Environmental Pollution 348 (2024) 123792

Contents lists available at ScienceDirect

Environmental Pollution

journal homepage: www.elsevier.com/locate/envpol

Disposable surgical/medical face masks and filtering face pieces: Source of microplastics and chemical additives in the environment☆

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ARTICLE INFO

Keywords:
MPs
Microfibre
Fragment
Plastic pollution
Bisphenols
Pandemic

ABSTRACT

The production and consumption of disposable face masks (DFMs) increased intensely during the COVID-19 pandemic, leading to a high amount of them being found in the terrestrial and aquatic environment. The main goal of this research study is to conduct a comparative evaluation of the water-leachability of microplastics (MPs) and chemical additives from various types of disposable surgical/medical face masks (MM DFMs) and filtering face pieces (FFPs). Fourier-Transform Infrared Spectroscopy was used for MPs analysis. Liquid Chromatography/High Resolution Mass Spectrometry was used to analyse analytes presented in the water-leachates of DFMs. FFPs released 3–4 times more microplastic particles compared to MM DFMs. The release of MPs into water from all tested DFMs without mechanical stress suggests potential MP contamination originating from the DFM production process. Our study for the first time identified bisphenol B (0.25–0.42 μg/L) and 1,4-bis(2-ethylhexyl) sulfosuccinate (163.9–115.0 μg/L) as leachables from MM DFMs. MPs in the water-leachates vary in size, with predominant particles <100 μm, and the release order from DFMs is MMIIR > MMII > FFP3>FFP2>MMI. The main type of microplastics identified in the water leachates of the investigated face masks was polypropylene, accounting for 93–97% for MM DFMs and 82–83% for FFPs. Other polymers such as polyethylene, polycarbonate, polyester/polyethylene terephthalate, polyamide/Nylon, polyvinylchloride, and ethylene-propylene copolymer were also identified, but in smaller amounts. FFPs released a wider variety and a higher percentage (17–18%) of other polymers compared to MM DFMs (3–7%). Fragments and fibres were identified in all water-leachate samples, and fragments, particularly debris of polypropylene fibres, were the most common MP morphotype. The findings in this study are important in contributing additional data to develop science-based policy recommendations on the health and environmental impacts of MPs and associated chemical additives originated from DFMs.

1. Introduction

The production and consumption of disposable face masks (DFMs), also known as single-use face masks, increased intensely during the COVID-19 pandemic, creating new challenges for local authorities due to the increasing volume of Personal Protective Equipment (PPE) waste (Selvaranjan et al., 2021; Benson et al., 2021; Roberts et al., 2022; Tesfaldet and Ndeh, 2022). During the COVID-19 pandemic, the World Health Organization (WHO, 2020) estimated a requirement of approximately 89 million medical DFMs every month for healthcare settings. Additionally, Prata et al. (2020) reported a global usage of about 129 billion DFMs per month during the same period.

Disposable face masks, classified as a type of PPE, serve as an effective barrier, providing highly effective filtration for particles and droplets emitted while coughing, sneezing, or speaking. DFMs help to limit the spread and reduce the risk of transmission of bacteria and virus (Chu et al., 2020; Barycka et al., 2020; Farzaneh and Shirinbayan, 2022). Table S1 in the Supplementary Material provides a description of common DFM types, typically made of polypropylene (PP), but other polymers such as polyethylene (PE), polyamide (PA)/Nylon, polystyrene (PS), polyester/polyethylene terephthalate (PET), polycarbonate (PC), polyphenylene oxide (PPO), and chlorotrifluoroethylene (CTFE) are also used in mask production (Akber Abbasi et al., 2020; Farzaneh and Shirinbayan, 2022). Masks consist of several layers, including filtering layers with a pore size of 1 μm or less, produced from conventional fabrication fibres (e.g., micro- and/or nano-fibre) using generally the

☆ This paper has been recommended for acceptance by Eddy Y. Zeng.

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https://doi.org/10.1016/j.envpol.2024.123792
Received 5 January 2024; Received in revised form 24 February 2024; Accepted 12 March 2024
Available online 20 March 2024

Spunbond-Meltblown-Spunbond (SMS) technology (Chellamani et al., 2013; SalehHudin et al., 2018; Leung and Sun, 2020). For instance, Jung et al. (2021) determined the weight percentage (wt.%) of all materials used for a typical filtering face piece (FFP) mask: PP (outer and inner layer; 73.33 wt%), PE (middle layer; 13.77 wt%), PA/Nylon (elastic cord; 8.27 wt%), and metal (pliable nose clip; 4.63 wt%). The main types of DFMs are as follows (Table S1): 1) surgical/medical masks (MM; Type I, Type II, and Type IIR); and 2) filtering face piece (FFP), also called respirator masks (FFP masks - N95 (US standards), FFP1, FFP2, and FFP3 (EU standards)). Both surgical/medical masks and filtering face pieces protect against transmitted droplets of infectious agents. Generally, closer-fitting FFP masks provide better protection against airborne pathogens compared to surgical/medical masks (Table S1). Additionally, FFPs can prevent inhalation of potential health hazards of dust, smoke, mist, vapor, and gases. The particular type (e.g., Type I, Type II, Type IIR, N95, FFP2, and FFP3) of DFMs must conform to certain minimum filtration standards (Table S1; BS EN 149, 2001+A1, 2009; ASTM F2100, 2007; BS EN 14683, 2019; BS EN ISO 15223-1, 2016). Based on the US standards from the National Institute for Occupational Safety and Health (NIOSH, US), there are several classes of FFPs depending on the degree of oil resistance: N - no oil resistance, e.g., N95, N99 and N100. The number after the letter indicates the percentage of filtration of suspended particles; R - mask resistant to oil for up to 8 h (e.g., R95, R99 and R100); P - a completely oil-resistant mask (e.g., P95, P99 and P100). FFPs such as N95, FFP2 and FFP3 can protect people against airborne infectious agents such as pathogens and viruses. Unfortunately, DFMs are presently non-recyclable through conventional routes and frequently encounter improper disposal, occurred extensively during the COVID-19 pandemic (Roberts et al., 2022; Oliveira et al., 2023; Hasan et al., 2023). The main areas of the littered DFMs were defined as street lanes, sidewalks, footpaths, parking areas, street gutter, waterways, parks, beaches, rural areas, etc. (Fig. S1 – this study; Aragaw, 2020; Shiferie, 2021; Tesfaldet et al., 2022; Amuah et al., 2022). Benson et al. (2021) indicated that around 3.4 billion of DFMs were disposed globally on a daily basis during the COVID-19 pandemic. Therefore, according to the research by Benson et al. (2021), it can be estimated that approximately 4.3 million tonnes of unrecyclable contaminated plastic waste of DFMs would be generated worldwide within a year. Roberts et al. (2022) analysed littering behaviour and the proportional composition of COVID-19-related litter (e.g. PPE waste including DFMs) for 14 months (September 2019–October 2020) for 11 countries (Australia, Belgium, Canada, France, Germany, the Netherlands, New Zealand, Spain, Sweden, the United Kingdom and the United States) using the open database Litterati on the litter collection (Litterati, 2020). Roberts et al. (2022) demonstrated that the quantity of DFMs discarded in the environment experienced a substantial exponential rise from March 2020 to October 2020 in response to the COVID-19 pandemic (an increase of nearly 9000%). A significant presence of DFMs has been reported recently in both terrestrial and aquatic environment (Fadare and Okoffo, 2020; Sangkham, 2020; Parashar and Hait, 2021; Amuah et al., 2022; Hasan et al., 2023; Fig. S1). Akber Abbasi et al. (2020) and Selvaranjan et al. (2021) have also noted the increase of the DFM wastes consisting of PP, PS, PE, PC, polyurethane (PU), and polyacrylonitrile (PAN), during the COVID-19 pandemic. Based on the population of coastal regions from 46 countries, Chowdhury et al. (2021) estimated that 0.15–0.39 million tonnes of discarded DFMs could reach the ocean annually. Those wastes can generate additional microplastics (MPs) pollution to the environment, which is one of a key global concern (Jambeck et al., 2015; Geyer et al., 2017; Schnurr et al., 2018; Akhbarizadeh et al., 2021; Shukla et al., 2022; Jiang et al., 2023b). DFMs can get to the environment through littering at public and countryside spaces (Fig. S1), disposal in landfill and dumpsites, and subsequently transporting to the terrestrial and aquatic (freshwater and oceans) environments (e.g., runoff, flooding, atmospheric deposition) (Hasan et al., 2023; Jiang et al., 2023b). Researchers have expressed concern over the improper disposal of used DFMs, such as littering, as it can have serious impacts on infrastructure and the environment (Hu et al., 2022; Hasan et al., 2023; Jiang et al., 2023b). Improperly discarded masks can block drains and sewage systems, potentially leading to pollution of soils and water systems like rivers, lakes, streams, and oceans. Moreover, they can also harm wildlife (e.g., living organisms might be entangled or trapped in DFMs; consumed plastic debris can damage organs), and leach toxic contaminants, including emerging pollutants, into the environment (Hasan et al., 2023; Oliveira et al., 2023; Jiang et al., 2023b). Additionally, infectious DFMs can release and spread pathogens such as bacteria, viruses, parasites, or fungi. Studies by Aragaw (2020), Shiferie (2021), Selvaranjan et al. (2021), Roberts et al. (2022), and Tesfaldet et al. (2022) have also highlighted those concerns. DFMs can be disintegrated into smaller particles like MPs (particle size <5 mm) through various physical, chemical, and biological processes (Akdogan and Guven, 2019; Selvaranjan et al., 2021; Oliveira et al., 2023; Jiang et al., 2023b) and can potentially be an additional source of MPs in the environment.

In the last three year, researchers have reported on the release of MPs and some chemical additives into water from DFMs (Table S2; Jiang et al., 2023b; Oliveira et al., 2023). Generally, a whole new or used DFM was immersed in water with shaking/agitation/rotation for a certain time period (from 4 h to 30 days) with subsequent filtration and drying steps before MP analysis (Table S2). Some researchers extracted MPs from the individual layers (Ma et al., 2021; Rathinamoorthy and Balasaraswathi, 2022) and small pieces (Wu et al., 2022; Wang et al., 2021) of DFMs, as well as from DFMs without the metal nose strips and the elastic ear loops (Dissanayake et al., 2021; Morgana et al., 2021). Different instrumental techniques were used to perform qualitative and quantitative analysis of the released microplastics/nanoplastics (MPs/NPs) and chemical additives from DFMs (Table S2; Jiang et al., 2023b). The majority of published studies have focused on the leachability of MPs/NPs and the transformation of DFM materials during physical (e.g., mechanical forces - agitation simulating wave action and sand abrasion; UV-light radiation; temperature effect) and chemical (e.g., effect of pH and different water compositions – freshwater, seawater, soil water) weathering processes (Table S2). The published studies have shown that DFMs can release MPs when submerged in water, particularly after undergoing various weathering processes (Table S2; Jiang et al., 2023b; Oliveira et al., 2023). The number of MPs released into deionised water (DI) from the entire DFM ranged from 28 MPs/DFM (Liu et al., 2022) to 5.02×10^4 MPs/DFM (Celik, 2023), which could be attributed to differences in the experimental leachability procedures/conditions, as well as variations in the application of different instrumental analysis and quantification methods (Table S2). Additionally, the inconsistency in the main quantitative characteristics, such as different units for MP concentrations such as mg/L, MPs/DFM, and MPs/cm^2 (Table S2), makes comparisons between studies challenging. It was observed that DFMs (MM and N95) released a higher quantity of NPs compared to MPs into water (Ma et al., 2021; Morgana et al., 2021). Some published studies have also focused on understanding the release of MPs from the individual layers of DFMs, mainly MM and N95 masks (Ma et al., 2021; Wu et al., 2022; Rathinamoorthy and Balasaraswathi, 2022). Wu et al. (2022) highlighted that the quantity of MPs released from different layers follows this order: middle layer > inner layer > outer layer. The published studies were in agreement that the release of MPs into water from DFMs increased significantly under various simulated environments and weathering conditions (Table S2; Jiang et al., 2023b). However, there is no general consensus in the published studies regarding the water-leachability of MPs from different DFMs (Table S2). For example, Chen et al. (2021), Jiang et al. (2022a), Liang et al. (2022), and Celik (2023) found no significant difference in the MP release into water among the MM and N95 types of DFMs. This contradicts the results of Delgado-Gallardo et al. (2022) and Ma et al. (2021), who reported that FFPs (e.g., N95, FFP2) released more MPs than MMs (e.g., MMI, MMIIR). Additionally, Dissanayake et al. (2021), Liu et al. (2022), Wu et al. (2022), and Gupta et al. (2023) reported opposite findings (Table S2). Therefore, further studies are essential in this area.

Face masks can also contain plastic additives, such as organic chemicals, dyes, and heavy metals, that can leach out into the environment (Pizarro-Ortega et al., 2022; Bussan et al., 2022; Oliveira et al., 2023). As summarised in Table S2, several studies have shown that masks, mainly the MM types, could release plastic additives into water, such as phthalate esters, ammonium perchlorate (AP), benzothiazole, di-tert-butyl peroxide (DTBP), bisphenol-A (BPA), phthalide (Liu et al., 2022), and heavy metals (e.g., Pb, Zn, Cu, Cd, Sb, Cr, Co, Ni) (Sullivan et al., 2021, 2021; Li et al., 2022; Delgado-Gallardo et al., 2022). Also, several researchers conducted screening analysis of the water-leachates to identify potential releases of the following chemicals from DFMs, mainly of the MM type: acetophenone (AP), 2,4-di-tert-butylphenol (DTBP), and bis(2-ethylhexyl) phthalate (Liu et al., 2022); surfactant molecules (e.g., Triton X), polyethylene glycol (PEG), dye-like molecules, polyamide-66 monomer, and oligomers (nylon-66 synthesis) (Sullivan et al., 2021; Delgado-Gallardo et al., 2022). Further research is still needed to understand the leachability of potentially toxic organic compounds from different types of DFMs.

To summarise, there is still a lack of knowledge and consistency in the published data regarding comparative studies on understanding the water-leachability of MPs and chemical additives from different types of DFMs. Additionally, it is pertinent to investigate whether MPs, possibly generated during the non-woven FM production process, are present in newly produced DFMs and, therefore, can be easily released into water without undergoing mechanical, chemical, or biological weathering. Therefore, the main goal of this research study is to conduct a comparative evaluation of the water-leachability of MPs and chemical additives from various types of disposable surgical/medical face masks (MMI, MMII, and MMIIR) and filtering face pieces (FFP2 and FFP3). This research is important in contributing additional data to develop science-based policy recommendations on the health and environmental impacts of microplastics and associated chemical additives originated from the DFMs.

2. Materials and methods

The laboratory work was conducted at the High-performance Analytical Hub of the Centre for Agroecology, Water and Resilience at Coventry University in the United Kingdom. Figure S2 illustrated the main steps taken in the experimental work and analysis.

2.1. Materials and chemicals

The main types of DFMs used for the water-leaching experiments were as follows (Table S1; Section S1): 1) Surgical/medical masks (MM) – MMI (Type I), MMII (Type II), and MMIIR (Type IIR - fluid repellent)); and 2) Filtering face pieces (FFP) – FFP2 (N95) and FFP3. The weight of used DFMs was: 3.89 ± 0.035 g for MMI, 3.25 ± 0.015 g for MMII, 2.765 ± 0.007 g for MMIIR, 5.775 ± 0.17 g for FFP2, 15.609 ± 0.11 g for FFP3 (n = 3). All DFMs were commonly used during the COVID-19 period and were purchased from UK pharmacies from different suppliers.

Reagents and chemicals used in this study were: Water, Optima™ LC/MS grade Water (Fisher Scientific); Methanol, Optima™ LC/MS grade (Fisher Scientific); Ammonium Acetate, Optima™ LC/MS Grade, (Fisher Chemical) and Formic Acid, 99.0+%, Optima™ LC/MS Grade (Fisher Chemical). Bisphenol B (BPB), ≥98.0% (HPLC) (Supelco), bisphenol A-d16, 98 atom %D (Sigma-Aldrich), and 1,4-bis(2-ethylhexyl) sulfosuccinate sodium salt, 96% (Sigma-Aldrich).

2.2. Sample collection and preparation

The one-flash water-leaching experiments without mechanical agitation were carried out for this research. The whole new DFM of each type was placed in a prewashed 200 mL glass beaker containing 150 mL of Optima™ LC/MS grade water (Fisher Scientific). To prevent microplastic contamination, all glass beakers were covered by aluminium foil. Then glass beakers were left without agitation at room temperature for 24 h. All water-leaching experiments were performed in triplicates. Each water-leachate was filtered through a membrane filter with a pore size of 0.2 μm and a diameter of 13 mm (AnoDisc™ 13, Whatman, GE Healthcare life sciences, Germany) using a prewashed vacuum-enhanced glass filtration system. The filter was then transferred to a prewashed glass Petri dish to dry at room temperature, and kept in it until Fourier-Transform Infrared (FTIR) analysis.

The water-leachates and water blanks were stored in the refrigerator until liquid chromatography-high resolution mass spectrometry (LC-HRMS) analysis. The water-leachates and water blanks were removed from the refrigerator, left to equilibrate at room temperature for at least 1 h, and sonicated for 30 min. 5 mL of each water-leachate or blank sample was transferred into a separate prewashed with methanol 10 mL head space vial (Thermo Scientific). 1 mL of methanol followed by 15 μL of internal standard (IS), bisphenol A-d16 in methanol, was added to each sample to give 0.6 μg/L of IS in each sample or blank.

2.3. Instrumental analysis

2.3.1. Analysis of microplastics in water-leachates from face masks

Each filter was analysed using the Nicolet iN10 MX FTIR Imaging Microscope fitted with a liquid nitrogen cooled MCT detector (Thermo Scientific, USA) in a transmittance mode and assisted with the Particle Wizard option in the Thermo Scientific™ OMNIC™ Picta™ Software (Thermo Scientific, USA) to identify the number, size (length and width of particles), morphology, and a polymer type of MP particles. To investigate the polymer types of DFMs, the layers of all investigated masks (Fig. S3-S5) were analysed using FTIR microscopy in Attenuated Total Reflectance (ATR) mode (Section S1 and Fig. S6-S11). The plastic spectra collected in a scanning range of 675–4000 cm^{-1} were added into the existing plastic specific spectral libraries (Thermo Scientific, USA) and used to identify plastic type of the MP found in the water-leachates. A microscopic image of each filter covering approximately 1 cm^2 was created and presented particles were automatically identified. Subsequently, a full spectrum within the FTIR scanning wavelength range of 675–4000 cm^{-1} was recorded for each identified particle (typically, 16 scans coadded at 8 cm^{-1}). An automatic correction for the background (an IR spectrum containing only filter material) was performed. The identification of plastic types was based on comparing the generated sample spectra with the IR spectra reference libraries (Thermo Scientific, USA), with only a matching confidence level above 80% considered for positive identification.

2.3.2. Analysis of organic analytes presented in water-leachates from face masks

Analysis of face mask water-leachates described above was carried out using EQuan MAX Plus Thermo Scientific™ Vanquish™ uHPLC system equipped with Q Exactive™ Focus Hybrid Quadrupole-Orbitrap™ Mass Spectrometer (Thermo Fisher, Bremen, Germany). The analytical procedure was described in Kourtchev et al. (2022). Acquisitions were performed in a negative ionisation mode where data were collected at a mass resolving power of 70,000, isolation window 1 m/z and AGC target 5e4. ESI parameters were as follows: spray voltage −3.5 kV; capillary temperature 325 °C; sheath gas flow rate 40 arbitrary units (AU), auxiliary gas flow rate 10 AU; sweep gas flow rate 0 AU.

The spiked with IS face mask water-leachates were initially screened using Full Scan (FS). Considering the variety of plasticisers and additives, including dyes and softeners, used in the manufacturing process of face masks, some of them can potentially leach out when exposed to the environmental or extreme conditions (Table S2). In this study, specific emphasis was given to bisphenols, and thus, the selected polar end-capped C18 stationary phase narrowed the analysis to moderately polar analytes (Layne, 2002), such as 1,4-bis(2-ethylhexyl) sulfosuccinate. It must be noted, however, that the full-scan chromatogram of the MMII and MMIIR mask water-leachates additionally contained an

early-eluting unresolved chromatographic 'hump'. Due to the poor chromatographic separation of that unresolved mixture and the complexity of the corresponding mass spectra, the emphasis was given to a major chromatographic peak with distinguishable MS features i.e., 1,4-bis(2-ethylhexyl) sulfosuccinte (observed in several samples). The LC/MS chromatograms and corresponding mass spectra for the 1,4-bis (2-ethylhexyl) sulfosuccinate standard and face mask water-leachates are shown in Figures S14 and S15. To improve measurement accuracy and precision, the samples then were reanalysed using Selected Ion Monitoring (SIM). Quantification of bisphenol B and 1,4-bis(2ethylhexyl) sulfosuccinate was done by preparing a five-point calibration curve in water:methanol (80:20 v/v) at the range of 0.01–0.2 μg/L for BPB and 1–20 μg/L, for 1,4-bis(2-ethylhexyl) sulfosuccinate.

For quantification of 1,4-bis(2-ethylhexyl) sulfosuccinate, the samples were diluted with water:methanol (80:20 v/v) 10 times to reduce chromatographic saturation from the 1,4-bis(2-ethylhexyl) sulfosuccinate peak.

Method LOD and LOQ were established using standard deviation of the response and the slope approach and calculated using Equation 1 and Equation 2.

Equation 1: LOD = 3.3 σ/Slope.

Equation 2: LOQ = 10 σ/Slope.

The method LODs are 0.075 μg/L and 1.65 μg/L for BPB and 1,4-bis (2-ethylhexyl) sulfosuccinate, respectively. The method LOQs are 0.25 μg/L and 5.495 μg/L for BPB and 1,4-bis(2-ethylhexyl) sulfosuccinate, respectively.

2.4. Quality assurance and quality control

To minimise external microplastic contamination during the experimental work, Quality Assurance and Quality Control (QA/QC) requirements were applied. The laboratory surfaces were cleaned with water and ethanol before experiments and analysis. Cotton clothing and cotton laboratory coats were worn in the lab. All solvents used were of analytical grade (>95%). The used glassware was pre-cleaning with methanol followed by thorough rinsing with Milli-Q water. Optima™ LC/MS grade water (Fisher Scientific) were used for the leaching experiments and for water-leaching sample preparation steps. Glass and stainless-steel equipment were used during the experimental work. The prewashed glassware and samples were covered with aluminium foil during the experiments and when they were not in use. Three procedural blanks were used during all experimental steps to account for any external contamination. The average number of MPs in the procedural blanks was 6.7 ± 1.5 MPs and it was subtracted from the results of the water-leaching experiments.

3. Results and discussion

3.1. Microplastics released into water from disposable face masks

Fig. 1 shows the microscopic images of each filter after filtering the water-leachate of different types of DFMs. It is apparent that all tested DFMs released MPs into the water. The highest concentration of MP particles (1067 ± 170 MPs/DFM) was observed in the water-leachate from the FFP2 mask, followed by the FFP3 mask (877 ± 11 MPs/DFM). The water-leachates from the surgical/medical DFMs contained a similar number of MP particles: MMI - 277 ± 8.7 MPs/DFM; MMII – 248 ± 11 MPs/DFM; MMIIR - 239 ± 20 MPs/DFM. Generally, the filtering face pieces (FFP2 and FFP3) released 3–4 times more MPs particles

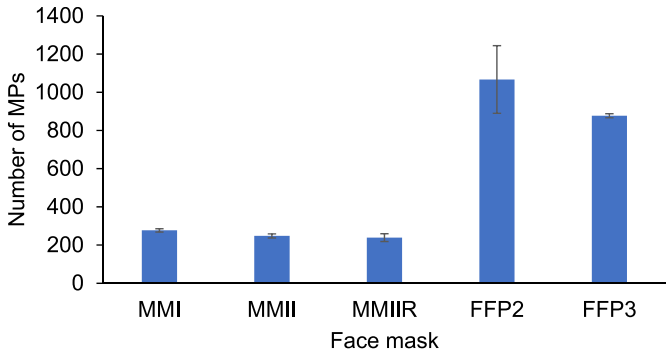

Fig. 2. Number of microplastic particles released from each tested type of the disposable face masks.

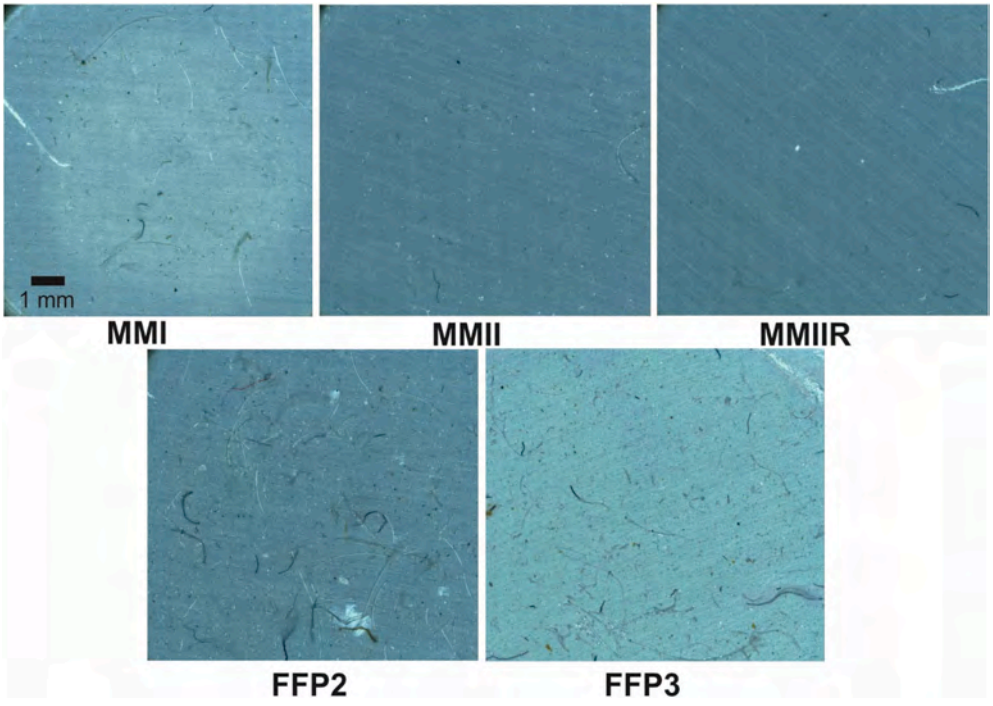

Fig. 1. Microscopy images of microplastics leached out from the disposable face masks.

compared to the medical/surgical DFMs (MMI, MMII, and MMIIR) (Fig. 2). This might be because filtering face pieces (FFP2 - 5.775 ± 0.17 g and FFP3 - 15.609 ± 0.11 g) have a higher weight than MM DFMs (MMI - 3.89 ± 0.035 g; MMII - 3.25 ± 0.015 g; MMIIR - 2.765 ± 0.007 g). The amount of released MP particles per g of each type of DFMs is generally similar for MMI - ~71 MP/g, MMII – ~76 MP/g, MMIIR – ~86 MP/g, and FFP3 - ~56 MP/g, but much higher for FFP2 - ~185 MP/g. The fact that all analysed DFMs released MPs under the static condition without mechanical weathering (e.g., agitation) can indicate that new DFMs may already contain MPs (MMI, MMII, MMIIR < FFP3<FFP2), probably generated during the non-woven DFM production process. Therefore, it is very important to improve the quality of produced DFMs to eliminate the presence of MPs in new DFMs.

Our results are also consistent with those reported by Delgado--Gallardo et al. (2022), where they estimated that the FFP2 masks emitted 4–6 times higher MPs than the MMIIR, based on the optical microscopy image analysis of the 3 μm^2 filter area. Jiang et al. (2022a) and Liang et al. (2022) demonstrated a higher quantity of MPs released from the MM and N95 DFMs (Table S2), possibly attributed to the fact that the authors shook the samples for 24 h, potentially enhancing the release of MPs. Chen et al. (2021) reported that MM and N95 DFMs released 183.00 ± 78.42 MPs/DFM after DFM water-leaching with shaking at 120 rpm for 24 h and using the stereomicroscope for MP counting. The authors did not observe a significant difference in MP release among the MM and N95 types of DFMs. The similar finding was also highlighted by Jiang et al. (2022a), Liang et al. (2022), and Celik (2023), who employed similar experimental designs (including shaking) (Table S2). Chen et al. (2021) also found that wearing DFMs before the leaching experiment could result in 6.0–8.1 times more MPs being released into water. Ma et al. (2021) also identified a larger quantity of MPs in the water-leachates (ranging from 1.3 to $4.4*10^3$ MPs/DFM) from the MM and N95 face masks. However, it is essential to consider that they collected ten water-leachates for their study, and each water-leachate was run for 3 min with vigorous shaking. However, upon estimating the number of MP particles released from MM face masks in water-leachates, we observed that the data (multiplied by 10: approximately 2390–2770 MPs/DFM) aligned with the findings of Ma et al. (2021). Ma et al. (2021) also observed that each mask (both MM and N95) released $1.6–3.8*10^9$ nano-plastics in the water-leachates. Morgana et al. (2021) also identified that the MM face mask released nano-plastics, when they mimicked mechanical weathering condition. Furthermore, in a study by Saliu et al. (2021) simulated the degradation of the MM DFMs in marine environments (such as artificial seawater) and demonstrated that MM face mask could release up to 173,000 fibres/day. Based on the literature data, it was shown that weathering caused by mechanical, UV-light, and chemical weathering processes in the environment can lead to an increase in MPs/NPs release from DFMs into water (Table S2). Additionally, our results indicate that approximately 255 MPs were released from a single MM DFM which was the most commonly used mask during the COVID-19 pandemic. Therefore, based on data from Benson et al. (2021), outlined that 3.4 billion DFMs were disposed daily during the COVID-19 pandemic, it was possible to estimate that 867 billion MPs were released into the environment every day from those masks. This estimation is alarming considering the monthly estimated use of 129 billion face masks worldwide (Prata et al., 2020), and highlights the potential for significant MP leaching into the environment due to improper disposal of used DFMs (Roberts et al., 2022; Tesfaldet and Ndeh, 2022).

The particle sizes of MPs varied greatly, ranging from around 10 μm–2082 μm, as shown in Fig. 3. The minimum size of the identified MPs particles was approximately 10 μm, which is due to the detection limit of the FTIR analysis used in this research. Fig. 3 displays the distribution of MP particles in five size ranges (<30 μm; 30–50 μm; 50–100 μm; 100–500 μm; >500 μm) that were found in the water-leaching samples of the investigated DFMs. The majority of the investigated DFMs released MP particles below 100 μm, accounting for

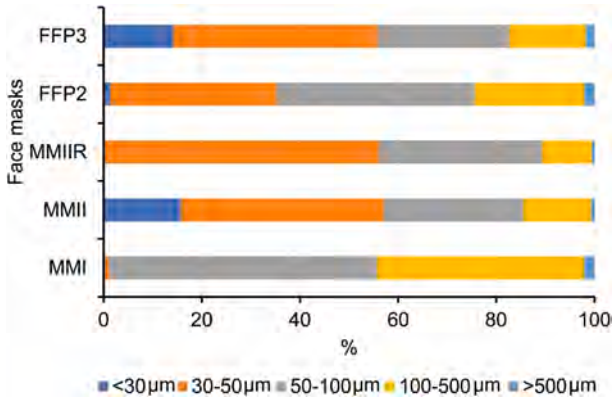

Fig. 3. Size distribution of MPs leached out from the disposable face masks.

approximately 75–90% (Fig. 3). However, the MMI face mask released around 56% of MPs below 100 μm, as shown in Fig. 3. Therefore, the DFMs releasing MPs with sizes <100 μm can be put in the following order: MMIIR > MMII > FFP3>FFP2>MMI. MMII, MMIIR and FFP3 released higher percentage (~56–57%) of MPs with size below 50 μm comparing to FFP2 (~35%) and MMI (~1 %). About 14–16% of MP size below 30 μm was water-leached from MMII and FFP2. Jiang et al. (2022a) identified that MMIIR face mask released MPs with size mainly below 100 μm (<30 μm - ~40%; 30–50 μm - ~25%; and 50–100 μm - ~25%) and less than 10% corresponded to MP size above 100 μm (100–200 μm - ~ 5%; 200–300 μm - ~ 3%), which is well correlated with our results for MMIIR. However, Chen et al. (2021), Liang et al. (2022), Wu et al. (2022), and Gupta et al. (2023) highlighted that MPs of 100–500 μm were predominant in the water-leachates from MM and N95 DFMs. Moreover, Liang et al. (2022), who investigated MP release kinetics, observed that the release rates of MPs <100 μm were highest on the first day of water-leaching experiment.

Two main MPs morphotypes, based on the analysis of data from FTIR microscopy (e.g., particle sizes - length and width), were identified in all samples: fragments and fibres (Fig. S13). The most commonly identified MP morphotypes were fragments, particularly that of MP fibres (e.g., debris of PP fibres), which accounted for 55.4–88.3% of all the samples with particle sizes smaller than 100 μm. It raises concerns regarding potential risks to both human health and the environment. The face masks of the MMI type were found to emit a comparable amount of microfibres and fragments. Most of the current studies have mainly focused on and expressed concern about the release of microfibres from MM face masks (Fadare and Okoffo, 2020; Saliu et al., 2021; Sullivan et al., 2021; Wu et al., 2022). Chen et al. (2021) reported that 70% of MPs related to microfibres. They found that the amount of MP fragments released into water was increased after wearing FM for one day, that might be due to mechanical stress. Therefore, future research is needed to understand the emission of MP fragments with sizes below 100 μm from DFMs (including different types of MMs and FFPs), as well as the assessment of the associated risks to human health and the environment. Also, a detailed investigation of micromorphology (e.g., particle shape and surface properties) of MPs released from face masks by scanning electron microscopy (SEM) would be useful to understand potential MP behaviour in the environment.

A higher number of fragments may be generated during the production process of DFMs due to using low-quality PP (such as low-quality recycled PP or damaged and fractured PP fibres), and contamination during production and packaging. Moreover, extensive use of non-woven fabrics in DFM production, known for their mechanical weakness compared to woven fabrics, may lead to a higher MP release (Chua et al., 2020). Additionally, the high demand for the DFM production during the COVID-19 pandemic might result in lower quality DFMs. For example, Jiang et al. (2022a) indicated scratches and

breakages on the fibre surfaces of new face masks using SEM. Furthermore, the fragmentation of mask fibres is expected in the environment due to physical, chemical, and biological degradation (Table S2). Wu et al. (2022) identified numerous cracks and protrusions on the surface of microfibres after sand abrasion. Li et al. (2022) showed that roughness of the surface of microfibres increased after immersing MM DFMs in the acidic (pH 4), neutral (pH 7), and alkaline (pH 10) water solution. Furthermore, Celik (2023) identified damages on the fibre surface of FFP2 DFMs after natural weathering (e.g. UV-sunlight, mechanical forces). In the study conducted by Ma et al. (2022), researchers examined the impact of DFM waste in the marine environment and discovered that the DFM fibres were fragmented just in one month in seawater. Similarly, Sun et al. (2021) investigated a leachability of MPs from MM DFMs into artificial seawater and showed that the masks released both fibres and fragments due to mechanical erosion.

Most of the identified MPs in the water-leachates from all the investigated DFMs are expectedly made of PP (82–97%) because it is the main polymer used in their production (Fig. 4 and Fig. S6-S12). Other polymers, such as PE, PC, polyester/PET, polyamide (PA)/Nylon, polyvinylchloride (PVC), and ethylene-propylene (EP) copolymer, were also identified in smaller percentages (1–5.6% for each plastic type) in the DFM water-leachates. Overall, FFP2 and FFP3 DFMs released a greater variety and a higher percentage (17–18%) of other plastic types compared to MM DFMs (3–7%) (Fig. 4). Generally, it can be due to difference in polymer compositions of the different type of DFMs (Section S1; Fig. S6-S12). There are only several published papers that have identified the polymer types (mainly PP) leached into water from DFMs (Table S2). Additionally, Chen et al. (2021) identified PET, while Liu et al. (2022) identified PA released from MM and N95 DFMs. Jiang et al. (2022a) investigated the water-leachability of MPs from MM DFM and identified that PP and PU were the main types of MPs released, accounting for 24.5% and 57.1%, respectively. Additionally, other types of MPs such as PET, PE, PA, and PS contributed to 18.4% of the total released MPs. The researchers noted that the mask ropes, which are often made from PU, were a major source of PU MPs released into water. However, it is important to consider that mask ropes can be made from different materials, such as PET (MMs and FFP3) and (PA)/Nylon (FFP2) (as shown in Table S1 and Section S1), which could influence the types of MPs released into water-leachates. Overall, the difference in polymer composition of different DFMs (see section S1 and Fig. S6-S12) and potential contamination during the DFM production process can lead in variation of MP types leached into water.

According to several studies, MPs released from DFMs into the terrestrial and aquatic environments can have a significant impact on living organisms (Haque and Fan, 2022; Jimoh et al., 2023; Hasan et al., 2023; Oliveira et al., 2023). Ma et al. (2021) showed that MPs from DFMs could be adsorbed onto diatoms and could also be easily ingested by marine organisms (e.g., rotifers, shrimps, copepods, scallops, groupers). Sun et al. (2021) demonstrated that the marine copepod *Tigriopus japonicus* could easily uptake MPs released from MM DFMs, leading to a substantial decrease in their ability to reproduce. Hu et al. (2024) showed that MPs and NPs, released from MM DFMs, could be accumulated into zebrafish embryos and larvae, and subsequently distributed to various tissues. Kwak and An (2021) observed that terrestrial organisms, such as earthworms and springtails, could easily absorb MPs released from DFMs, and it affected intracellular esterase activity in earthworm coelomocytes, and spermatogenesis in earthworm seminal vesicles, and the number and size of juvenile springtails. Exposure to MPs, whether through inhaling contaminated air or consuming polluted beverages and food, can lead to adverse effects on human health. This exposure may result in physical harm to human cells and organs, as well as the leaching and accumulation of potentially toxic chemicals such as bisphenols and phthalates from certain plastics, and the potential infectious by pathogens transported on MP surface (Galloway, 2015; Vethaak and Leslie, 2016; Hwang et al., 2019; Ma et al., 2021). Ma et al. (2021) identified MPs in the nasal mucus when people wearied DFMs. This is concerning because previous research by Hwang et al. (2019) has indicated that the accumulation of MPs in the human body could pose various health risks. Hwang et al. (2019) specifically mentioned that the accumulation of MPs could potentially lead to cytotoxicity, hypersensitivity, unwanted immune response, and acute response such as hemolysis. They showed that direct interaction of PP particles (<20 μm) and peripheral blood mononuclear cells (PBMCs) as well as human mast cells (e.g., HMC-1) might potentially develop hypersensitivity by elevating the levels of cytokines and histamines. Vethaak and Leslie (2016), Wang et al. (2018), Oliveira et al. (2023), and Hasan et al. (2023) highlighted the potential for plastic debris to act as a carrier for chemical pollutants (e.g., heavy metals, polycyclic aromatic hydrocarbons (PAHs),

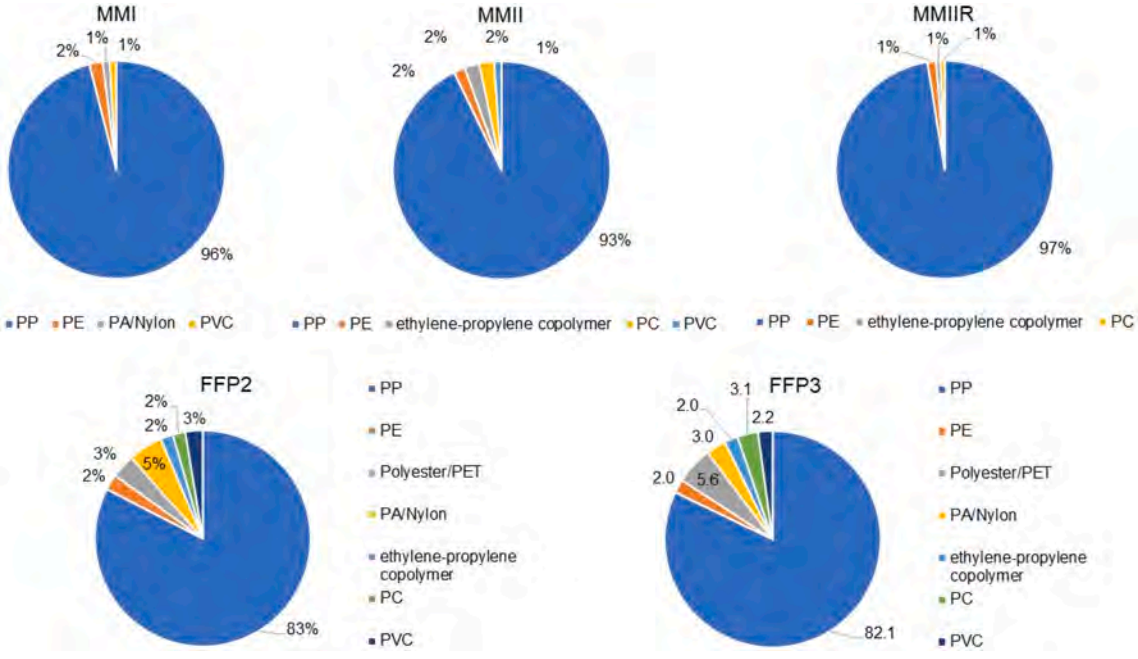

Fig. 4. Distribution of MP types leached out from each type of the studied disposable face masks.

polychlorinated biphenyls (PCBs), perfluorooctanoic acid (PFOA), antibiotics), and as a surface for the proliferation of pathogens and antibiotic resistance genes. This can increase harmful risk for the living organisms in the terrestrial and aquatic environments. Additionally, prolonged use of the same DFMs allows pathogens to colonise the surfaces of MPs presented within the masks. As a result, upon inhalation, those MPs can transport the pathogens into the human respiratory system and increase the risk of infection. Despite this potential risk, there remains insufficient understanding of the toxicity of different type of MPs released from different type of DFMs and their impact on living organisms in the terrestrial and aquatic environments and humans, emphasising the need for further investigation.

The presence of MPs in water leachates poses potential risks to the terrestrial and aquatic ecosystems and human health, highlighting the importance of understanding and mitigating this source of MP pollution (Hu et al., 2022; Hasan et al., 2023; Jiang et al., 2023b). Furthermore, there is a significant knowledge gap in plastic regulation and plastic waste management related to DFMs (Fadare & Okoffo, 2020; Hu et al., 2022; Hasan et al., 2023). Hasan et al. (2023) outlined an integrated 5R (Reduce, Reuse, Recycle, Redesign, and Restructure) strategy consisting of monitoring, policy, and legislation as key elements for managing PPE interventions. It is important to enhance collaboration among researchers, PPE producers, waste managers, government, policymakers, and society (Haque and Fan, 2022; Hasan et al., 2023). It remains important to create clear, simple, and straightforward guidelines for the safe disposal of DFMs and other PPEs within public communities. It is also essential to establish robust infrastructures for proper waste management and utilisation, for example, introduce clearly labelled bins in public areas (e.g., hospitals, schools, airports, shops, and transportation hubs) and a clear guideline on DFM disposal at designated waste management facility. The findings from previous and current research studies also can support the transition to circular economy initiatives such as: develop safe methods and processes for sustainable design and better quality DFM production, decontamination and reuse of DFMs, and DFM recycling; use recycled material in various applications (e.g., building and construction sector); develop alternative products such as easily disinfected and high quality reusable face masks; develop environmentally friendly face mask alternatives; and develop sustainable strategies to mitigate PPE pollution during future pandemics. Additionally, the methods for removing MPs from the aquatic environment are urgent. Several potential methods, such as filtration, adsorption, coagulation, froth flotation, and magnetic separation, have been highlighted and discussed in review papers (Zhang et al., 2021a; Abuwatfa et al., 2021; Jiang et al., 2023b). Abuwatfa et al. (2021) outlined that the MP removal by biochar might be a promising low-cost, efficient, and environmentally friendly method. Wang et al. (2022) proposed a sustainable strategy involving iron coagulation to remove MPs from the aqueous environment, followed by the carbonisation of the coagulated MPs. This process synthesises magnetic carbon/iron nanocomposites, which can be served as heterogeneous catalysts for pollutant degradation. Shi et al. (2022) developed an efficient and environmentally friendly method for removing MPs from aqueous solutions using magnetic sepiolite for magnetic separation. Zhang et al. (2021b) and Jiang et al. (2022b, 2022c, 2023a) proposed froth flotation as an effective and promising technology for the removal of MPs from aqueous systems. Further research is needed to optimise and scale up those technologies for the water treatment industrial applications.

3.2. Bisphenols released into water from disposable face masks

In this study, the water-leachates of several types of DFMs (MMI, MMII, MMIIR, FFP2, and FFP3) were screened for selected bisphenols i.e., bisphenol A (BPA), bisphenol B (BPB), bisphenol S (BPS), bisphenol E (BPE), which have similar chemical characteristics and are suspected of having damaging effects (e.g., endocrine disruption, may damage fertility or the unborn child, etc.) (Vandenberg et al., 2012; Hahladakis et al., 2018). Out of four tested targets, BPB was identified in MMII and MMIIR water extracts at concentration 0.25 ± 0.02 μg/L and 0.42 ± 0.02 μg/L, respectively (Fig. S16). The amount of released BPB per g of the MMII type is 0.012 μg/g and the MMIIR type is 0.023 μg/g. Based on data from Benson et al. (2021), it is possible to estimate that approximately 128–214 kg of BPB were released into the environment each day from those masks. BPB, a substitute of BPA, is widely used in the polycarbonate plastic and resins production (e.g., epoxy resins that coat the insides of canned goods and water supply pipes). To the best of our knowledge, bisphenols are not used in PP production, however, their presence in the leachates from some DFMs might be due to usage of other plastic materials such as PC that can contain BPB, and/or potentially cross-contamination during products' manufacturing process (e.g., insufficiently cleaned processing machinery, using recycled PP that can have chemical impurities transferred from other waste material, etc.). In the environment BPB is one of the least investigated and detected bisphenols (Noszczyńska and Piotrowska-Seget, 2018). There is no data for direct comparison of detected concentration of BPB from our study with that from literature; however, BPB was previously observed in municipal sewage treatment plants (STP) influents in India (Karthikraj and Kannan, 2017) and in industrial STP effluents in Slovenia (Cesen et al., 2018) at 2.5 ng/L and 8.46 ng/L, respectively. BPB is included in The Endocrine Disruptor Exchange list (TEDX, 2018) of potential EDCs, and in vitro results of the U.S. EPA Endocrine Disruptor Screening Programme (EDSP; US EPA, 2023). Information on BPB levels in European freshwater ecosystems is scarce (Serra et al., 2019). Further research is still needed to understand the leachability of potentially toxic organic compounds like BPB from different types of DFMs, and their impact on living organisms.

3.3. 1,4-bis(2-ethylhexyl) sulfosuccinate released into water from disposable face masks

Although the main goal of the LC/MS analysis was to screen DFM water-leachates for bisphenols, presence of a saturated peak at *m/z* 421.2271 in the negative ESI-LC/MS chromatograms (when analysed in FS) from MMII and MMIIR face masks water-leachates drew our attention. The molecular assignment from the accurate mass measurement of this peak resulted in $C_{20}H_{37}O_7S$ and was assigned to 1,4-bis(2-ethylhexyl) sulfosuccinate (<2 ppm mass error) using a Compound Discover software. The proposed assignment was confirmed by analysis of 1,4-bis(2-ethylhexyl) sulfosuccinate authentic standard and comparing its mass spectrum and retention time with that in the mask sample (Fig. S16). 1,4-bis(2-ethylhexyl) sulfosuccinate, also known as dioctyl sulfosuccinate (DOSS) belongs to the chemical family docusates. Docusate salts are used as food additives, emulsifiers, dispersants, and wetting agents (Ash and Ash, 2004). The concentration of 1,4-bis(2-ethylhexyl) sulfosuccinate in MMII and MMIIR water-leachates was 163.9 ± 1.4 μg/L and 115.0 ± 3.9 μg/L, respectively. The amount of released 1,4-bis(2-ethylhexyl) sulfosuccinate per g of the MMII type is 7.56 μg/g and the MMIIR type is 6.24 μg/g. To authors knowledge there is no literature data for direct comparison of observed concentrations of DOSS. DOSS is also used as a dispersant to emulsify spilled oil and was a major component of the dispersants most used in the 2010 Deepwater Horizon Oil Spill incident response (Mathew et al., 2012). Although this compound is considered to be relatively safe and even approved for use in pharmaceutical and cosmetic industry (Fiume et al., 2016), its wide application for oil spillage removal raised some concerns regarding its toxicity to fish (MacInnis et al., 2018). Moreover, recent studies indicated that exposure of vertebrates to DOSS can potentially lead to endocrine disruption by altering thyroid hormone (TH) function (Corrie et al., 2021).

4. Conclusion

The release of MPs into water from all tested DFMs under static

conditions without additional mechanical stress (MMI, MMII, MMIIR < FFP3<FFP2) suggests that new DFMs may contain MPs originating from the production process of non-woven DFMs. FFPs released 3–4 times more MPs particles comparing to MM DFMs. Additionally, MM DFMs released BPB and 1,4-bis(2-ethylhexyl) sulfosuccinate. Therefore, DFMs might be considered as potential source of MPs and BPB contamination in the environment, particularly, when they dispose of incorrectly, such as through littering or/and landfilling. The particle sizes of MPs varied greatly, ranging from around 10 μm–2082 μm, but MP particles below 100 μm were predominant in the water-leachates. Additionally, the order of DFMs releasing MPs with sizes <100 μm is as follows: MMIIR > MMII > FFP3>FFP2>MMI. PP is a main type of microplastics identified in the water-leachates from the investigated face masks. Fragments and fibres were identified in all water-leachate samples, and fragments, particularly debris of PP fibres, were the most common MP morphotype among all investigated types of DFMs (>60%). Surgical/medical masks released a higher percentage of PP MPs (93–97%) compared to filtering face pieces (82–83% PP MPs). Additionally, other MP polymers such as PE, PC, polyester/PET, PA/Nylon, PVC, and EP copolymer were also identified. FFP2 and FFP3 DFMs released a wider variety and a higher percentage (17–18%) of other plastic types compared to MM DFMs (3–7%). Overall, the difference in polymer composition among different DFMs and potential contamination during the DFM production process can lead in variation of MP types leached into water.

CRediT authorship contribution statement

Anna A. Bogush: Writing – review & editing, Writing – original draft, Visualization, Validation, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Ivan Kourtchev:** Writing – review & editing, Methodology, Formal analysis.

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.

Acknowledgements

This work was financially supported by the QR Strategic Priorities Fund (2021–2022) from Coventry University (United Kingdom).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envpol.2024.123792.

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