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Application of membrane filtration-ozonation (MFO) system for additional water treatment

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--*ABSTRACT*--
Treatment of municipal wastewater has excellent potential in the circular economy, especially for water reuse. When we would like to reuse treated wastewater, we should additionally purify WWTP's effluent. Our research work was introducing membrane filtration with an ozone generator (MFO) to treat the effluent from municipal wastewater treatment plants. We used a SiC membrane filter with pores of 100 nm. The main goal was to remove as many suspended solids, organic matter, and bacteria as possible to obtain water that could be reused for various purposes. After membrane filtration of treated water (WWTP's effluent), the following treatment efficiency was achieved, namely: 55% of COD, 92% of total suspended solids (TSS), 88% of total coliform bacteria (MPN), and 93% based on Escherichia coli (E. coli). The membrane itself does not allow sufficient removal of pathogens, so we further ozonated the filtered water. After ozonation of filtered water, we achieved the following additional effects: 5% of COD, 100% of total coliform bacteria (MPN), and 100% of E. coli. The study confirms that treated wastewater with membrane filtration and ozone disinfection meets the minimum requirements for reusing treated water from municipal wastewater treatment plants regarding the new EU Regulation [1].
KEYWORDS; municipal wastewater, MFO system, water reuse, ozonation, SiC membrane filter
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I. INTRODUCTION

In the last decade, membrane bioreactors (MBRs), which include the biochemical decomposition of waste materials and activated sludge suspension separation by membrane filtration, have been enforced very intensively [2-4]. SBRs are useful in removing organic and inorganic pollutants and, above all, in separating suspended solids. In membrane biological reactors, we have better control over biological activities, and we get a high-quality effluent at a higher level of organic load [1].

Researchers in Delhi treated wastewater from a Luxury Hotel because of a lack of water. They wanted to get water for reuse. MBR technology was used to treat wastewater to a suitable recycling level [5]. Membrane bioreactors operate most often in the range of microfiltration and ultrafiltration [2, 6-8].

In environmental protection, the profession focuses on resources management and water management by treating wastewater to such an extent that it can be reused [9-16]. It is related to the circular economy model, including water consumption by including water reuse in the environment and the cycle's shape in production processes [17-19]. Wastewater recycling and reuse technologies represent a new alternative to reducing pressures on water resources. Treated wastewater can only be reused under certain conditions by legal regulations and other environmental guidelines [1]. The regulation's primary objective is to protect the environment and human health from the harmful effects of possible contamination of water reuse. This goal can be achieved by setting minimum water quality requirements and monitoring preventive measures and comprehensive risk management at the EU level. The objectives align with other Union policies [20] and the United Nations Sustainable Development Agenda [21].

Two objectives are particularly crucial for the water strategy, which aim to improve water quality by 2030 by reducing pollution, eliminating, and discharging hazardous chemicals and materials, halving the share of untreated wastewater, and increasing the recycling and reuse of safe water [22, 23]. Legislation on minimum water quality requirements [1] can ensure that part of the treated wastewater is reused for purposes such as irrigation in agriculture, process water for industrial and recreational purposes, and in the field of the environment. The reuse of treated urban wastewater is also defined in the United Nations Report [21], stating

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that the reuse of water in agriculture has great potential, as agriculture is an essential water source. Data show that in spring 2014, the agricultural sector consumed around 66% of all water consumed in Europe. If more than half of the water treated from municipal wastewater treatment plants were reused for irrigating agricultural land in the European Union, water stress would be reduced by more than 5%. It is also one of the ways to protect water resources [24]. Four countries in southern Europe (Greece, Italy, Portugal, and Spain) have already adopted reusing treated wastewater regulations [15]. Practice so far shows that treatment plants have been built near the sea and rivers, which is not economically optimal in water reuse [16]. Discharges from treatment plants affect the change of parameters in the aquatic environment to which they flow, e.g., suspended solids containing bacteria settle on the water bottom and harm the environment and aquatic organisms [25-27]. We cannot remove some heavy-degradable and non-degradable organic substances used in medicines, cosmetics, and various plant protection products with conventional biological wastewater treatment. The products of their decomposition usually remain in treated water, which is transferred to the aquatic environment. Procedures are being developed to remove these substances, such as membrane technologies, advanced oxidation processes, and electrochemical treatment processes [28, 29]. Wastewater treatment depends mainly on the pollution and flow of wastewater, and treatment processes affect the quality of treated water [30, 31]. For water reuse, wastewater from the treatment plant must be additionally treated and discharges monitored by monitoring individual parameters: the amount of total suspended solids (TSS), chemical (COD) and biochemical oxygen demand (BOD_5), pollution with nitrogen and phosphorus compounds, and bacteriological contamination. For the removal of substances that conventional biological processes cannot remove, sound systems are being developed, such as membrane technologies, advanced oxidation processes and electrochemical treatment processes [6, 32].

In our study, the so-called MFO system is introduced for additional wastewater treatment. The abbreviation MFO means wastewater treatment by microfiltration (SiC membrane, pore size 100 nm) with additional ozone treatment (O_3). The system is simple, as it does not require high filtration pressure, and in parallel, allows adequate disinfection-ozonation. So far, a similar type of technology has been introduced by Japanese scientists [7], who have developed a wastewater reuse system that has included ozonation, coagulation, and ceramic membrane filtration. Dongbum with co-authors [14] and Im with co-authors [33, 34] used ceramic membrane microfiltration in wastewater reuse and compared ozonation and coagulation. The results showed that removal of viruses by coagulation was more efficient than pre-treatment with ceramic membrane filtration, while ozonation proved to be more effective after membrane filtration. It means that treated water's hygienic safety can be further improved if ozonation is used after membrane filtration.

The authors performed modern wastewater treatment in the production of crude oil production [7, 35], who used a combined system of pre-zoning and microfiltration. The results showed that ozone pre-treatment changed the chemical composition (pH and conductivity) of wastewater. Additional ozone treatment was effective in reducing membrane resistance and reducing chemical oxygen demand (COD).

A similar hybrid system (ozonation, coagulation, and membrane filtration) has also been shown to be effectively removed the organic matter from wastewater, e. g. COD Mn by 50% and colours by about 80% [36].

Recent studies have shown [7, 35-37] that membrane filtrations are useful in many areas. However, they also mention the disadvantages and limitations of membrane fouling and clogging [14, 37].

Clogging depends on the particles being filtered and the material from which the membrane is made. Clogged membranes can be cleaned by a backflow of filtrate or chemically with soda or citrate and by acid (HCl, H_2SO_4) and oxidant (hypochlorite, H_2O_2) EI EU [38]. The study [39] confirms that pre-treatment of wastewater with ozone (O_3) and biologically activated carbon (BAC) can reduce the frequency of dry cleaning, thereby extending the life of membranes. In our study, we performed a backflow filter cleaning that proved to be appropriate.

Slovenia has a large amount of water at its disposal, as it has the second most water per capita in Europe. In 2018, the amount of water consumed per capita was 59.3 m^3, the European average. In the same period, 766 million m^3 of wastewater was discharged from industrial activities [40]. Water consumption, however, has increased sixfold in the last 100 years. In addition to high water consumption, emissions of hazardous substances into water also increase, deteriorating its quality and the suitability of available water resources for use [40]. Although there is a lot of water available in Slovenia, due to the uneven distribution of water (especially in summer) and climatic conditions (drought, floods), it is necessary to ensure that part of the treated water is reused. The minimum requirements for the safe reuse of treated municipal water reflect available scientific knowledge and internationally recognized practices regarding the reuse of water and ensure that such water can be used safely for irrigation in agriculture, including industrial, recreational, and environmental purposes [1]. The Annex to Regulation 2020/741 sets out the quality requirements for processed water defined in quality classes A to D. Limit values: for *E. coli* is $\leq$ 10 MPN/100 mL, for $BOD_5 \leq 10$ mg/L, for TSS $\leq$ 10 mg/L and turbidity $\leq$ 5 NTU. These values correspond to quality class A by including technology's framework objectives: secondary treatment, filtration, and disinfection. Limit values for quality class B (secondary treatment and disinfection) are set for: *E. coli* ≤ 100 MPN/100 mL; other parameters comply with Directive 91/271/EEC (EU Directive 91/271/EEC, 1991). For quality class C the limit value for *E. coli* is ≤ 1000 MPN/100 mL, and for class D the value of *E. coli* is ≤ 10 000 MPN/100 mL. The other parameters are following the Directive [38].

This study introduced a new experimental method of additional wastewater treatment (after biological treatment) with the MFO system (Micro Filtration and Ozonation). The goal of the method was to obtain potentially usable water by meeting the minimum requirements for reuse of treated water from municipal treatment plants, which, in addition to other parameters (COD, TSS), also include microbiological elements (e. g. coliform bacteria, *E. coli*) as defined in EU Regulation [1].

II. MATERIALS AND METHODS

Our research work was based on the additional modern treatment of treated water (effluent) from municipal wastewater treatment. The purpose of our research work was to obtain usable water with a SiC membrane filter and ozone generator (MFO), which would remove part of the remaining contaminants (COD, total suspended solids (TSS), and total coliform bacteria - MPN/L).

Treated water (effluent) was obtained at the Shalek Valley municipal wastewater treatment plant, which operates on the attached biomass principle. Near the wastewater treatment plant, agricultural areas could be irrigated, especially in the summer months, with treated water from the treatment plant. To achieve adequate water quality, we constructed a filter device to analyse the water parameters and determine their reuse suitability. Treated water (effluent) was filtered through a silicon carbide (SiC) membrane with pores of 100 nm. The unique feature of this membrane is that it has a negative charge, so the possibility of organic contamination, oil contamination, microorganisms' development on the surface of the filter's surface is reduced. At the same time, the selected ceramic filter does not require high pressure.

Treated water filtered through a membrane filter was also treated with ozone (disinfection). Chemical analysis showed that treated water could be reused for irrigation, rinsing surfaces around the treatment lant, rinsing roads, watering golf courses or football fields, and firewater.

Based on the results, we found that treated wastewater with membrane filtration and ozone disinfection meets the minimum requirements for reuse of treated water from municipal wastewater treatment plants, which, among other parameters, include microbiological elements (e. g. coliform bacteria, *E. coli*), defined in EU Regulation of the European Parliament and the Council [1].

Materials

The tests were performed at a municipal wastewater treatment plant (WWTP) with a capacity of 50,000 population units (PE), which operates by a fixed biomass biofiltration process. We sampled treated water (effluent after the biological treatment), which flows from the WWTP to the nearby river Paka after treatment. The effluent was analysed before and after membrane filtration on the parameters: concentration of organic matter (COD), total suspended solids (TSS). After ozonation, we also performed tests for the presence of total coliform bacteria and *E. coli*.

Development of MFO pilot plant

Our first step was to assemble a membrane filter with an ozone generator (MFO) that would be easy to use and require minimum filtration pressure. For the research work, we constructed a pilot plant for membrane filtration with additional ozonation. The MFO pilot plant consists of the following parts (Figure 1): 1.) Reservoir 1 (R1), 2.) Filter (F1 and F2), 3.) Reservoir 2 (R), 4.) Microfilter (MF), 5.) Control panel, 6.) two pumps (P1 and P2) power 30 W (15 V, 1.6-2.5 A), 7.) Diaphragm pump (2.4 bar), 8.) Valves (V1, V2, V3, V4) and corresponding connections.

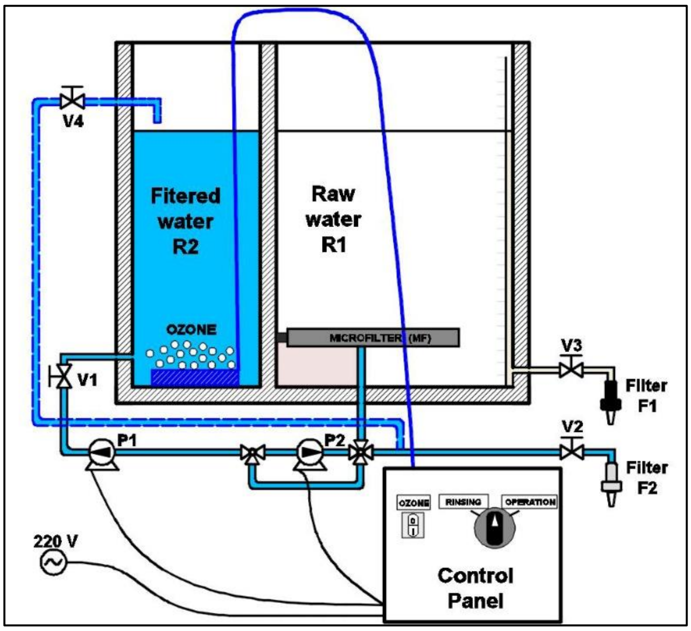

Figure 1: Scheme of the MFO pilot plant

We prepared 20 L of treated water (effluent) and analysed samples before and after filtration on the parameters: chemical oxygen demand (COD), suspended solids (TSS) and microbiological analysis of water - coliform bacteria and *E. coli*. A certain amount of water flowing out of WWTP was poured into reservoir 1 (R1), in which a microfilter made of silicon carbide (SiC) with pores of 100 nm, manufactured by OVIVO (Denmark), and constructed by TF LAB d.o.o. The active area of the microfilter is 462 cm^2. The purpose of MF is to retain suspended solids more significant than 100 nm (activated sludge residues, microfibers, bacteria). A microfiber sampling filter is installed behind the V3 valve. For microfiber analysis (size 300 nano-bubbles up to 5 mm), take a certain amount of water sample from R1 and drain some of the water through the filter by inserting a filter cloth (e.g., felt of a specific gradation) into the device, open the valve (V3) and filter a certain amount of water. From R1, the water is pumped (filtered) into tank 2 (R2), volume 8 L, where the filtered water flowing through the microfilter is collected. The control panel is multi-purpose. It contains a) ozone generator, b) a switch for pump drive 1 (P1), and c) a switch for pump drive 2 (P2). From the control panel, the pipe leads to tank R2, through which ozone is conducted. At the bottom of R2 is a ceramic diffuser. The exact time required for microfiltration of a certain amount of water from R1 to R2 was measured. The process of water passage through the membrane took 3.5 min / 5 L of water. After treating filtered water and taking samples for microscopic analysis, the ozone generator was turned on for 2 minutes. 107 mg of O_3 was used for 8 L of filtered water ozonated for 2 minutes. We performed the same analyses as before, including microbiological analysis (coliform bacteria, *E. coli*).

Complete operation conditions of MFO system were:

- complete surface of the SiC filter: 462 cm^2 = 0.0462 m^2
- filter pore size: 100 nm
- flow rate through filter: 0.6 min/L = 21.645 L/h = 21,645 m^3/h
- ozonation: 13.4 mg O_3/L = 13.4 g/m^3
- filter lifetime: 20 years.

Methods

The following standard methods we used: ISO 15705 [41] for COD; ISO 11923 [42] for TSS; EN ISO 9308-1 [43] for coliform bacteria and *Escherichia coli*; EN ISO 6878 [44] for total P, and ISO 29441 [45] for total N.

III. RESULTS

Microfiltration was performed with a water sample. The results contain the following definitions:

- Effluent - treated wastewater flowing from the treatment plant,
- Filtered water, which passes through the membrane (from R1 to R2) and
- Ozonised water - water that passes through the membrane (from R1 to R2) and additionally treated with ozone in R2.

COD reduction from biological treated water (effluent)
The effluent was passed through a membrane filter. The COD value of effluent ranged from 39 mg/L to 45 mg/L (average value 41 mg/L). After membrane filtration, the value of COD was from 13 mg/L to 15 mg/L (average value 15 mg/L). It means that the average treatment efficiency was 64.1%.
EU Directive [38] sets COD limits for effluents at 110 mg/L. The results showed that microfiltration could achieve values that are much smaller than prescribed.
TSS reduction from biological treated water (effluent)
The statutory limit value for TSS at the outlet is 35 mg/L [38]. In the WWTP's effluent, the amount ranged from 6.6 mg/L to 14.0 mg/L (average value 9.75 mg/L). After microfiltration, the value decreased to 0.3 mg/L and to 0.5 mg/L (average value 0.4 mg/L), which means that microfiltration was successful. Treatment efficiency was 98.9%.
The results show that microfiltration can meet the quality requirements of processed irrigation water in agriculture. Regulation [1] sets the TSS value < 10 mg/L, which means that the processed water belongs to quality class A and applies to all irrigation methods for food plants consumed raw, the edible part of which is in direct contact with processed water, and roots and tubers for eating raw.

Microscopic images of TSS
The analysis of TSS was performed by filtrating 2 L of concentrate from R1. The effluent was filtered through a felt filter. The filter was photographed under a microscope (1600 x magnification). The microscopic image of the effluent was polluted with suspended solids and plastic microfibers (Figure 2).

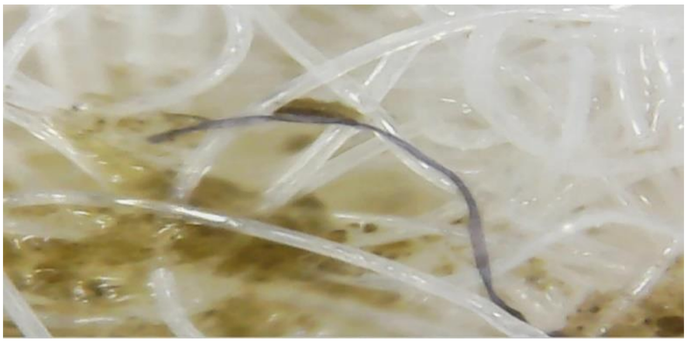

Figure 2: TSS on the felt filter after filtration of the effluent (1600 x magnification)

After membrane filtration of the WWTP's effluent, there were no more visible suspended solids (Figure 3) on a felt filter.

Figure 3: TSS on the felt filter after using MFO system (1600 x magnification)

Reduction of N and P compounds after microfiltration of effluent
The total nitrogen value at the effluent ranged from 13.9 to 15.8 mg/L (average value: 14.7 mg/L), and after microfiltration 5.8 to 12.0 mg/L (average value: 8.3 mg/L). The total phosphorous value at the effluent ranged from 1.08 to 1.43 mg/L (average value: 1,24 mg/L), and after microfiltration 0.44 to 0.49 mg/L (average value: 0.47 mg/L). The main reason for such relatively high total N and P reduction is the decrease of TSS after microfiltration. The average total N reduction was about 44% and about 58% for total P.

COD of effluent after MFO Process
The COD value at the effluent ranged from 39 mg/L to 45 mg/L (average value 41 mg/L). After microfiltration, the value decreased to 12 mg/L and 14 mg/L, respectively (average value 13 mg/L), which

means that the average treatment efficiency was 68.3%. After disinfection with ozone, the COD fell to 11.6 mg/L and 12.4 mg/L (average value 12,0 mg/L). We can conclude that there was slight oxidation; the COD was partially removed after the ozonation step. The average treatment efficiency was 7.7% (Figure 4).

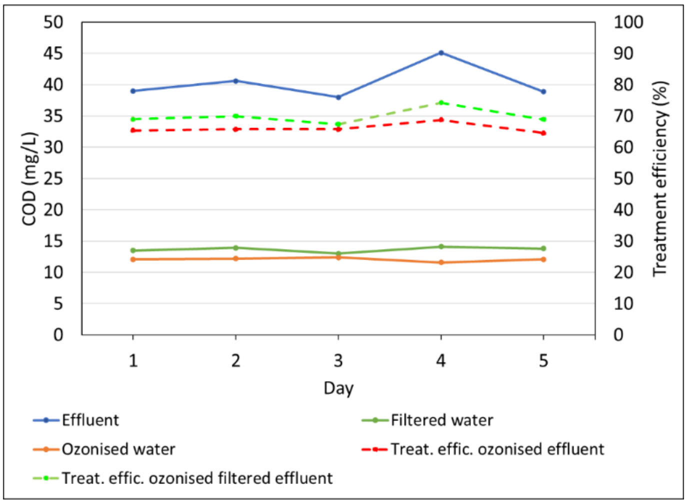

Figure 4: COD concentration of effluent using the MFO process.

Total coliform bacteria (MPN/100 m/L) in effluent using the MFO process

The value of total coliform bacteria at the effluent was > 200.5 MPN/100 mL. The amount was reduced to 23.1 MPN/100 mL to 40.6 MPN/100 mL after filtration, indicating a treatment efficiency of 79.7% to 88%. After disinfection with ozone, coliform bacteria's value decreased to <1 MPN/100 mL, which means that the treatment efficiency was close to 100% (Figure 5).

The Regulation (Union, 2020) determines the bacteriological value of quality classes of processed water from A to D. The results show that microfiltration can achieve water quality corresponding to quality class B, i.e., up to a value of <100 MPN/100 mL. This water can be used in agriculture in all irrigation of food plants that are eaten raw and the edible part of which grows above the ground and is not in direct contact with processed water and processed food and non-food plants, including cultivated plants used to feed animals for milk and meat production. After ozonation of the filtered water, the processed water corresponds to quality class A, i.e., up to a value of <10 MPN/100 mL, which means that it corresponds to the use of all irrigation methods for food plants consumed raw, the edible part of which is in direct contact with the processed water, and roots and tubers were eaten raw.

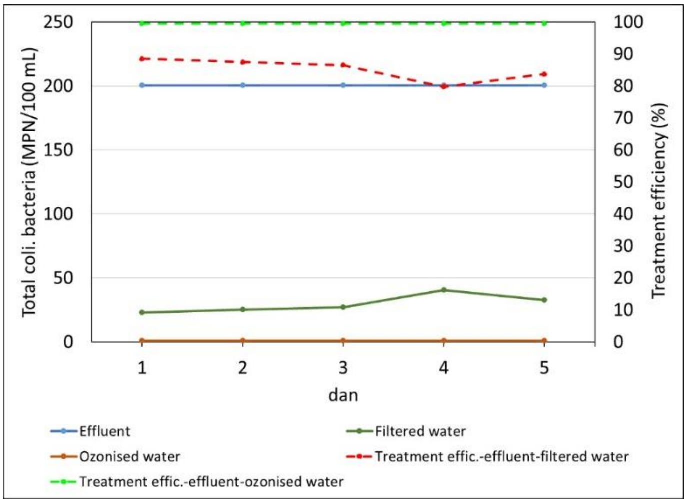

Figure 5: Total coliform bacteria in effluent using the MFO process, and treatment efficiency.

Escherichia coli (MPN/100 mL) in effluent, using the MFO process

The value of Escherichia coli at effluent was > 200.5 MPN/100 mL. The value decreased from 13.2 MPN/100 mL to 14.8 MPN/100 mL after filtration, meaning that the treatment efficiency was about 93%. After disinfection with ozone, the value of E. coli decreased to <1 MPN/100 mL, which means that we removed 100% of E. coli (Figure 6).

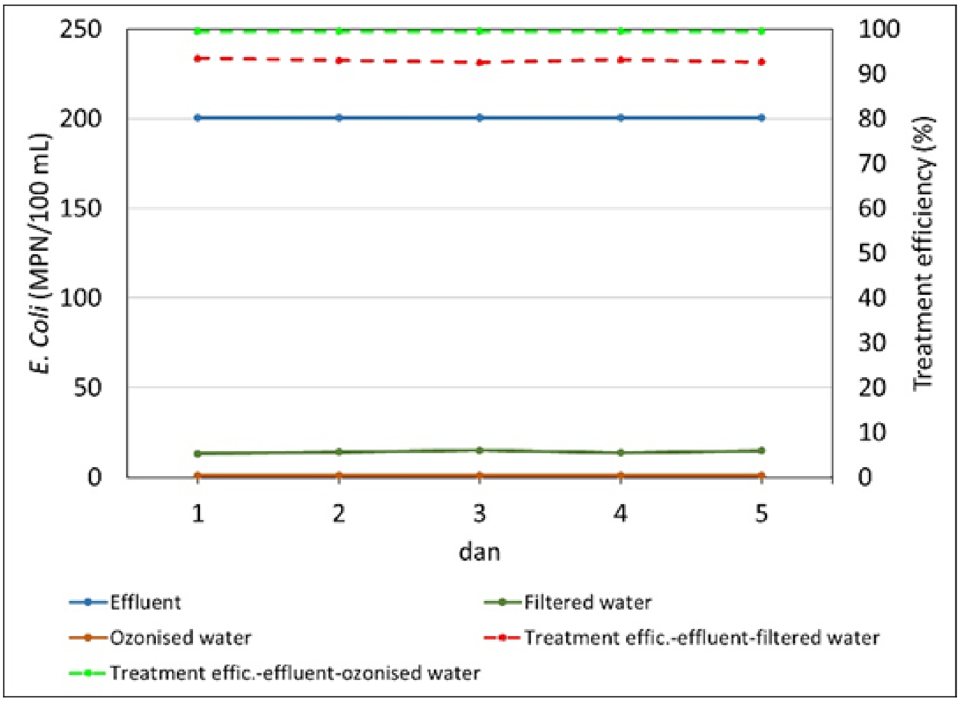

Figure 6: *Escherichia coli* (MPN/100 mL) in effluent using the MFO process, and treatment efficiency.

Regulation (Union, 2020) determines the bacteriological value (*E. coli*) of quality classes of processed water from A to D. The results show that microfiltration can achieve water quality corresponding to quality class B, i.e., up to a value <100 MPN/100 mL. This processed water can be used in agriculture in all irrigation methods for food plants that are eaten raw and the edible part of which grows above the ground and is not in direct contact with processed water and processed food and non-food plants, including cultivated plants used to feed animals for milk and meat production. After ozonation of the filtered water, the processed water corresponds to quality class A, i.e., up to a value of <10 MPN/100 mL, which means that it corresponds to the use of all irrigation methods for food plants consumed raw, the edible part of which is in direct contact with the processed water, and roots and tubers were eaten raw.

IV. CONCLUSION

In the study of additional treatment of municipal wastewater effluent from a municipal treatment plant with membrane filtration, which was performed with a SiC membrane filter with pores of 100 nm and ozone generator (MFO), we can conclude the following:

- Flow rate through membrane filter was 21,6 m^3/h.
- Ozone consumption was for disinfection was 13,4 g/m^3.
- Total suspended solids (TSS) are removed by membrane filtration up to 99%.
- COD is removed from treated water (effluent) up to 55%.
- N-total and P-total are removed from wastewater treatment plant effluent by membrane filtration up to 44% and 62%, respectively. Most of the N and P compounds are part of removed suspended solids.
- By membrane filtration, it is possible to remove practically all suspended matter present in municipal wastewater and biologically treated water. Microfiltration alone showed that it does not remove all microorganisms, and therefore, we used additional ozonation.
- With additional ozonation (disinfection), some organic matter is removed, especially over 100% of total coliform bacteria (MPN) and *Escherichia coli*.

The main advantages of MFO are small pump pressure for managing filtration, easy cleaning of the filter, and long-life resistant.

Filtered water (effluent) thus complies with the legal definition under the EU Regulation (EU Regulation 2020/741, 2020) on minimum water reuse requirements. With microfiltration, we can get water quality B; after additional ozonation, we get water quality A. The MFO system could be introduced at treatment plants, as it would improve the outflow into the watercourse (less polluted watercourse - suspended solids), or the water would be reused for specific purposes, e.g., irrigation of agricultural land, process water of business facilities, horticultural and recreational purposes, accumulation (firewater, construction), or for groundwater enrichment.

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