Statement

by

Industrievereinigung Chemiefaser e. V. (IVC)

Microplastic And Textile Fibres
Review: Release Of Synthetic Fibres From Washing

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Abstract

Published and reported examinations of the release of fibre particles from the washing of synthetic textile products are reviewed and evaluated. The results of the studies scattered with differences of four orders of magnitude!

Unfortunately many reports about the release of fibres from washing miss detailed information about the tested textile products, have even shortcomings in the design of the study and use flawed or questionable methods.

Currently only the study by Pirc et al. (2016) can be regarded to present reliable results of the shedding from washing and tumble drying which was achieved from a PES plush (polarfleece) blanket regarded as a shedding sensitive product. The used more fold washing treatment allows a distinction between the shedding due to mechanical treatment and the release of remaining or weakened fibres from the manufacturing of this product. The loss during washing after a tumble drying resulted in < 40 fibres per g of this plush fabric with double sided high floor from 1 dtex microfibres under mild washing conditions.

However, this and other results of fibre release from any individual textile product are absolutely not suitable for any extrapolations of released fibres into sewage water. Too many factors have an impact and textiles are too variable and complex.

Our assumption of few synthetic fibres in textile constructions which might be partial sensitive to the shedding during washing needs still to be confirmed by professional studies. The regarding examinations need to differentiate between release of left-over fibres and shedding from abrasion and to develop simple methods for these tests.

Introduction

The term “microplastic” is used for solid particles of synthetic polymers, insoluble and persistent in marine environment. The dimension is not undisputed yet, often in use are diameters below 5 mm, even without a lower boundary and including nanoparticles (GESAMP, 2016), see also in Appendix 1.

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Fibrous fragments from synthetic man-made fibres are generally considered as "microplastic".[1] These are also more easily detectable already by visual inspection with stereomicroscope and reported in many studies as most abundant form of synthetic particles in environmental samples.[2]

The shedding of fibres during washing from synthetic garments was assumed to be the most common source of synthetic fibres in the marine environment and huge volumes of synthetic fibres were estimated to be emitted in the aquatic environment and oceans, as they are not retained (sufficiently) in waste water treatment plants.

But results of the examinations of fibre release from washing differ with factors of up to four orders of magnitude. Some differences in the extent of the shedding of fibres from washing of the mostly tested plush textiles (or polarfleece) are expected, as these might be manufactured in different constructions and with different yarn specifications. However, we noticed also shortcomings in the design of some studies and even in the weighing procedures which relegates many results.

The reasons and sources of these huge and incredible differences are analyzed in this critical review surveying and discussing the regarding reports and publications in more detail.[3] The need for reliable studies which differentiate the different textile products and distinguish between the shedding of the fabrics from washing treatments and the extraction of loose or damaged fibres in new garments is emphasized.

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[1] These particles are termed in some studies erroneously as "microfibres"; a term which is used in the textile industry since few decades for thinner fibres including continuous filaments (typically < 1 dtex independent from the length, see Appendix 1.

[2] Some studies relying on spectroscopical analytical identification methods ignored to count fibres assumed to be biased from contaminations.

[3] The results from the "Mermaid" project sponsored by the EU Life program could not be covered in this review, as the project responsible person refused to provide the submitted but not officially released reports about simulated washings and in wash machines of woven and knitted fabrics with different auxiliaries (http://life-mermaids.eu).

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Examinations

Overview

Figure 1 summarizes the results from examinations of released fibres from the washing of PES textiles and garments as mass ratios: spreading of four orders of magnitude. The studies are grouped in the discussion below in reports without description of the analytical procedures, full papers with methodological details and simulated washing procedures.

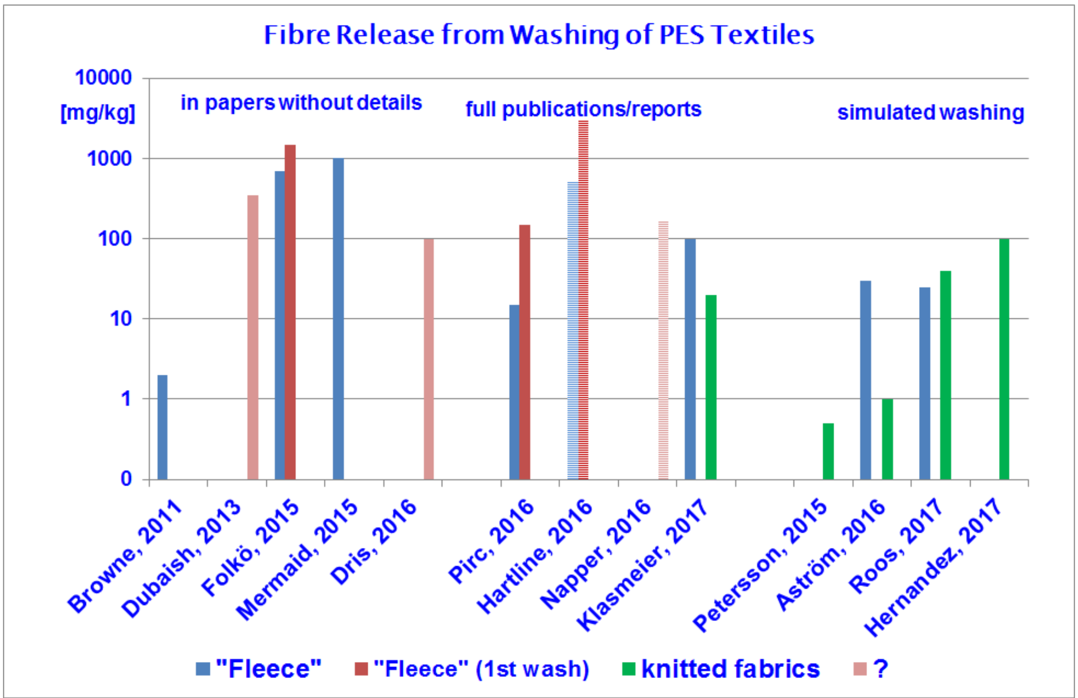

Fig. 1: Reported fibre releases from washing of PES textile products as mass ratios [mg per kg product]; in a logarithmic scale; explanations, see fig. 2 and 3, tables 1 – 3.

Our Expectations

The release of fibre fly from clothes and textiles during wear as well as from washing is common observation and well-known experience. The shedding of fibres is a helpful tool for forensic scientists interested in that subject already before (Watt et al., 2010).

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This fibre release is more prominent from articles made with natural fibres as cotton or wool, particularly in unfinished state. In an earlier forensic examination of outdoor fibre particulates only 5 % represented synthetic origin (Grieve and Biermann, 1997). A screening examination of the fibres on the fluff sieve of a household washing machine with polarized light microscopy and thermoanalytical method concluded a content of less than 5 % as synthetic fibres (PES, PP and PA), whereas most fibres found were cotton, besides natural animal fibres (wool, but also hairs) (BAM, 2017).

Man-made synthetic fibres are less susceptible to this loss and textiles from continuous (or "endless") filament yarns are used therefore for clean room clothing. However, some textile products are expected to be more prone to sheddability as plush fabrics with PES microfibres (i.e. ≤ 1 dtex, diameter: ≤ 10 µm for round filaments) also called polarfleece or simple "fleece", manufactured with cutting or ripping the loops of velour type fabrics. Textiles from staple fibre yarns, particularly those in low-pilling quality – even these might have only a very low market share – should also tend to shedding.

We assume furthermore that the extent of the shedding is influenced by the quality of fibres and particularly the spun yarns, but also on the construction of tested textile products. Even the polarfleece textiles might show differences: as these are manufactured with different yarns and technologies: with texturized or flat yarns in different fineness's for the loop (often with low tensile strength microfibres); manufactured by loop weft or warp knitting technique, in various heights of the loops and the loops might remain intact, cut or even ripped with a kind of a rake. All these different technologies might have some impact on the shedding behavior. Therefore results achieved from the testing of an individual textile product or garments are meaningless without this information about the textile construction.

The peculiar sensitivity of plush textiles and spun yarn fabrics was mentioned by the forensic scientists Lunstroot et al. (2016) in a study about fleece garments: "Other common polyester sources (sports clothes, lining) have either a lower sheddability or occur in combination with cotton fibres". In this publication results of the "tape test" (pressing a tape on the surface of the cloth) of nearly 200 various dark colored garments from PES polarfleece was reported with mark 4 for new and mark 3 of used garments in the scale of 0 – 5 for none to heavy fibre loss. The higher level of new garments is explained with remaining sheared fibres from manufacturing of the

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textile. Textured PES yarns were used in 2/3 of the tested garments for the loops with diameters of 10 µm to 17 µm (Lunstroot et al., 2016)[4].

These results suggest also the importance of a differentiated approach for the question of fibre release from textile products during use and washing: differencing analyses for the shedding from fabrics (as results from abrasions and mechanical stress) versus the release of already loose fibres (as left-over from manufacturing of the fabric or presumably more relevant from making the garment).
And between might be the shedding of fibres damaged during manufacturing.

Poorly described examinations

The first report about the release of synthetic fibres from washing of PES "fleece" garments appeared in 2011 (Browne et al., 2011). The main subject of this publication was the finding of fibrous particles in beach sediments, but the result of 1900 lost fibres per washed "fleece" garment received major attention, is often cited and used for extrapolations. The release of fibres from the washing of synthetic garments was a side aspect and only briefly described without details of tested materials or analytical procedure. A fibre content of 120, 160 and 300 fibres per liter wash water was reported for PES "blankets, shirts or fleece".

Further examinations found in publications or reports containing only minimal information are included in table 1. Some of these results are reported in mass ratios and other in number of lost fibres per garment, which are converted in the table 1 in mass ratios.

The extreme differing results are shown in the graph of figure 2 in a logarithmic scale.

[4] Circular 1 dtex PES microfibres have a diameter of 10 µm; the false twist texturing results in some flattening of the circular into a more oval cross section which might explain the mentioned range of the diameters up to 17 µm.

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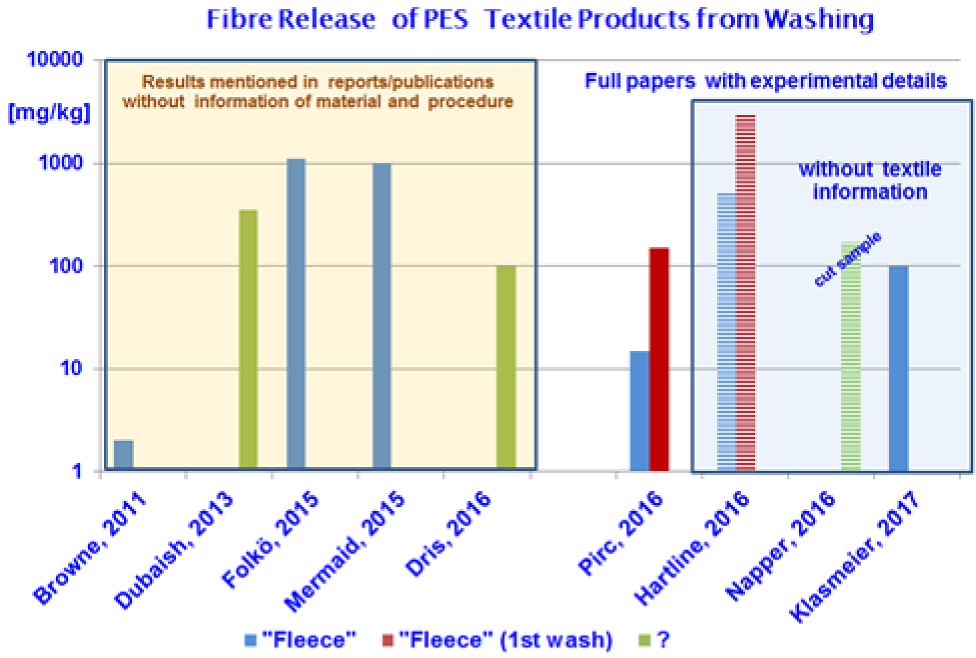

Fig. 2: Released fibres from washing of PES garments and textiles as mass ratio [mg/kg] in a logarithmic scale; blue bars: "fleece" garments, red bars: results from initial washings; green bars: not described products; dashed bars with questionable mass determinations.

Table 1: Studies without reported experimental details: Release of fibres from washing; conversion of fibre numbers with mass of 0.5 µg per fibre (for 2.5 mm long 2 dtex fibre)

Study	Tested product	Results reported in fibre [f] numbers	converted in mass ratio [g/kg]	Results reported in mass ratio [g/kg]	
Browne, 2011	PES fleece garment	1900 f/article or 300 f/L	0.002 (0.15 mg/ L)		a)
Dubaish, 2013	PES garment			0.3 – 0.4	b)
Folkö, 2015	new PES fleece shirt			0.7 – 1.5	c)
Mermaid, 2015	PES fleece jacket PAN scarf	1 000 000 f/article 300 000 f/article	1		
Dris, 2016	?	8 800 – 18 700 f/L	0.06 – 0.14		d)

a) Very fragmentary information about the method and tested articles; mass of garment not reported (assumed with 0.4 kg).
b) Even less information; casually: "Own experiments show that between 220 mg and 260 mg fibres were released from a single 660 g PES garment/washing".
c) Values taken from the IVL study (IVL, 2016); the release dropped with multiple washings.
d) Tested PES textile not described. Mass concentration is calculated with a liquor ratio of 15:1.

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Simulated washings

Some studies used a simulated washing procedure with laboratory color fastness testing instruments and smaller fabric samples. The mechanical treatment in the washing machines should be simulated by added metal balls. The results of these examinations are reported in two student's theses, a report and one publication of a Swedish respectively a Swiss research institute which differed also extremely.

The unexpected differences provoke a preliminary conclusion below about this test method.

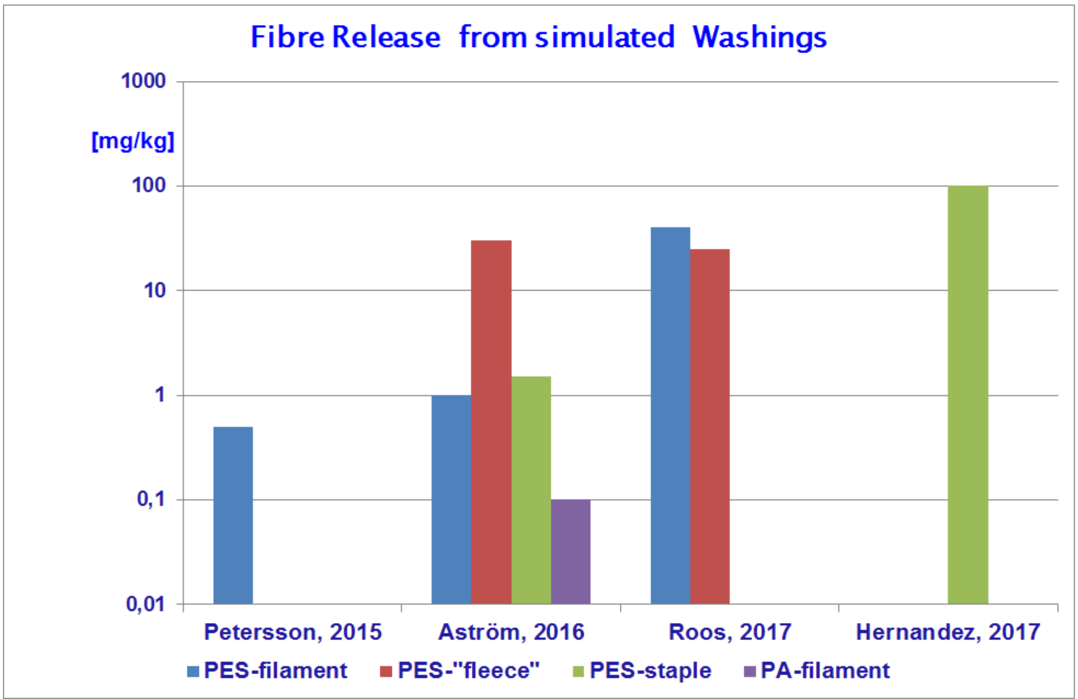

Fig. 3: Fibre releases from different fabrics expressed as mass ratio (calculation see table 2).

Swedish School for Textiles, Boras *(Peterson and Roslund, 2015; Aström, 2016)*

The Swedish School for Textiles in Boras compared different textiles in the colour fastness tester, the studies and results are reported in two theses of the University Boras and Göteborg University.

Textile samples of 100 cm² were cut and welded with laser and prewashed for 15 min in a washing machine. The treatment in the GyroWash tester lasted 30 min at 60 °C with liquid detergent in contact with 25 small metal balls. The sample is rinsed

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and squeezed by hand; wash water filtered and fibres inspected microscopically. These samples were washed only once in six-fold repetition per textile product.

The first study used PES knitting goods including frottage fabrics and compared fabrics from microfibre with those from fine (continuous) textile yarns: 0.7 dtex (diameter: ~ 8 µm) with 3 dtex (17 µm), presumably flat and not texturized.

12 fibres from the microfibre and 5 fibres from the other product were found as average of 18 samples in three variants each (corresponding to about 0.5 mg per kg fabric – calculated with an assumed square weight of 200 g/m² (Petersson and Roslund, 2015).

11 different textile products (institute-made laboratory fabric and commercial "fleece" textiles) were compared in the second study with single washing treatment in six-fold repetitions. From the commercial fleece sample 1000 fibres to 1200 fibres were counted, from the other fabrics 2 fibres to 17 fibres; the fabrics from PES staple fibre yarn was with an average of 15 fibres at the upper end. The variations between the six-fold testing of same fabrics were in both series low. The lengths of the counted fibres were reported in case of the fabric from PES staple fibres to 1 mm to 3 mm for 70 %, whereas of the fabrics from PES filament yarns to < 1 mm for approximately half of the fibres.

The results expressed in mass ratios: 50 mg/kg to 100 mg/kg for the fleece products and 0.2 mg/kg to 1 mg/kg for the institute fabrics (calculated with a mass of 0.2 µg per fibre and 2 g sample).

Samples of the institute fabrics were polished additionally with a sander which resulted in emissions up to 5 fold higher with larger variations of the individual results (Aström, 2016).

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Table 2: Release of fibres from laboratory washing (simulation with metal balls) of various textiles with counting of fibres.

Study	Tested Fabric	Results reported in fibre [f] numbers	converted in mass ratio [g/kg]	
Petersson, 2015	PES – knitted fabrics – 3 dtex - with 0,7 dtex filaments	5 f/ 100 cm² 12 f/100 cm²	0.000 5	α)
Aström, 2016	4 PES – knitted fabrics (filaments) PES (staple yarn) PA 3 commercial PES "fleece"	3 – 17 f/100 cm² 15 f/100 cm² 2 f/100 cm² 1 100 f/100 cm²	0.000 3 – 0.002 0.001 5 0.000 1 0.03	β)
Roos, 2017	PES Jersey (1.2 dtex) PES "microfleece" (0.6 dtex)	1000; 2000 f/150 cm² 1850; 2600 f/190 cm²	0.030/0.050 0.015/0.035	γ) δ)
Hernandez, 2017	2 PES knitted fabrics (~4 dtex staple) + detergent	not reported/300 cm²	0.025 0.1	ε)

α) 100 cm² samples, cut and welded with laser, prewashed in washing machine; assumption for the conversion in mass ratios: 1 mm long fibres and 2 g per sample,

β) 100 cm² samples, cut and welded with laser, prewashed in washing machine; assumption: 0.2 µg per fibre and 2 g per sample

γ) 150 cm² samples cut with ultrasonic; pre-cleaned; filtration with 5 µm / calculation with 1 mm length fibres (most of the counted fibres < 100 µm);

δ) 190 cm² samples (7.8 g and 4.4 g), cut with scissor; pre-cleaned, filtration on 100 µm filter; calculation with 1 mm fibre length

ϵ) 300 cm² samples with folded edges; prewashed; mass ratio reported (calculated from counted fibres).

SWEREA (Roos et al., 2017)

The Swedish Research Center Swerea IVF started a project for analyzing the shedding from PES fabrics sponsored by the Mistra Future Fashion Initiative.[5] The development of suitable methods was the first task of this project and some preliminary results achieved with commercial textile samples were reported already.

The textile samples were cut either with scissor or with ultrasonic and folded to bags with edges differently sealed. Vacuum cleaner or roller was used for pre-cleaning of the surface. The simulated washings with and without detergents were done in a

[5] See www.mistrafusionfashion.com

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GyroWash color fastness testing instrument with metal balls inside and outside of these bags. Released fibres were filtered from the wash water and inspected microscopically with an automatic analysis of fibre number and dimension. The tests were done in quadruplicate.

The preparation of the sample has a major impact: cut with scissor provided twice the fibre numbers than with ultrasonic. Two different brushed PES "microfleece" fabrics manufactured with 0.6 dtex filaments released 400 fibres per g fabric and 1000 fibres per g fabric (cut with scissor, vacuum cleaned, and treatment without detergent). Jersey knitting goods from 1.2 dtex PES continuous filaments reached nearly the same counted fibres (cut with ultrasonic, mildly brushed, vacuum cleaned, treatment with detergent, with a smaller filter mesh size).

The conversion to mass ratios gives 24 mg/kg and 60 mg/kg for the fleece fabrics (calculated with 0.06 µg/fibre for a fibre length of 1 mm[6] and the given square weight of the fabrics).

The authors did not comment the discrepancy of their findings to the results of the Textile School of Boras which is cited in the paper.

***EMPA** (Hernandez et al., 2017)*

Researchers of the Swiss Materials and Science Institute examined the release of fibres from two Jersey type knitting goods manufactured with 4 dtex PES staple fibre yarns in simulated washing treatments. 7 g samples cut from garments, with folded and sewn edges and pre-rinsed were treated in a laboratory washing apparatus in a 30:1 liquor ratio with steel balls. The washing liquor was filtered, fibres counted and their lengths determined initially with an imaging program (a lower detection limit of 40 µm was mentioned). Later, the fibre masses were extrapolated from the coverage of the filter with fibres. Up to five wash cycles were performed per sample with an air drying in between. Tests were performed as triplicates and loss of counted fibres is expressed as mass ratio.

The treatment with deionized water yielded in a release of 0.025 mg/g fabric without a considerably reduction from the more fold (up to five times) washing of the same

[6] S Roos personal information, June 30th, 2016: typical length of released fibres: 90 % < 100 µm.

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fabric, but with larger variations within the same treatment. The range of counted fibres was 20 fibres/g to 210 fibres/g sample.

The use of a detergent increased the number of counted fibres to a mean yield of 0.1 mg/g about the threefold and a range of about 50 fibres/g to 500 fibres/g. Most fibres were detected in the 2nd and 3rd washing (but also with larger variations between these triplicates) and a tendency could be assumed for declining numbers with iterative washings.

The average fibre size was shown as 0.5 mm (corresponding to 0.2 µg/fibres)[7]; ratio of fibre above 1 mm should be less than 2 %.

The results of further tests for understanding the source of these fibres are not conclusive: longer duration of this washing simulation treatment did not increase the number of released fibres. This finding was explained in the publication that their mechanical action during the yarn spinning would create shorter fibres remaining in the yarn which would be extracted during the simulated washing.[8]

Preliminary Conclusion

These simulated washings resulted not only in divergent results but also in unexpected comparative findings, as the negligible effect of washing duration and that the use of detergent released the threefold amounts of fibres in the EMPA study. The opposite is expected for shedding from any mechanical stress and abrasion. Equally not expected are similar amounts found for knitted fabrics from continuous fine filament yarns and the plush microfibre fabrics as in the SWEREA study.

These observations raise doubts about the suitability of this metal ball-test for the simulation of the washing machine (mechanical forces from drum and co-washing garments, a major effect is expected from the spin drying when the cloth will be pressed against the holes of the drum).

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[7] About 0.4 µg/fibre might be used in the publication (average of individual fibre numbers in table S3 and the reported mass ratio)?

[8] But this interpretation misses the probably more relevant source of these fibres: the preparation of the tested sample by cutting and not sufficiently protecting the edges.

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Therefor we believe these reviewed results need to be interpreted differently: the shedding of the fabric by any abrasion might be of lower relevance for some of these results, but left-over fibres from sample preparation might be responsible (the larger spreading between the triplicate samples in the EMPA examination might support that fear).

We consider following aspects relevant for the interpretation of these results and to be considered in further studies with simulated washing treatments (despite still some inconsistent results)[9]:

- The cutting of the fabric for preparing smaller samples generates shorter fibres at the edges which could be extruded from the yarn structure during washing.[10] The extent of this generation should depend also on the cutting method.
- We assume that hemming with sewing does not prevent the extraction of short fibres from the edges (compare the results of staple fibre fabrics from the EMPA study with those of the Swedish Textile School with laser welded edges).
- Released fibres might stick to the surface of the fabric depending on their structure. This might be reduced with detergents, explaining also the higher number of fibres found in the treatment with detergent.

The suitability of the laboratory color fastness tester for the evaluation of the shedding propensities needs to be confirmed. To overcome the possible bias we consider following measures necessary:

- The edges of the fabrics (from thermoplastic fibres) should be welded or heat sealed.
- The likely retention of shed fibres on the surface of the fabric need to be minimized by proper rinsing to exclude the findings of left over fibres.
- Iterative washings of the same sample are necessary for a better attribution to shedding respectively to leave over / damaged fibres.

[9] Different lengths of released fibres are not understood: Swedish Textile Institute: about 1mm; EMPA: average of 0.5 mm; SWEREA: < 0.1 mm.

[10] Not only for staple fibre yarns, but also for continuous textured and bulky yarns with filaments not vertical to the cut might be cut twice.

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- This test should be compared with the more simple forensic tape test (Watt et al., 2005) and with high and low shedding fabrics.

Examinations of Fibre Release in full Publications

Three studies about fibre release form washing were published – five years after the initial report – in September 2016 as full papers with more detailed information about the used procedures.

Unfortunately only the study by the Laboratory of Polymer Chemistry and Technology of the National Chemistry Institute in Ljubljana (Pirc et al., 2016) used a meaningful design and a sufficient precise weighing procedure for achieving reliable results. Both are missing in the publications from the Bren School in California (Bren School, 2016; Hartline et al., 2016) and from the Marine Biology and Research Centre in Plymouth (Napper and Thompson, 2016). The huge discrepancies between these three studies are shown in figures 2 and listed in table 3.

Particularly the described determinations of the masses of lost fibres are a major cause of concern and doubts in both studies.

Examination by Bren School (Bren, 2016; Hartline et al., 2016)

The fibre release from the washing of Patagonia's outdoor apparels was the subject of a student project from the master course of the Environmental Science and Management Program of Bren School, University of California, Santa Barbara. Five different garments should be washed and the shedding from two different washing machines should be compared (but instead new with already once washed jackets in the different machines were compared). The results are presented in the student's report (Bren School, 2016) and in a publication (Hartline et al., 2016) which differ in some values of the results.

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The most relevant shortcoming of this study is the weighing procedures: Use of moisture sensitive polyamide filters with an estimation of the net weight of self-cut filters by their area instead of pre weighing.[11]

Four commercial PES jackets and pullover and one PA jacket were washed individually without pretreatment first in a top-load thereafter in a front-load washing machine with synthetic program (but with a higher spin of up to 1200 rpm).

Proportions of washing water effluents were filtered with 333 µm and 20 µm polyamide filters. The filters dried in desiccators were weighed, net weights of these hand cut PA filters calculated from the area of the used filters (diameter: ~ 11.5 cm) with an image analysis program.

The publication reports an average fibre loss of 1 174 mg per garment; 1 906 mg respectively 220 mg were reported as medians for the findings with top-load (initial washing) respectively with consecutively used front-load machines. The fibres were described with a maximal size of 5 mm (report) or as "pills" (publication), most fibres were retained on the larger 333 µm filter.

Comment

The design of this study was already not well considered and has some shortcomings:

- for a comparison of different washing machines[12] comparable garments should be used and not juvenile and once washed garments
- the filtration of only aliquots (3.7 % respectively 14 %) and the use of two filters in different sizes multiplies the analytical weighing errors;
  - The non-precise and non-reliable weighing procedure is assessed to be the most severe problem of the examination[13].

[11] These problems were obviously the reason for some negative weighing differences and different results of lost fibres in the report and in the publication.

[12] The spin of 1200 rpm in the used washing program of one machine is higher than usually applied for synthetics (600 rpm); not mentioned for the other machine! The spinning is the most abrasive treatment during the washing.

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- Instead of any pre-weighing of the hand-cut filters, the net weight was estimated with the filter area determined with an imaging program.
- The moisture regain of polyamide (with a hysteresis) is another possible source of wrong determinations, as the drying of many app, 1 g filters in the desiccator might not achieve a constant level.
- All over 10 % of all mass "determinations" gave negative results (up to 39 mg!) which were set to zero for the statistical analyses.[14]
- Other positive results are also questionable (the number of fibres shown in figure S3 of the publication is lower than > 100 000 fibres (calculated from the minimum weight in table for the <u>aged</u> garment D and with a fibre mass of 0.5 µg/fibre).
- Different results are presented in the student's report and the later publication.

Due to these reasons the results are not reliable and without some more iterative washings absolute useless for any conclusion.

Patagonia, the sponsoring company of this study, started in 2017 a new study with the North Carolina State University.

Examination by Marine and Biology Centre Plymouth *(Napper and Thompson, 2016)*

The Marine Biology and Research Center of Plymouth University examined the fibre release by washing from three different fabrics described only with the textile label information. The effect of different washing conditions (with and without the use of detergents and conditioners) should be tested.

The description of the analytical procedures used, the only very few values presented in the whole publication (despite many performed analyses) do not allow a final statement of the precision, but leaves at least the perception that the analytic technique described in the methods could not achieve reliable results (presented values seems to be also not coherent and could not be recalculated).

[13] A simple error calculation would be more helpful for the students for analyzing critically their methods and their errors, instead of unduly statistical analyses.

[14] It is very honest to confess such analytical error and bias, but that observation should be the cause to repeat the study with better techniques and not to publish non reliable data!

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Fabric samples of 20 x 20 cm² cut from PES, PAN and PES/CO "jumpers" were hemmed and seamed with a thread. These samples were washed individually in a standard front-load washing machine (spin of 1400 rpm!) fivefold consecutively and with four replicates each. Variations of temperature, use of detergents or conditioner resulted in 12 different treatments (overall 720! individual analyses). The whole wash water was filtered on a 25 µm filter, residue transferred to a paper filter which was dried at "30 °C to constant weight". The fibre lengths was determined with an image program from the microscopically inspection of 10 fibres each.

This publication contains only few values: the dimensions of fibres and the masses of retained particles from one treatment with fivefold launderings:

- the dimensions of the released fibres: average lengths 5 mm to 8 mm (diameter: 12 µm (PES), 14 µm PAN, 18 µm PES/CO;
- the effect of more fold washing on the determined filtered masses is shown in a figure and described in the text (for one not disclosed treatment): decrease for PAN and PES fabrics (PES: 2.79 mg to 1.63 mg for 1st to the 5th laundering), stable for blended PES/CO yarn (~ 0.4 µg per sample).

The different treatments (temperature variations, use of detergent or conditioner) should have only negligible effects; PES/CO blend fabric gave lower releases than the pure synthetic fabrics in 9 from 12 treatments, according to a tabular comparison.

The doubts and critique to the design and the reliability performance of this study:

- smaller cut fabric samples (we assume < 10 g) with hemmed edges; shorter fibres from staple fibre yarns generated by the cut might probably be extracted with the iterative washings, as the hemming might not close the edges tightly (the average length of some millimeter is also indicative for extraction and less for shedding);
- the washing conditions for this single small sample consisted on an exaggerated spin of 1400 rpm (instead of typically approx. 600 rpm for synthetics);
- only 10 fibres each were analyzed for length and diameter;

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- the weighing procedure is not sufficient for these small masses – lowest reported mass: 0.30 mg (paper filters are also hygroscopic and exhibit a hysteresis behavior; the used filter should have a mass of ~ 150 mg).

The missing description and characterization of the fabric and textile construction makes this publication anyway useless.

Table 3: Examinations reported in full publications (with experimental details): Fibre release from washing; conversion of fibre numbers reported with mass of 0.5 µg per fibre (for 2.5 mm 2 dtex fibre)

Study	Tested product	Results reported in fibre [f] numbers	converted in mass ratio [g/kg]	Results reported in mass ratio [g/kg]	
Pirc, 2016	new PES "fleece" blankets double side 10mm plush 5 an old jacket. not described	(<25 000 f/kg)		1st washing: 0.1 – 0.2 >6th wash.: 0.012-0.015 0.012	e)
Hartline, 2016	PA jacket 4 new PES fleece jackets			3.8 (t-l) / 0.18 (f-l) 2.2–4.5 (t-l) / 0.1–0.8 (f-l)	f)
Napper, 2016	PES – "jumper" PES/CO –" jumper" PAN - "jumper "	83 000 f/kg 23 000 f/kg 121 000 f/kg	0.172 0.068 0.158		g)

e) Each fabric was tumble dried between iterative washings (three-fold masses of fibres separated in the dryer); the numbering result in bracket was calculated for a mass of > 0.53 µg/fibre.

f) Initial washing with top-load (t-l) and subsequent treatment with front-load (f-l) washing machines. Results from the publication differ from the values in the former study report; questionable weighing procedures (estimation of the net weight of the filter from the area of the filter manually cut).

g) 20 x20 cm² cut samples, hemmed and seamed with thread, individually washed;
– numbers of released fibres per 6 kg textiles are reported only; for the back calculation in mass ratios the reported conversion were used (for PES: 476 fibres/mg[15] as reciprocal fibre mass; for the given dimension of 7.79 mm 11.91 µm fibres; the correct reciprocal fibre mass would be: 909 fibres/mg. Similar error for the calculations of the other fibres.

Examination by Laboratory of Polymer Chemistry, Ljubljana *(Pirc et al., 2016)*

The Slovenian study was sponsored by the EU funded project DeFishGear and compared the release from more fold washings using a short, mild synthetic washing program. The used fabric was a complete, well described PES "fleece" blanket, which

[15] The printed "475,998 fibres/mg" is assessed as typing error.

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was washed ten times with tumble drying after each washing treatment. The whole washing water was filtered through (non-moisture sensitive) metal filters.

The structure of the 380 g/m² red blanket is described as double sided plush made from weft knitted ~ 2.5 dtex texturized PES yarns as ground textile and with textured 1 dtex PES yarns as 10 mm high loop fibres. The edges of the blanket are sealed and hemmed.

Fig. 4: Microscopic view on a sample of the tested blanket

The whole 320 g blanket was washed in a new front-load washing machine with a short and mild synthetic program (15 min, 600 rpm spin). The whole effluent was filtered over 200 µm stainless steel filters. The blanket was dried after each washing in a tumble dryer at 40 °C for 18 min equipped with inbuilt plastic filters of 180 µm openings. The procedure was repeated ten times for each blanket and in duplicate for each treatment. Further new untreated blankets were used for these treatments with detergent as well as with detergent and softener.

The microscopic inspection of the filters detected nearly only the 1 dtex microfibres with lengths in the range of 0.3 mm to 25 mm and in average of > 5.3 mm (most likely as underestimation as the longer entangled fibre would be difficult to separate). Nearly all fibres were retained on the 200 µm filters, as an additional fine filtration of the effluent resulted in a negligible amount of some and shorter fibres only.

The masses of released fibres decreased considerably from the 1st washing to the > 5th washing, from 80 mg/kg and 290 mg/kg (as individual values of the untreated blankets) to an average of 13 mg/kg, see figure 5. The use of detergent with or without softener did not affect the outcome.

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The tumble drying generated 3.5 fold more released fibres than the washing.

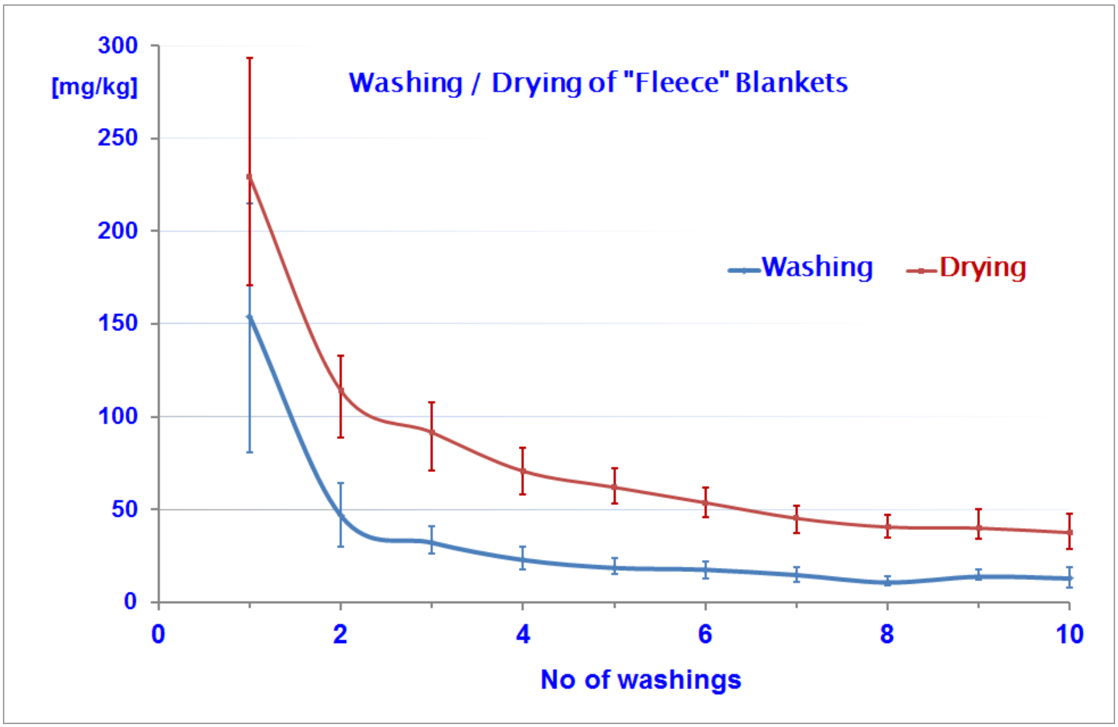

Fig. 5: Released fibres from washing and tumble drying as average of tested 6 blankets (values taken from the supplement of the publication by Pirc et al., 2016)

The release of 12 mg/kg corresponds to < 22 000 fibres per kg fabric, calculated with a length of > 5.3 mm of 1 dtex fibres (i.e. > 0.5 µg/f).

A five years old PES fleece jacket (not further described) was also washed and dried under these conditions resulting in the release of 12 mg/kg and 11 mg/kg from the washing respectively from the tumble drying as average of the 3 treatments.[16]

Comment

The design of this study is already convincing as it addresses a more fold washing (also under the assumption of left fibres from the manufacturing in new fleece garments), examines the more abrasive tumble drying (water might be considered as

[16] The filter sized of 200 µm might not be sufficient for retaining fibres shed from this "fleece" jacket which misses a description of its textile construction and possibly shorter fibres.

lubricant) and operates with a short but otherwise usual washing conditions for synthetics.

The weighing procedure is precise enough for masses of released fibres of > 3 mg and by using filter materials which is not sensitive to ambient humidity. The consistent masses with low variations between the various blankets from more often washings (i.e. the iterative > 5th washing; with or without detergent) can be assigned as proof for the performance of the analytical work of the students.

Regarding the relevance of these results to an evaluation of the shedding of fibres from washing of "fleece" synthetics:

- the used new washing machine of a supreme manufacturer might be less abrasive; and the blanket was washed alone without other products;
- the washing conditions were short and mild, but not un-typical for synthetics; and confirm with the gentle conditions of ISO 6330 (ISO, 6330);
- the fleece" blanket - plush on both sides, with texturized 1 dtex microfibres and 10 mm high loop - is particular sensitive to the shedding;
- the high floor of the "fleece" blanket is responsible for larger lengths of fibres, allows also the use of larger filter (200 µm), which might be not sufficient for examinations of other textile products;
- the release from this iterative washing could be regarded as shedding from washing (or from transferred left-over respectively damaged fibres from tumble drying).

These most reliable results might be considered as the typical loss by shedding of a most sensitive fleece garment under mild washing conditions (program and machine).

Conclusions and Outlook

The Slovenian study by Pirc et al. (2016) is considered to present up to now the only reliable study for the examination of shed fibres during washing: from the design (more fold testing the same material), analytical performance (less critical weighing procedure), product description and repetitions (6 fold including variations of

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detergent and softener which should also have no major effect on the shedding). The tested material is also sufficiently described and represents also a more sensitive polarfleece product (texturized microfibres in a double sided plush fleece construction with a high floor). It has to be taken into account that these results were achieved under gentle – but not un-typical washing conditions for synthetics – in a new washing machine without co-washing of other textiles.

The loss of < 20 mg/kg (or < 40 fibres/g) from iterative washings might be representative for the shedding of this fabric quality.

But despite this acknowledged performance of this examination, questions remain still open:

- are these lost fibres (from iterative washings) those which were shed during washing treatment or left over from the more abrasive tumble drying before each iterative washings?
- originate the losses found in the initial washings from weakened / damaged fibres of the polarfleece fabric or from loose fibres from the edges of the blanket sourced from insufficient seam?

The other studies reviewed are useless regarding the questions for the quality of fabrics and for an understanding of shedding or release of loose fibres due to missing descriptions of tested textile products, partly without iterative washings, questionable analytical methods. Furthermore we doubt additionally the suitability of simulated washing examinations.

We consider two synthetic textile products to be sensitive to shedding as these contain fibres which have reduced tenacity either conditionally upon the processing or intentionally and their laundry might contribute to the occurrence of synthetic fibres in the sewage water:

- "polarfleece" products, particularly with textured microfibres,
- fabrics made from staple fibres, particularly those in a low-pilling quality

This presumption needs to be confirmed with professional examinations by textile institutes with analyses of the shedding from washing and wear (in comparison with

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suitable abrasion tests) which should be well differentiated from analyses of the release of loose fibres.

Any result of numbers of mass ratio of lost fibres from washing or wearing of individual textile products is meaningless for any estimations of the amount of fibres released into the environment, as too many factors have an impact. The individual result is meaningful for the evaluation of the quality of the regarding textile product.

The forensic tape test (De Wael et al., 2010) might be more suitable for the purpose of quality testing and for an evaluating of the shedding of textile fabrics (after washing) and the release of damaged and weakened fibres in the fabrics (test before washing).

Further and other tests needs to be developed for the question of the release of fibres generated during the making of the garments.

Proposals for Research

- The release of remaining fibres from the manufacturing of the garments and the shedding of the textile fabric by any mechanical treatments should be differentiated more clearly in all future examinations.
- The shedding behavior of synthetic fabrics should be regarded as quality criteria, for which simple tests needs to be implemented. The forensic tape test might be suitable for that purpose, should be compared with laundering in simulated laboratory equipment and in washing machines.

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Appendix: Terms and Definitions

Fibre Terms

General Distinction Of Textile Fibres (BISFA; 2009)

fibre (textile):
: morphological term for materials characterized by their flexibility, fineness, and high ratio of length to cross-sectional area;

man-made fibres:
: covers synthetic and cellulosic fibres (as viscose, lyocell or cellulose acetate)
differentiation in (endless) filament, staple fibre and flock

synthetic fibres:
: manufactured from synthetic polymers, as the most relevant classes: polyester (PES; most important PET); polyamide (PA), polyacrylnitrile (PAN), polypropylene (PP)

cellulosics:
: includes man-made (as viscose, modal, lyocell or acetate) and natural fibres (as cotton, flax or jute)

natural fibres:
: covers vegetable (as cellulosic cotton, flax, …), animal (as wool or silk) and also inorganic fibres (as asbestos)

Table 4: Coding system of synthetic man-made fibres (selection)

Code	Generic name	Polymers (at least 85 %)	Density [g/cm³]
EL	elastane	polyurethane (alternate elastic and rigid elements), stretchable	~ 1.2
PA	polyamide	polymer with amide linkages; as PA6 ("Perlon") or PA66 (Nylon)	1.14
PAN	acrylic	polyacrylnitrile and acrylic copolymers	1.18
PES	polyester	polyester of a diol and terephtalic acid as poly(ethylene terephthalate) or poly(butylene terephthalate)	1.38
PLA	polylactide	linear macromolecule of lactic acid esters (from natural sugars)	1.26
PP	polypropylene	polypropylene	0.91

Fibre types

Filament (or endless):
: a fibre of very great length considered as continuous;

staple fibre:
: textile fibre of limited but spinnable length
(typical for spun yarns – cotton type: 30 mm – 50 mm; wool type: > 50 mm)

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flock:

very short fibres intentionally produced for other purposes than spinning (typical: 1 mm)

Fibre fineness (Falkai, 1981)

Linear density (or "titre")

mass per unit length of an essential linear structure, as staple fibres, filament yarn or tow

tex-system

linear density for man-made fibres (replaced the former used denier system)
tex = g per 1 000 m length or dtex = g per 10 000 m

denier-system

den = g per 9 000 m (initially introduced for silk yarns)
replaced by the metric tex system, but is still in use in USA.

Microfibres

term introduced in the 90ties for fibres with linear densities of < 1 dtex or diameter < 10 µm for fine and dense fabrics or functional clothes (as polar fleece textiles) example: 1 dtex corresponds to a diameter of 9.7 µm for round PES fibres

fine fibres:

1 dtex – 5 dtex: for apparels – similar fineness as natural fibres as cotton: 2 dtex – 3 dtex)

coarse fibres:

> 10 dtex: for interior applications (as carpets) or technical applications (as safety belts); monofilaments for filtration

Categories for marine environment

microplastic:

solid particles of synthetic polymers, insoluble in water and persistent[17] (not (bio)degradable)

sizes of microplastics:

- commonly used:
  - < 5 mm (including nano range) (GESAMP, 2016),
  - but lower limit is "defined" often by the analytical method applied
- in discussion by an ISO working group of TC 61 ("Plastics"):
  - "microplastic" 1 µm – 1 000 µm
  - "large microplastic": 1 mm – 5 mm

forms of microplastic

usually differentiation in: fragments, beads or granules, pellets, films, fibres

primary microparticle:

particles originally produced to that size

secondary microparticle:

resulting from degradation, fragmentation, weathering of larger plastic items

[17] As persistence criteria often the criteria from REACH (Annex XIII) for substances are considered. But these developed for water soluble substances should not be applied for solid structures as the microplastic. As even natural biodegradable material will fail these tests criteria.

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particles

small, solid material, covering different forms: as films, beads, granules fragments or fibres;

microparticle:

0.1 µm – 100 µm (defined by IUPAC, 2012)

nanoparticle:

1 nm – 100 (500) nm (including fibres with this diameter)

fibrous particles from synthetic fibres (or fibrous synthetic particles)

the preferred term of short synthetic fibres in the current definition of "microplastic": < 5 mm

<u>Dust (airborne)</u>

particulates:

airborne particles (below the visibility to the naked eye)

fibre fly:

airborne fibres (or parts) visible to the human eye,
diameter corresponds to diameter of the original individual fibres or filamentsp.,

fibre lint:

short fibre fly at the threshold of visibility

fibril

subdivision of a fibre

particulates from fibres:

fibrous (i.e. with a minimum aspect ratio of 3:1) or spherical dust consisting of fibre material

respirable fibre-shaped particulates (RFP):

Airborne particulates fulfilling the following dimensional conditions: length (L): > 5 µm and diameter (D): < 3 µm and length / diameter (L/D) ratio of > 3:1

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