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The release process of microfibers: from surgical face masks into the marine environment

Francesco Saliu[a,*], Maurizio Veronelli[b], Clarissa Raguso[a], Davide Barana[c], Paolo Galli[a], Marina Lasagni[a]

[a] Earth and Environmental Science Department, University of Milano Bicocca, Piazza della Scienza 1, 20126 Milano, Italy
[b] RDLAB137 srl Via Solari, 32 - 20144 Milano, Italy
[c] Loestrasse 63, 7000 Chur, Switzerland

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ABSTRACT

Due to the Covid-19 pandemic, the use of disposable face masks has been adopted worldwide as a precautionary measure to slow down the transmission of the virus. This has determined an unprecedented rise in the production of these protective equipments, and unfortunately to a new form of environmental contamination due to the improper disposal. To provide a preliminary estimation of the release of microfibers by a surgical mask dumped in the marine environment, we carried out artificial weathering experiments. Results indicated that a single surgical mask submitted to 180 hours UV-light irradiation and vigorous stirring in artificial seawater may release up to 173,000 fibers/ day. Moreover, SEM and micro-FTIR analysis carried out onto surgical masks collected from Italian beaches highlighted the same morphological and chemical degradation signature observed in the masks subjected to the artificially weathering experiments, confirming the risks of a similar microfiber release into the marine environment.

1. Introduction

The ubiquitous presence of plastic in the marine environment is one of the unfortunate signs of our current era (Waters et al., 2016; Efimova et al., 2018). The adverse effect of this form of pollution has been widely investigated in the recent literature (Browne et al., 2008; Cole et al., 2014; Fossi et al., 2012; Gall & Thompson, 2015, Galloway et al., 2017; Rist et al., 2018; Saliu et al., 2021; Wright et al., 2013; Taylor et al., 2016).

After the outbreak of the COVID-19 pandemic (Elachola et al., 2020), disposable face masks have turned from a specific tool designed for healthcare professionals (National Institute for Occupational Safety and Health (NIOSH) 2020) to a generalized measure to mitigate the propagation of the virus (World Health Organisation (WHO) 2019; Coronavirus disease (COVID-19) 2021; Esposito et al., 2020). As consequence, common people who do not have proper skills regarding handling and disposal has started to use masks for everyday life, and this has led to an unprecedented increase in production, estimated between 2,4-52 billion pieces in the 2020 (Eurorostat 2021; Phan & Ching, 2020; Prata et al., 2020) that is added into the global plastic production estimated at 368 million ton in 2019 (PlasticEurope, 2020(Plastics - the Facts 2019), causing around 8 million tonnes of plastic lost from the world's coast annually (Jambeck et al., 2015).

Unfortunately, many surgical masks wasted have already turned into a new environmental threat for the marine environment, since their presence in the marine litter have already been reported by several environmental organizations *i.e.* the World Wildlife fund (WWF), Greenpeace, and Marevivo (COVID-19 2021).

In the scientific literature, the possible risks posed by face masks as a source of microplastics and/or nanofibers have already been highlighted by several authors (Okoffo & Tadele, 2020; Aragaw 2020, Fadare et al., 2020). The need to carry out research to assess the comprehensive data on their abundance and thorough estimation of the related environmental risks, comprised possible impacts onto marine wildlife, was also highlighted (Aragaw, 2020).

At the present time, no data regarding the number of microfibers released by the surgical masks into the marine environment are available. For this reason, here we provide a first and prelimary evaluation, obtained by submitting commercially available surgical masks to artificial aging experiments, including UV irradiation and application of mechanical stress into artificial seawater. Moreover, we analyzed these samples together with masks collected from Italian beaches by means of infrared spectroscopy (IR) and scanning electron microscopy (SEM), aiming to provide comparison of the degradation extent with.

* Corresponding author.
E-mail addresses: francesco.saliu@unimib.it (F. Saliu), davide.barana@hotmail.it (D. Barana).

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Fig. 1. Pictures from the experimental procedure. From the left to the right: (a) a surgical mask after prolonged UV light exposition (b) surgical mask stirred inside a bottle filled with artificial seawater solution (c) appearance of the microfibers "soup" on the top of the artificial seawater solution at the end of the artificial weathering treatment.

2. Materials and methods

2.1. Samples

Seven surgical masks (from *SM1* to *SM7*) produced by different vendors and found available to be purchased from Italian supermarket, from a local pharmacy, and through an online shopping platform, were submitted to the tests. Table 1 reports the list of the masks and the main information regarding their composition and certification (as provided by the vendors). Additionally, we collected infrared spectra from the nose strip and for each of the three layers of every single mask (two replicates from each layer) to confirm the declared composition. Five masks (from *SM8* to *SM12*) sampled from an Italian beach were also analyzed also (location 39°08'4.88" N 8°17'53.30" E, Sardinia, Italy).

2.2. Artificial aging

Artificial aging experiments were carried out by submitting the selected masks to UV light expoition and by soaking in artificial seawater under stirring. The intent was to simulate the combined weathering effect of sun irradiation and the mechanical solicitation caused by breaking waves in the surf zone (Fig. 1). More specifically, two set of different experiments were carried out in order to mimic both "gentle" and "extreme" weathering condition: (a) soaking of the surgical mask in artificial seawater for a prolonged time and without UV irradiation (in this case motion was applied by using a orbital shaker (b) exposition of the surgical mask to UV light and then to turbulent flow generated by magnetic stirring inside a glass bottle filled with artificial seawater.

2.2.1. UV-light exposition

The samples were subjected to UV irradiation / condensation cycles following the indication of ISO 4892-3 which "specifies methods for exposing specimens to fluorescent UV radiation, heat and water in apparatus designed to simulate the weathering effects that occur when materials are exposed in actual end-use environments to global solar radiation, or to solar radiation through window glass" Specifically, irradiation was applied by using a UV-A lamps (340 nm; 0,76 $W{\cdot}m^{-2}{\cdot}nm^{-1}$ for 10 hours at temperature of 65°C. This ster was alternated with a condensation step of two hours at 50°C with light switched off. This entire cycle was repeated 15 times, for a total exposure time of 180 hours.

2.2.2. Seawater exposition and mechanical stress

Each surgical mask previously subjected to UV aging was placed inside a 1 L glass bottle already provided with a magnetic stirring bar.

Table 1
Description of the surgical masks submitted to the artificial weathering experiments. The description is based on the indication provided by the vendor.

Sample	Weight (mg)	Description
SM1	4334	Disposable surgical mask. Class I medical device, EN14683:2019 Type IIR BFE≥98%. TNT polypropylene without glass fiber, heat-sealed, hypoallergenic, nonsterile, 3-ply, 70% spunbond, 30% melt blown fabric
SM2	4237	Disposable surgical mask non-sterile,3-ply with earloop Type 1, EN14683:20019, china standard YY/t0969-2013,
SM3	3792	Disposable surgical mask non-sterile,3-ply with earloop directive 93/42/EEC Annex v certificate EN 149:2001+A1:2009. CLASS I > 95% - filtration (AORN 95%). 1° layer 25g/m2 - Spunbond PP 2° layer 25g/m2 – PP melt blown (filtration are) 3° layer 25g/m2 - Spunbond PP .
SM4	4873	Class I Disposable surgical mask non-sterile,3-ply with ear-loop. EN14683 Type II
SM5	3907	Type II surgical mask in polypropylene. Disposable. Non-woven. GMP, CE, FDA, EN 14683
SM6	4305	Disposable Medical Surgical Mask 3-ply, non-woven with CE (Regular CE certification, China export white list) Executive standard: EN 14683:2019 Type IIR. Material: 25 g non-woven + 25 g fabric Melt blown cloth + 25 g non-wove; Sonic welded
SM7	4145	three layers of soft non-woven polypropylenes that prevent the inhalation or exhalation of respiratory droplets. Polypropylene spunbond and meltblown nonwoven fabrics. Passed YY/T0969-2013 standard for single-use face mask

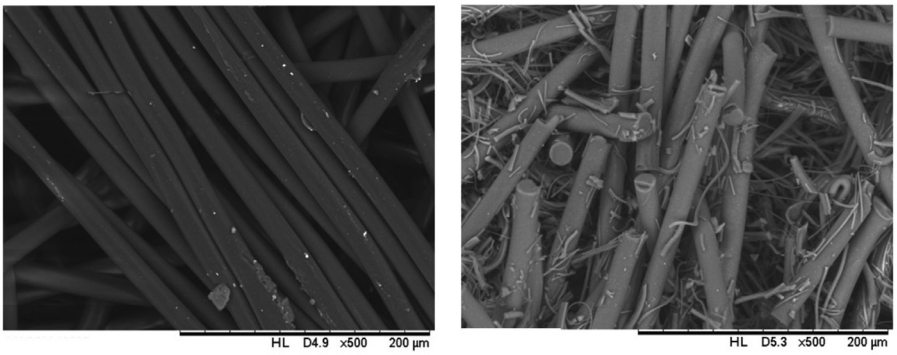

Fig. 2. SEM images of surgical mask *SM3* taken before and after application of 180h UV irradiation treatment.Scale bars were re-drawn for clarity. The imagines show fibers, fragments, and particles in micro and nanosized ranges that are abundant and loosely attached to the products' structural fibers. After the application of the aging procedure cracks and crazing are also evident.

Artificial seawater, prepared following the indication of ASTM D1141-98, was poured into the bottle until filled. The bottle was then sealed with a polyethylene screw cap (DIN GL45), and the whole system was placed under magnetic stirring at 4000 rpm for 24h at room temperature.

2.3. Microplastic analysis

At the end of the seawater exposition test, the mask was gently extracted from the bottle. Then the artificial seawater solution was sieved through 500 μm stainless steel mesh and filtered through a Whatman nitrocellulose filter (pore size 0.45 μm, Ø 4.5 cm) by using a glass filtration apparatus attached to a vacuum pump, model V600 provided by Buchi (Buchi Italia srl, Cornaredo, Italy). The filters were left to dry for 24 h inside an aluminum protective case to avoid contamination with airborne impurities and then submitted to particle counts. Plastic fibers fragments and aggregates deposited onto the surface of the sieve and visible by the naked eye were retrieved by using tweezers and placed onto stainless steel filters DIN ISO 9044 (Ø =2.5 cm, 25*25 μm) provided by PACO filters (Paul GmbH & Co. KG, Postfach, Germany). Infrared microscopy was then carried out to confirm the polymeric origin of the collected aggregates and to determine the chemical degradation that occurred due to the weathering (for this confirmation we analyzed one fiber every 25 counted fibers). The filters obtained at the end of the treatment were firstly observed at the stereomicroscope to check for the homogenous distribution of the fibers. Then the total number of fibers was determined according to the method described by De Falco et al. (2018). Infrared spectra were collected by considering a 10*10 mm filter portion as described in previous paper (Saliu et al., 2018).

2.4. Instrumental

SEM images (Fig. 2) were obtained by using a HITACHI TM3030 Plus instrument with a backscattering detector. Samples of pristine and UV-A treated surgical masks were cut into approximately 5 mm × 5 mm pieces. The specimens were mounted on aluminum stubs using carbon tape. The weathered surfaces were analyzed directly (without coating) at 15 kV at a magnification of 500x.. Infrared analysis were carried out by using a Perkin Elmer Spotlight 200i instrument (Perkin Elmer, Milano, Italy) equipped with a diamond "attenuated total reflectance' (ATR) unit and liquid nitrogen cooled mercury cadmium telluride (MCT) single detector was used to record spectra in the wave-number range 4000–400 cm^{-1} with a resolution of 4 cm^{-1} and 32 co-added scans. A background measurement against air was recorded prior to each sample measurement using the same acquisition set up. Patented COMPARE^{TM} spectral comparison algorithm (Perkin Elmer) was used for spectral searching in the commercially available library.

Table 2
Release of microfibers obtained after ten day soaking of surgical masks into artificial seawater. Values indicates the number of microfibers identified onto nitrocellulose filter after filtration of the artificial seawater solution.

Sample	Microfibers
SM1	403
SM2	297
SM3	445
SM4	387
SM5	411
SM6	396
SM7	447
Mean	398
SD	46

3. Results

3.1. Microfibers counts

The counts related to the microfibers realease from the unaged surgical masks soaked into artificial seawater solution are reported in Table 2. The experiments showed that after ten days of exposition, no fragments neither fiber aggregates were present on the sieved fractions, whereas an appreciable number of microfibers was collected after filtration. Specifically. the highest number of fibers released by a single mask was 447, while the minimum amount was 297. Overall, the mean value for the seven masks was 398 fibers with an SD of 46. These numbers were considerably higher than the limit of quantification of 7 PP determined from replicated blank runs (by considering the average of the blanks plus six times their standard deviation). This background contamination was most probably due to the widespread use of surgical masks in the daily life (for instance, we were all asked to access the university buildings and labs by wearing the masks). The second set of experiments carried out by submitting the surgical masks to accelerated aging and mechanical stress into seawater showed overall a larger release of material (compared to the unweathered masks) In this case, a very large number of fiber aggregates and fragments resulted already visible by naked eye after one day of seawater exposition, floating on the top of the water solution in the notorious form of a"plastic soup"(see Fig. 1). Hence, these large aggregates were sieved and collected from the sieve surface by using tweezers to perform counts and characterization. Results showed a maximum of 82 fiber aggregates for mask *SM3* and a minimum of 29 for mask *SM2*. After that, the sieved solution was left to settle and a fraction (1/20 in volume) of the supernatant was filtered through a 47 mm diameter filter to perform the counts of the particles under the 500 µm size range. This procedure was followed to ensure that an optimal quantity of fibers was deposited onto the filter for per-

Table 3
Release of microfiber from surgical mask observed after application of 180 h UV-light irradiation and 24 h stirring into artificial seawater. More detail regarding the experimental procedure are available in the experimental section of the paper.

Sample	1–5 mm (number of aggregates)	500–25 μm (number of fibers)	Total mass (mg)	Mass loss (%)
SM1	43	78,000	10.8	0.25
SM2	29	55,000	7.4	0.17
SM3	81	173,000	10.4	0.26
SM4	71	110,000	6.3	0.12
SM5	67	95,000	8.7	0..23
SM6	58	67,000	4.8	0.09
SM7	79	142,000	11.2	0.26
Mean	61	117,400	8.0	0.19
SD	19	42,345	2.7	0.07

form the estimation (avoiding accidental superimposition and collecting a reasonable number of fiber to be counted). Mass measurements were then carried out by filtering the remaining portion of the supernatant solution onto a different filter, previously weighted with analytical balance (E 50/s3, Gibertini, Italy). Results (Table 3) showed an average of 135,000 particles released per mask (RSD = 24%). The maximum amount was 173,000 registeted from surgical mask *SM3*. Mass analysis indicated an average mass loss of 0.19% estimated for the seven surgical masks submitted to the artificial aging procedure.

3.2. SEM analysis

Fig. 2 shows the SEM micrographs of surgical masks before and after the exposition to the UV-A aging cycles. Before the treatment, the outer layer's fibers appear to be intact and display a diameter of *ca* 15–20 microns. No signs of degradation or fracture were noticeable, considering also that a fraction of powder residues may come from background contaminations. After application of UV irradiation, the pronounced damage caused to the fibers structure due to the photo and thermal degradation of the material was instead clearly observable (right side of Fig. 2). Moreover, a large amount of smaller fiber fragments, presumably originating from the meltblown inner layer's cracking, was clearly visible along the entire surface of the observed sample. Finally, the SEM inspection of the surgical mask collected from local beach showed smaller fiber fragments and crakings similar to those observed in the artificially weathered masks (Supplementary, Fig. S7)

3.3. MicroFTIR analysis

Analysis by MicroFTIR carried out on the fibers sampled form all the three fibrous layers used to fabricate the masks showed signs of oxidation. These signs were specifically detected by considering the broad peak around 3400 cm^{-1}, corresponding to hydroxyl groups and to multiple overlapping carbonyl peaks centered around 1720 cm^{-1} that may originate from γ-lactones (1780 cm^{-1}), esters and/or aldehydes (1733 cm^{-1}), ketones (1714 cm^{-1}), and carboxylic acids (1700 cm^{-1}), that are known to be formed during the radical chain oxidation process. A similar chemical signature of the degradation process was found in the "environmental" surgical mask (Fig. 3, C) collected from an Italian beach. Carbonyl peak index values calculated accordingly to the indication in Almond et al. (Almond et al. 2020) ranged from 0.2 to 0.6 depending of the mask and the fiber aggregates analyzed (Supplementary, Table S9).

Fig. 4.

3.4. Discussion

Overall, the experiments here reported showed that a single surgical mask exposed to weathering may severely degrade and release massive amounts of microfibers in the seawater (that can even be easily seen by naked eye). Polypropylene, the polymer used for the surgical

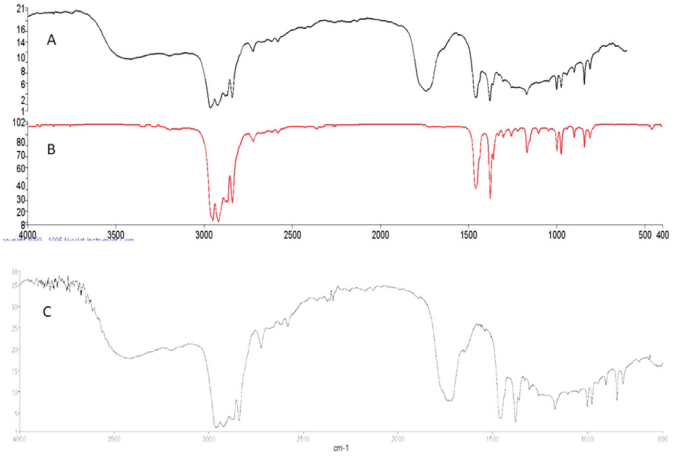

Fig. 3. infrared spectra collected from fibers isolated from surgical mask (A) after the application of artificial weathering experiment. (B) before the application of artificial weathering experiment (C) from an surgical mask collected from a local beach.

mask fibers fabrication, is known to display low photooxidative stability (Andreassen, 1999), and it is susceptible to oxidation in the presence of air (Almond et al., 2020). The UV-A (315-400 nm) portion of the ultraviolet radiation which is emitted from the sun, and reach the ground with minimal losses may interact with defects and impurities such as hydroperoxides, carbonyls, instaurations, and catalysts residues onto polypropylene surface that act as chromophores: as a result, the polypropylene activation spectrum exhibits few maxima in the spectral region (Tocháček & Vrátníčková, 2014) evenf if, independently from the molecular weight, paraffinic hydrocarbons should not absorb radiation in the UV-A range. In addition, the excited chromophores promote the photooxidative degradation of the polymer chains, ultimately leading to the breakage of the material (LaCoste et al., 1993). More specifically, free radicals generated in the initiation stage react with other macromolecular backbones causing chain cleavage and crosslinking reactions, with the former dominating the latter (Mylläri et al., 2015). These changes at the molecular level are responsible for a loss of mechanical properties favoring the material's degradation at the macroscopic level. When radicals are extinguished in termination reactions, they form different oxygenated species, including several carbonyl containing groups (e.g., ketones, esters, etc.) that can be traced by FTIR spectroscopy, observing the changes in the carbonyl band (1850-1650 cm^{-1}) and evaluating the related carbonyl index (CI) as described by Almond et al. (Almond et al.2020).

In our specific case, the seven masks submitted to the articial weathering showed CI values ranging from 0.3 to 0.5 (Supplementary, Table 9). Since the correlation between laboratory and natural weathering test results has long been a source of debate and controversy

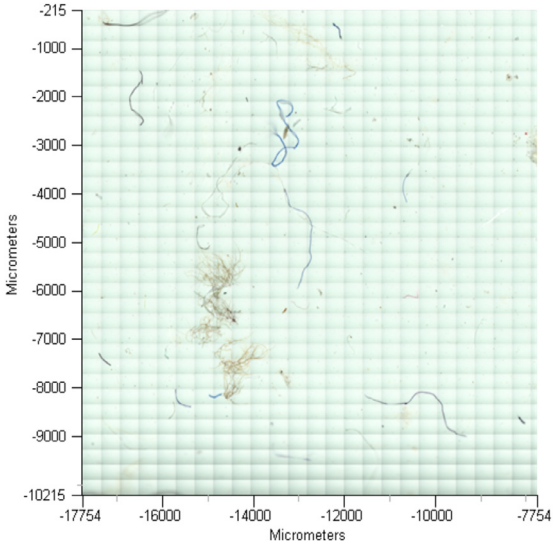

Fig. 4. 10 × 10 mm section of a filter used for the filtration of artificial seawater exposed to a surgical mask. Fibers are uniformly distributed and not superimposed, enabling counts and verification of the polymeric composition by infrared microspectroscopy.

(Fedor&Brennan, 1996), conditions may by highly variable and it is widely accepted that no single test or device can reproduce all the variables that may be found in different climates, altitudes and latitudes; we compared these values with the values obtained by analyzing five masks sampled from Italian beaches. Results showed values ranging from 0.1 to 0.4 (Supplementary, Table 10). As indicated by previous authors, these values are a clear sings of embrittlement and dramatical change in the tensile strength of the original material, since elongation of break may be reduced from 400% to 50% (Rajakuma et al., 2009). In the artificial surgical masks, this deterioration of the materials resulted clearly recognizable by SEM analyses: an extensive fragmentation of the original fibers following the weathering process was in fact observed (Supplementary, Fig. S6-S8). On the other hand, similar signs of degradation were observed also on the masks collected from the beaches, confirming that our weathering procedure provided a reliable description of what really occurs outdoor under prolonged sunlight expostion. This is in agreement also with recent findings related to general mechanism of microplastic formation, which highlight how common polymers get initially degradated mostly in the terrestrial environment (land and/or on the beach surface) due to the exposition to the UV radiation (Andrady, 2011; Duis and Coors, 2016) that makes the plastic material brittle and easily subjected to fragmentation. Other factors that may facilitate plastic's chemical degradation are indicated to be the exposition to high temperature, thaw-freeze cycles, and microbial attack (Andrady, 2011; Moore, 2008). These mechanisms are absent or less effective in water, where the chemical degradation is much slower (Shah et al., 2008; Barnes et al., 2009). However, in the sea, mechanical degradation is enhanced due to the abrasion induced by sediments and the fragmentation occurring at the sea swash and wave breaking zone (Efimova and Chubarenko, 2018), especially during a stormy event (Shah et al., 2008; Chubarenko et al., 2016). Thus, the combination of the chemical and mechanical degradation ultimately leads to the formation of microplastics. In our experiments, the mechanical stress on the UV exposed masks was simulated by the vortex induced by the circular motion of the stirring bar. Is it is plausible that the obtained results may still represent an underestimation of the actual release of fibers taking place in the "real" marine environmental conditions: even if the turbulence applied was high, there was no simulation of the abrasion induced by contact with sediments that normally occur in the marine environment, and that was instead taken in account in previous works devoted to study the formation of microplastic from larger plastic items (Efimova et al., 2018; Jungnickel et al., 2016). Our intent was in fact to perform a preliminary evaluation of the degradation of the masks due to the exposition to sunlight and high temperature (as it may happen when a surgical mask is dumped in the beach in warm areas). This exposition may be prolonged for several weeks before the same masks is moved forwards into the marine environment, where is subjected to the mechanical stress induced by the waves, and where the mayor release of the fragmented microfibers may take place.

According to these premises, and because we observed that the artificial weathering caused average mass loss of 0.2% it is plausible that a mask dumped at the beach may completely degrade into microscopic fiber fragments and aggregates in less than two years (further experiments carried out at prolonged time scale are needed to verify this assumption). Since the global production of facial masks for 2020 is estimated between 2.4 and 52 billion pieces (Panda et al., 2020; OceanAsia, 2020), corresponding to approximately 7200–312,000 ton, and it can be estimated that 1-10% are released in the environment (as for other single use plastic materials), this accounts potentially from 72 to 31,200 tons of microplastic waste adding up in to 2020 to our oceans.

4. Conclusion

Single use surgical masks have become in some way the standard of protection during the COVID-19 pandemic. The main reason is that they are very practical: they are lightweight, flexible, and comfortable to wear, offering a right balance between filtration efficiency, cost, and ease of breathing. However, the staggering increase in the number of disposable masks being used globally on a daily basis exacerbates the environmental risk connected to improper disposal. As shown by the preliminary results outlined in this paper, a surgical mask has the potential to release in the marine environment thousands of microscopic fibers. Action is therefore urgently needed to limit the amount of discarded surgical masks reaching the marine ecosystem, *i.e.,* campaigns to promote the correct disposal and improvements in managing these new waste streams. Furthermore, research efforts may provide more eco-friendly alternatives based, for example, on biodegradable components or higher quality solutions that can be used over extended periods.

Declaration of Competing Interest

The authors declare no competing interest.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.envadv.2021.100042.

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