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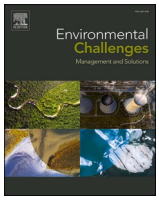

Washing machine filters to mitigate microplastics release: Citizen science study to estimate microfibers capture potential and assess their social acceptability

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ABSTRACT

The accumulation of microplastics in the environment, particularly due to the significant contribution of synthetic clothing washing water, leads to a need for developing source-based retention solutions. The project's objective is to assess the capability of washing machine filters to capture microfibers from domestic wastewater under real laundry conditions. This involves developing a protocol for quantifying microfibers tailored to washing machine lint samples, estimating the potential microfiber capture by washing machine filters, and evaluating the social acceptability of using such filters in households. Volunteers were recruited and they used their washing machines normally, collecting lint in the filter over a 6-month period and documenting information for each load. Various reagents were tested using plastic controls and lint samples to select the most efficient one for removing organic matter without affecting microplastics. Microscopic visual alterations, chemical signatures via FTIR, and the efficacy of the digestion protocol were assessed through mass balance and visual counting. After discarding digestion protocols affecting plastic integrity, those ensuring the most efficient mass loss of organic matter were identified as H_2O_2 30 % for 5 days at room temperature (RT), NaOCl 3 % for 24 h at RT, and H_2O_2 30 % at 40 °C for 24 h. Polyester, acrylic, and polyamide controls were tested with NaOCl 3 % for 24 h and H_2O_2 30 % for 5 days, showing no changes in SEM images. The FTIR successfully recognized the chemical signature. More significant alterations on positive samples (cotton and cellulose) were observed after NaOCl exposure. The mean mass of microfibers remaining after digestion was estimated at 4.62 mg per liter of washing water or 61 mg/kg of washed garment. The survey revealed that 67.8 % of volunteer participants found the filter installation challenging, and 21.4 % had to hire a plumber, highlighting potential challenges associated with implementing washing machine filters on a large scale.

1. Introduction

Microplastics (MPs) are generally defined as small debris of plastic that are less than 5 mm in size. They can be classified into two types Boucher (2017): Primary microplastics, released into the environment at the micro scale, and secondary microplastics, resulting from the fragmentation of plastic waste in the environment. The durability of these plastics, ranging from several decades to millions of years, remains an open question. With the exception of what has been incinerated, it is believed that the majority of plastics introduced into the environment persist in their integral form or as fragments (Thompson et al., 2005).

Synthetic microfibers have been identified as a major contributor to primary microplastic pollution (Boucher, 2017). When synthetic fabrics are washed, microfibers (MF) are produced through abrasion and fiber loss. Thousands to millions of microfibers are estimated to be released during laundry (Browne, Crump et al., 2011; De Falco et al., 2018; Lassen et al., 2015; Napper & Thompson, 2016).

Microfiber shedding rates are influenced by multiple factors. Studies have shown that microfibers release rates depend on fabric type, detergent type or fabric softener, temperature, the type of washing machine (De Falco et al., 2018) while shedding is greater for new garments according to Pirc et al. (Pirc et al., 2016). The methodologies proposed to estimate microfibers detachments are mostly conducted in laboratory under simulated conditions. These studies have the

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advantage of identifying certain factors that affect their release but do not reflect it under real household laundry conditions.

Microfibers released during laundry enter wastewater collection systems and are transported to wastewater treatment plants or discharged directly in some cases, into the receiving environment. Microplastic concentrations of 1–10,044 particles per liter have been reported in the influent of wastewater treatment plants (Sun et al., 2019). Microplastics are captured in biosolids via settling, biological treatment, or other processes (Liu et al., 2021). Reported microplastic sludge concentrations vary widely, from 1000 to 170,900 particles/kg of dry sludge, with fibers accounting for the vast majority. The spreading of sludge constitutes a pathway for the penetration of microplastics into the soil and subsoil layers. However, despite the high percentages of microplastic removal reported in wastewater treatment plants (95–99 %), it is estimated that up to 10^8 microplastics are discharged per day in the effluent from a single wastewater treatment plant that treats between $0{,}26.10^6$ m^3/d and $1{,}51.10^6$ m^3/d (Nguyen et al., 2019). Thus, it is reported that microplastics released from wastewater treatment plants still account for 25 % of the total flow of plastic pollution to the oceans (Boucher, 2017). More specifically, the fluvial corridor of the St. Lawrence River between Montreal and Quebec City presents among the highest concentration of microplastics recorded in the world's freshwater and marine systems (Crew et al., 2020). For the moment, sludge and the intercepted microplastics can be incinerated or used as fertilizer in agriculture. As well as looking at new treatment processes, it is desirable to reduce microfibre emissions at source, which is the aim of washing machine filters.

The relative effectiveness of washing machine filters in capturing microfibers has been demonstrated at laboratory scale (McIlwraith et al., 2019; Napper & Thompson, 2016) where washing machine filter showed a capture potential up to 87 %. When scaled up to a community (Erdle et al., 2021) the effectiveness of machine filter was significant because the installation of washing machine filters in 10 % of the homes in town reduced the quantity of microfibers in the effluent from the treatment plant by an average of 41 %.

The objective of this project was to characterize the potential of washing machine filters to capture microfibers from laundry wastewater, under real laundry conditions. The specific objectives of the projects were to 1) develop a protocol for microfibers quantification adapted to washing machine lint samples, 2) estimate the potential microfibers capture by washing machine filters, and 3) evaluate the social acceptability of using such filters in households.

As previous rare social science study noted (Herweyers et al., 2020), the issue of microfibers, unlike the more widely recognized problem of microplastics such as the plastic soup, remains relatively obscure. This has led to an underestimation of the impact of people's washing habits on environmental pollution. Effectiveness stands out as the primary criterion for products aimed at reducing microfiber release during washing. It's not just about scientific validation but also about providing visible results that users can perceive, crucial for ensuring sustained usage. Alongside effectiveness, durability plays a key role in the adoption of these products, ensuring they can be relied upon for the long term. Simplicity and user-friendliness are equally important; products need to be easy to operate to encourage consistent use. While cost is a factor, its importance is secondary to ensuring affordability across different socio-economic groups to achieve widespread adoption. Cleaning these products should be quick, easy, and foolproof, making it achievable within regular washing routines.

In this study, 30 volunteers were hired to participate to the project in a citizen science mode. Each volunteer participant installed a washing filter in his/her household. Volunteer participants used their washing machine in a normal way and collected lint accumulating in the filter over a 6-month period, and documented information regarding each load. After, using plastic controls and lint samples collected by volunteer participants, different reagents were tested in order to select the most efficient to remove organic matter without altering the microplastics. Microfibers were quantified after digestion by weighting and counting. Finally, feedback from volunteer participants provided insights on the feasibility and practical implications of such a measure on a larger scale. The project was realized in collaboration with GRAME (French name: Groupe de Recommendations et d'Actions pour un Meilleur Environnement) organization (initiating the project and coordinating the logistic of the citizen science phase).

2. Materials and methods

2.1. Collect of lint by citizen science

Washing machine filters were provided to 30 volunteer participants from 30 households located in the Greater Montreal area. Volunteer participants installed their filter on their washing machine, to capture the microfibers released during their laundry in a citizen science mode. The collection began in May 2021 and ended in December 2021. The recruitment of volunteer participants and collection of lint was coordinated by GRAME. Guidelines provided to participants are shown in supplemental information, Fig. S1.

The filter used to collect the lint was LUV-R model (from Environmental Enhancements), made of stainless-steel mesh with hole diameters of 667 μm (0.0625 inches). A picture of the filter is shown in supplemental information, Fig. S2.

When the bowl of the filter was visually obstructed by lint, volunteer participants were instructed to clean the filter by opening the bowl, collecting the lint retained by the filter and keeping it in a small resealable bag to be stored in their home freezer, until sent by mail in prepaid envelope to our laboratory. To do so, they proceeded by pouring the contents of the bowl in a coffee filter or a tightly woven cloth and brushing the filter with a toothbrush or a metal brush. Volunteers were supported by sharing between them the techniques they employed to collect lint. The frequency of emptying the filter varied among participants based on how much lint built up and the number of wash cycles. This schedule depended on individual habits and household size. Samples received were placed in our freezer until analysis started in January 2022.

A key indicator for changing the filter was visible lint accumulation in the bowl, along with clothes being wetter than usual after washing or the washing machine not draining properly. Each participant adapted their method of collecting lint. Lint deposited on the filter mesh was collected by households either by a dish brush, knife blade, toothbrush or by hand. The lint present in the filter bowl water (e.g., water mixed with lint) was collected by filtration using either a coffee filter, nylon socks, or a piece of fabric. The date of cleaning operations of the filter was recorded on a calendar provided to volunteer participants.

Additionally, at each wash, volunteer participants provided laundry settings regarding washing water volume (low, medium, high), temperature (cold, warm, hot), duration (min), intensity (regular, fast, intense), percentage of filling of the machine with garments (%) and the use of detergent. This information was documented in a table provided to volunteer participants (shown in Supplemental information, Table S1).

Since the brands and characteristics of the washing machines differed from one volunteer participant to another, the following hypotheses were taken to process and convert non numerical data:

The values assigned to determine the average volume of washing water were 40, 60 and 80 liters corresponding to low, medium, and high settings reported by households.

Values of 20 °C, 40 °C and 60 °C were attributed to cold, warm, and hot to express water temperature (CDA Buying Advice, 2023).

Factors of 1, 2 or 3 were attributed to fast, regular, and intense, respectively regarding washing intensity.

According to estimations, washing machine with a capacity of 7 kg is suited for a small sized family ("https://hernebaydomestics.co.uk/2023"). Considering an average household size of 2.2 persons in

2021 for Quebec (Canada, 2022), a capacity of 7 kg was assumed corresponding to 100 % filling of the washing machine.

Although 30 households originally participated to the citizen science phase, the data analysis concerned 19 volunteer participants. Within volunteer participants that were not considered in the analysis, some samples were used to carry method development tests, while others were omitted due to missing or incomplete information and withdrawal of some volunteer participants from the project due to problems encountered with the washing machine filter.

2.2. Methods for estimating microfibers mass and count

The subsequent steps were followed to estimate microfibers mass and count after digestion in lint matrices:

a) Collect lint accumulated on the washing machine filters and document washing parameters about laundry (carried out by the volunteer participants).
b) Classify reagents according to mass loss of digested samples.
c) Assess the alterations of plastic controls due to exposure to reagents during the digestion phase.
d) Chose optimal reagent to digest lint samples based on steps b and c.
e) Quantify via two approaches (weighting and counting) microfibers in lint samples after applying selected reagent.
f) Estimate the number of microfibers diverted from discharge into sewer using a washing machine filter. The quantity of avoided microfibers has been expressed with respect to the captured lint dry weight (MF/ g lint), liter of washing water (mg MF/L) and washed garment (mg MF/Kg garment).

2.3. Method development and quantification

The procedures followed for method development and quantification are shown in Table 1

Four types of tests were carried out. Two method development tests were used to select the digestion protocols best suited to purifying the samples and preserving the microplastics. The first test (Plastic integrity in Table 1) was used to determine which digestion protocols would

Table 1
Summary of procedures followed in a step-by-step format.

Test progression ↓

	Tests for method development		Tests for quantification	
Type of test	**Plastic integrity**	**Reagent efficiency classification**	**Mass**	**Count**
Type of sample material	plastic controls: pellets or fibers from clothes	Lint	Lint	Lint
Total Nb of tests (one per sample)	48	24	157	3
Sample preparation	None for pellets; Tweezers to collect fibers	40°C oven drying	40°C oven drying	40°C oven drying
		Blender; approx 100 mg sub-sample	Whole lint	Tweezers to compose approx 4g sample
		Weighing	Weighing	Weighing
Digestion method	20 ml of either : - 5M HCL, RT, 24h - 10 M NaOH, RT, 24h - 10 M NaOH, 40°C, 24h - 10% (w/v) KOH, RT 24h - 10% (w/v) KOH, 40°C 24h - 3% (w/v) NaOCl, RT 24h - 30% H_2O_2, RT, 5d - 30% H_2O_2, 40°C 24h		250 ml NaOCl 3%, 24h	500 ml NaOCl 3% 24h
Sub-sampling	Whole pellet or fiber	Whole sub-sample	Whole lint	collection of 3 sub-samples of 5ml from digested sample solution
Filtration	vacuum filtered on 1.5 um fiber glass filters (diameter 47 mm)		vacuum filtered on 1.5 um fiber glass filters (diameter 90 mm)	vacuum filtered on 0.45 µm cellulose nitrate membrane (diameter 47 mm)
Analysis	Visual observation; SEM imaging; FTIR	Weighing	Weighing; FTIR	Binocular imaging (9 sub-samples; 6 images per sub-sample); ImageJ

preserve the integrity of microplastics, so as not to underestimate them (by testing laboratory and consumer-goods polymer controls in the various reagents chosen (digestion method) and observing the size and shape of the polymer controls before and after). The second protocol determines the effectiveness of the chosen protocols (see digestion method in Table 1) in terms of organic matter reduction (based on mass measurements of lint samples before and after digestion).

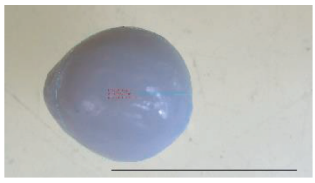
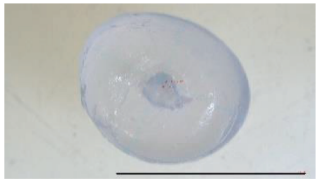
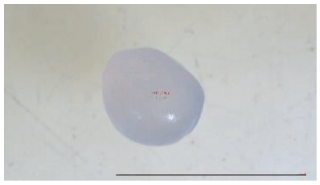
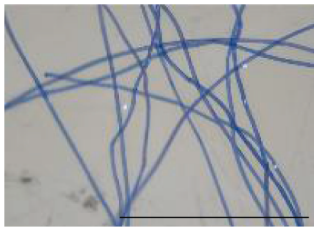
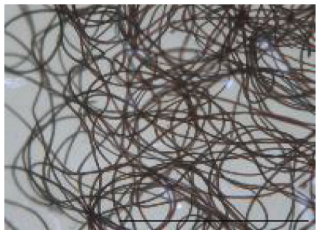
Once the protocol had been chosen on the basis of the method development tests, two tests were carried out to quantify the microplastics in the lint samples (Mass and Count in Table 1). The mass test assessed the mass of plastic microfibers produced by the various washings, assuming that the rest of the organic and inorganic matter had been produced by the digestion methods used. In addition, the counting test enabled the number of microfibers produced by the washes to be established by microscopy. Note that in our context, density separation using a saturated NaCl solution was not an efficient method to separate microfibers from other organic residues, since fibers and other matter were highly entangled.

2.3.1. Sample preparation

Different procedures were used to prepare samples for method development and quantification.

For plastic integrity tests, plastic pellets of polyethylene terephthalate (PET), polypropylene (PP) and polyamide (PA6), along with

Table 2
Test controls charateristics (the scale bar represents 5 mm).

	Controls	Shape	Size (μm)	Color	Source
	Polyethylene terephthalate (PET)	Spherical	3600	Opaque grey	Laboratory plastic pellets
	Polypropylene (PP)	Oblong	3600/4500	Semi transluscent	
	Polyamide 6 (PA 6)	Oblong	2200/3100	Semi transluscent	
	Polyester (PES)	Fiber	25 (diameter)	Blue	New beanie 100 % polyester
	Acrylic (PMMA)	Fiber	16.5 (diameter)	Pink	New beanie 100 % acrylic
	Polyamide (PA)	Fiber	13.5 (diameter)	Black	New socks 100 % polyamide
	Cotton	Fiber	ND	White	T-shirt 100 % cotton
	Cellulose	Fiber	ND	Brown	Paper towels
	Wool	Fiber	ND	Purple	Undergloves 100 % wool

fibers from a blue beanie (100 % polyester (PES)), acrylic fibers from a pink beanie (100 % acrylic (PMMA)) and nylon fibers from black socks (95 % nylon (PA)) were chosen as plastic control samples (Table 2) to assess plastic integrity after reagent exposure.

Control natural fibers consisted of cotton from a white t-shirt (100 % cotton), cellulose fibers (from brown paper towels) and wool from purple undergloves (65 % wool). All fiber samples were collected from new garments purchased in January 2022 in stores in Montreal city.

We selected polyester (PES), polyamide (PA) and acrylic (PMMA) as synthetic control fibers. Polyester and nylon were selected because they are the most produced polymers in the fiber industry, representing 52 % and 5 % of the 2020 global fiber market, respectively (Exchange, 2021; TextileExchange 2021). Acrylic represents 5.2 % of the market along with polypropylene and elastane.

Prior to digestions, lint samples collected from volunteer participants were transferred to labeled containers and air dried. They were placed after in a drying oven at 40 °C for 4 h to remove excess moisture. Dried lint samples were weighed. Plastic bottles containing dried lint were then sealed and stored in the laboratory at room temperature until digestion and quantification analysis.

For reagent efficiency classification, 24 sub-samples of approximately 100 mg were collected from the lint of 3 volunteer participants. A blender (magic bullet) allowed to homogenize lint clusters prior to collecting the sub-samples. The blending time was 20 s.

For quantification tests by mass, 157 whole lint samples were used, corresponding to the production of 19 volunteer participants.

For quantification tests by counting, different fragments of lint were collected with tweezers from 3 volunteer participants to form 3 samples of approximatively 4 g.

2.3.2. Digestion

For method development tests, plastic controls and lint samples were added to 20 ml of reagent solutions (either 5 M HCl, 10 M NaOH, 10 % (w/v) KOH, 30 % H_2O_2 or 3 % (w/v) NaOCl) in 100 ml beakers. For thermal digestions, samples were transferred to 250 ml glass tubes and placed in a digester (Foss™ Tecator™ 2508 Digestion System, Fisherscientific).

Digestions were performed for 24 h at room temperature (25 °C) with 5 M HCL, 10 M NaOH, 10 % KOH, 3 % and 6 % NaOCl solutions and 5 days for 30 % H_2O_2. The digestion temperature was set to 40 °C for an additional set of tests to determine temperature effect during exposure to 10 M NaOH, 10 % KOH and 30 % H_2O_2 reagents.

Digestions for quantification tests were performed for 24 h at room temperature using either 250 ml of 3 % NaOCl for quantification by mass, or 500 ml of 3 % NaOCl for quantification by count (see results and discussion for the analysis that led to this choice of reagent).

2.3.3. Subsampling

For quantification by count, digested lint solutions were placed on a stirring plate. Under continuous stirring, 3 sub-samples of 5 ml from each digested lint solution were transferred to a 25 ml graded cylinder that were then vacuum filtered before analysis.

This step of subsampling was carried out to better distinguish the microfibers under binoculars once filtered and to avoid the formation of a filtration cake on the filter.

2.3.4. Filtration

Solutions were vacuum filtered on 1.5 µm Whatmann fiber glass filters with either 47 mm diameter for plastic controls and lint used for reagent efficiency classification or 90 mm for digestions used for mass quantification.

Filtration was carried out so as not to transfer sand particles that remained in the bottom of the beakers and tubes. They were rinsed with demineralized water to collect all remaining residues through the filtration system and make sure they reach the filter surface.

For counting quantification tests, sub-samples were vacuum filtered on a 0.45 µm cellulose nitrate membrane (Pall GN-6 Metricel membranewere).

2.3.5. Analysis

2.3.5.1. Visual observation. For plastic integrity tests, alterations were noted visually immediately after exposure to the reagents and then after 24 h for controls tested with HCl, NaOH, KOH, NaOCl, and every 24 h for H_2O_2 exposed samples at room temperature. The purpose was to assess visible surface alterations of synthetic microfibers before and after reagent exposure.

2.3.5.2. SEM and FT-IR. SEM and FT-IR analysis were performed on plastic controls (polyester, acrylic and polyamide) to determine if significant physical or chemical alteration have occurred that were not detected during visual examination. This analysis was performed for samples treated with the two highest ranked reagents identified after reagent efficiency classification.

Scanning Electron Microscopy (SEM) was used to perform further analysis of synthetic controls fibers surface. Images were captured using a HITACHI TM3030Plus tabletop scanning electron microscope. Samples were placed on a circular stage using a double-sided adhesive tape. No metal coating was used. Observations were performed in low vacuum and an accelerating voltage of 15 kV.

Fourier Transform Infrared (FT-IR) spectroscopy analysis was used i) as qualitative approach to determine whether a reagent had reacted with synthetic polymers and ii) to check if non plastic fibers such as cellulose or cotton remained in lint after digestion. For the latter, 38 FT-IR analysis were performed on fibers collected from filtered digested samples. Spectra were acquired by a PerkinElmer Spotlight™ 400 µFT-IR imaging system, in transmission mode, covering the IR spectral range from 4000 to 700 cm^{-1}, using four scans per pixel of 32 µm. Spectra were then compared to the FT-IR database for identification.

2.3.5.3. Weighing. Lint obtained after the digestion step were oven dried at 40 °C for 24 h and weighed.

The mass loss of each sample was calculated using Eq. (1):

$$Mass\ loss\ (\%) = 100\ x\ ((Mi - Md)/Mi) \quad (1)$$

Where Md = Mass of dry lint after digestion, Mi = Mass of dry lint before digestion. Weighting was performed using a precision balance (Ohaus Explorer scale - 210 gx0.1 mg).

For reagent efficiency classification, digestion protocols were classified according to the average measured mass loss for each digestion method.

2.3.5.4. Counting. Microfiber counting was used to estimate the number of microfibers that were released during the washings.

For each filtered sub-sample, 6 images were randomly acquired on the filter surface with a binocular integrated camera to a stereomicroscope system (LAXCO) as shown in Fig. 1. Each image covered a rectangular area (Ai) of 25 mm^2.

Images were processed using IMAGEJ software to count and measure microfibers length. Average number of microfibers per subsample (N) was calculated using Eq. (2):

$$N = \frac{\sum_{i=1}^{6} Ni}{(6\ .\ Ai/Af)} \quad (2)$$

Where N_i is the number of microfibers per image, A_i the area of one image (25 mm^2) and A_f, the total filtration area (770 mm^2). The 6 Images captured covered 19.8 % of the total filtration area. This method was repeated for two other samples.

As the filters were rapidly clogged before they could be read and counted, a fraction of the sample was quantified, and the number of fibers extrapolated based on dilutions. Therefore, in total, 54 images (6

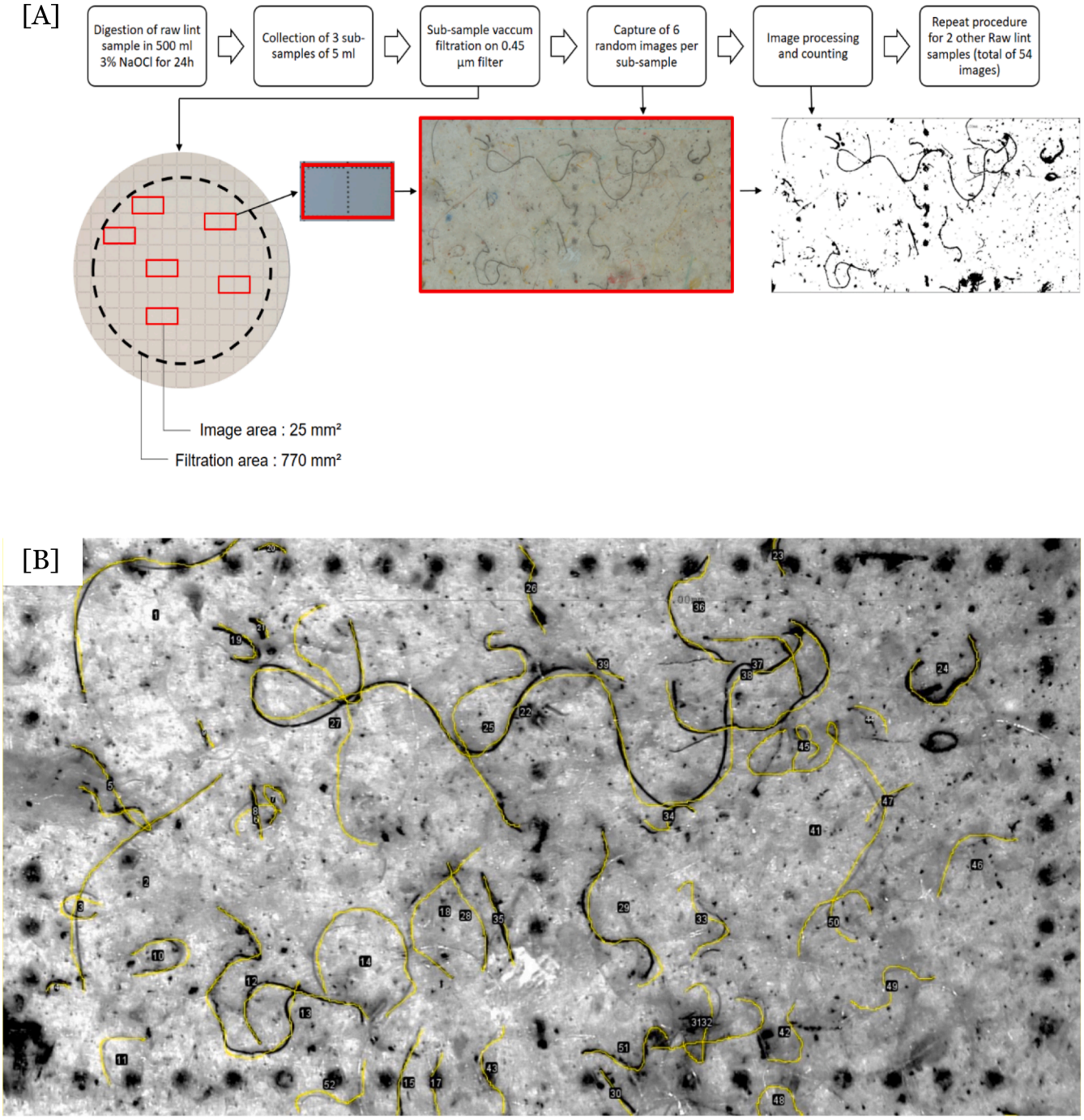


Fig. 1. Sub-sampling and counting process (A) and detailed view of microplastic counting in one example (B).

images per sub-sample) were processed to obtain the average count and length of microfibers scaled to the project. Count of fibers per gram of lint for each subsample (Nss) was calculated using Eq. (3)

$$Nss = \left(\frac{Vss}{Vs}\right) \cdot \left(\frac{N}{Mdl}\right) \quad (3)$$

Where *Vss* is the volume of the subsample (5 ml), Vs the volume of the digested lint solution (500 ml), and *Mdl*, the mass of the dry lint sample before digestion. Average number of fibers per gram of lint (N_{avg}) was then calculated as $Navg = \left(\frac{1}{9}\right) \cdot \sum_{i=1}^{9} Nssi$, as 9 subsamples were used in total.

3. Results and discussion

Raw data of laundry habits and lint mass are provided in Supplemental Information, Table S2.

3.1. Laundry habits of volunteer participants

The analysis of data collected in the citizen science phase showed that 1110 washes were performed over a period of 6 months by 19 households. In average, 7 washes were completed between two filter cleanings for a total of 58 washes per household.

Loading levels ranged from 25 % to 100 % with an average loading of 65 %. Washing cycles took between 26.8 min and 88.2 min to complete,

with an average of 50.3 min.

Regarding the intensity of washing, volunteer participants used a regular intensity in 46 % of the cases. The average score was 1.2, corresponding to a wash intensity set between regular and fast.

Using the same approach for water temperature, washing with warm water setting was preferred in 42 % of the cases. The average was established at 1.7, representing a water temperature of 34 °C.

Based on the assumptions established regarding water levels, about 66 600 liters were used for the washings.

The total dry mass of lint collected (before digestion) was 496 g.

3.2. Reagent efficiency classification by samples mass loss

The assumption that lint samples mass loss after their digestion allowed us to classify reagents efficiency was made as we sought to remove the organic matter contained in the lint samples (dust, hair, etc.) without altering the plastic microfibers (confirmed by plastic integrity tests). The resulting average mass reductions for different reagents used in digestion are given in Fig. 2.

Lint samples digested with 10 M NaOH and 10 % KOH solution at room temperature registered 25 % and 23 % mass loss respectively. In addition, a slight impact of temperature (40 °C instead of room temperature) was observed for both NaOH and KOH digested samples on their mass loss, which increased to 28 % for 10 M NaOH digested sample. The effect of temperature on mass loss was more noticeable for 10 % KOH digested samples which increased to 33 % in average. Additionally, slight discoloration of the fibers tending toward white was observed when treating with 10 % KOH at 40 °C.

In 5 M HCl, the lint mass decreased only slightly, by 7 %. Hair present in lint digested samples, digested at 5 M HCl, was still clearly identifiable after digestion.

Hydrogen peroxide (H_2O_2 30 %) and sodium hypochlorite (NaOCl 3 %) digested samples showed a significantly higher mass reduction compared to HCl, NaOH and KOH digestions. The mass loss averaged 46 %, 44 % and 41 % respectively for digested lint samples with H_2O_2 30 % for 5 days, NaOCl 3 % and H_2O_2 30 % for 24 h at 40 °C. Thus, it was noticed that H_2O_2 digestion during 5 days at room temperature was relatively more effective than for 24 h at 40 °C.

Overall, the most effective reagents in terms of mass loss were found to be H_20_2 30 % for 5 days at room temperature, NaOCl 3 % for 24 h at room temperature and then H_2O_2 30 % at 40 °C for 24 h.

In general, these observations are in accordance with literature findings. Compared to acidic or alkaline treatments, hydrogen peroxide (H_2O_2, 30–35 %) is more effective in digesting organic matter (Nuelle et al., 2014; Qiu et al., 2016; Zhao et al., 2017). Highest rates of biogenic organic matter removal were attributed to 7 % and 10 % NaOCl after 6 h of exposure at 50 °C when testing digestion solutions including Fenton's reagent (Monteiro et al., 2022). Although digestion protocols and samples differ from the ones used in our experiments, these results show that H_2O_2 and NaOCl are effective reagents to digest and remove organic matter from different types of sample matrices compared to acid or alkaline reagents.

3.3. Effect of the digestion protocol on plastic integrity

Digestions for 24 h at room temperature with HCl, NaOH, NaOCl and H_2O_2 showed no noticeable degradation of PES fibers, PET, PP and PA beads. No changes in shape or size were visible with naked eyes.

Under 5 M HCl, nylon fibers underwent significant degradation. Almost instantly, the fibers contracted and dissolved. Similar observations were reported in literature, where PA (PA6, PA66) completely dissolved when treated with concentrated acid solutions at room temperature (Karami et al., 2017).

However, PA pellets were not degraded like the PA fibers during the 5 M HCl treatment. From this finding, the pellets were deemed unrepresentative and only the plastic fibers were retained for further analysis.

Under digestions with NaOH, KOH, and HCl, changes in observable shape of natural fibers were less apparent than when using hydrogen peroxide H_2O_2 30 % and NaOCl 3 %.

Cellulose fibers were visually slightly degraded with H_2O_2 after 5 days but stayed visually detectable. Comparatively, cellulose reduced relatively with 3 % NaOCl while size contraction of the cotton sample was also noted in the case of 3 % NaOCl.

However, of all the digestion methods tested, none resulted in the complete degradation of the cotton fibers.

Based on the results of reagent efficiency classification tests, SEM analysis was performed on polyester, acrylic (Fig. 3) and polyamide fibers before and after exposure at room temperature to NaOCl 3 % for 24 h and H_2O_2 30 % for 5 days. SEM analysis did not reveal any visual surface degradation, change in size, shape, or alteration.

FT-IR was used as complementary analysis to examine potential impacts on the chemical signature of plastic. Spectra of the synthetic controls (PET, PA, PMMA) exposed at room temperature to 3 % NaOCl for 24 h and 30 % H_2O_2 for 5 days showed no significant changes compared to the untreated samples (Fig. 4). The characteristic peaks were generally maintained when exposed to both reagents, indeed The Perkin-Elmer software was able to recognise the plastic at stake in its database which indicates that digestion protocols did not alter the plastic signature.

In a previous study, Lenz et al. analyzed Raman and FT-IR spectra to

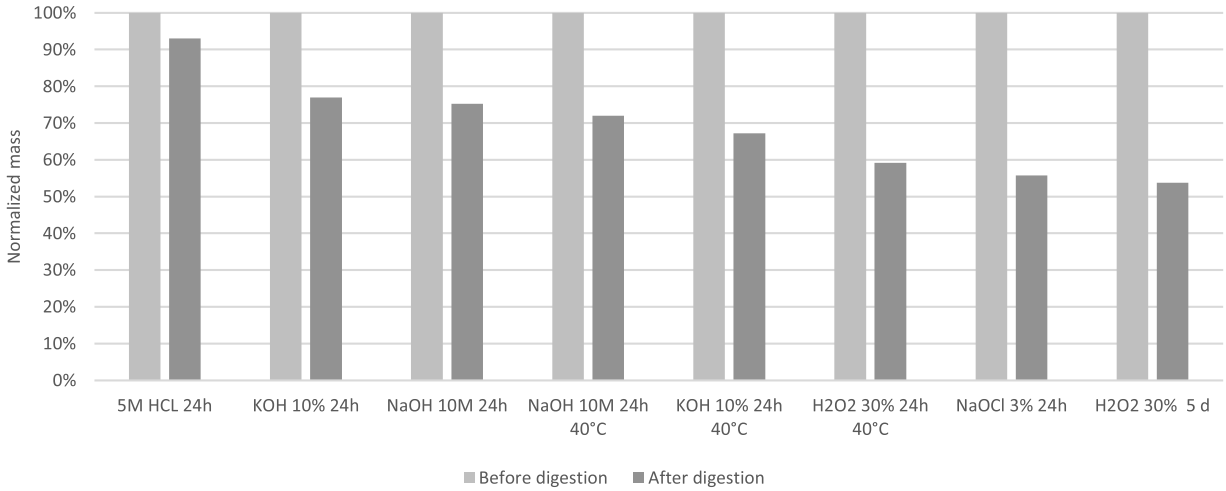


Fig. 2. Mean Reagent efficiency classification following digestion tests.

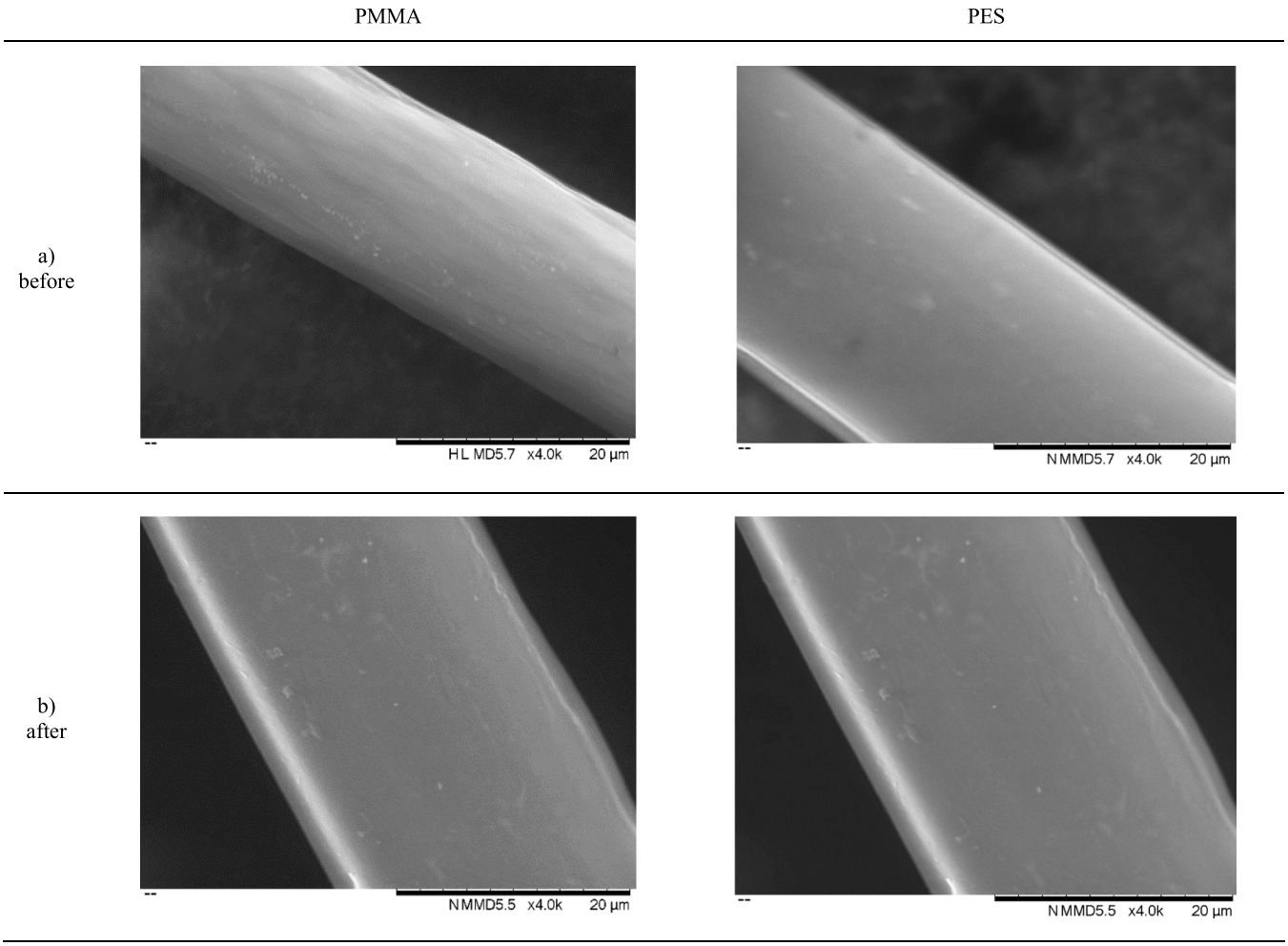


Fig. 3. Scanning electron micrographs of acrylic and polyester a) before treatment b) after exposure to 3 % NaOCl for 24 h.

assess whether a reagent has reacted with a polymer to such a degree that it would no longer be recognized by its spectroscopic signal (Lenz et al., 2021). Minor degradation observations were reported for some polymers exposed to H_2O_2 treatment for 14 days at room temperature. According to their observations, new bands could be attributed to carboxylic acids that could result from the oxidation of polymer chain ends.

3.4. Selection of digestion protocol

The diversification of the types of polymeric controls used is a novelty brought about by this article, but also influences the choice of protocol according to the controls used. In summary, after conducting reagent classification by samples mass loss, SEM observations and FT-IR analysis, the digestion with 3 % NaOCl was found to be the most satisfactory among those tested. While purification of lint samples from all organic matter and natural fibers was not possible in our experiment, plastic integrity was preserved and thus, quantification concerned plastic fibers and the remaining non-plastic fraction that could not be quantified.

Digestion with H_2O_2 was also interesting as it offered a slightly higher mass loss but the availability of NaOCl 3 % and shorter digestion time to achieve similar mass loss rates (24 h for NaOCl vs 5 days for 30 % H_2O_2), along with its low cost were additional advantages compared to H_2O_2 30 %.

In future studies, further experiments to determine the amount of reagent (or combination of reagents) to be used per sample mass can be conducted to aim for higher rates of organic matter and natural fibers removal. Depending on the exposure durations and concentrations, additional testing on control samples should be conducted to ensure the integrity of the plastic fibers. The list of plastic and natural controls should be expanded to include and test a wide range of polymers of the textile industry.

3.5. Quantification of microfibers emitted by household in the region of Montreal

After digestion and filtration, the total dry mass of all collected lint was 308 g, compared to 496 g before digestion. The corresponding average mass loss during digestion was 38 %. The lint (dry weight before digestion) captured by the filters was equivalent to a mean value of 7.44 mg per liter of washing water. This amount expressed per kilogram of garment washed is equivalent to 98 mg/kg. The mean mass of microfibers remaining after digestion was 4.62 mg per liter of washing water or 61 mg/kg of washed garment.

Using the counting method, the number of microfibers ranged from 18 to a maximum of 73 per captured image of 25 mm^2. The resulting number of microfibers per subsample was 1 169 MF in average per subsample (using Eq. (2)). Each subsample consisted of a 5 ml aliquot taken from a 500 ml lint digested solution. Based on the average weight of the digested lint and using Eq. (3), 27 280 MF were calculated per g of lint dry weight (27 280 MF/ g lint). Assuming the validity of our counting method, a total number of 8 402 240 MF (after digestion) was estimated to be captured by the filters of all volunteer participants for 6 months. Raw data of count tests are provided in Supplemental

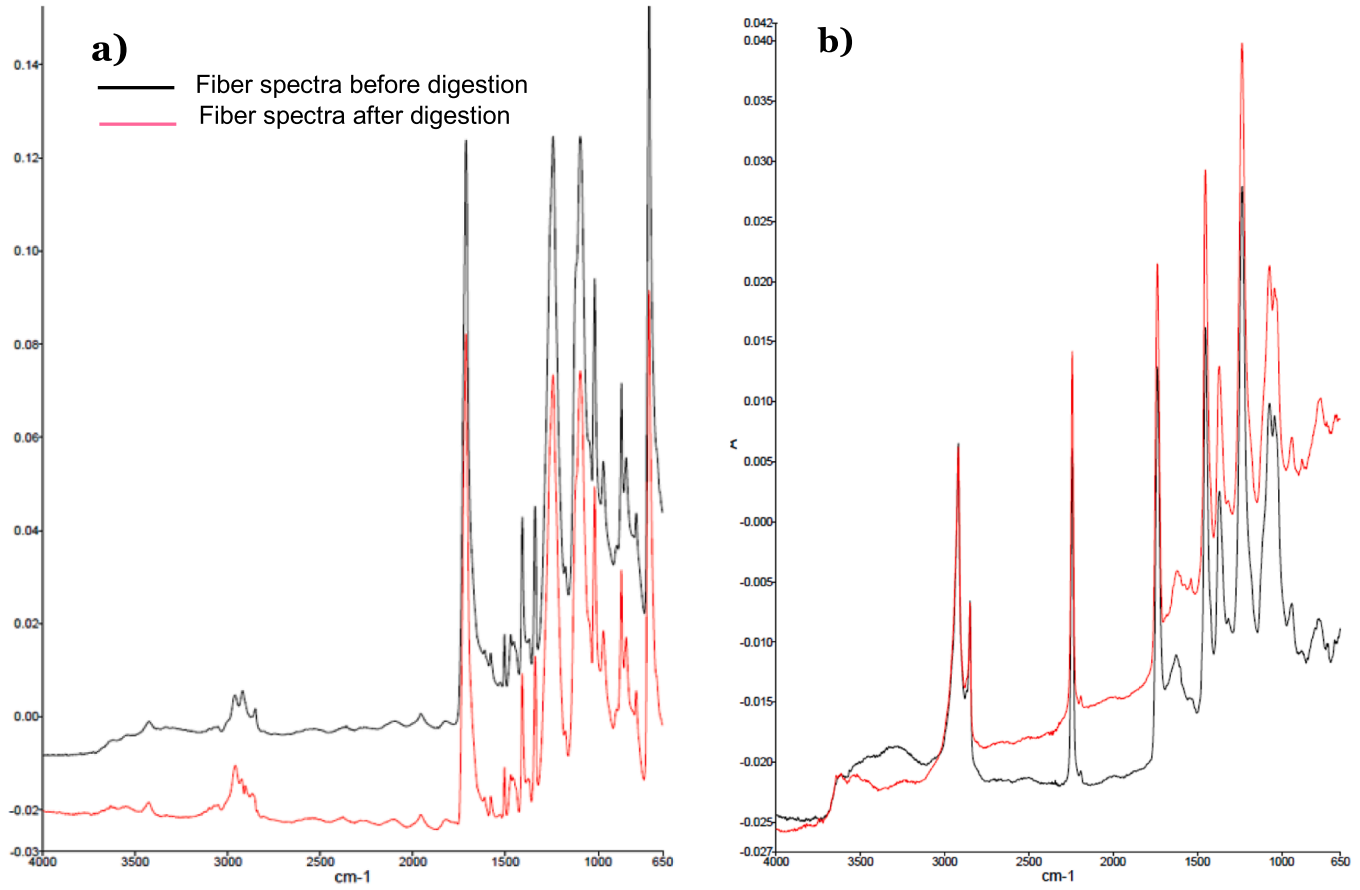


Fig. 4. Spectra before and after treatment with 3 % NaClO for 24 h at RT of a) polyester and b) acrylic controls.

Information, Table S3.

It is important to report that the composition of the lint differs from sample to sample, even if it originates from the same household. After digestion, cellulosic and other unidentified organic matter were still detected in some samples during FT-IR analysis.

A recent study in Ontario documented microfibers captured by washing machine filters using a similar approach based on citizen science. The authors reported microfiber count ranging from 28 to 423 MF per mg of lint. It also reported an average household weekly lint capture of 6.4 g (wet weight). In our study, we found an average weekly lint capture mass of 1.04 g per household (dry weight before digestion), which is six times lower than the value reported by Erdle et al. Our counting process yielded in average of 27 MF per mg of lint (dry weight after digestion).

Differences in the methodologies may explain the different outcomes from the two studies. For example, we dried out the lint samples before weighting and used a digestion treatment before counting to reduce the mass of non plastic MFs. Also, the LUV-R filter, according to the manufacturer's recommendations, requires cleaning approximately once every 2–3 weeks (10–15 loads of laundry). In our study, samples were collected in average after 7 loads.

This may reduce the optimum capture rate of the filter, as its action is described as « dynamic, meaning that as the filter collects lint, it becomes more efficient and the trapped lint acts as a filter itself » (environmental enhancement). Besides, samples were collected by the households with a flexible protocol regarding collection method. Thus, variations in collection methods may greatly influence the mass of lint collected. Also, the time and diligence devoted to this task might influence the amount of lint recovered.

Both studies were based on samples collected from households having installed external filters. However, the filters used in both studies have different mesh size, and thus capture rates. While it was demonstrated that an important fraction of microfibers (by weight) was removed by LUV-R filter (McIlwraith et al., 2019), efficiency reported in the other study was less significant (Napper & Thompson, 2016).

In our case, the intent was not to assess the efficiency before and after installation of the filter, but rather to estimate the number of microfibers that could be removed from the wastewater by the implementation of an external filter on a domestic washing machine.

Except for this Ontario study that used lint collected under real household laundry conditions, most experiments reporting MF detachments were conducted under simulated laboratory conditions and their applicability to real-life conditions of fiber release during normal domestic laundering remains unaddressed. However, they have the advantage of identifying or isolating specific factors that may affect fiber release. For instance, top loading machines have been reported to be more abrasive on garments as compared to the rotating drum of the front-load machine, causing higher microfibers shedding (Hartline et al., 2016). Moreover, decreasing washing load is reported to increase the release of microfibers (Volgare et al., 2021). The use of a softener during washes reduces the number of microfibres released by more than 35 % (De Falco et al., 2018). Other parameters such as higher temperature, washing time and mechanical action produced an increase of microplastics release (De Falco et al., 2018). Also, depending on their composition and the way they were constructed, textile shed fibers differently (Carney Almroth et al., 2018).

Yet, there is no general consensus on methodologies for quantifying and reporting the results of detached MFs from textile garments, and the degree of similarity between published studies remains limited (Belzagui et al., 2019). The different conditions under which these experiments were conducted, in addition to the diversity of textile properties, load mix, fiber shape and washing conditions limit the interpretation

and generalization of the emission and detachment properties of microfibers during domestic washing.

In our study, lint was collected over several washes, often with a modification of one or a combination of parameters (percentage of filling the washing machine, temperature, washing water volume, textile composition, washing duration and intensity, etc.). As a result, the correlation coefficients between the mass of captured lint and laundry parameters were low. Hence, it was not possible to point out which parameter influences and promotes the release of microfibers. However, positive correlation coefficients of 0.44 and 0.33 respectively for the number of washes and the average wash time demonstrated moderate correlation with the amount of lint retained by the filters. The diversity of machines, collection methods and washing habits were a challenge in this study. This has a direct influence on the lack of correlation between washing habits and the quantification of microfibers found, compared with a controlled laboratory study with a strict protocol.

It is necessary to deepen the knowledge of the average composition of a typical load to reproduce and conduct the washing cycles in the laboratory under well-defined conditions and collection protocols. Key indicators provided in our study about households washing habits regarding laundry parameters can be used as average settings to conduct those laboratory experiments.

Modification of one parameter at a time could be analyzed under these conditions to determine the real impact of each factor, in an effort to understand and reduce further plastic microfiber pollution emitted during household laundry.

3.6. Social acceptability of washing machine filters for the capture of microfibers

While our study demonstrates that the filters can certainly capture a significant amount of microfibers at the household level, it also revealed that their installation and use might be challenging.

A survey of participating households conducted by GRAME about the installation of the filter, its management, and their feedback on the experience, showed that 67.8 % of volunteer participants found the installation difficult and 21.4 % had to hire a plumber to install the filter. Regarding use and maintenance, 39.2 % took less than 10 min to wash their filter. Cleaning the filter was deemed difficult by 71.4 %. Also, 64.2 % reported poor water drainage and clothes still wet after washing. Additionally, 46.4 % reported bad odors from the filter. Only 53.5 % of volunteer participants were planning to continue using the filter after the completion of the project. To encourage households to adopt washing machine filters, several effective strategies could be proposed such as advocating for integrating filters directly into machines to reduce maintenance, or emphasizing designs that minimize user burden and do not solely rely on technical skills. Education efforts should focus on raising awareness about how filters work and their benefits for water quality. Tailored recommendations are crucial, recognizing that not all filters fit every machine or user, which can mitigate issues like load loss and adaptation challenges. By addressing these factors clearly, users could be empowered to manage filters effectively, promoting broader adoption for enhanced water quality and longer-lasting appliances.

This puts into perspective the potential challenges that can be associated with the implementation of washing machine filters on a large scale since the maintenance and use of the filter, in addition to the method of collecting and disposing of the captured lint, strongly influence the efficiency of such a measure.

4. Conclusion

The objective of this project was to characterize the potential of washing machine filters to capture microfibers from domestic wastewater, under real laundry conditions. 30 volunteer participants collected lint from domestic laudry operations during 6 months. Our study provided a suitable digestion reagent to reduce organic matter in the lint samples and an estimation of plastic microfibers that could be diverted by the installation of a washing machine filter under normal household washing conditions. The NaOCl reagent was used for digesting lint samples, based on the degradation efficiency of the organic matter contained in samples and on the resistance of synthetic controls to exposure to reagents given by FT-IR spectroscopy and SEM. The simplicity of implementation, cost and availability were considered in the choice. Quantification of the flow of microfibers retained by LUV-R filters installed in participating households was realised by weighing the lint samples after digestion, and by counting the synthetic microfibers retained on the filters through microscopy. Data collected during the citizen experiment showed an average of 7 washes between two filter cleanings, resulting in one lint sample. The total quantity of wash water used was in the order of 66,600 liters, reducing the amount of raw lint intercepted by the LUVR by 7.44 mg/L of wash. A survey of participating households about the installation of the filter, its management, and their feedback on the experience, showed that 67.8 % of volunteer participants found the installation difficult and only 53.5 % of them planned to continue using the filter after the completion of the project. This puts into perspective the potential challenges that can be associated with the implementation of domestic washing machine filters on a large scale.

CRediT authorship contribution statement

Mohammed Abourich: Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Conceptualization. **Ambroise Bellamy:** Writing – review & editing, Visualization, Validation, Methodology. **Abdellah Ajji:** Writing – review & editing, Supervision, Resources, Conceptualization. **Dominique Claveau-Mallet:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.envc.2024.100984.

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