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Microfibres and coliforms determination and removal from wastewater treatment effluent

Jolanda Rihter Pikl [a], Aleksandra Lobnik [a], Milenko Roš [b,*], Hakim El Khiar [c], Nataša Uranjek [d]

[a] University of Maribor, Faculty of Mechanical Engineering, Smetanova 17, 2000, Maribor, Slovenia
[b] Faculty of Environmental Protection, Trg mladosti 7, 3320, Velenje, Slovenia
[c] AQUA LAB d.o.o., Verdijeva 1, 6000, Koper, Slovenia
[d] Municipality of Velenje, Koroška cesta 17b, 3320, Velenje, Slovenia

A R T I C L E I N F O

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A B S T R A C T

The research aim was to remove as many microfibres, microplastics and harmful bacteria as possible from the polluted water to produce suitable water for reuse. The test water was the effluent from the municipal wastewater treatment plant in Shalek Valley. A pilot plant with a ceramic SiC filter for membrane filtration and ozonation of filtered water was set up to remove suspended solids, micro-fibres, microplastics, and harmful microorganisms. The Microfibers Detection System was developed to identify microfibers on-site. The results showed that the microfiltration system combined with ozone treatment effectively removed total suspended solids, microfibres, microplastics and microorganisms. A detection system method for identifying microfibres and microplastic particles was used to determine how many microfibres and microorganisms were identified by membrane filtration and ozonation. The study showed that membrane filtration successfully removed all microfibres, 88% of total coliforms and 93% of *E. coli*. After additional ozonation, we achieved a 100% removal rate of total coliforms and a 100% removal rate of *E. coli*. The treated water (effluent from the municipal wastewater treatment plant) can be used for specific purposes, such as agricultural irrigation or enhancing bathing waters near the plant's water effluent.

1. Introduction

The primary sources of microfibres include laundries, households (Henry et al., 2019; Le et al., 2022), and wastewater treatment plant effluents (Carr et al., 2016; Jiang et al., 2022; Reddy and Nair, 2022; Sadia et al., 2022). These microfibres pose a significant and growing environmental risk, emphasising the need for protective measures and sustainable practices in the textile and fashion sectors. Approximately 9 million tonnes of microfibres are produced annually, with synthetic fibres accounting for 60% and non-synthetic or natural fibres accounting for 25% (Das et al., 2023). Various methods for removing microfibres from wastewater have been proposed, such as filtration technology (Jiang et al., 2022; Liu et al., 2021; Nakao et al., 2021), pulse clarifiers (Sarkar et al., 2021), membrane bioreactors (Bayo et al., 2020; Lares et al., 2018; Li et al., 2020), oxidation processes (Easton et al., 2023), coagulation (Li et al., 2022), bioremediation (Li et al., 2022), or constructed wetlands (Sotiropoulou et al., 2023). While conventional wastewater treatment can remove suspended solids, including microplastics, it is crucial to address the presence of plastic fibres in watercourses due to their significant risk to aquatic organisms (Masiá et al., 2020). The efficacy of wastewater treatment depends on factors such as pollution levels, wastewater flow, and treatment processes, all of which impact the quality of the treated water (Afolalu et al., 2022). Additionally, attention must be given to monitoring and disposing of microplastics, mainly polyester and polyethylene, found in sewage sludge (Alavian Petroody et al., 2021). Most microplastic particles and fibres detected in wastewater samples are polymers such as polyethylene terephthalate (PET), polystyrene (PS), polypropylene (PP), and polyethylene (PE) (Sun et al., 2019). Research suggests that many particles are removed during the primary and secondary treatment stages, with the membrane bioreactor (MBR) being the most efficient technology for the tertiary treatment (Prata et al., 2019). The research conducted by Nguyen et al. (2021) demonstrated the effectiveness of using a stainless steel mesh filter with a pore size of 45 µm for removing microplastic particles, specifically from polyethylene (PE) and polypropylene (PP), in wastewater. Their findings suggest that filtration with this type of filter

* Corresponding author. Ziherlova ulica 6, 1000, Ljubljana, Slovenia.
E-mail address: milenko.ros@fvo.si (M. Roš).

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can significantly reduce the presence of microplastics in wastewater. The identification of nanostructured particles was carried out using scanning electron microscopy (Zhou et al., 2020) and presented methods for the identification of microplastics using a stereomicroscope and micro-Fourier transform infrared spectroscopy. They investigated the accumulation of microplastic particles in seed soils on agricultural land resulting from contaminated water. The study revealed the presence of microplastic particles ranging from 1 to 3 mm in the soil, with the majority falling within the size range of 90 μm to 1 mm in the water. Predominant types of microplastics detected were polyethylene (PE), polypropylene (PP), polyester (PES) and polyamide (PA). A similar experimental approach was employed in a study by Chen et al. (2020). FT-IR and micro-FTIR are the most effective methods for identifying microplastics due to their short analysis time. Spectra obtained from FT-IR analysis is highly accurate in determining microplastics down to a size of 6.6 μm (Kirstein et al., 2021). FT-IR can be used for spectroscopic analysis of wastewater samples to detect microplastic particles with a size of ≤20 μm (Xu et al., 2021). Another study by Franco et al. (2020) examined microplastic particles in wastewater using attenuated total reflection-Fourier transform infrared spectroscopy. Their findings highlighted that the most common fibrous forms of microplastics were within the size range of 100–355 μm, followed by 355–1000 μm, and finally between 1000 and 5000 μm. Libraries of spectra dominated by polyethylene (PE), polypropylene (PP), and polystyrene (PS) are commonly employed for the identification of microplastic particle spectra (Aliabad et al., 2019; Jung et al., 2018). The increasing pressures of a growing population, urbanisation, and consumption pose challenges to the carrying capacity of our aquatic environments. Implementing a circular economy model that emphasizes water reuse is crucial to address these challenges. Voulvoulis (Voulvoulis, 2018) highlights the transition to a circular economy as a critical strategy for promoting water reuse. Through an assessment of existing systems and regulations, it is emphasised that a circular economy approach should prioritise ensuring the safety of water reuse by meeting stringent water quality standards. Additionally, it should aim to guarantee the efficient and reliable operation of water reuse systems. Chen et al. (2021) provide a comprehensive overview of the various aspects and regulations of pursuing innovative and unconventional water reuse technologies. Their work serves as a valuable resource for promoting the adoption of sustainable practices. However, successfully implementing water reuse strategies necessitates an integrated approach that considers political, social, economic, technological, and environmental factors (Sgroi et al., 2018). Effective water reuse requires careful consideration of these diverse aspects to ensure a balanced and sustainable approach. Furthermore, it is essential to note that the reuse of treated water is subject to specific conditions, which must be met to ensure its safe and appropriate application. The European Directive (EU Regulation, 2020/741) establishes minimum requirements for the safe use of wastewater from urban wastewater treatment plants for agricultural purposes, aiming to protect both humans and the environment. This regulation specifies quality requirements for treated water in terms of parameters such as suspended solids (TSS), biochemical oxygen demand (BOD5), and bacteriological contamination (*E. coli*). In response to the increasing demand for water and sustainable solutions, there is a significant potential for recycling and reusing treated wastewater. Supporting and promoting the reuse of water for various purposes, including agriculture, industry, recreation, and environmental activities, is contingent upon monitoring and compliance with legal requirements (EU Regulation, 2020/741) and guidelines (ISO 16075:2015 - Guidelines for the Use of Treated Wastewater for Irrigation Projects). These guidelines emphasise the importance of assessing the risks of using treated water, including micropollutants and microplastics. Modern technologies are emerging as viable solutions to address the need for additional wastewater treatment. One such technology is hybrid pilot technology, which combines microfiltration using SiC membranes with ozone (O3) treatment. This system offers simplicity, low-pressure filtration, and the ability to simultaneously perform effective ozonation or disinfection (Rihter Pikl et al., IJES, 10, 2021). Similar technologies involving ozonation, coagulation, and ceramic membrane filtration have also demonstrated promising results in wastewater reuse systems, with coagulation proving more efficient in virus removal and ozonation after membrane filtration improving hygienic safety (He et al., 2013, 2019; Li et al., 2020). Scientific publications by Zhang and coauthors (Zhang and Chen, 2020) provide insights into various physical and biological treatment methods, membrane and carbon treatment methods, membrane bioreactors (MBR), flotation techniques, microbial degradation, and enzymatic digestion.

2. Materials and methods

We developed a hybrid filter unit that combines a SiC membrane with a pore size of 100 nm and an ozone generator, known as microfiltration with an ozonation system (MFO). This system has been designed to efficiently filter wastewater from wastewater treatment plants through membrane filters, aiming to remove contaminants such as suspended solids (TSS), fibres, microplastics, total coliforms and *E. coli*.

The SiC membrane utilised in our system is designed to facilitate high flow rates and possesses a negative charge, which helps to minimise organic contamination and the growth of microorganisms. Membrane technology offers a new and efficient approach to further wastewater treatment. When combined with ozone treatment, the microfiltration with an ozonation system further enhances the removal of microorganisms. Through our research, we have demonstrated the effectiveness of this approach in removing various pollutants, ultimately producing treated water that meets the necessary standards for safe water reuse.

2.1. Experimental methods

To conduct our research, we collected samples of treated water (effluent) from the Shalek Valley wastewater treatment plant, which has a 50,000 population equivalents (PE) capacity. This treatment plant utilises a biofiltration process with attached biomass. The samples were collected continuously over 24 h using an automatic sampler and stored in specialised refrigerated containers to ensure their integrity.

From the average 24-h samples, we extracted 10 L of water for further processing in the microfiltration and ozonation system. The water underwent pre-treatment by passing it through a microfilter, and then the filtered sample was subjected to ozonation treatment.

Both the current samples, obtained from the 24-h sample and the sample post-membrane filtration of the effluent, were then analysed for suspended solids, including fibres and microplastics, using analytical techniques such as Fourier Transform Infrared Spectroscopy (FT-IR) and the microfibres detection system.

Furthermore, after the ozonation process, the effluent and filtered water were examined for total coliform bacteria and *Escherichia coli* (*E. coli*). For this examination, the 1-L portions of each sample were passed through a specialised filter, a component of our microfibers detection system, which has been tailored for microfibers and microplastics analysis.

2.2. Analytical methods

Our analysis adhered to industry-standard methods to ensure accuracy and reliability. The suspended solids were determined using the ISO 11923:1997 method, which involves filtration through glass fibre filters. To assess the presence of coliform bacteria and *E. coli*, we followed the EN ISO 9308–1:2014 protocol.

The FT-IR (Fourier Transform Infrared) method was utilised for microplastic analysis. This technique allows for identifying microplastic particles based on their unique infrared spectra. Our reliance on the FT-IR method is supported by the works of Huppertsberg and Knepper

(Huppertsberg and Knepper, 2020) and Tagg et al. (Tagg et al., 2015), which have established its effectiveness in microplastic detection and characterisation. By employing these established methods, we ensure robust and standardised procedures for determining the presence of microplastics in our samples.

2.3. Pilot plant for microparticle removal

The research entailed the development of a pilot unit called the microfiltration with ozonation system (MF), which combines microfiltration and ozonation to remove microparticles and disinfection (Fig. 2). The MFO unit was utilised to treat biologically treated water, and additional ozonation was applied to the treated water for further analysis.

The MFO pilot plant consists of the following parts: reservoir 1 (volume about 20 L), membrane filter (surface area 462 m^2), reservoir 2 (volume about 15 L), ozoniser (capacity of 13.4 mg/L of O_3) and corresponding valves and connections (Fig. 1).

The research sought to provide a comprehensive solution for removing microparticles and disinfection in wastewater treatment by utilising the MFO system, which combines microfiltration and ozonation. The treated water from this system was subjected to further analysis to evaluate its effectiveness in removing the targeted suspended solids and improving the overall quality of the treated water.

The characteristics of MFO operation are:

- The total surface area of the SiC membrane is 462 cm^2
- The pore size of the membrane is 100 nm
- Flow through the membrane: 0.6 min/L = 21.645 L/h = 21.645 m^3/h
- Water permeability is > 3000 LMH/bar at 20 °C.
- Ozonation: 13.4 mg O_3/L = 13.4 g/m3
- The lifetime of the membrane is 20 years.

During the process, a slight vacuum is applied to the membrane, creating a pressure difference and allowing water to be drawn through the membrane's pores. SiC (silicon carbide) membranes are particularly advantageous in this application as they are naturally and permanently hydrophilic. This hydrophilicity means that SiC membranes repel organic contaminants, such as oil while attracting water molecules.

2.4. Microfibres detection system

The microfibres detection system was explicitly designed to analyse the presence of microfibre particles, such as fibres and other microplastics. This innovative system, depicted in Fig. 1, provides a rapid and accurate method for determining the levels of microplastics in various samples. At the heart of the microfibres detection system is a membrane-supported filtration system. This integral component plays a critical role in the analysis process by effectively capturing and isolating microplastic particles from the tested sample. Utilising a membrane-supported filtration system, the microfibres detection system ensures high filtration efficiency, allowing for reliable and precise identification of microplastics. This system enables researchers, scientists, and environmental professionals to gain valuable insights into the presence of microplastics in different environments, ultimately contributing to efforts towards preserving our ecosystems.

We employ a technique where about a 1-L sample (depending on the amount of microfibres) is pumped through the membrane to expedite identifying microfibres. Subsequently, microfibres and microplastics can be evaluated and analysed. A microscope and a smartphone or tablet are necessary for this evaluation to be practical. Hence, we utilise a wireless microscope connected to the phone or tablet via Bluetooth for seamless and convenient microplastic characterisation (Fig. 3).

3. Results and discussion

During the microfiltration process, we conducted experiments using wastewater from the biological stage and treated water from the wastewater treatment plant. To facilitate clarity in our discussions, we have provided definitions for the following terms:

- *Effluent*: Refers to the treated wastewater discharged from the treatment plant post-treatment.
- *Filtered water*: Indicates the sample that has traversed the membrane.
- *Ozonated water*: Denotes the filtered water undergoing an additional treatment step involving ozone.

3.1. Removal of total suspended solids

After treatment, the effluent still contains a minor bioburden of total suspended solids (TSS). It may also contain bacteriological contaminants such as total coliforms and *E. coli*. Our study aimed to analyse the

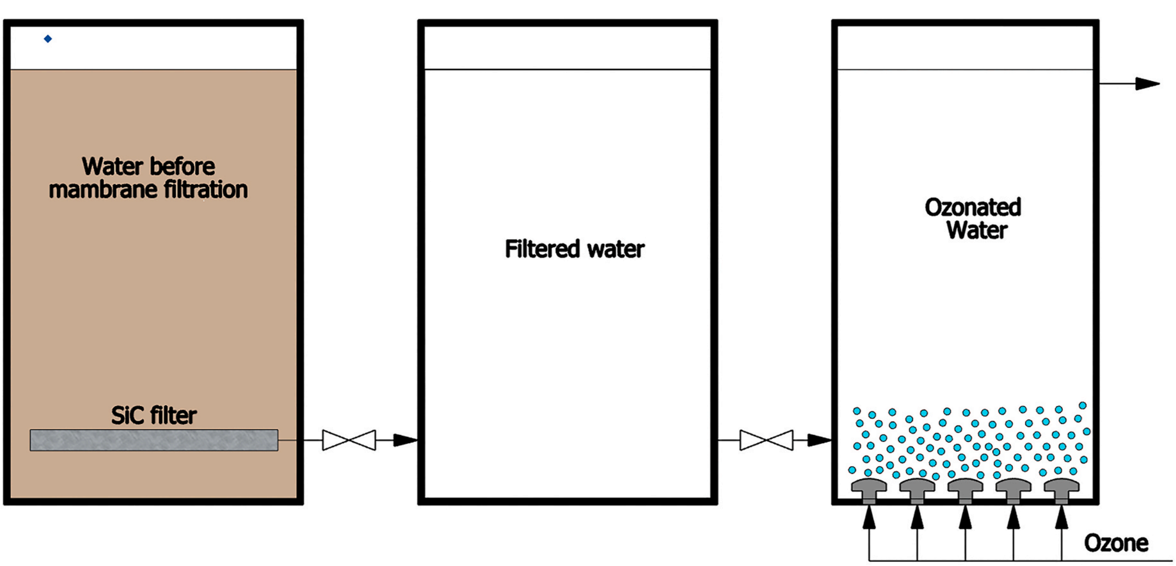

Fig. 1. System for membrane filtration and ozonation.

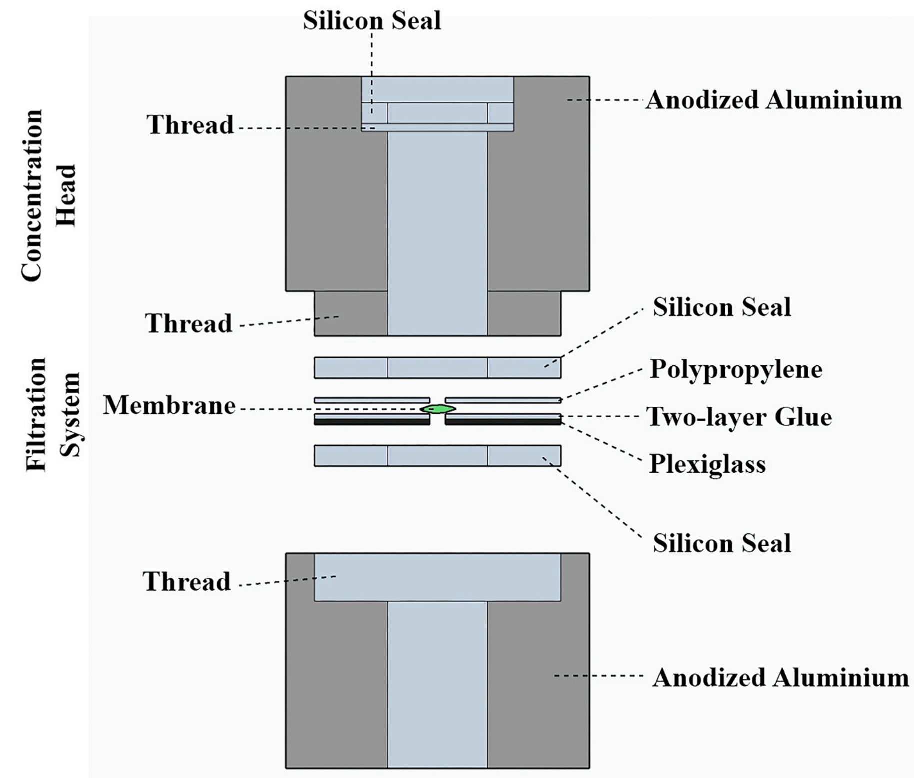

Fig. 2. Microfibres sampling scheme.

Fig. 3. Microfibres Detection System (a – sampling filter; b – microscope; c –smartphone).

composition of the current effluent sample.

The TSS concentrations in the effluent range from 0.9 mg/L to 3 mg/L. However, after implementing microfiltration, the concentration decreased to below 0.01 mg/L (detection limit). This demonstrates that microfiltration achieved an impressive treatment efficiency of up to 97 %, highlighting the effectiveness of this filtration method.

We visually inspected the filter surface after subjecting the effluent to microfiltration. We observed that the membrane effectively retained almost all of the microfibres, indicating the high efficiency of microfiltration in removing microfibres from the wastewater.

3.2. Analysis of microplastic particles and fibres with a microplastic detection system

We used a filtration device with a concentration disc to analyse the presence of microplastic particles and fibres in the effluent. This enabled us to pass 1 L of the sample through it and collect the filtered water in a measuring vessel. The filtration device's membrane effectively captured the wastewater's microplastic particles and fibres.

The following are the results of two samples taken at different times. We visualised the filter surface and identified particles and microfibres within 1 L of influent water. Additionally, we examined the shape and colour of the microfibres, as shown in Figs. 4 and 5. These observations verified that the influent was contaminated with various suspended solids, such as microfibres, microplastics, and organic suspended solids.

After subjecting the samples to microfiltration, we performed another visual examination of the filter surface (Figs. 6 and 7). We discovered that the membrane successfully captured nearly all of the microfibres, demonstrating the high efficiency of microfiltration in eliminating microfibres from the effluent.

3.3. FT-IR spectra of microplastic particles

We used an FT-IR spectrophotometer to perform a more precise analysis of the microplastic particles in the effluent.

Smaller signals were observed at the wavenumber 3298 cm^{-1}, indicating the presence of a hydrocarbon-polyamide (PA) material in the effluent. A more significant signal was detected at wavenumber 1377 cm^{-1}, characteristic of polypropylene (PP). However, upon consulting the literature, we were unable to conclusively identify a specific material corresponding to the signal at the wavenumber 872 cm^{-1}. It is possible that this signal could be a mixture of different polymeric materials, or it may be a result of noise (Jung et al., 2018).

After applying microfiltration to the effluent, only two smaller signals were obtained at the wavenumbers 3243 cm^{-1} and 1373 cm^{-1}. This

Fig. 4. Microfibres and other particulate residues present – sample 1; a – 100× magnification, b, c – 400× magnification.

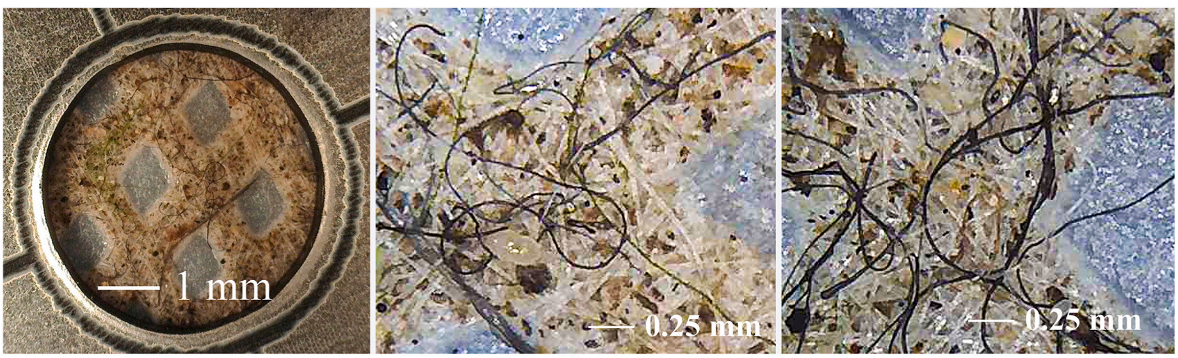

Fig. 5. Microfibres and other particulate residues present – sample 2; a – 100× magnification, b, c – 400× magnification.

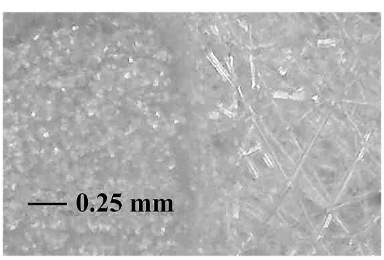

Fig. 6. Sample 1 – Microfibres and other particulate residues present observed after microfiltration (no residues present).

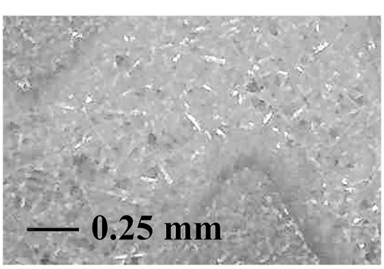

Fig. 7. Sample 2 – Microfibres and other particulate residues present observed after microfiltration (no residues present).

indicates that the membrane effectively retained most of the particles described in this analysis. However, due to the low intensity in the spectrum, we could not identify a precise material based on existing literature. These signals could potentially be impurities of other materials or simply noise.

3.4. *Values of total coliform bacteria using the MFO system*

We analysed the current effluent sample to evaluate its quality. Initially, the total coliform bacteria in the effluent measured exceeded 200.5 MPN/100 mL. However, with the implementation of microfiltration, the coliform bacteria count decreased from 18.3 MPN/100 mL to 40.6 MPN/100 mL, indicating a treatment efficiency of approximately 80%–90%. According to EU Regulation 2020/741, this water would be classified as quality class B for treated water.

Ozone disinfection was implemented to enhance the treatment process further. This additional step significantly reduced coliform bacteria levels to less than 1 MPN/100 mL (Table 1). This demonstrates a treatment efficiency of 100%, and according to the regulation, the water would now be classified as the highest quality class A for treated water.

The treated water meets all the criteria specified for agricultural irrigation, which are established by law in categories A to D. Additionally, the law permits using this water for other purposes, including industrial applications, recreation, and fulfilling environmental needs (EU Regulation, 2020/741).

4. Conclusions

Adhikari and Halden (Adhikari and Halden, 2022) report that in the developed world, over 109,000 WWTPs are inventoried in 129 countries, serving 2.7 billion people worldwide. This means that over 50 million m^3 of treated water flows daily from such plants into watercourses containing suspended solids, many of which are microparticles, microplastics, and harmful microorganisms. Our research has shown that it is possible to completely remove microfibres and harmful microorganisms from the effluents of biological wastewater treatment plants.

In our quest to tackle this challenge, we harnessed the power of a unique membrane filter system complemented by an ozone generator

Table 1
Total coliform bacteria (MPN/100 mL) and *E. coli* in the effluent, filtered and ozonised effluent using the MFO system.

Sample	Total coliform bacteria			*E. coli*		
	Effluent	Filtered water	Ozonised water	Effluent	Filtered water	Ozonised water
1	200,5	23,1	<1	200,5	13,4	<1
2	200,5	25,2	<1	200,5	14	<1
3	200,5	27,1	<1	200,5	15	<1
4	200,5	40,6	<1	200,5	13,7	<1
5	200,5	32,7	<1	200,5	14,8	<1
6	200,5	29,9	<1	200,5	34,4	<1
7	200,5	18,3	<1	200,5	16,4	<1

(MFO) tailored to our research objectives. The SiC membrane filter with a pore size of 100 nm demonstrated exceptional effectiveness and practicality. Its design sets it apart, featuring high flow rates (Water permeability >3000 LMH/bar at 20 °C) and a negative charge to minimise the risk of organic contamination and microbial growth. Moreover, the MFO system paves the way for innovative ozonation as an additional treatment approach.

By utilising the MFO system, we successfully removed microplastics and suspended solids from the WWTP effluent. Furthermore, the filtration process through the SiC membrane filter eliminated over 88% of coliform bacteria and up to 93% of *E. coli*; when targeting microorganisms incorporating additional ozonation, we achieved complete removal (100%) of total coliform bacteria and *E. coli*.

To monitor suspended solids in the water, we developed a microfibre detection system that allowed us to quickly monitor the quality of the effluent for suspended solids and microfibres.

Our findings demonstrate that the treated WWTP effluent, processed through the MFO system, complies with the minimum requirements for reusing treated water from municipal wastewater treatment plants, as defined by EU Regulation 2020/741 and ISO standard 16075:2015. These investigations will offer valuable insights into the efficiency and suitability of our treatment approach for producing high-quality treated water for irrigation.

CRediT authorship contribution statement

Jolanda Rihter Pikl: Writing – review & editing. **Aleksandra Lobnik:** Writing – review & editing. **Milenko Roš:** Writing – review & editing. **Hakim El Khiar:** Writing – review & editing. **Nataša Uranjek:** Writing – review & editing.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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