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Microplastics' Emissions: Microfibers' Detachment from Textile Garments

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Abstract: Microplastics (synthetic polymers <5 mm) have been recently recognized as a big environmental concern, as their ubiquity is an undeniable fact. Their wide variety regarding shapes, sizes, and materials turn them into an intrinsically risky pollutant capable of causing several environmental impacts. Textile microfibers (MF) are a microplastic sub-group. These are mostly shed when a normal laundry of any garment takes place. Special attention has been put onto them, as high concentrations have been found in products for human consumption as shellfish and tap water. However, as there is no consensus on the methodologies to quantify and report the results of MFs detached from textile garments, the degree of similarity between published studies is very low. Hence, the aim of this research was to evaluate the microfibers' detachment rates of finished garments and to provide a set of comparable units to report the results. These were found to range between 175 to 560 MF/g or 30000 to 465000 MF/m^2 of garment. In addition, there was a high correlation between the MF detachment and the textile article superficial density. Finally, our results were compared with a recent paper that estimated the annual mass flow of MFs to the oceans. This previous publication is 30 times higher when related to the mass but 40 times lower if related to the number of MFs.

Keywords: Microplastic; Microfiber; Pollution; Textile.

Capsule: This work provides new insights with respect to microplastic pollution. It also establishes a method for the quantification of textile microfibers and recommends comprehensive and comparable units to be used when publishing the results.

1. Introduction

The globally widespread plastic pollution is a well-known environmental concern that has been even suggested as an indicator of the *Anthropocene* period.[1] Synthetic polymers debris sized at <5 mm, generally defined as microplastics (MPs from now on), have been recently recognized as an important and abundant pollutant.[2] MPs occurrence is increasingly growing in freshwaters, terrestrial and atmospheric ecosystems,[3–5] and have even reached remote places far from anthropogenic influence.[6] However, the major sink seems to be the marine environment, where these pollutants are ubiquitous, as they are found from the top to the bottom[7,8] and from the equator to the poles.[9,10] Last estimations reported 15 to 51 trillion *buoyant* MPs in the oceans,[11] but these are believed to be only the "tip of the iceberg".[12]

The ingestion of these plastic particles by biota is well registered,[13] especially in marine organisms, where MPs have been identified in all levels of the trophic chain.[14,15] However, it also extends to organisms of other ecosystems.[16,17] Observed possible impacts in biota are MPs' retention[18] and trophic transfer,[19] reduced vital functions capacity,[20–22] translocation to other organs,[23] gene exchange,[24] endocrine disruption,[25] increased mortality,[26] bioaccumulation of toxic chemicals,[27] altered sinking rates for fecal pellets,[28] etc. MPs are also known to act as vectors for alien species and for hydrophobic contaminants (either added in the plastic manufacturing process or adsorbed once in the environment),[29,30] and to alter physical properties of beach sediments.[31] Moreover, there is evidence of MPs presence in products for human consumption as seafood,[32,33] tap and bottled water,[34] and table salt.[35] Nevertheless, the human health risks still remain unclear.

The MPs' sources are usually classified into two main groups (adopted for this work): *Primary MPs* are those emitted to the environment in an MP size range (e.g., textile microfibers, microbeads); and, *Secondary MPs* are those originated once in the environment from the degradation and fragmentation of mismanaged plastic waste.[2,36] However, there are still no accurate estimations of the contribution of each MP source; hence, it is necessary to elaborate tools that enable us to achieve a better knowledge of the importance of each contributor.

Textile microfibers (MFs from now on) are detached among every step of a textile article life cycle, especially when its laundry takes place.[36] Many publications have reported high concentrations of MFs in aquatic environments,[37] hence, they appear to be one of the most important primary MPs contributors.[36,38] A few studies have proposed different methods to quantify detached MFs.[37,39–44] These usually applied indirect methodologies, e.g., estimating the amount of MFs from their weight and length. However, the accuracy between these studies is still low, and the units used to express the results are different, making their comparison even more difficult. These methods were tested in our laboratory, but the estimations were not in accordance with our visual quantification of MFs (discussion in section 3.1). Therefore, in this work, it was developed and applied a direct and reliable method to determine the ***microfibers' detachment rates*** (**MFDR** from now on) when washing textile articles. In addition, a relation between the number of MFs and their mass, and also a set of comparable units are provided. Finally, from this quantification, an estimation of the amount of MFs released to the environment is carried out and compared with previously published works.

2. Materials and Method

2.1. Materials

Brand new garments of polyester, polyester-elastane, and polyamide-elastane were bought from different fashion stores in Spain and tested. The characteristics of the selected garments are described in Table 1 (*photographs in SI, 1a*):

Table 1. Characteristics of the tested garments.

Material	Group	Naming	Type	Mass [g]	Area [m^2]	Type of Fabric
100%	F	F1	"Fluffy"	603	1.63	Woven fabrics, with the

Polyester		F2		723	1.95	exception of F4 which is knitted.
		F3		643	1.60	
		F4		396	1.08	
		F5		728	1.50	
	P	P1	Shirt (1)	101	0.55	Knitted fabric.
		P2	Nightgown	241	1.50	Woven fabric.
80% Polyester 20% Elastane	PE	PE1	Shirt	134	0.35	Knitted fabric.
		PE2	Gym pants	193	0.57	
		PE3	Jacket	305	0.90	
70% Acrylic 30% Polyamide	PAC	PAC	Woolen cap	74	0.09	Knitted fabric.

(1) Recycled polyester.

A front-load conventional washing machine (*FAGOR Innovation F-2810*, Spain) was used. In order to save energy and water, the *superquick* program was chosen (15 minutes, 22 liters of effluent, 1000 revolutions per minute, ambient temperature). Tap water from *Terrassa* was supplied to the washing machine. A common detergent ("*Bosque Verde*", Spain) was selected, where the quantity applied was in accordance to the specifications written on the container as a function of the weight of the garment and the hardness of the water (349 mg $CaCO_3$/L [45]). An explanation of the operational parameters selection is described in Section 2.2.

Polyamide filters (*Millipore NY20*, Ireland) with a 20 µm pore diameter were used to retain the MFs. The filtering system consisted of a flask connected to a vacuum pump. A stereomicroscope (*Carton Stereo Zoom SC*, Japan) and an electronic microscope or SEM (*PHENOM ProX Desktop*, The Netherlands) were used to analyze the MFs.

2.2.Methodology

The developed method is constituted by the following steps:

a. The garments were weighted, measured and characterized with respect to the material before starting the procedure.

b. A commercial washing machine was selected to execute the trials, as laboratory washing machine simulators might not produce the same effects (e.g., the centrifugal operation step is not simulated). An empty washing cycle was done to clean residual dirt between launderings of different garments.

c. One of the selected garments was independently washed 10 times to determine the number of washing cycles required to achieve a stationary situation. According to the result of this test and to other previously published works, the rest of garments were only washed 5 times (also independently).

d. For each washing cycle, the washing effluent was completely collected (22 L) in a closed container. A 10 L sample was taken, while stirring, using a hose assembled at the bottom of the closed container.

e. From the 10 L sample, three smaller aliquots of 10 mL were taken, while stirring, and rinsed up to 100 mL with distilled water to get a more homogeneous MF distribution on the filters. The purpose of such smaller aliquots is to be able to visually count the MFs retained after the filtration, as previous trials showed that major volumes made it impossible due to MFs overlapping.

f. The filters, which were always kept in Petri dishes to avoid contamination, were carefully placed on the filtration system with a clamp and the small water aliquots were filtered. Then, distilled water was used to drag all the MFs retained in the sample collector.

g. Subsequently, the filters were dried at 60ºC for 24 hours. Once cooled, these were placed under the stereomicroscope where the visual counting of the MFs was done.

h. The background on which the Petri dishes with the filters were posed was alternated between light and dark-colored depending on the color of the garment.

A simple diagram of the described methodology is shown in Figure 1:

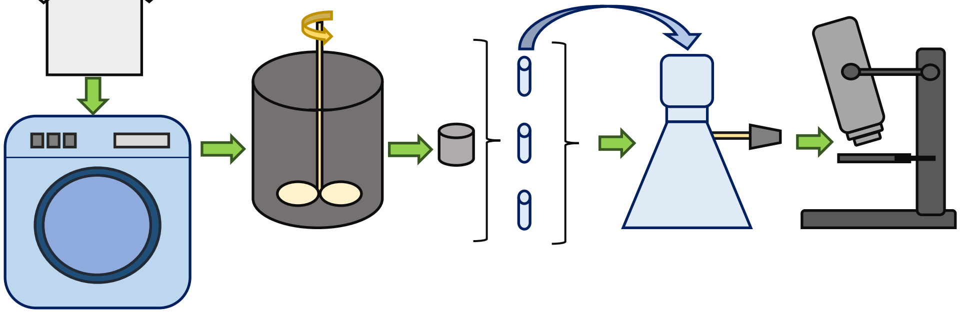

Figure 1. Scheme of the developed method.

It must be clarified that the operational parameters were also chosen expecting the minimum MF detachment from the garments. Previous publications have reported that front-loading washing machines,[41] liquid detergent[44], and lower temperatures[44] produce less mechanical stress than their opposites; the same was expected from washing the garments for a short period of time. Still, further work is needed to determine the relevance of these parameters in the MFDRs.

The observed amount of detached MFs will be expressed with respect to the garment weight ($W_G: MF/g$) and surface ($A_G: MF/m^2$). Additionally, an evaluation of these results and the superficial density ($SD_G = W_G/A_G$) of each garment was done, from where a positive correlation was expected.

Furthermore, a scanning electron microscope was used to evaluate the MFs' morphology.

2.3.Calculations

The repeatability of the method for the quantification of the detached MFs (steps a-h) was evaluated with the average error and the average coefficient of variation of all the samples. The equations used are described in SI, Table S1.

On the other hand, a relation between the quantity of MFs and their mass, MF_W (MF/mg), has been established. This can be obtained from the fiber linear weight, usually named yarn count C ($dtex$), and the MFs' average length, $\bar{L}_{MF}$ (mm/MF), by using the following equation (1):

$$MF_W = \frac{1}{C \cdot \bar{L}_{MF}} \quad (1)$$

A decitex (dtex) is a unit of measurement of the linear weight of a filament textile fiber. It is expressed in grams of filament fiber per 10000 meters. The equation (2)[46–48] used to estimate the linear weight (C) is described hereunder:

$$C = \emptyset^2 \cdot \frac{\pi \cdot \gamma}{400} \quad (2)$$

Where the average diameter $\emptyset$ (μm) of the MFs was obtained from SEM observation, and γ (g/cm^3) is the specific weight of the fiber material. In this study, the specific weight of the polyester (1.38 g/cm^3) was used in all calculations as it is the predominant material.

From equation (1), a table with typical *dtex* values versus a range of MF lengths between 0.1 to 5 mm was plotted to facilitate the estimation of the mass of the MFs (Table S2. SI).

Finally, MF_W from equation (1) was afterward applied to estimate the mass loss of MFs by multiplying its inverse with the quantity of MFs detached from the garments.

3. Results and Discussions

3.1. Microfibers' Detachment Across the Washing Cycles

In order to determine the required number of washing cycles to apply across this work, the garment F1 was washed 10 consecutive times (as indicated in section 2.c). It was found that between the 4th and 5th washing cycle the MFDR stabilized (SI, Figure S2). This result is in accordance with the publication of Napper and Thompson (2016).[39] Henceforth, the rest of the garments studied in this work were only submitted to 5 washing cycles. All the observations gathered from the washing trials are included in SI. As an example, some of them are shown in Figure 2, where confidence intervals for each trial are also plotted.

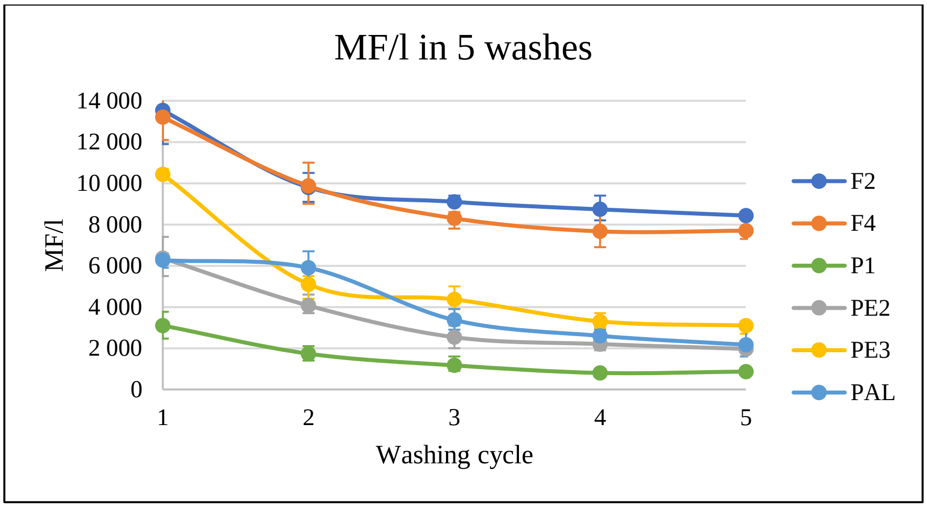

Figure 2. The MFs detachment decreases from the 1st to the 5th washing cycle in all tested garments.

From the washing trials observations, it can be noticed that the garments shed more MFs in the first washing cycles, which is probably due to the presence of leftovers from the garment manufacturing process. Hence, the application of MFs' retention mechanisms in industrial stages, as in the textile dyeing and finishing processes, could easily help to reduce a considerable amount of MFs from reaching the environment.

However, no clear trend was found between the garment material and the progressive reduction from the 1st to the 5th wash of the MFs' detached. Fluffy garments reduced the MFDR from 20 to 40%, other polyesters (P1, P2) from 10 to 70%, polyester mixed with elastane (PE1, PE2, PE3) from 70 to 80%, and PAC 40% (data in SI, Table S3). Also, the type of fabric (knitted or woven) does not seem to have a relevant influence on MFDR results.

On the other hand, on the bases of equations described in Table S1, the average error (calculated for all samples and 5 washing cycles) was E = 8% and the coefficient of variation was CV = 10% (data in SI, Table S4), which indicates that the method has a high repeatability. It should be mentioned that methods proposed in previous publications[39–41] were tested. Nevertheless, overestimated results were found when comparing the data obtained from those methodologies with the visually counted MFs. A feasible explanation comes from the impurities that were found on the filters and within the MFs. These might come from the detergent, from additives un- or intentionally applied to the garments during the manufacturing process, and/or from the tap water used for the washing machine trials. Hence, the reliability of the method developed and applied in this work is generally higher than that of previously published ones because possible interferences implied in the weighting process of the MFs are eliminated.

3.2.Morphological Aspects of Detached MFs (length and shape)

By means of the stereomicroscopic observation, it was found that the length of the detached MFs decreased from the 1st to the 3rd wash in every tested garment. This behavior was also confirmed by determining the trend of the ratio MF/mg, which was seen to increase from the 1st to the 3rd washing cycle. In this way, longer and more MFs are detached in the firsts washing cycles, strengthening the greater ease and effectiveness that the early-stages MFs' retention systems mentioned in section 3.1 could have.

In the last washing cycle, all MFs were visible under the stereomicroscope. The average length was between 0.2 to 0.4 mm, and the minimum found was of ~ 0.08 mm. However, a smaller fraction < 20 µm to nanoscales might exist but was not evaluated. As an example, the evolution of the trend of the P1 garment MF length across 5 washing cycles is shown in SI, Table S3.

Furthermore, from SEM observation, two possible causes for the detachment of these MFs were identified. A first group corresponds to MFs already attached or entangled with the fibers' grid of the garments (Figure 3A), which have a regular tail-ended shape. In contrast, the other group appears to be MFs that were ripped-off from the fibers' grid (Figure 3B) as a consequence of the mechanical stress suffered by the garment throughout the launderings. The garment UV degradation[49] and its use might also debilitate the fibers and facilitate the MFs' ripping.

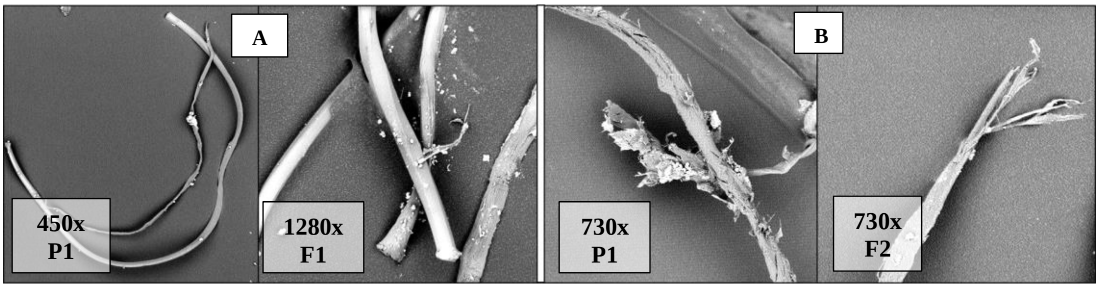

Figure 3. A) Microfibers with a regular end-tailed shape, and **B)**, microfibers with a ripped-off end-tailed shape.

Hence, the application of a biodegradable coating could help to reduce these MFs by enhancing the grid connections and/or the garments' resistance to mechanical stress.

Finally, as seen in Figure 3A and B, there is more material detached from the garments, which are thought to be oligomers from the fiber manufacturing process.[50] As these microparticles are also released to the environment, an evaluation of an inclusive definition that contemplates the "total released material to the environment" should be considered in case of a terminology standardization.

3.3.Detached MFs

As previously indicated, based on previous publications, the operational parameters described in Section 2.2 were selected to get the lower MFs' detachment conditions for the tested garments.

Total MFs detached per garment is plotted in Figure 4. The evaluation of the detached MFs is referred to the 5th washing cycle, as it is the point where the MFDR stabilizes.

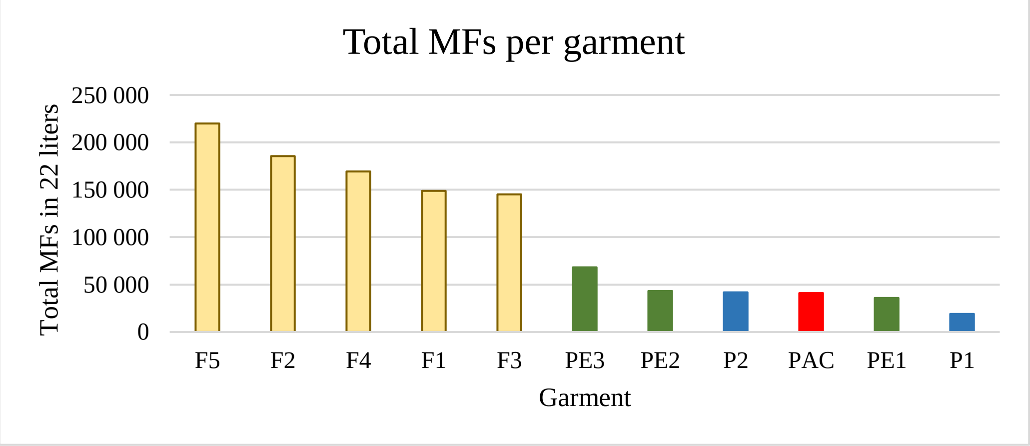

Figure 4. Total amount of detached MFs per garment for a total effluent of 22 liters (5th washing cycle).

When considering absolute values, fluffy garments detached the most MFs, followed by a non-obvious pattern of garments. However, absolute values are only useful to appreciate the difference between whole finished garments and to efficiently inform about this issue to the consumers, but should be avoided when the objective is to achieve fundamental conclusions on the MFs' detachment behaviors of the textiles. For this reason, the detachment of MFs was also expressed in two other different units (MF/m^2 and MF/g) and evaluated with the superficial density of each garment (Figure 5A and 5B):

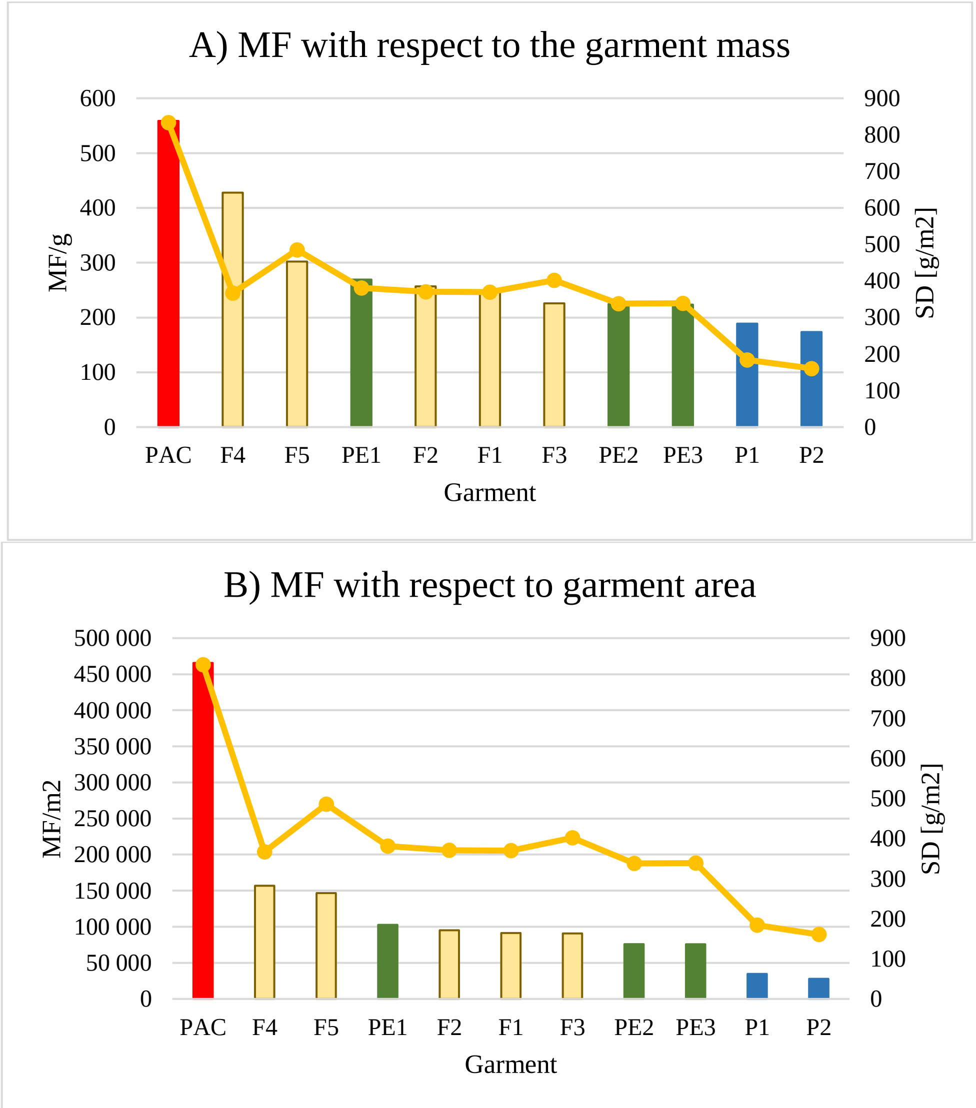

Figure 5. The MFDR divided by the garment **A)** mass and **B)** area. The Superficial Density (SD) of each garment is indicated by yellow points. (results expressed for a total effluent of 22 liters and taken from the 5th washing cycle).

As expected, both units allow better comparability of the results and should be used when evaluating MFs' detachment trends. Firstly, from Figure 5 it can be seen that the acrylic/polyamide garment (PAC) had by far the highest MFDR (560 MF/g or 465 000 MF/m^2), which might be a consequence of the textile type (knitted fabric and a tassel formed by a low twist yarn, which seems to have a major role in the MFDR); whereas fluffy garments situated at the middle, followed by polyester/elastane and other polyesters (175 MF/g or 30 000 MF/m^2).

However, it should be noticed that different operational conditions of the washing machine (e.g., washing time, temperature, etc.) than the ones used for our trials might give other results, although the relative detachment rates between garments should remain constant.

In addition, a positive correlation was found between the surface density (SD) and the MFDRs. Adjusted R^2 was 0.71 for MF/g and 0.89 for MF/m^2. Also, the relative MFDRs between the garments remained unchanged when unifying the results with respect to the garment mass or area. This means that although the SD is an important predictor of the MFDR, other factors also influence it, as e.g., the garment material and fabric type. For this reason, it is a purpose for future works to make an exhaustive evaluation of them.

Finally, by using equations (1) and (2) with an average length of 0.3 mm and a diameter of 20 µm, results were transformed to mass loss of MFs. In this way, ranges of 2 to 29 mg/garment, 23 to 73 mg/kg of garment and 4 to 61 mg/m^2 of garment were obtained.

3.3.1. Comparison of MFs' Detachments Published by Other Works:

Some authors have published estimations of the detachment of MFs, although their results are difficult to compare because they are expressed in different units. In addition, methods used for the estimation of these MFs are not always available or reliable. A summary of these previously published works, expressed in their corresponding units, is presented in Table 2. In order to facilitate the comparison, we also included our results but expressed in the same units.

Table 2. Previous published works information and comparisons with the present work.

Work	Comments on the analytical method	Bibliographic results	Our results expressed in the same units
Browne et al., (2011) [37]	No clear information of the methodology used. Conservative estimations reported.	130 – 280 MF / L per garment > 1 900 MF / garment	1 500 – 10 000 MF / L per garment > 30 000 MF / garment

Napper and Thompson (2016) [39]	Indirect method [(a)]. Fibers with a mean length > 5 mm considered.	140 000 – 730 000 MF / 6 kg of washed garments 500 000 MF / mg	1 000 000 – 6 500 000 MF / 6 kg of washed garments 5 760 – 11 521 MF / mg [(b)]
Pirc et al., (2016) [40]	Indirect method [(a)]. Filters used of 200 µm; mean length considered > 5 mm.	135 000 MF / 6 kg of washed garments	1 000 000 – 6 500 000 MF / 6 kg of washed garments
Hartline et al., (2016) [41]	Indirect method [(a)].	29 – 431 mg of MF / garment washed (front-load) 1 471 – 2 121 mg of MF / garment washed (top-load)	2 – 29 mg of MF / garment washed [(c)] (front-load)
Äström (2016) [42]	*Gyrowash* to simulate washing machine.	7 360 MF / (m^2 L)	1 200 – 33 000 MF / (m^2 L)
Cesa (2017) [43]	Used Napper & Thompson (2016) and Pirc et al. (2016) methods.	184 000 – 250 000 MF / garment	30 000 – 230 000 MF / garment
De Falco et al., (2018) [44]	*Linitest* apparatus used to simulate washing machine. Direct quantification.	6 000 000 – 17 700 000 MF / 5 kg of washed garments	1 000 000 – 6 500 000 MF / 6 kg of washed garments

(a) Indirect method: the quantification is estimated from the weight, the length, and/or the density of the MFs.
(b) Estimated by applying the calculation methodology explained in Section 2.3 and using an average MF diameter of 20 µm and a MF length between 0.2 to 0.4 mm.
(c) Same procedure than (b) but using an average MF length of 0.3 mm.

As seen in Table 2, when referring to the number of MFs, our results are mostly higher than previous publications (Ref. 37, 39 and 40), although in some cases they are similar (Ref. 43) or even lower (Ref. 44). However, when related to the weight, our results are always lower (Ref. 39 and 41). These discordances could be because different methods and factors were used. For instance, particles that are not MFs might have been weighted and reported as MFs. Also, in some cases, the units used to report the results were confusing or even useless. Hence, it is recommended to standardize a method with clear parameters and to unify the observations with respect to the garment area and/or weight (MFs and/or milligrams of MFs per unit of area and weight of the garments). Finally, it should be pointed out that a strict comparison is always affected by the intrinsic inter-laboratory variability, due to factors such as the washing machine and washing cycle, water quality, etc.

3.3.2. Estimation of the Textile Microfibers' Global Input to the Oceans:

The last estimation of the global textile MFs' flow to the environment was published by Boucher and Friot (2017).[36] They approached three scenarios (minimum, central and maximum) based on previously published works. However, the quantity of textile MFs reaching the oceans (Table 3) was estimated using data from works[37,51–53] in which their purpose was not to evaluate the MFDRs.

Hence, in this work, the textile MFs' flow to the oceans is re-estimated on the bases of the following assumptions:

a. The approaches of the annual laundry cycles per capita, load per standard wash, and regional availability of wastewater technologies and population are still the ones proposed by Boucher and Friot (2017).[36]

b. The data for Boucher and Friot (2017) was obtained by using a MF linear weight of 300 *dtex*. In the present work, the linear weight of the MFs was estimated by applying the methodology described in Section 2.3. In this way, MFs were considered to have an average length of 0.3 mm and a maximum diameter of 20 µm (SI, Figure S3). Applying those values, a linear weight of 4.34 *dtex* was obtained and adopted to proceed with the analysis. This value is consistent with ranges reported for typical polyester filament yarns. [54]

c. As an uneven number of garments of each group were tested, the same weight was applied to every group in order to homogenize the observations. This was done by calculating the MFDRs' average within the garments of each group (refer to Table 1); those outcomes were used to determine the resulting average between the groups, which was considered as the central value. We considered that garment PAC is not a representative sample for the purpose of this analysis, as this type of garment is less frequently used and/or laundered. Hence, it was removed from the data.

Therefore, we recalculated the different scenarios of MFs reaching the oceans. The results are indicated in Table 3:

Table 3. Recalculated values for textile microfibers reaching the oceans.

Results		**Boucher and Friot (2017)[36]**		**Present Work**	
Values (mg of MFs per kg of garments)	Minimum	300		23	
	Central	**900**		**33**	
	Maximum	1 500		56	
Total MFs reaching the oceans using central values		**Tons MF/year**	**MF/year [(a)]**	**Tons MF/year**	**MF/year**
		520 000	**$3.6•10^{15}$**	**17 830**	**$1.4•10^{17}$**

(a) Estimated using the 300 ***dtex*** and 5 mm for a MF length assumed in Boucher and Friot's work**.**

From Table 3 we can conclude that, based on Boucher and Friot's[36] calculations and with respect to our results, they might have overestimated the mass flow rate of MFs to the oceans. This discrepancy is mainly because the linear weight applied to calculate the mass of the MFs is presumably overestimated. In fact, the calculations of Boucher and Friot (2017) derive from using a linear weight of 300 dtex, which is a common value of yarns composed of a group of individual filament fibers.[46,54] This factor was firstly applied in a report of the Norwegian Environmental Agency[52] and later assumed as appropriate in most of the subsequent publications.[36,41,51,55] However, in this particular case, less does not necessarily means better, since using the values of Boucher and Friot a particle flow of $3.6•10^{15}$ MFs/year can be estimated, which is a 2.7% of the $1.4•10^{17}$ MFs/year calculated with our results. Moreover, it should be underlined that the values reported by Boucher and Friot (2017)[36] were estimated with an assigned MF length of 5 mm, in contrast with the average of 0.3 mm measured in this work. As a consequence, according to our results, smaller and more easily ingestible MFs are heading towards the oceans.

4. Conclusions

A direct and highly reliable method to quantify the detachment of textile microfibers from whole finished garments was developed and applied. In order to normalize the microfiber detachment rates results, comprehensive and comparable results are needed. In this way, we recommend a set of units that give fundamental conclusions of the microfiber detachment with respect to the textile article. In addition, a methodology to estimate the relation between the number of MFs and their mass was developed.

From consecutive washing trials, it was found that the microfiber detachment rate (MFDR) decreases until stabilization is reached in the 5th washing cycle. The MFDR in that point is between 175 to 560 MF/g or 30000 to 465000 MF/m^2 of garment. It was also found a high and positive relation (R^2 = 0.71 to 0.89) between the MFDR and the superficial density (g/cm^2) of the garment. Transforming the results into units of mass, we estimated a MF loss between 23 to 73 mg/kg of garment or 4 to 61 mg/m^2 of garment.

Moreover, the morphology of the microfibers was analyzed, and two different shapes were found: one group that comes from microfibers that were already loosely entangled with the fibers' grid of the garments, while the other corresponds to microfibers that were ripped-off from the fiber grid as a consequence of the mechanical stress suffered in the launderings. This latter case could be perpetuated by the garment use and its UV degradation. With respect to the microfiber length, it was found that it decreases from the 1st to the 3rd washing cycle. Both findings are helpful to evaluate the applicability of new microfibers' reduction solutions in different steps of the garment life cycle.

Finally, our results were used to re-estimate the mass flow of microfibers to the oceans, which was found to be overestimated by other authors. However, according to our results, the amount of MFs reaching the oceans is $1.4 \cdot 10^{17}$ MFs/year, which is higher than the value obtained when our calculation methodology is applied to the previously published data. This implies that a higher quantity of smaller and more easily ingestible microfibers is heading towards the oceans.

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