# Performance of filters applied for removal of microplastics from water – testing methodology

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#### ABSTRACT

The separation of microplastics from water using the filtration process was studied in this work. The deep-bed filter media were made of polypropylene using the melt-blown technique. The separation performance of these filters was compared to the pleated cellulose elements (surface filtration) for the prepared suspension of fragmented polystyrene particles. No pretreatment nor additives to enhance the separation process were applied upstream the filtration unit. With the increased filter efficiency (rating of depth filters ranging from 20 to 5  $\mu$ ), the cut-off dimeter of removed microplastic also decreased. The experiments confirmed that deep bed filtration is capable to remove efficiently very fine microplastic particles (as small as 1  $\mu$ ), while maintaining relatively low pressure drop. The capacity of the deep bed filter will guarantee relatively long operation time for suspensions having a low concentration of MPs, however in real process this will probably be predominantly affected by the simultaneous deposition of other solid contaminants present in the water.

Keywords: Microplastics in water; Water filtration; Deep bed filters; Melt-blown filter media

# 1. Introduction

Microplastics are small pieces of plastics (solid synthetic organic polymers), the dimension of which is in the 1  $\mu$ m-5 mm size range. Depending on their origin, two categories are distinguished: primary and secondary microplastics. The first group includes items that are deliberately manufactured in the form of particles with dimensions within the given range, such as microbeads added to cosmetics and personal care products or plastic pellets. The second category refers to plastic fragments resulting usually from the degradation of larger objects, which are often mismanaged waste, such as plastic bottles, bags or fishing gear [1,2]. Although microplastics mainly originate from land-based sources, it is the aquatic environment, in particular

the marine environment, that is considered to be its final recipient [3]. The main transport routes responsible for the migration of these micropollutants from terrestrial to water areas include, among others, surface runoff, atmospheric transport and sewage discharges [1–3]. Reported microplastic concentrations in water vary within very wide ranges. For example, based on literature data, average values of microplastic content in freshwater environments range from almost zero to even several million pieces per cubic meter (for small objects) [4]. The spatial distribution of plastic particles is related to many factors, both anthropogenic and environmental, such as winds or currents, which makes it difficult to determine their abundance in a specific area [1].

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In addition, sampling approaches and sites also have a significant impact on the obtained results [4]. However, regardless of the abundance of microplastics, the fact is that they are present in the aquatic environment and thus have a negative impact on the ecosystem. When ingested by aquatic organisms, they can enter the food chain, posing a potential risk to people consuming contaminated food (e.g., seafood) [5].

Although the negative effect of microplastics on the human body has not been confirmed, there is a growing interest and ongoing research studies to verify this hazard [6-9]. Moreover, adsorption of hazardous compounds such as metals or organics can significantly increase their chemical toxicity. It has also been proven that a long-term weathering of microplastics in aquatic systems create active sites for the sorption of various compounds, including pollutants [9]. Another important issue refers to the colonization of microplastic by various aquatic microorganisms. Apart from carrying over the attached bacteria or algae, the biofilm formation can enable enrichment of bacterial pathogens [7]. Importantly, the presence of microplastics in human lungs and blood has recently been found. This indicates that after ingestion or inhalation, they can undergo translocation and accumulation in various organs and tissues [10,11]. Thus, considering the fact that microplastics can act as vectors of toxic pollutants, their presence in the human body may be associated with a serious health risk.

In recent years various processes has been experimentally studied to determine suitable and economic techniques capable to remove microplastics from water [12–22]. Of course, the efficacy strongly depends on the form of this contaminant, mainly the size and morphology of polymer object. Various methods have been tested and their efficiencies reported. The processes involve the sedimentation, the flotation, the filtration, where typically a coagulation step is used upstream the separation. The simultaneous removal of microplastics and other contaminants in WWTP has also been studied, and it shows capability of so called CASP (conventional activated sludge process) to separate this pollutant by microorganisms [23]. Other non-conventional and advanced methods such as electrocoagulation [19], magnetic seeded filtration [15] or ingestion and excretion of polymer particles by selected mussels have also been demonstrated [24].

Among others, the filtration process, which is widely used for removal of dispersed solid particulates and droplets, can be one of the most efficient for very small polymer particles. Other afore-mentioned methods are effective only to a limited extent in terms of retaining very small solid contaminants (objects of the size of few micrometers) [12,13]. In particular, processes such as coagulation or sedimentation are not suitable for the elimination of fine plastic items, not only because of their sizes, but also low densities (density difference with comparing to water) [12,13,17]. A more robust process for the removal of MPs in terms of efficiency is filtration using microfiltration membranes or depth filters. These processes are very reliable when properly designed to capture very small objects (with dimensions of the order of 1 um or even smaller), such as membrane techniques or depth filtration with the use of fine filters, which allows to achieve efficiencies significantly exceeding 90% [12,13,25]. The typical municipal water supply and wastewater treatment systems remove majority of suspended solids, but fine microplastics can still potentially penetrate through the existing separation equipment such as sand filters, adsorption beds, ion exchange columns etc., which have not been designed to separate this contaminant [13]. In general, the filtration process can be divided into two main groups: (i) a cake or surface filtration, and (ii) a depth filtration. In the first one, a uniform layer of deposit is formed on the filter media and grows until a critical dP is reached. Then the deposit is removed (for example using reverse flow, mechanical scrapping etc.) and usually the filter can be reused. In depth filtration, the particles are collected not only on the inlet surface, but they penetrate the porous material and are collected on the fibres. Therefore, the entire specific surface area is available for the deposition, provided the filter is properly designed for the process (contaminant properties), that is, largest particles are collected on the inlet and do not penetrate into the media, the moderate size object enter the filter volume but not very deep, and the smallest particles reach the deepest layers of filter material. For such operation the material must be properly designed for a size-selective deposition of particles depending on position inside the fibrous bed, which requires a gradient structure (a negative porosity gradient along the flow, and usually the average diameter of fibres also decreases from the inlet to the outlet). Although the depth filters have typically a higher initial pressure drop comparing to surface filtration of the same size (cartridge dimensions), they are usually more efficient and can accommodate more contaminants (so called "dust holding capacity" is higher). Moreover, the pressure drop increase in time is less steep, and very often the dP at the cleaning/ changeout is on a similar level for both types of filtration elements [25].

Literature data on the effectiveness of MPs elimination during individual water treatment processes, including various filtration methods, are very limited and relate mainly to bench-scale experiments [16,26]. Measurements of MPs abundance at one of the drinking water treatment plants located in China showed a high removal rate of relatively large particles (>10 µm) during sand filtration. The smallest and most numerous fraction, that is, 1-5 µm, was retained in the filter bed only to a slight extent. As a consequence, the overall efficiency of this process was quite low, ranging from 29.0% to 44.4% [16]. On the other hand, according to Zhang et al. [12], filtration can be considered as the most effective method of removing both micro- and nanoplastics. Laboratory-scale test results indicate that the filtration process is able to completely remove polymer microbeads larger than 100  $\mu m$  from the water, while particles as small as 1.2 µm can be retained up to 95% [12]. A high MPs removal rate was also observed for rapid sand filters used in tertiary wastewater treatment [26]. As reported by Talvitie et al. [18] MPs content after the filtration process was reduced by 97.1%, however, only particles  $>20 \ \mu m$  were considered in this study.

It should be noted that only a few publications are available on the elimination of plastic particles by filtration. Moreover, in the case of depth filters, only granular beds are considered, and there is no information on the effectiveness of fibrous beds in retaining MPs. Therefore, it is reasonable to carry out research in this area, focusing primarily on the smallest plastic particles (with sizes of several  $\mu$ m), which are the greatest challenge in terms of their removal from water. Hence, in this work the separation performance of polypropylene depth filters (various grades) was determined and compared to the pleated cellulose elements (surface filtration) for the in-house prepared suspension of fragmented polystyrene particles.

# 2. Materials and methods

#### 2.1. Preparation of microplastics

The microplastics were produced in-situ, that is, in the laboratory, using impact mills equipped with four stainless steel blades. In order to obtain particles with sizes from several to several dozen micrometers, the base material (plastic pellets) was subjected to grinding processes with simultaneous cooling with the use of dry ice added directly to the inside of the mill. The aim of this procedure was to limit the excessive heating of the device, and thus its contents, which could lead to a counterproductive effect, that is, the conversion of pellets into a soft, plastic mass instead of their fragmentation. As a result of grinding the base material, which was polystyrene pellets, particles in the form of microgranules were formed. The morphology of microplastics was identified based on the analysis of microscopic images using a scanning electron microscope model Phenom G2 and an optical microscope Nikon Eclipse E200 equipped with a digital camera. By measuring the perimeter (P) and the surface area (A) of the particles, the shape factor (SF) was determined using the circularity Eq. (1). The shape factor values ranged from 0.588 to 0.928, with the average 0.766.

$$SF = \frac{4\pi A}{P^2}$$

In turn, the measurements of the diameters allowed to plot the size distributions of polystyrene particles obtained by grinding (Fig. 1). Furthermore, in order to determine the agglomeration tendency of microplastics, zeta potential measurements were also performed, as detailed in section 2.4.2.

## 2.2. Specification of filter media

One of the most widely used methods of fabrication of fibrous polymer structures is the melt-blow process. The deep bed media used in presented research were manufactured in-house using an automated system, which guaranties a good reproducibility of produced structures. The material used was the polypropylene (Borealis Borflow HL504FB).

Tracking the path of polymer from an granulate to the form of final product, the following steps of the process can be distinguished:

- feeding the screw extruder with a granulate,
- melting of polymer in the heating sections and its transport towards the dye,
- extrusions of the melt through the multichannel dye, where the sheath hot air is simultaneously supplied,
- elongation of the polymer streams due to shear stresses and subsequent solidification of fibers resulting from cooling down the filaments,
- collecting of formed fibers on the receiver, usually rotating cylinder (a drum or directly the support core of filter cartridge) with a reciprocating movement along the axis of symmetry.

A scheme of the melt-blow system for the manufacturing of fibrous media used in this work is presented in Fig. 2.

The presented melt-blow system is capable to manufacture a wide range of fibrous media – with various diameter and porosity – by controlling the operation parameters such as polymer flow rate, temperature and flow rate of air, position and rotational speed of the receiver. In addition, a fully programmable process enables to fabricate complex structures, for example, with various diameters and porosity gradient across the porous media in a single run (a robust alternative to structures assembled manually from separate layers as a "sandwich").



Fig. 1. Particle size distribution histogram of polystyrene after milling.

Table 1 presents the structural characteristics of the tested depth filters, which are schematically depicted in Fig. 3. To determine the diameters of the fibers forming the outer and inner layers, each filter was slit to the core depth. Individual layers (I and II as presented in Fig. 3) at different depth of filter media were observed using a scanning electron microscope Phenom G2 (Fig. 4). Diameters of fibers were sized using the NIS-Element image analysis software from Nikon. The second parameter, filter porosity, was provided by the manufacturer (Amazon Filters Ltd.).

# 2.3. Experimental set-up

The experimental setup was assumed that the pollutant particles (microplastics) were dispersed in the water and injected upstream the circulation pump, that is, on the suction side. The prepared suspension of fragmented polystyrene particles was mixed to prevent sedimentation or flotation of dispersed plastics, which allowed to obtain a uniform concentration in the feed tank T1. The variable speed motor enabled to adjust the desired flow rate based on the signal from the flow meter located downstream the filter test housing F1 (closed loop control). Apart from the flow, the pressure drop (dP) during the test was monitored. The rig used in water filtration experiments is schematically shown in Fig. 5.

## 2.4. Testing methodology

The first series of experiments on the effectiveness of microplastics removal from water was carried out for depth



Fig. 2. Scheme of the melt-blow system for the production of fibrous media: 1-motor, 2-screw extruder, 3-cooling zone, 4-heating sections, 5-granulate feed, 6-compressed air heater, 7-multichannel nozzle, 8-receiver drum.

# Table 1 Specification of the fibrous media used in experiments

filters with nominal removal ratings of 20 (DF20), 10 (DF10) and 5 (DF5) microns. In the test installation, the water flow rate was set at 300 L/h. The polystyrene suspension of approx. 12.5 g/L was injected to the main water stream at a rate of 200 mL/h, thus achieving the initial microplastic concentrations (at the filter inlet) of a few milligrams per liter. It should be noted that the inlet concentration in performed testing (calculated value: approx. 8 mg/L) although very low, was still significantly higher than the values reported in the literature for surface waters, which are usually only a few milligrams per m3 [27,28]. Throughout the experiment, the homogeneity of suspension of polymer particles in water was maintained by agitation using the magnetic stirrer. Importantly, the RO water was used to eliminate any contamination that could interfere with the subsequent visual identification of the microplastics. The test time was 30 min, while filtrate samples (1 L each) were collected every 10 min, and the inlet sample was taken only once at the beginning of the experiment. The sampling time was kept as short as possible and did not exceed 30 s.

In the second series of tests, two pleated cellulose elements (PE1, PE2) made of commercially offered materials were used (Table 2). In order to obtain the same inlet velocity as for depth filters (0.0016 m/s), and thereby maintain similar operating conditions, it was necessary to adjust the water flow rate in the rig taking into account the total filtration area of the pleated elements. As a result, the water flow rate increased to approx. 600 L/h for both PE1 and PE2. Other test conditions, such as suspension concentration and dosing rate, as well as the duration of the experiment and sampling frequency, were kept the same to enable a reliable comparison of various filters.

The collected samples were filtered in a vacuum system using PES filtration membranes with a pore size of 0.45  $\mu m.$  The volume of filtered water was determined

#### Table 2

Specification of the pleated cellulose elements used in experiments

	PE1	PE2
Number of pleats	45	45
Height of pleats, mm	15	15
Total height of element, mm	75	75
Maximum pore size*, µm	19	25

\*Based on the material specification.

Filter symbol		DF20	DF10	DF5	DF20	DF10	DF5	
Filtration rating		20 µm	10 µm	5 µm	20 µm	10 µm	5 µm	
Layer			Outer (I)			Inner (II)		
	min.	37.8	24.7	17.5	12.8	2.98	1.99	
Fiber diameter, µm	avg.	62.3	43.5	31.4	22.5	12.2	4.94	
	max.	120.2	72.8	52.8	40.5	26.2	16.4	
Porosity, %		70.6	71.9	72.2	74.6	76.5	78.2	

experimentally, taking into account the concentration of microplastics (i.e., was different for inlet and outlet samples) to make feasible to observe and size particles collected on the membrane. Therefore, 100 and 200 mL of water were filtered from upstream and downstream of the filter, respectively. A grid was pre-printed on each membrane to facilitate microscopic analysis of the retained particles. To ensure an even distribution of the microplastics on the membrane, the samples were constantly stirred during the filtration using a glass rod.

# 2.4.1. Microscopic analysis

The PES filters were analyzed under the optical microscope – image analysis using the Nikon NIS Element software. As previously mentioned, a grid was printed on the membranes dividing each of them into 1,590 square areas with dimensions of 800  $\mu$ m × 800  $\mu$ m. Polystyrene particles from randomly selected squares were sized and counted, and then extrapolated over the entire membrane surface. Based on the collected data, particle size distributions were determined and the fractional – number and volumetric (mass) – efficiencies of microplastics removal on the tested filters, were calculated. To obtain the volumetric efficiency, for each sample, the cumulative volume of the identified particles was estimated taking into account the shape factor. The results are presented and discussed in section 3.

# 2.4.2. Zeta potential

A Zetasizer Nano (Malvern Instruments Ltd.) compatible with the Dip Cell Kit (ZEN1002) was used to determine the surface charge of the polystyrene particles, and therefore their ability to aggregate in water. For this purpose microplastics were mixed with ultrapure water (pH = 7).

#### 3. Results and discussion

All depth filters used in the experiments showed a high level of microplastics removal from the water, as evidenced by the fractional efficiency values (Table 3). For samples taken after 30 min, the numerical efficiency was about 80%, while the volumetric efficiency exceeded 95%.

As expected, the removal of the smallest particles proved to be the most problematic issue. For microplastics with sizes  $<2 \mu m$ , a clear decrease of number efficiency with decreasing particle size was noted. For example, after 30 min of filtration on DF20 (Fig. 6), polystyrene granules

Table 3

Number and volume (mass) efficiencies of depth filters as a function of filtration time

	DF20	DF10	DF5	DF20	DF10	DF5	
	Number efficiency (%)			Volumetric (mass) efficiency (%)			
10 min	72.8	67.0	48.6	99.7	99.1	89,8	
20 min	74.8	71.8	68.3	99.8	99.7	99.4	
30 min	78.4	82.1	78.1	99.9	99.8	97.2	

with sizes from 2.5 to 3.0 µm were retained in 95%, for the range of  $1.5-2.0 \mu m$  the value dropped to 80%, while for the finest measured particles (<1.5 µm) the elimination efficiency decreased drastically to 27%. The two remaining depth filters DF10 and DF5 having better removal ratings (i.e., 10 and 5  $\mu$ ) showed significantly better results in capturing the smallest micropollutants (Figs. 7 and 8). The degree of particle elimination in the size range of 1.0–1.5 µm on DF10 and DF5 (after 30 min of the experiment) reached 84% and 70%, respectively. Fiber diameter and porosity are important parameters affecting the effectiveness of depth filters. DF20 is characterized by relatively coarse fibers, both in the outer and inner layers, and thus less specific surface area and larger pore sizes, which explains its low capability to retain the smallest particles compared to the other two tested filters. The high volumetric (mass) efficiencies obtained in all depth filter experiments indicate that larger microplastics (>5 µm), which contribute predominantly to the total particle mass, were removed with nearly 100% efficiency (i.e., in Figs. 6-8 particle fraction on the outlet approaching or reaching zero).

Table 4 shows the results of the comparative tests carried out with the use of surface filtration. It can be noted that in terms of numerical efficiency of polystyrene granules removal, pleated cellulose elements demonstrated similar performance to depth filters. However, the volumetric efficiencies achieved were lower – 95.7% and 92.7% (after 30 min) for PE1 and PE2, respectively – indicating that surface filtration is not such effective at capturing larger



Fig. 3. (a) Dimensions of the element (on the left) and (b) real image of the polypropylene filter cartridge used in experiments (on the right).



Fig. 4. Example scanning electron microscopy images of fibers from the outer (on the left) and inner (on the right) layers of a 10  $\mu$  depth filter.



Fig. 5. Flow diagram of the rig used in water filtration experiments: RO-reverse osmosis system, T1-agitated tank containing microplastics dispersed in water, P1-peristaltic dozing pump, P2-centrifugal pump with a controlled speed, F1-housing of tested filter, F2-cleaning filter, FT-flowmeter, DPI-differential pressure sensor, S1,S2-sample valves.

particles as depth filtration. This finding is somehow counterintuitive to commonly observed tendencies of solid filtration, where bigger particles are usually captured and remover with a higher efficiency. Although the aggregation of particles was not noticed on the collecting membrane, this effect can lead to spontaneous coagulation, which is more intense for smaller objects when driven by Brownian diffusion (co called perikinetic aggregation regime). Such cluster grow to the size bigger than the pore of the flat filter media and are retained, while bigger single granules (still smaller in size than pores) penetrate through the filtration barrier. This also can occur in depth filter media, but due to a high specific surface area the probability of deposition on the fibers by inertial or interception mechanism, the probability of passing the filter is much less. The above hypothesis explains observed tendencies when comparing depth and surface filters.

Moreover, it was observed that the filter operation time did not have a significant impact on the efficiency of the filtration process, both deep bed and surface – the microplastic content in the subsequent samples (taken 10 min apart) slightly decreased in most cases, however, in general, it remained at a very similar level (Tables 3 and 4). However, the process time was relatively short, the concentration of contaminants very low and no significant filter loading took place (which is confirmed by a constant value of the pressure drop over the test time as shown in Table 5).

For each filter, the experiments were performed in duplicate (while maintaining the adopted process conditions), and the presented results are the average values obtained for the microscopic analysis (counting and dimensioning of plastic granules identified on the membrane) carried out for two replicates. Standard deviations determined for the number of MPs at the filter outlet did not exceed 3.5%.



Fig. 6. The size distribution of particles (by number) upstream and downstream for filter DF20.



Fig. 7. The size distribution of particles (by number) upstream and downstream for filter DF10.



Fig. 8. The size distribution of particles (by number) upstream and downstream for filter DF5.

Table 4

Number and volume (mass) efficiencies of pleated cellulose elements as a function of filtration time

Table 5 Pressure drop for the face velocity (on filter inlet) equal to 0.0016 mm/s

	PE1	PE2	PE1	PE2	Filter symbol	Pressure drop (dP), Pa
	Number ef	ficiency (%)		Volumetric (mass)	DF20	250
		, , , ,		efficiency (%)	DF10	1,200
10 min	70 0	75 5	05.4	79.6	DF5	1,800
	70.0	75.5	95.4	78.8	PE1	1,100
30 min	79.0	78.4	95.9	92.7	DED	820



Fig. 9. The size distribution of particles (by number) upstream and downstream for filter PE1.



Fig. 10. The size distribution of particles (by number) upstream and downstream for filter PE2.

Despite the high concentration of microplastics at the inlet of the filters (higher than those actually observed in the aquatic environment, as reported in the literature), no noticeable loading of the filters was observed (the total mass of retained particles was very low). For all tested filters the pressure drop remained constant throughout the entire test time (Table 5). In all cases, the standard deviation for this parameter did not exceed 1.5% of the mean value.

The measured value of zeta potential is very low. According to the literature [29], for the solution to be physically stable, the values of this parameter should be below –30 mV or above +30 mV. The obtained values were

about –5 mV. It implies that the polystyrene microgranules may have a tendency to agglomerate. This can potentially explain the phenomenon described above related to retaining small particles and penetration of large ones for surface filtration.

## 4. Conclusions

The results demonstrate that even the smallest microplastics, that is, with a size of 1–2  $\mu$ m, can be effectively eliminated from the water by the deep bed filtration process. Despite the "difficult" experimental conditions (high concentrations of microplastics and a very small particle sizes), high removal rate of the tested micropollutants was achieved for a relatively coarse filter media (having relatively low values of the pressure drop). However, it is assumed that the use of depth filters with lower nominal removal ratings (<5  $\mu$ m) or the modification of the test system, for example, by including pre-coagulation or a second stage filtration, could result in a significant improvement in fractional efficiencies, which is crucial for capturing the smallest microplastics that pose the greatest challenge in terms of their removal from water.

It is worth mentioning that the presence of other solid contaminants is an important issue in research on the elimination of microplastics from water (and it creates a massive challenge in instrumental analysis of such mixed solids in a sample). As previously described, in the conducted experiments a medium devoid of any impurities was used. However, in real conditions (at water treatment stations), the presence of various contaminants in the treated water is inevitable, which may significantly affect the effectiveness of the filtration process, due to enhanced filter loading. In a properly performed deep bed filtration process (characterized by an appropriately designed element structure), the removed pollutants, depending on their size, are retained in subsequent layers of the filter material. Importantly, depth filters can operate at high efficiency without the risk of clogging the surface by the deposit layer (so-called filter cake), even if the characteristics of the incoming contaminants have not been identified and their dimensions vary widely. In turn, in the case of surface filtration, the different sizes of the retained particles contribute to the formation of a thick layer of impurities, which can be compressible and have a low permeability. As a consequence, the resulting filter cake may cause a significant increase in flow resistance in a relatively short time and thus necessitates the replacement or cleaning of the filter cartridge. Taking this into consideration, laboratory tests call for long-term studies as well as on the filter performance under real (environmental) conditions, for example, by adding mineral particles to the water (such as test dust), which has been planned for the future work.

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