

Microfiber Masses Recovered from Conventional Machine Washing of New or Aged Garments

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Supporting Information

ABSTRACT: Synthetic textiles can shed numerous microfibers during conventional washing, but evaluating environmental consequences as well as source-control strategies requires understanding mass releases. Polyester apparel accounts for a large proportion of the polyester market, and synthetic jackets represent the broadest range in apparel construction, allowing for potential changes in manufacturing as a mitigation measure to reduce microfiber release during laundering. Here, detergent-free washing experiments were conducted and replicated in both front- and top-load conventional home machines for five new and mechanically aged jackets or sweaters: four from one name-brand clothing manufacturer (three majority polyester fleece, and one nylon shell with nonwoven polyester insulation) and one off-brand (100% polyester fleece). Wash water was filtered to recover two size fractions ($>333 \mu\text{m}$ and between 20 and $333 \mu\text{m}$); filters were then imaged, and microfiber masses were calculated. Across all treatments, the recovered microfiber mass per garment ranged from approximately 0 to 2 g, or exceeding 0.3% of the unwashed garment mass. Microfiber masses from top-load machines were approximately 7 times those from front-load machines; garments mechanically aged via a 24 h continuous wash had increased mass release under the same wash protocol as new garments. When published wastewater treatment plant influent characterization and microfiber removal studies are considered, washing synthetic jackets or sweaters as per this study would account for most microfibers entering the environment.



INTRODUCTION

Microplastics (plastics sized at $<5 \text{ mm}$) are environmental pollutants¹ in freshwater,^{2,3} marine,⁴ and terrestrial⁵ environments and can directly impact local organisms^{6–8} by enabling their uptake of toxic chemicals via consumption.^{9,10} To date, microplastic origins are incompletely understood, but they include primary and secondary sources, with the latter including breakdown of products upstream of, or within, the environment.³ In situ environmental fragmentation¹¹ is a difficult-to-control microplastic source because it stems from larger plastic debris continuously entering the environment.³ However, the release of microplastics upstream conceivably could be managed if sources are identified and controlled.

Plastic microbeads entering wastewater treatment plants (WWTPs) via sewage are well-recognized pollutants and are considered microplastic pollution.¹² Microplastics also include synthetic microfibers,^{13,14} i.e., submillimeter-sized polyester, acrylic, and nylon fibers which, similarly to microbeads, are found in various environmental compartments including marine sediments.^{14,15} Furthermore, microfibers may be entering the human food supply via environmental reservoirs^{16,17} and may negatively impact marine⁷ or terrestrial organisms.¹⁸

In addition to entering marine environments, microfibers can be introduced to terrestrial soils⁵ via land-applied WWTP biosolids.^{19,20} This implies that WWTPs can be microfiber

pathways into the environment; a corollary is that untreated sewage, septic tanks, or graywater are also environmental pathways. Regardless, WWTPs receive microfibers from upstream sources: thousands of microfibers are released into wash water during conventional machine washing of common garments,²¹ and washing machine effluent either discharges to soils directly as graywater or out of septic tanks,²² to surface waters as untreated wastewater, or to sewers destined for WWTPs.

Synthetic fibers are produced in quantities that vastly exceed even the most highly produced manufactured nanomaterial, i.e., carbon black,²³ with polyester fibers alone produced at over 40 million tons per year.²⁴ Within polyester production, the largest application of this material is clothing, accounting for 54% of the market use distribution in 2011.²⁵ Yet microfiber shedding during the conventional washing of synthetic fiber clothes is inadequately characterized,²⁶ including mass concentrations that could be accounted for and potentially controlled. Jackets provide a particularly prime target to study because they represent the broadest range in construction, allowing for

Received: June 17, 2016

Revised: September 26, 2016

Accepted: September 30, 2016

Published: September 30, 2016

potential changes in manufacturing as a microfiber pollution mitigation measure.

In this study, commercially available polyester jackets or sweaters, including name- and off-brand models, were washed as either new or mechanically aged garments, and the released fibers of two size classes ($>333\ \mu\text{m}$ or between 20 and $333\ \mu\text{m}$) were captured and massed. Recovered microfiber masses were compared for top- versus front-load washing machines, for each filter size, by garment brand, and by garment age. The recovered microfiber masses were contextualized relative to published studies of microfiber loading to, and removal from, WWTPs. This allowed for analyzing the importance of such garment washing as a microfiber source to the environment and for consideration of the implications.

MATERIALS AND METHODS

Garments. Five widely commercially available jacket types were selected to test a range of garment constructions (Table S1). Of particular interest were the two synthetic fleeces, jackets D and E, because these allowed a comparison of name-brand (D) to off-brand (E) jacket microfiber shedding. Individual garments were either provided by the manufacturer (A, B, C, and D; Table S1) or purchased (E; Table S1). All garments were men's "medium" size outerwear and were numbered individually such that microfiber masses recovered after the first wash trial (for new garments) and after the second wash trial (for aged garments) could be compared.

New Garment Washing. Two types of wash trials were conducted. The first type was for new garments in either top- or front-load washing machines. The second type was for jackets that, having been washed once as "new" garments, were then mechanically aged (protocol below) before washing under conditions similar to the first trial. Wash trials were conducted with a top-load (Whirlpool model WET3300XQ) or front-load (Samsung model WF42H5000AW/A2) residential-type washing machine in Ventura, California. Tap water from the Ventura Water domestic water supply was used in all wash experiments (Table S2).

For the first wash trials, jackets were tested directly after removal from their original packaging to minimize possible environmental contamination. Protective (against abrasion and contamination) packaging of jacket types A–D takes place immediately after manufacturing, reducing the possibility of attached material or generation of fibers during transport. However, the packaging procedure for jacket type E was unknown; therefore, jacket type E was gently shaken to remove loose fibers prior to the wash trials. Quadruplicates ($n = 4$) of new jackets of each type were washed, without detergent, one at a time in the top-load machine (43 L capacity; settings: extra small capacity, $29.6\ ^\circ\text{C}$ warm cycle, 30 min; 12 min wash, 14 min rinse, and 4 min spin). For each trial, 136 L of output water was collected in a clean 227 L polyethylene rain barrel. The fluid was then stirred 20 times clockwise with an ABS 2 in. pipe before using two partial scoops of a 4 L glass beaker to pour 5 L of collected water through a filtration column with two inline hand-cut Nitex nylon filters, $333\ \mu\text{m}$ followed by $20\ \mu\text{m}$ (Figure S1). Following each filtration event, the filters were removed to individual polystyrene Petri dishes (138.9 mm diameter, triple vent, 21.2 mm height, aseptic) for later massing. To avoid carryover mass between garments (including of the same garment type), the filtration column and polyethylene barrel were rinsed with distilled water, followed by conducting an empty 10 min wash cycle (settings: hot, extra small capacity,

regular cycle). To minimize contamination, a blue cotton lab coat and nitrile gloves were worn during the wash experiments, and samples were exposed to the ambient environment only during the brief transfer from the filter column to the Petri dishes. The order of garment washing was recorded to allow for evaluating the effect of washing order and to test for possible carryover despite cleaning procedures between wash trials.

The above protocol was repeated using a front-load machine, except with triplicate ($n = 3$) garments and with front-load settings: extra small capacity (45 L), $29\text{--}41\ ^\circ\text{C}$ warm cycle, 24 min; 8 min wash, 10 min rinse, and 6 min spin (1200 rpm). For each trial, 36 L of output water was collected in a clean 45 L polyethylene storage container, which was stirred 20 times clockwise and then sampled, filtered, and stored in the same manner as for the top-load wash procedure.

Mechanically Aged Garment Washing. Following the first wash trials, the garments were mechanically aged with their triplicate or quadruplicate treatment group through a process developed by the garment manufacturer (of jacket types A–D) that is used to test the effects of wear on their textile products. This aging process was a previously developed protocol in accordance with best practices for textile testing while laundering²⁷ and involved a wash cycle without detergent in a top-loading, commercial, heavy-duty washer (Speed Queen model AWN432SP113TW04), through a 24 h no spin agitation wash cycle with settings for cold wash temperature ($20\ ^\circ\text{C}$), large capacity (61 L), and regular cycle. Each jacket was then fully dried in a home-style dryer (Whirlpool model WET3300XQ) and then thoroughly dusted in a clean environment, including pocket interiors, to remove any remaining loose fibers.

Following the aging procedure, each aged then dried garment was washed in a second trial following the same procedures as the new garments, including filtration, filter recovery, and apparatus cleaning for the same machine type as before aging (front- or top-load).

Filter blanks ($n = 4$), prepared for each filter size (333 and $20\ \mu\text{m}$ filter) and for each washing machine type, were collected at the end of the experiment as controls to account for mass that could have been contributed by the water source. The blank collection and processing was conducted identically to the wash trials, except that blanks were retrieved from inline filters following a complete machine and filter column wash procedure to eliminate carryover from prior washes.

Filter Massing. Filters with recovered microfibers were moist and thus required desiccant drying prior to massing. The filters, maintained in closed but not gastight Petri dishes, were placed on a metal rack above the desiccant (Damp-Rid Moisture Absorbers; calcium chloride, sodium chloride, and potassium chloride) within a sealed 62 L polyethylene box. The samples were dried for 3 days, during which time the individual Petri dish plus filter masses ceased declining. Each dried specimen (filter with microfiber) was then photographed using a tripod-mounted digital SLR camera (Nikon D3200). Using clean forceps and only edge handling, each specimen was folded twice and transferred into a premassed and -labeled Celltreat polypropylene 50 mL centrifuge tube, which was then capped and massed in a draft-free Mettler Toledo AB104-S balance. Transfer to premassed tubes was necessary because of the wide variations in Petri dish masses. Personnel wore blue or white cotton lab coats over nonsynthetic clothing, and exposure time was limited to minimize contamination.

Table 1. Average Fiber Mass (Milligrams, with Standard Deviation in Parentheses) and Median Fiber Mass (with Range in Square Brackets) Recovered Per Wash on Either 333 or 20 μm Filters, Across Measured Variables for the Five Tested Jacket Types^a

filter size		20 μm				333 μm			
machine type		top-load ($n = 4$)		front-load ($n = 3$)		top-load ($n = 4$)		front-load ($n = 3$)	
age		new	aged	new	aged	new	aged	new	aged
jacket A	mean	333	407	69	383	1141	1264	0	296
	st. dev.	(100)	(172)	(20)	(604)	(353)	(660)	(0)	(116)
	median	319	397	74	71	1237	1248	-62	252
	range	[243; 449]	[249; 584]	[46; 86]	[-82; 1080]	[640; 1450]	[581; 1981]	[-113; -29]	[208; 427]
jacket B	mean	526	381	25	98	1475	988	58	136
	st. dev.	(129)	(175)	(22)	(7)	(521)	(523)	(51)	(21)
	median	538	337	35	95	1596	887	80	126
	range	[378; 652]	[223; 628]	[-136; 39] ^b	[94; 106]	[740; 1969]	[489; 1691]	[-128; 95]	[123; 161]
jacket C	mean	419	414	29	92	1332	1227	0	161
	st. dev.	(73)	(124)	(26)	(20)	(308)	(489)	(0)	(8)
	median	386	370	37	102	1425	1173	-90	163
	range	[376; 529]	[322; 596]	[-19; 51]	[68; 105]	[885; 1593]	[687; 1876]	[-91; -88]	[152; 168]
jacket D	mean	449	420	122	92	1119	2018	0	139
	st. dev.	(100)	(92)	(120)	(22)	(406)	(574)	(0)	(39)
	median	440	429	121	98	1103	2077	-88	154
	range	[337; 580]	[300; 523]	[3; 242]	[68; 112]	[690; 1580]	[1276; 2643]	[-283; -54]	[95; 169]
jacket E	mean	687	1773	199	111	1434	853	232	277
	st. dev.	(42)	(228)	(34)	(17)	(348)	(492)	(232)	(34)
	median	691	1703	189	105	1481	932	110	265
	range	[636; 730]	[1582; 2102]	[171; 236]	[98; 130]	[1014; 1757]	[182; 1364]	[87; 500]	[250; 315]

^aThe number of independent replicates of each jacket within each wash mode (top- or front-load) was $n = 4$ for top-load and $n = 3$ for front-load.

^bNegative values were converted to 0 prior to calculating the mean and for use in making comparisons between means.

Filter Image Analysis. Because hand-cutting filters was required for the custom inline filtration column, filter areas varied slightly. This resulted in slight variations in the pristine filter masses. Thus, it was necessary to image filter areas so that pristine filter masses could be calculated which in turn allowed for calculating the recovered microfiber masses from the difference of dried specimen (filter plus microfibers) less the pristine filter.

Photographs of dried individual specimens (filters with recovered microfibers) were analyzed for overall area calculations using ImageJ (Image Processing and Analysis in Java version 1.49), calibrating measurements to the ruler captured within each photo (Figure S2). Using area measurements and masses for the filter blanks, a reference mass-to-area ratio was calculated for each filter size (333 or 20 μm); these reference ratios were averaged across washing machine types. The average reference ratio for each filter size was used to calculate the approximate mass of each pristine filter, which was then subtracted from the mass measurement of the dry specimen (filter plus recovered microfibers) to obtain the mass of fibers on each filter. The microfiber mass was then scaled up by the ratio of total liters in the wash cycle to liters filtered to obtain the total mass of fibers that would have been recovered per each filter had all of the wash water been filtered.

Data Analyses and Statistics. To determine if microfiber masses for individual garments were biased by wash order, which would indicate that the cleaning procedure between washing trials was inadequate, the microfiber mass was regressed against trial number (i.e., 1 for first wash and 2 for the wash trial postaging) and all other variables (i.e., front- or top-load and garment type), and a t test ($\alpha = 0.05$) was performed.

Variability in individual area measurements was also examined to determine its impact on fiber masses. Ten randomly chosen samples were thus selected for remeasuring the filter areas, which were then compared to their initial values. This allowed for assessing bias in the image analysis method used to determine filter areas.

Nonparametric tests ($\alpha = 0.05$) were used as robust comparisons of central tendency (mean or median) because of small sample sizes and non-normal distributions for measured variables (washing machine type, aging, jacket type, and filter size). Wilcoxon signed-rank tests were used to determine if median microfiber masses recovered differed between filter sizes, and sign tests were used to determine if the median difference in microfiber masses after aging were significantly greater than zero. Wilcoxon rank-sum tests were conducted to determine significant differences in jacket type and washing machine type medians. For interactions between variable groups, a multiway analysis of variance (ANOVA) ($\alpha = 0.05$) was used. Interpretation of significance of variable interaction was limited by non-normality (strong positive skew), unequal variances, and small sample sizes. Data were analyzed using R 3.2.3 (<http://www.r-project.org/>) and Excel 2013.

Assessment of Environmental Implications. To estimate the relative contributions of synthetic microfibers released during conventional washing to overall microfibers received at wastewater treatment plants, a model was developed to interpret the data collected herein (fiber mass released per wash) relative to a recent study¹² regarding WWTP influent concentrations of synthetic microfibers. Although other studies have quantified the presence of microfibers in WWTP influent,^{28,29} the study by Murphy et al.¹² verifies the fibers as synthetic using Fourier transform infrared spectroscopy

(FTIR) analysis. A notable difference in the fiber removal method of the latter WWTP study and the wash experiments herein was the use of different filter mesh sizes (20 μm for the wash experiments herein and 65 μm for the WWTP study¹²). This would result in an overestimation of the relative contribution of synthetic jacket washing to influent microfibers at WWTPs.

The model assumed a population of 100 000 individuals (N), each with a single synthetic jacket with average shedding mass (m , fiber mass per wash), and a range of possible washing frequencies (f , wash per day), from once per month (every 30 days) to twice per year (every 180 days), to estimate the fiber masses released in laundry effluent each day by the number of jackets (eq 1).

$$\text{Fiber mass per day for } N \text{ jackets} = \frac{N \cdot m}{f} \quad (1)$$

The mass of fibers released to the WWTP per day from the population (N) of 100 000 individuals was estimated using an average per capita daily sewage influent flow rate (S) of 0.35 $\text{m}^3 \text{person}^{-1} \text{day}^{-1}$, calculated from 13 European WWTPs.³⁰ A typical decitex or linear density (D) of 0.03 mg mm^{-1} for polyester³¹ and an assumed average fiber length (L) of 0.300 mm fiber^{-1} was used to convert the WWTP influent fiber count concentrations (C , number per m^{-3}) from units of fibers per cubic meter into mass-based concentrations. The fiber length assumption of 0.3 mm per fiber was based on the top end of mesh sizes used to filter WWTP influent.²⁹ From these data and assumptions, the fiber mass entering the WWTP per day for a population of 100 000 was estimated (eq 2).

$$\text{Fiber mass per day for } N \text{ population} = N \cdot S \cdot C \cdot D \cdot L \quad (2)$$

The output of eq 1 was then compared to that of eq 2 to calculate the percentage of fibers in WWTP influent that could originate from the laundering of synthetic jackets based on the range of possible average washing frequencies from once per month to twice per year.

RESULTS AND DISCUSSION

Overall Microfiber Recovery. Across all treatments, within all replicate groups, there was an average coefficient of variance in recovered fiber masses of 0.40%. However, the recovered microfiber masses across the treatment types varied widely, with an average of 1174 mg ($n = 70$, 95% CI [−651, 3000]) of total fibers (across the 20 and 333 μm filters) recovered with each wash (Table 1), accounting for an average of approximately 0.2% of the unwashed garment mass (Table S3). By comparison, a study by Van Amber et al.³² found that mass loss (as a percentage of silk or silk-blend knit garment mass) was up to 1% after six wash cycles (0 to 0.29% for dyed fabrics). Compared to our findings, after a single front-load wash cycle, new garments had a combined (20 μm + 333 μm) mass loss of 0 to 0.078% (Table S3).

A regression of top-load fiber mass with filter size, age, washing machine load, jacket type, and trial number included as predictor variables explained a significant proportion of the variance in fiber mass ($R^2 = 0.55$, $F(8,131) = 22.21$, $p < 0.001$), but trial number was not a significant or positive predictor ($t(72) = -0.20$, $p = 0.84$) (Table S4). These results strongly suggest that there was no positive relationship between trial number and fiber mass, indicating that significant fiber

carryover between wash trials was unlikely and thus that the cleaning procedure between wash trials was effective.

The variability in area measurements was tested to understand its effect on the calculated fiber masses. From the ten randomly selected samples with remeasurements, area variations were low, all within $\pm 1\%$ of the original measurement. This amounted to a variation of about 10% in fiber masses. Of the 140 samples, 13 had negative calculated fiber mass after subtracting the estimated filter mass. The observed variation in area measures possibly contributed to this result. However, visual inspection of the filters found that these samples had few to no apparent fibers on them. Thus, the values were retained for rank-based statistical tests but set to zero for mean-based analyses.

Microfiber Recovery Variation by Filter Size. Across all wash trials and both washing machines (front- and top-load), median fiber masses recovered per garment for 20 μm (median = 302 mg, $n = 70$) and 333 μm (689 mg, $n = 70$) filter sizes were compared using a Wilcoxon signed-rank test. Median fiber mass was found to be significantly greater on the 333 μm versus the 20 μm filters for paired filters (Figure 1; $Z = 4.57$, $p <$

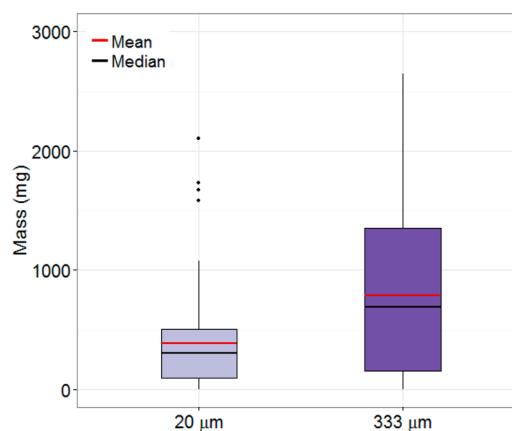


Figure 1. Distribution of fiber mass recovered, across all jacket types and all treatments, on the 20 μm ($n = 70$) and 333 μm ($n = 70$) mesh filters. Red lines indicate means, black lines medians, and black dots outliers (beyond 1.5 times the interquartile range).

0.001). Visual inspection of the filters identified the presence of pills on the 333 μm filter along with smaller fibers, which could partially explain the higher mass observed on the 333 μm filters (Figure S3).

Median fiber masses recovered per garment in top-load washing machines for 20 μm (median = 442 mg, $n = 40$) and 333 μm (median = 1294 mg, $n = 40$) filter mesh sizes were compared (Figure 2). A Wilcoxon signed-rank test found significantly greater median fiber mass on 333 μm filters than on 20 μm filters for paired filters ($Z = 4.13$, $p < 0.001$). For front-load washing machines, a significant difference between 20 μm (median = 94 mg, $n = 30$) and 333 μm (median = 116 mg, $n = 30$) median microfiber mass recovered was not observed ($Z = 1.30$, $p = 0.90$). To further test an effect of washing machine mode on fiber mass recovered, a multiway ANOVA was performed on the interaction of washing machine and fiber mass, with garment age and type included as covariates. A significant interaction was found between machine type and fiber size ($F(1) = 30.68$, $p < 0.001$). Fewer pills were observed on the top-load 333 μm filter samples despite higher recovered fiber mass, suggesting that pilling may have a small

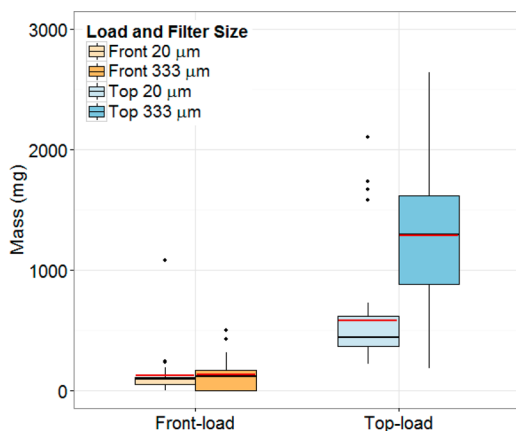


Figure 2. Distribution of fiber mass recovered on the 20 and 333 μm mesh filters, across all jacket types and aging categories for front-load ($n = 30$ each box) versus top-load ($n = 40$ each box) washing machine modes. Red lines indicate means, black lines medians, and black dots outliers (beyond 1.5 times the interquartile range).

impact on the mass of recovered fibers. The central agitator of the top-load washing machine, which moves clothes vigorously through the water, may have influenced the difference in recovered masses, potentially causing a higher proportion of larger fibers to be released. Further testing top-load machines with versus without a central agitator could clarify this relationship.

Ageing Effects on Microfiber Recovery. Median differences between new and aged treatments were tested to determine if total recovered microfiber mass (across the 20 and 333 μm filters) increased after garment aging. A sign test of mass before and after aging was conducted, showing that the mass of recovered fibers increased significantly after aging ($p < 0.001$). On average, aging resulted in 25% more fibers recovered. Visual inspection of the jackets indicated that there was fraying on the aged jackets, which could lead to the increased mass of recovered fibers. Another possible variable influencing the shedding of mechanically aged garments is the process of drying the garments after aging. However, it is yet unknown how drying could have affected the shedding characteristics of the jackets.

The fiber masses recovered per wash on the 20 μm filter for new jackets ($n = 35$) and aged jackets ($n = 35$) were compared (Figure 3). A sign test found no significant increase in mass after aging ($p = 0.250$). For the 333 μm filter, however, there was a significant increase in recovered fiber mass after aging ($p = 0.0205$). This may indicate that aging has a greater effect on larger fiber release, i.e., those retained on 333 μm filters. However, a multiway ANOVA on the interaction of aging and filter size, with jacket type and load included as covariates, found no significant effect ($F(1) = 0.486, p = 0.487$).

Effect of Front- versus Top-Load Washing Machine. Median overall total fiber mass recovered per garment for front-load (median = 220 mg, $n = 30$) and top-load (1906 mg, $n = 40$) washing machines were compared using a Wilcoxon rank-sum test. Median fiber mass was found to be significantly greater for the top-load machine ($Z = 8.74, p < 0.001$). As discussed in the previous analysis of fibers recovered on each filter type, the central agitator of the top-load washing machine may be more abrasive on garments as compared to the rotating drum of the front-load machine, leading to the significantly higher observed shedding overall (Figure S4). Further testing

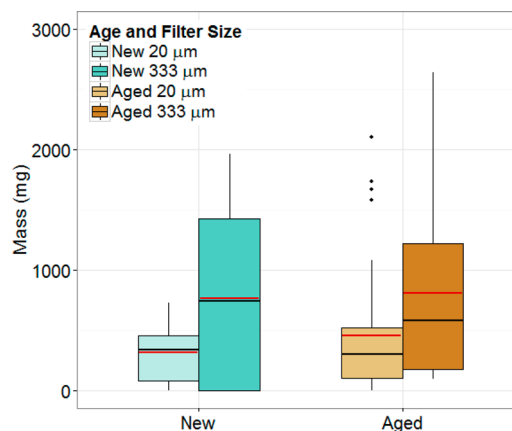


Figure 3. Distribution of fiber mass recovered for new versus aged jackets on the 20 and 333 μm mesh filters ($n = 35$ each box), across front- and top-load washing modes. Red lines indicate means, black lines medians, and black dots outliers (beyond 1.5 times the interquartile range).

with additional models of washing machines could improve understanding of the connection between fibers released in wash and type of agitation of washing machines.

Effects of Jacket Type on Recovered Microfibers. For jacket E (off-brand), the median total fiber mass recovered across aging and machine loading treatments, and across both fiber size classes, was found to be 41% higher than the similarly constructed jacket D (brand name) (Figure 4). However, the

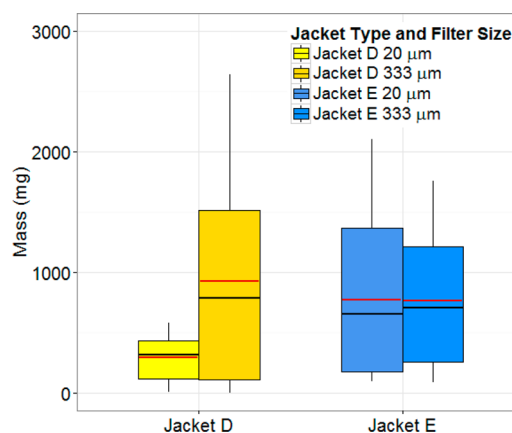


Figure 4. Distribution of fiber mass recovered for jacket D versus jacket E on the 20 and 333 μm mesh filters ($n = 14$ each box), across all treatments including aged or new and front- or top-load washing modes. Red lines indicate means, black lines medians, and black dots outliers (beyond 1.5 times the interquartile range).

median total fiber masses per wash for jacket D (median = 1300 mg, $n = 14$) versus jacket E (1831 mg, $n = 14$) were compared using a Wilcoxon rank-sum test, finding no significant difference in fiber mass recovered between the two jacket types ($Z = 0.92, p = 0.178$) overall. A multiway ANOVA test of the interaction between the two jacket types and fiber mass according to filter size, including washing mode and age as covariates, found a significant interactive effect ($F(1) = 7.53, p = 0.0084$). It is unclear what factors alter the apparently differing fiber size classes (according to differing recovered masses by filter size) from washing the two jackets, but it appears that jacket brand, perhaps related to manufacturing

processes or variations in materials of construction, can influence the characteristics of fibers recovered. Further research on yarn and fabric construction could clarify the fiber size differences between jacket types.

Assessment of Environmental Implications. Assuming that the masses recovered in this study are indicative of actual masses released, the modeled microfiber release for 100 000 jackets washed either once per month or twice per year would result in an average mass of 0.65 or 3.91 kg, respectively, of synthetic microfibers released to a hypothetical WWTP each day. On the basis of a WWTP microplastic removal rate reported by Murphy et al. of 98.4%,¹² which is similar to other removal percentages reported for microfibers that were not chemically characterized,^{28,33,34} this would result in 0.64 or 3.85 kg of microfibers being retained in WWTP solids each day, and 0.01 or 0.06 kg released to the aquatic environment each day. It is important to note that variations in WWTP design and operation would significantly alter such estimates.³⁵ In the WWTP assumed for this analysis, the facility was considered a secondary WWTP, which removes a greater amount of microplastics from the influent before discharge. If the facility instead discharged the effluent after only primary treatment, it could release greater than 20% of influent microplastics to the aquatic environment.¹²

Assuming 0.35 m³ of sewage per person per day as influent to the WWTP³⁰ and an influent fiber concentration of 2905 fibers m⁻³,¹² a population of 100 000 people would produce approximately 1.02 kg of fibers each day. Assuming once per month to twice per year washing with all released microfibers transporting to the WWTP, laundering of synthetic jackets would account for approximately 71 to 428% of the fibers observed in WWTP influent. Under the more frequent washing scenario of once a month, the model is clearly overestimating the contribution of laundering synthetic jackets to the load observed at the WWTP. Additionally, there are many other synthetic garments that would likely contribute to the fibers observed at the WWTP, meaning that the expected contribution of synthetic jackets would be even lower.

A study by Baldwin et al.³⁶ regarding the Great Lakes watersheds found that the abundance of synthetic fibers was not significantly correlated with the amount of treated wastewater effluent in streams, suggesting that other pathways of transport may play a larger role in the dispersal of microfibers through the environment. Besides possible entry to the water (via WWTP effluent) or soil (via biosolids), microfibers eroded from soils via runoff, or microfibers carried to the air in aerosol droplets, could also be deposited to land or surface waters through dry or wet deposition from the atmosphere.³⁷ Furthermore, where graywater is applied directly to land, microfibers would be directly released.

Overall, this study documents synthetic microfiber shedding via common washing of garments and indicates that clothing age and washing machine type can alter recoverable microfiber masses. Further testing with the apparel washing protocol reported in this study could clarify factors influencing shedding dynamics, such as water temperature, detergent, machine model, and clothing construction. On the basis of the environmental assessment model, common jacket washing could account for a substantial proportion of synthetic microfiber load into WWTPs. Quantifying household effluent fiber content in comparison to WWTP influent could be a next step to refining the understanding of residential synthetic microfiber sources and transport to local WWTPs. The mass

balance approach paired with size fractionation, as per this study, can be used to effectively evaluate the relative magnitude of microfiber sources, which could assist mitigation measures to reduce microfiber pollution.

■ ASSOCIATED CONTENT

📄 Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.est.6b03045.

Description and composition of jacket types tested (Table S1), water quality metrics (Table S2), regression of wash order and covariates against fiber mass (Table S4), average percent of jacket mass recovered across treatment types (Table S3), filtration column with size specifications (Figure S1), example ImageJ measurement (Figure S2), image of pilling (Figure S3), average top- and front-load mass recovered (Figure S4), and average mass recovered across measured variables (Figure S5) (PDF)

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

Patagonia Inc. financially supported this research and provided or purchased all garments used in this study. Sage Davis assisted with the design and construction of the filtration column.

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1 Supporting Information: Microfiber Masses Recovered From
2 Conventional Machine Washing of New or Aged Garments

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23 **Number of pages: 12**

24 **Number of figures: 5**

25 **Number of tables: 4**

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29 **Table S1.** Description and composition of each jacket type tested.

Jacket	Body Fabric Composition	Description
Type A	100% nylon	Technical non-fleece synthetic jacket
Type B	85% recycled polyester, 15% polyester	Synthetic fleece pullover
Type C	63% recycled polyester, 33% polyester, 3% spandex	Synthetic fleece midlayer jacket
Type D	100% polyester	Synthetic sweater fleece jacket
Type E	100% polyester	Budget synthetic sweater fleece jacket

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32 **Table S2.** Tap water quality metrics as reported by Ventura Water for 2012-2014.¹

Constituent	2012	2013	2014
pH	7.4	7.3	7.3
Hardness (ppm)	619	671	645
Calcium (ppm)	162	182	169
Magnesium (ppm)	52	49	42
Manganese (ppb)	ND	0.41	0.41
Sodium (ppm)	134	140	129
Phosphate (ppm)	0.1	0.07	ND
Potassium (ppm)	4.8	4.97	4.74
Total Alkalinity (ppm)	269	265	266

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35 **Table S3.** Average percent and standard deviation percent of jacket mass recovered per wash
 36 across treatment types.

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Filter Size Machine Type Age	20 μm				333 μm			
	Top-load		Front-load		Top-load		Front-load	
	New	Aged	New	Aged	New	Aged	New	Aged
Jacket A	0.086% (0.026%)	0.106% (0.045%)	0.018% (0.005%)	0.1% (0.157%)	0.296% (0.092%)	0.328% (0.171%)	0% (0%)	0.077% (0.03%)
Jacket B	0.099% (0.024%)	0.072% (0.033%)	0.005% (0.004%)	0.019% (0.001%)	0.278% (0.098%)	0.187% (0.099%)	0.011% (0.01%)	0.026% (0.004%)
Jacket C	0.107% (0.019%)	0.106% (0.032%)	0.008% (0.007%)	0.023% (0.005%)	0.341% (0.079%)	0.314% (0.125%)	0% (0%)	0.041% (0.002%)
Jacket D	0.077% (0.017%)	0.072% (0.016%)	0.021% (0.02%)	0.016% (0.004%)	0.191% (0.069%)	0.344% (0.098%)	0% (0%)	0.024% (0.007%)
Jacket E	0.126% (0.008%)	0.324% (0.042%)	0.036% (0.006%)	0.02% (0.003%)	0.262% (0.064%)	0.156% (0.09%)	0.042% (0.042%)	0.051% (0.006%)

38 **Table S4.** Regression model of wash order against recovered fiber mass (mg), with other
 39 variables included as covariates. Wash order did not significantly predict fiber mass: $t(131) = -$
 40 $0.20, p = 0.84$.

Variable	Estimate	Standard Error	t-value	p-value	R ²
(Intercept)	-4.7	426	-0.011	0.99	0.55
Type: B	15	179	0.083	0.93	
Type: C	-0.1	130	-0.0010	1.0	
Type: D	67	130	0.51	0.61	
Type: E	279	239	1.2	0.25	
Age: New	-164	362	-0.45	0.65	
Load: Top	947	699	1.4	0.18	
Filtersize: 333	406	69	5.9	< 0.001	
Wash order	-4.0	20	-0.20	0.84	

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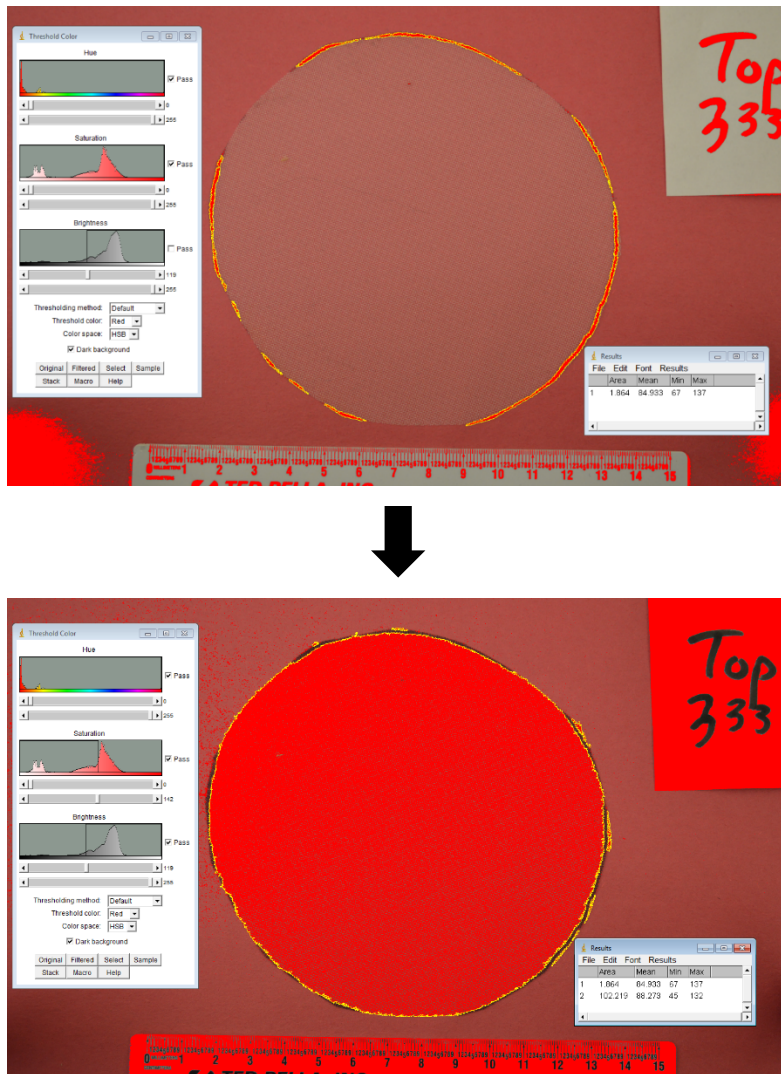
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52 **Figure S1.** Filtration column with size specifications. Each filter housing contains a Nitex®
53 nylon filter manufactured by Aquatic Research Instruments roughly cut to a circle about 11.5
54 centimeters in diameter. The filtration column was constructed out of 10.16 cm diameter
55 schedule 40 ABS piping. These were connected with schedule 80 PVC 10.16 cm diameter pipe
56 compression union with Viton o-ring. The filters were secured between the halves of the unions.

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61 **Figure S2.** ImageJ measurement of a top-load 333 μm sample. The 15 mm ruler at the bottom of
 62 each image is used to set the scale. For 333 μm filters, areas were calculated in two parts to
 63 isolate both the black marker lines (0 to 255 Hue, 0 to 255 Saturation, and 94-100 to 255
 64 Brightness) and white filter (0 to 255 Hue, 0 to 130-138 Saturation, and 0 to 94-100 Brightness).
 65 For 20 μm filters, areas were calculated with one measurement (0 to 255 Hue, 0 to 130-135
 66 Saturation, and 0 to 255 Brightness).



77 **Figure S3.** Image of aged top-load Jacket D 333 μ m filter showing the pilling effect of smaller
78 fibers, causing them to catch on the larger filter.

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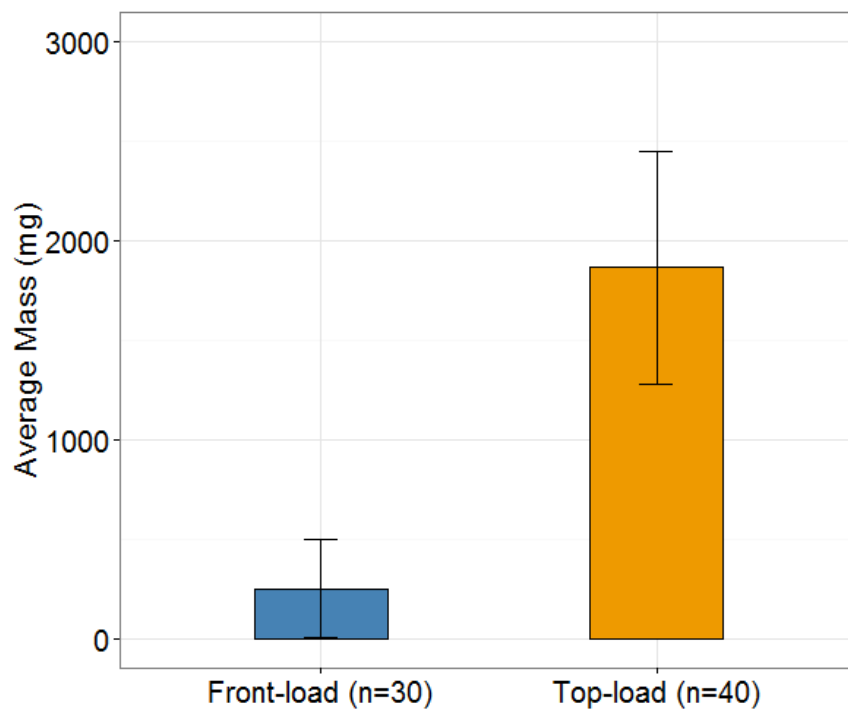
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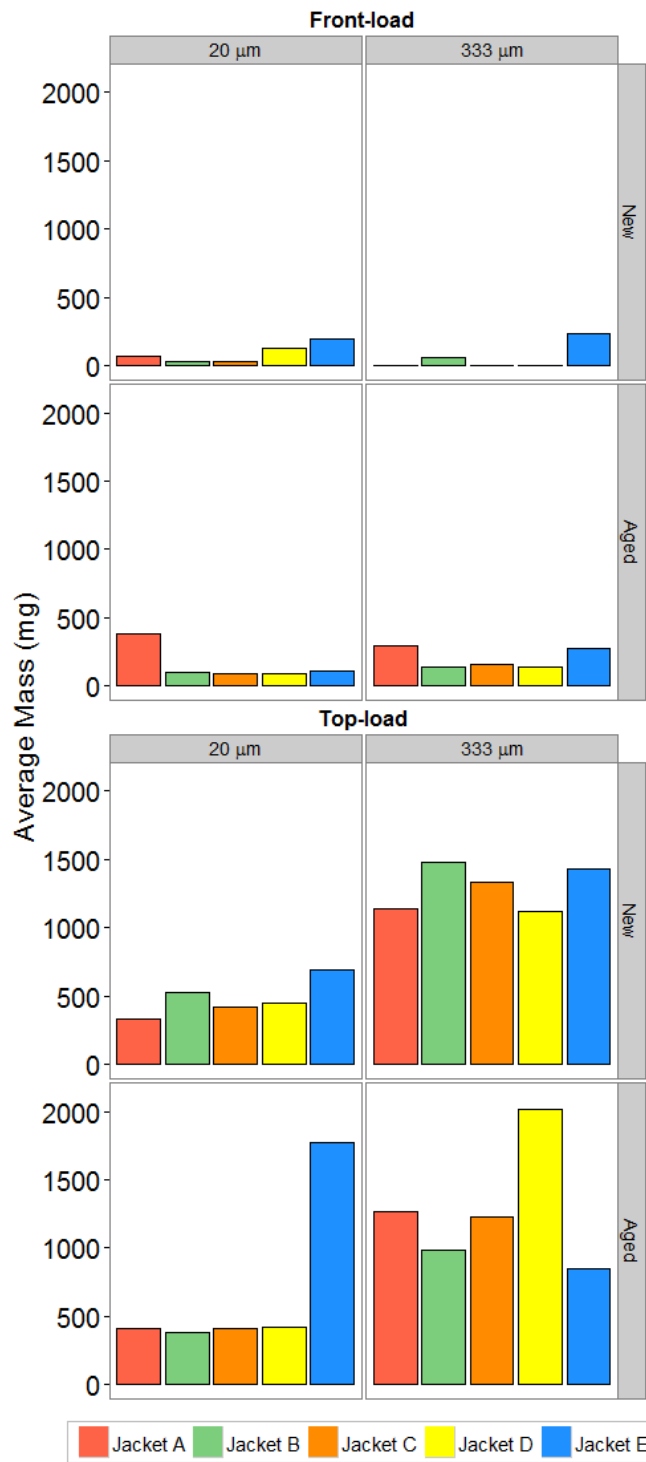
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98 **Figure S4.** Average total ($20\mu\text{m} + 333\mu\text{m}$) fiber mass recovered in Front-load and Top-load
99 washing machine effluent. Error bars are \pm one standard deviation.

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102 **Figure S5.** Average fiber mass recovered across all measured variables: age, washing machine

103 load type, filter sizes, and jacket types.

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