 The decrease of microfibers with the number of cycles observed in the present study (see Table
292 1) is in agreement with other works [7],[19],[20],[15]. For instance, Pirc et al.[19] showed that
293 the emissions from the washing of a polyester fleece (no additive) initially decreased from 0.016
294 wt% (161 mg.kg⁻¹) to 0.00239 wt% (23.9 mg.kg⁻¹) for the fourth cycle and then stabilized at
295 approximately 0.0011 wt% (11 mg.kg⁻¹) after 8-10 cycles. The large openings of the filter (200
296 x 200 μm) used to evaluate the mass of microfibers could explain the lower values obtained
297 compared to the present work.

298 Regarding results from the counting method (Table 1), one can also see the decrease of
299 microfibers release from the first (690 000 μfibers.kg⁻¹) to the fourth cycle (220 000 μfibers.kg⁻¹)
300 of PET-1 garment. Fig. 4c shows the effect of the number of washing cycles on microfibers
301 distribution. The reduction of small microfibers (<200 μm) is strongly related with the number
302 of cycles whereas emissions of long microfibers (>600 μm) are relatively less impacted. This
303 shows the initial presence of small loose microfibers that may be simply attached to the textile
304 surface rather than an inherent part of the matrix. One consequence is that the average length
305 tends to increase with the number of cycles (from 194 μm for the first cycle to 301 μm for the
306 cycle number 4).

307

308 **3.2.3 Textile effect (PET-1 C1, PET-2 C1, PET/PA C1, PAN C1 and CO C1)**

309 Filtration results ranged from 305 to 1520 mg.kg⁻¹ for the first washing of the five different
310 fabric types tested in this study (Table 1). The lowest mass was obtained for the cotton towels
311 (CO) and the highest for the 88% polyester/12% polyamide towels (PET/PA). Similarly to PET-
312 1 garment, particles other than microfibers can be identified on the surface of the filter by SEM
313 (Fig. A.3). These filtration results should not be interpreted as microfibers mass. The mass

314 obtained by Eq.1 (Table 1) and based on microfibers distributions determined by the counting
315 method is a better evaluation of microfiber mass.

316 For PET/PA and CO garment, it was not possible to perform the counting method due to a high
317 level of entanglement of the microfibers. Fig. 6 illustrates this phenomenon on PET/PA (left)
318 and CO (right) textiles. Despite dilution and specific ultrasound treatment, the entanglement
319 was still incompatible with the post-processing image techniques used in this study. For other
320 textiles, the counting method was performed without any issue.

321 Microfiber emissions for PET-1, PET-2 and PAN garments during the first washing were
322 respectively found to be 690 000, 1 570 000 and 1 390 000 microfibers per kg, which
323 correspond to 87, 149 and 273 mg.kg⁻¹ respectively (Eq. 1). A higher release of microfibers was
324 also found by Sillanpää and Sainio [20] for a polyester T-shirt compared to a polyester fleece
325 garment. However, Almroth et al. [45] determined that polyester fleece fabrics shed the greatest
326 amount of microfibers while Napper and Thompson [7] found that microfibers emissions from
327 acrylic garments are higher than those of polyester. The variety in analytical methods, washing
328 conditions and garment types is probably responsible for the discrepancy between studies and
329 highlights the difficulty to conduct a rigorous comparison between literature results in the
330 absence of normalized procedures.

331 Fig. 4d, shows similarly shaped distributed microfibers for the three garment types. Microfibers
332 smaller than 200 µm in length represent 73, 67 and 78% of the distribution for PET-1, PET-2
333 and PAN, respectively. This agrees with Hernandez et al. [23] that showed that the overall
334 microplastic fiber length profile remains similar regardless of the wash condition or fabric
335 structure, with the vast majority of fibers ranging between 100 and 800 µm in length.

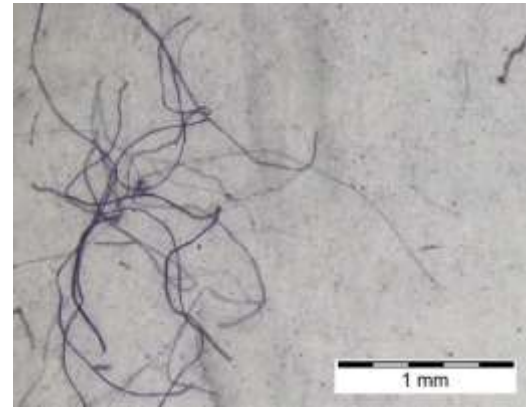
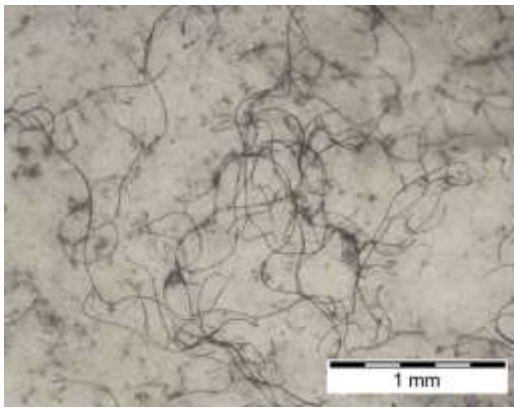
336

337

338

(a) PET/PA C1

(b) CO C1



339 Fig. 6. Optical microscopy images of the surface of glass fibers filters after the filtration of
340 wastewater samples from (a) PET/PA C1 test and (b) CO C1 test.

341 3.2.4 Detergent effect (PET-1 C1 and PET-1 C1-D)

342 Detergents may contribute to the mass of filtering residues because of the insoluble inorganic
343 compounds they contain [46]. These compounds will account in mass determination by
344 filtration method and can also create a thick layer that may induce an error (underestimation)
345 in the counting method since the microfibers can be partially or completely embedded in the
346 detergent compounds.

347 From Table 1, one can see that a high value of 1751 mg.kg^{-1} is obtained for the filtration result
348 with detergent (PET-1 C1-D) which is probably due to the presence of water insoluble
349 compounds at the surface of the filter. SEM observations confirm the presence of a thick layer
350 on the surface of the filter (Fig. A.2).

351 Detergent was found to cause a drastic increase of around 300% in the number of microfibers
352 emitted during the first cycle (see Table 1, 2 820 000 for PET-1 C1-D versus 690 000 for PET-
353 1 C1). This is even more relevant for small microfibers ($<200 \mu\text{m}$), as shown in Fig. 4b, which
354 tends to decrease the average microfiber length.

355 Several studies have studied the impact of detergent on microfibers emissions, but no clear
356 conclusion seem to emerge. Hernandez et al. [23], showed that among different parameters such
357 as the number of cycles or the washing temperature, the use of detergent appeared to impact the

358 release of microfibers the most. Falco et al. [46] came to a similar conclusion for polyester and
359 polypropylene fabrics. Carney Almroth et al. [45] showed that the use of detergent increase the
360 microfibers release for 3 out of 4 of their tested fabrics. However, Pirc et al [19] mentioned that
361 the use of detergent and softener did not significantly influence emission and Cesa et al. [17]
362 even recommend the use of detergent to diminish microfibers pollution. These inconsistencies
363 in conclusions are likely due to the different methodologies used in each study (analytical
364 method, washing conditions, garment type, detergent type etc...) and highlight the need for
365 setting standards in the community.

366

367 **3.2.5 Design of microfibers capture technologies and environmental aspects**

368 To prevent microfiber pollution, it is important to develop technologies that will efficiently
369 retain these particles during textile washing. Currently there is no regulation already in force
370 with respect to microfibers capture, but, for instance, the French government has recently voted
371 a law that will impose the implementation of a microfibers capturing device for all new
372 professional and domestic washing machines by 2025 [14]. So far, no retention efficiency target
373 has officially been set, but one should expect ambitious goals to effectively limit this pollution
374 and justify the deployment of such technologies.

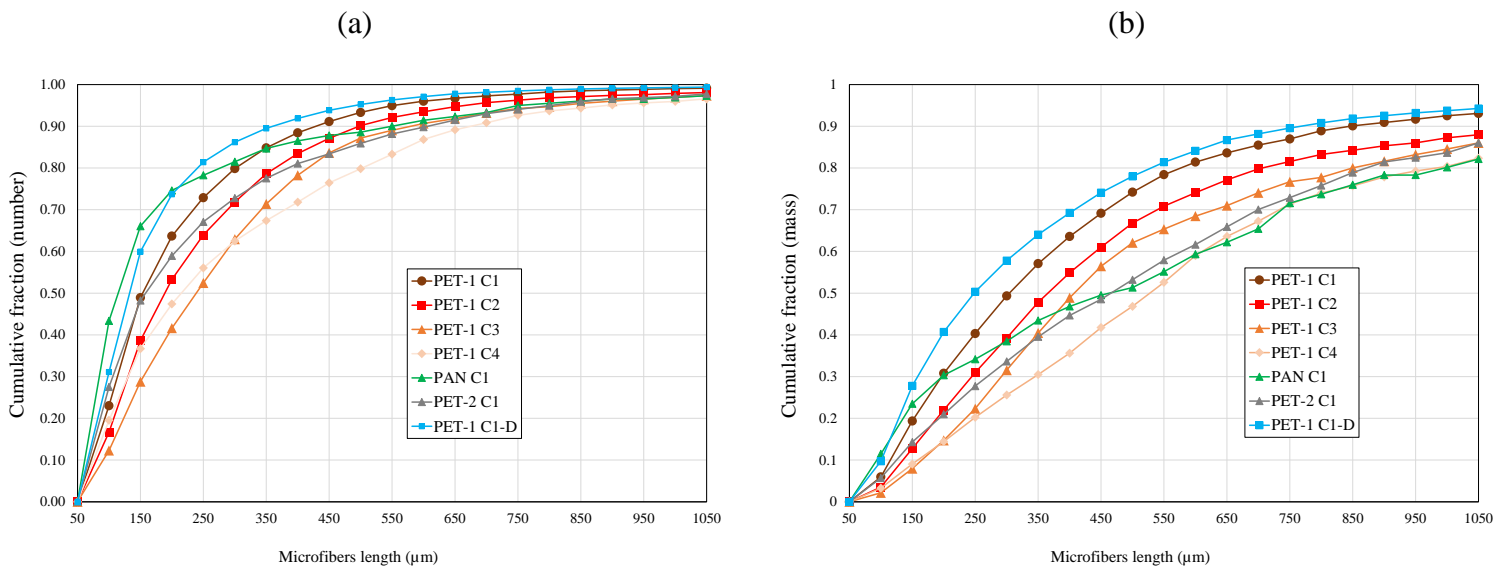
375 The analytical procedure developed in present study allows to access the detailed morphology
376 of microfibers released from textile washing. Therefore, it is a tool to better apprehend the
377 object size that must be captured by the microfibers capturing device. Fig. 7 presents the
378 microfibers cumulative length distributions (in number and in mass) for the different
379 experiments conducted in present work. If one considers a target of 90% retention efficiency in
380 number, from Fig. 7a it can be inferred that the future system should be able to stop microfibers
381 with a length lower than 100 μm and probably close to 50 μm . If the mass is chosen as the
382 criterion, then the 90% efficiency would lead to consider a minimum microfiber length to be
383 retained of around 100 μm based on Fig. 7b. One should also keep in mind that the small

384 diameter of microfibers (8 to 16 μm , see Table. A.2) must be accounted in the design of the
 385 retention technology. Indeed, depending on the approach angle, a microfiber can pass a filter
 386 through its diameter section rather than its longitudinal section.

387 Several microfibers filter technologies are available at commercial stage for domestic washing
 388 machines such as PlanetCare [47], Lint-LUV [48], GuppyFriend [49] or XfiltraTM [50]. These
 389 technologies were tested and characterized by Napper et al. [22]. The largest pore size was
 390 found for the filters Lint LUV-R (2 pore sizes: 285 μm and 175 μm) and PlanetCare (pore size
 391 of 200 μm). Low microfiber retention efficiencies were found for these two technologies (<30%
 392 in mass [22]) which seems consistent with the small dimensions of microfiber described in the
 393 present work. The bag device (Guppyfriend) and the Xfiltra filter showed higher efficiency (54
 394 and 78% in mass respectively [22]) due to their smaller pore size of respectively 50 and 60 μm .

395 This confirms the necessity of a fine filtration technology to effectively retain microfibers from
 396 washing machines.

397



398 Fig. 7. Microfibers cumulative length distributions – a) in number, b) in mass – PET-1: 100%
 399 polyester fleece jacket, PET-2: 100% smooth polyester T-shirt, PAN: 100% acrylic sweater–
 400 horizontal axis was cut at 1050 μm for visualization purposes.

401 While the description of the morphological properties of the microplastics delivered by the
402 washing of synthetic cloth is essential for the design of capture technologies, it is also of prime
403 importance for the good understanding of their environmental impact. Indeed, the
404 environmental impacts of microplastics are related to their chemical composition, and to the
405 morphology of the individual particles that compose them and that are released in ecosystems.
406 The environmental persistence is, for a specific chemical composition of plastic, directly
407 correlated to the surface to volume ratio of its individual particles. The environmental penalty
408 of a microplastic particle is not only related to the persistence of that particle, but also to its
409 bioavailability to living organisms throughout the food chain, which has been recently shown
410 to be shape dependent [51], [52]. These arguments reinforce the need to characterize in detail
411 the morphology of microplastics in order to better understand and control their impact. The two
412 aspects of capture and environmental impact will certainly be confronted in regulatory
413 measures, so that the technologies reach reasonable remediation levels. In this perspective, the
414 measurement of fibers size distributions should concentrate necessary efforts of normalization.

415

416 **4 Conclusions**

417 We present in this paper a novel analytical protocol that consists in characterizing microfibers
418 in number and size through filtration techniques, optical and electronic microscopy and
419 automatic image post-processing. The filtration process allowed to estimate the mass of
420 particles larger than $0.7\mu\text{m}$ while depositing them on a surface that could be easily characterized
421 by microscopy. However, it did not distinguish the mass belonging to the microfibers from the
422 mass coming from other compounds potentially present in solution, especially during the first
423 wash. Scanning electron microscopy provided more precise information on the morphology of
424 all the products retained by the filtration and on the diameter of the microfibers, showing that
425 other amorphous organic compounds are also retained during filtration. However, the nature of
426 these products is currently unknown. Optical microscopy and image post-processing using the

427 plug-im! platform provided reliable and detailed results for counting and size distribution of the
428 microfibers emitted from washing machines.

429 The analytical method was successfully applied on different washing conditions: 3 different
430 synthetic garments (PET-1, PET-2 and PAN), several cycles and in the presence or absence of
431 detergent. The automated image processing was however not successful to characterize cotton
432 and a blend of PET/PA due to a high level of entanglement between the microfibers.

433 The results obtained by this method provide an alternative to the current characterization
434 protocols. The proposed characterization protocol of microfiber length can also be extrapolated
435 to the counting of several other objects that have a similar morphology to the analyzed fibers.

436 The data can be helpful to the development of new microfibers capture technologies as well as
437 better quantifying the environmental impact of textiles waste, and could pave the way for
438 providing reference methodologies in the future.

439

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595 **Appendix A**

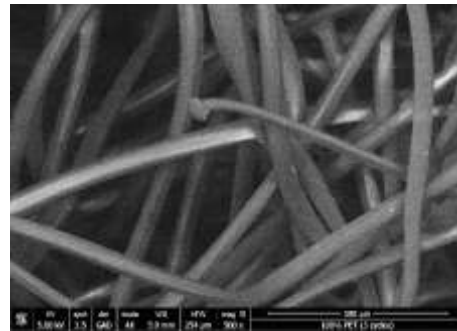
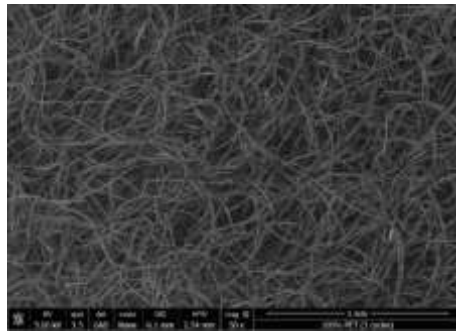
596 **Table A.1. Garment characteristics.**

	PET-1	PET-2	PET/PA	PAN	CO
Color	black	black	black	black	navy-blue
Composition	100% PET	100% PET	88%PET/ 12%PA	100%PAN	100%CO
Garment model	fleece jacket	T-shirt	towel	sweater	towel

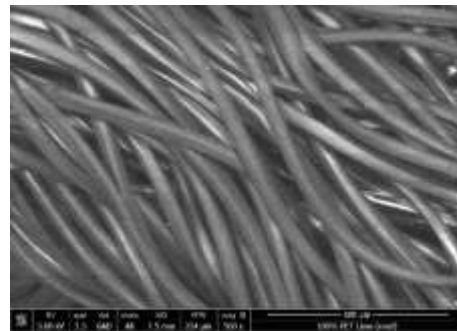
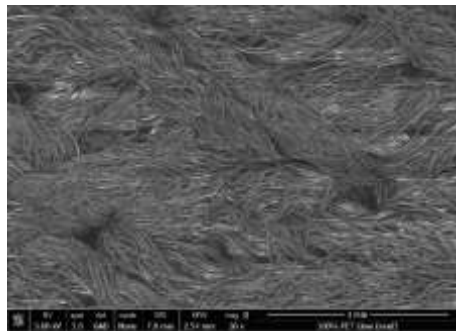
Garment Size	Extra Large	Large	-	Medium	-
Garment weight (g)	294	112	327	531	470
Fabric weight per unit area (g.m ⁻²)	194	117	166	543	374
Fabric thickness (mm)	1.6	0.4	0.6	4.5	2.4
Fabric type	knitted	warp knit	woven	ribbing knit	terry cloth
Yarn type	Filament	Filament	Filament and staple	two ply-yarn (staple)	two ply-yarn (staple)

597

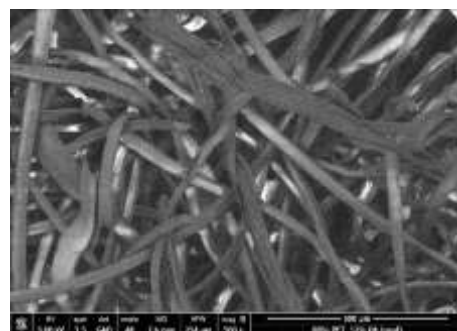
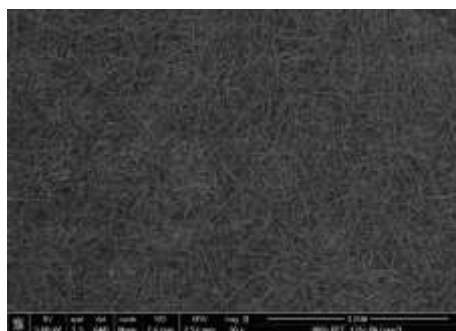
PET-1



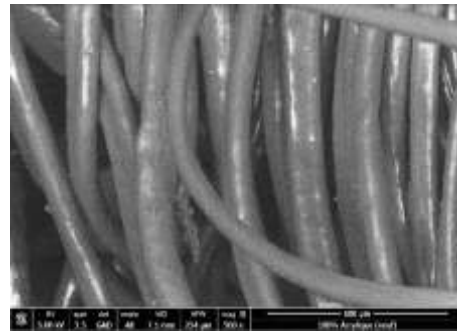
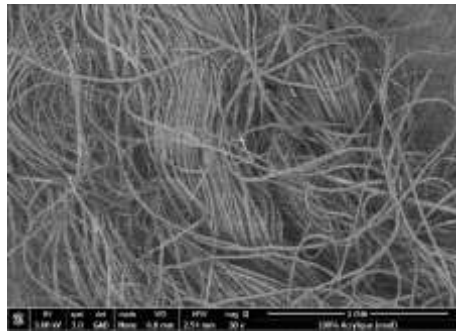
PET-2



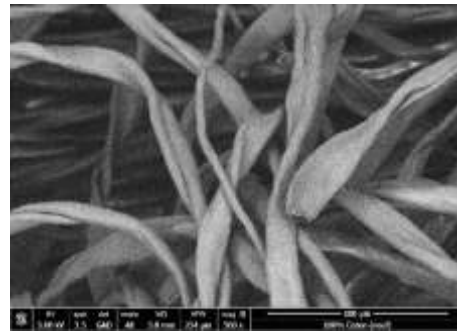
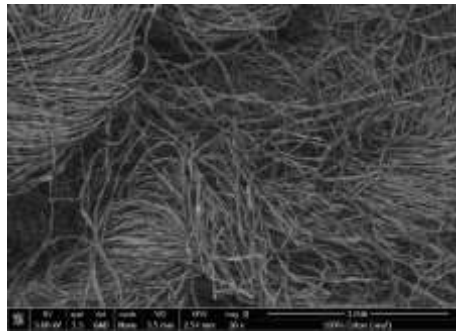
PET/PA



PAN



CO



598

Fig. A.1. SEM micrographs of garment fibers.

599

600

Table A.2. Average diameter of garment fibers determined by SEM.

Type of fabric	Average diameter (μm)
PET-1	12,32 (\pm 2,90)
PET-2	9,59 (\pm 1,67)
PET/PA	8,20 (\pm 2,84)
PAN	16,99 (\pm 4,10)
CO	13,21 (\pm 4,94)

601

OM

SEM

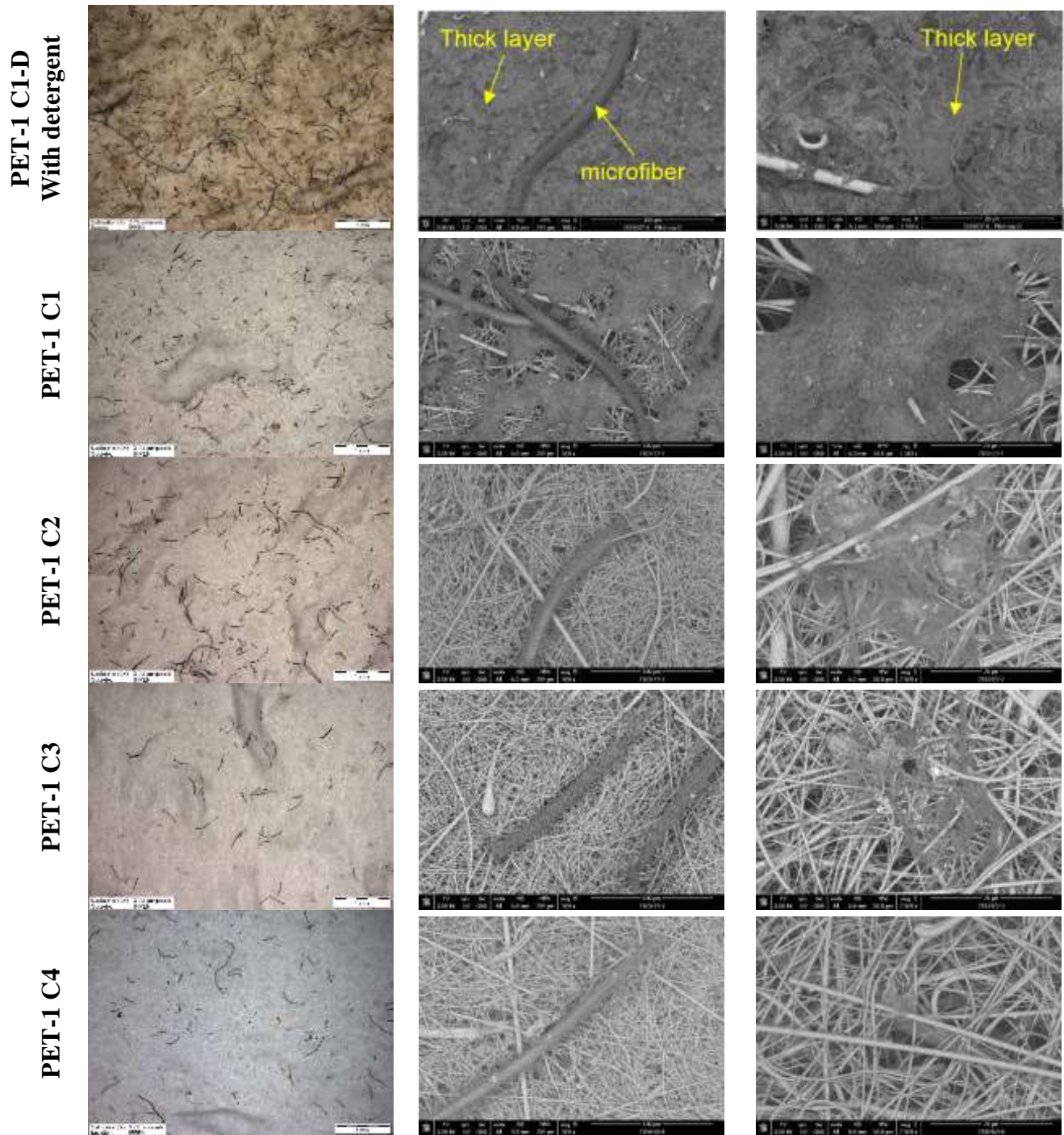
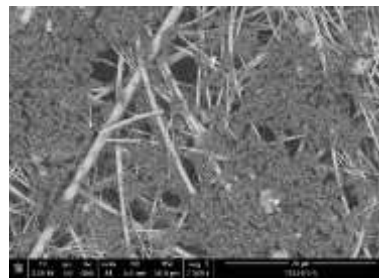


Fig. A.2. Optic Microscopy (OM) and SEM pictures – effect of washing cycles.

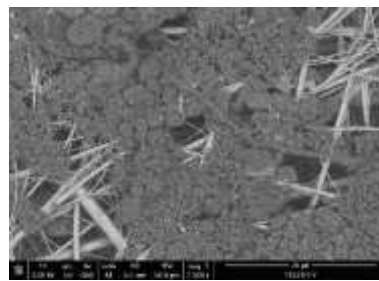
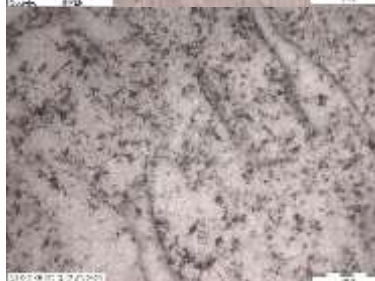
602



PET-2 C1



PET/PA C1



PAN C1



CO C1

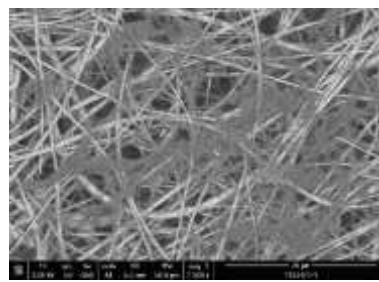


Fig. A.3. Optic Microscopy (OM) and SEM pictures – effect of textile type.