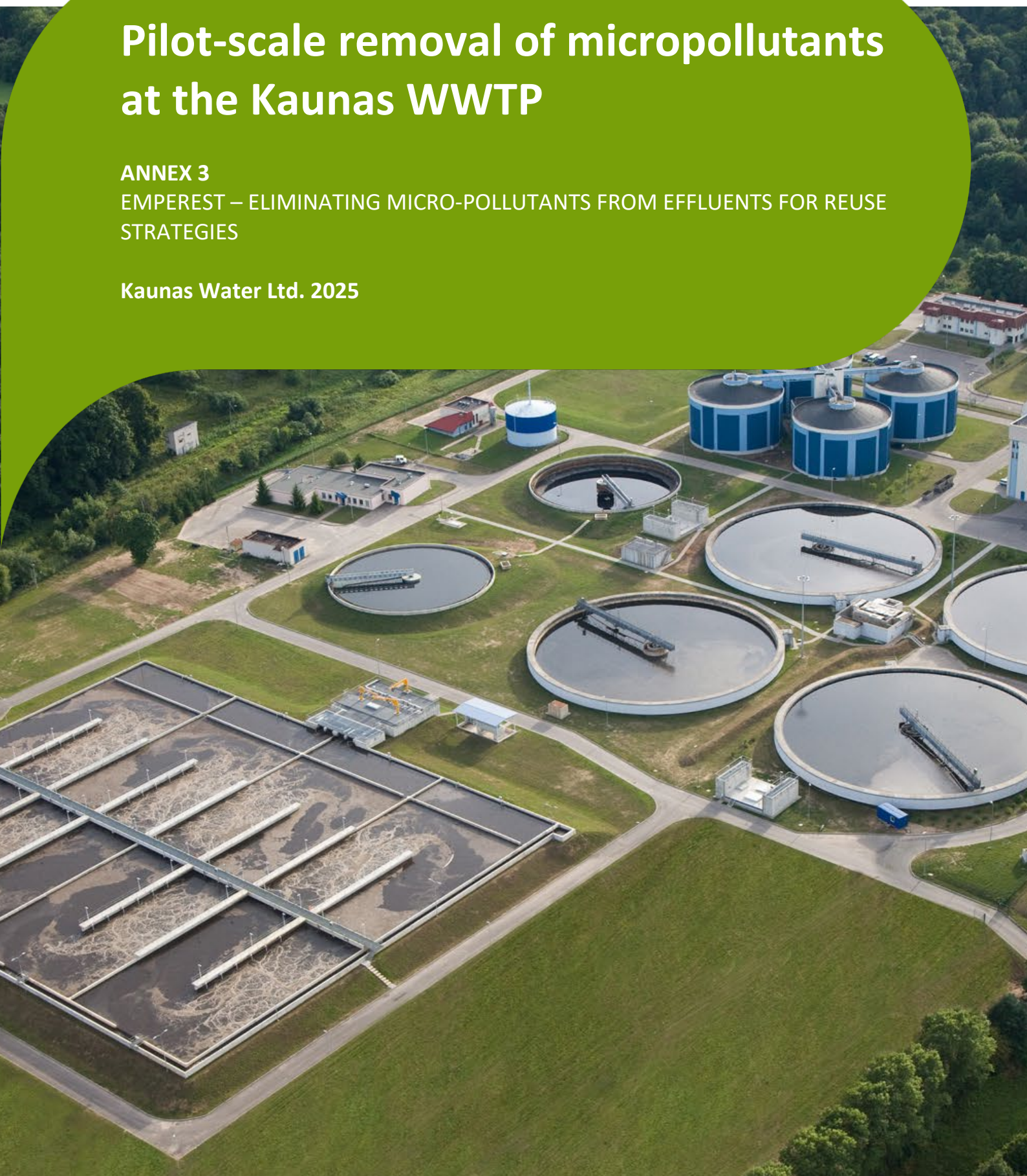


Pilot-scale removal of micropollutants at the Kaunas WWTP

ANNEX 3

EMPEREST – ELIMINATING MICRO-POLLUTANTS FROM EFFLUENTS FOR REUSE STRATEGIES

Kaunas Water Ltd. 2025



Imprint

This publication has been developed within the project **EMPEREST – Eliminating Micro-Pollutants from Effluents for Reuse Strategies**, co-financed by the Interreg Baltic Sea Region Programme 2021–2027, and helping to drive the transition to a green and resilient Baltic Sea region.

This report forms Annex 3 of the overarching study “Strategies and technological means for minimising organic micropollutant emissions from WWTPs”. Each annex presents a site-specific sub-study conducted within the broader framework.

EMPEREST consortium: Union of the Baltic Cities Sustainable Cities Commission c/o City of Turku (FI), Baltic Marine Environment Protection Commission – Helsinki Commission (HELCOM) (FI), University of Tartu (EE), Berlin University of Technology (DE), Turku University of Applied Sciences (FI), Gdańsk Water Utilities (PL), Water and Sewage Company Ltd of Szczecin (PL), Tartu Waterworks Ltd (EE), Tallinn Water Ltd (EE), “Kaunas water” Ltd (LT), Turku Region Wastewater Treatment Plant (FI), DWA German Association for Water, Wastewater and Waste DWA Regional group North-East (DE), Environmental Centre for Administration and Technology (LT), City of Riga (LV).

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Project note

The EMPEREST project supports local authorities, service providers and policy-making community in finding ways to reduce PFAS (Per- and polyfluoroalkyl substances) and other organic micropollutants from the water cycle. The project has four activity strands to fulfil its aims. First, in close cooperation with HELCOM EMPEREST prepares methodological recommendations to monitor PFAS group in the aquatic environment. Second, local authorities address the subject on the city level by developing a PFAS risk assessment framework to identify and assess PFAS-related risks and propose relevant risk mitigation strategies. Third, EMPEREST supports water utilities in making informed decisions about cost-effective treatment strategies and investments for removing micropollutants from wastewater. Finally, capacity building takes place for both local authorities and public service providers to inform them about the recent developments in the field and train them with tailored materials and tools.

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List of Abbreviations

AC	air compressor	SS	suspended solids
AOF	adsorbable organic fluorine	TCOD	total chemical oxygen demand
AOPs	advanced oxidation processes	TN	total nitrogen
A2/O	anaerobic/anoxic/oxic system	TOC	total organic carbon
BOD ₅	biochemical oxygen demand	TP	total phosphorus
COD	chemical oxygen demand	TSS	total suspended solids
DE	Germany	UV	ultraviolet
DF	drum filter	UVA	ultraviolet absorbance
DOC	dissolved organic carbon	UVA254	ultraviolet absorbance at 254
DOM	dissolved organic matter	wt%	weight percentage
EBCT	empty bed contact time	UWWTD	urban wastewater treatment
EE	Estonia	WWTPs	directive
EU	European Union	4-MTB	wastewater treatment plants
FI	Finland	6-MTB	4-methyl-benzotriazole
GAC	granular activated carbon		6-methyl-benzotriazole
GC-MS	gas chromatography-mass spectrometry		
HELCOM	Helsinki Commission		
HMI	human-Machine Interface		
IX	ion exchange		
IXR	ion exchange resin		
LC-MS	liquid chromatography-mass spectrometry		
LC-MS/MS	liquid chromatography-tandem mass spectrometry		
LT	Lithuania		
LV	Latvia		
OC	oxygen concentrator		
OG	ozone generator		
OMPs	organic micropollutants		
PE	population equivalent		
PFAS20	list of twenty PFASs		
PFBA	perfluorobutanoic acid		
PFBS	perfluorobutane sulfonic acid		
PFDA	perfluorodecanoic acid		
PFDoDA	perfluorododecanoic acid		
PFDoDS	perfluorododecane sulfonic acid		
PFDS	perfluorodecane sulfonic acid		
PFHpA	perfluoroheptanoic acid		
PFHpS	perfluoroheptane sulfonic acid		
PFHxA	perfluorohexanoic acid		
PFHxS	perfluorohexane sulfonic acid		
PFNA	perfluorononanoic acid		
PFNS	perfluorononane sulfonic acid		
PFOA	perfluorooctanoic acid		
PFOS	perfluorooctane sulfonic acid		
PFPeA	perfluoropentanoic acid		
PFPeS	perfluoropentane sulfonic acid		
PFTTrDA	perfluorotridecanoic acid		
PFTTrDS	perfluorotridecane sulfonic acid		
PFUnDA	perfluoroundecanoic acid		
PFUnDS	perfluoroundecane sulfonic acid		
PL	Poland		
PLC	programmable logic controller		
PSA	pressure swing adsorption		

1. Introduction

Organic micropollutants (OMPs) in wastewater are an emerging environmental concern due to their widespread presence and adverse effect on human health and the environment. OMPs are defined as trace-level contaminants, typically present in very low concentrations ranging from nanograms to micrograms per liter. They originate from various industrial and human sources and pose significant ecological and health challenges due to their persistence, bioaccumulation potential, and biological activity at low concentrations.

Wastewater treatment plants (WWTPs) are considered one of the main source of OMPs in aquatic environment. Although WWTPs are designed to remove organic matter and nutrients, they are often not fully effective at eliminating micropollutants, which include pharmaceuticals, personal care products, pesticides, hormones, and microplastics. The problem of OMPs in wastewater highlights the need for improved treatment technologies, stricter regulatory measures, and better public awareness regarding the disposal of chemicals and pharmaceuticals. Improvements including advanced oxidation processes, membrane filtration, and activated carbon adsorption are being explored to mitigate this growing threat, ensuring the protection of both human health and aquatic ecosystems.

To better protect human health and the environment, in November 2024, the European Union (EU) adopted the revised Urban Wastewater Treatment Directive (UWWTD), which mandates enhanced nutrient removal and imposes stricter requirements for monitoring and eliminating micropollutants from urban wastewater. Urban WWTPs serving 150,000 population equivalents (PE) or more must implement quaternary treatment to remove a broad spectrum of micropollutants by 2045. The UWWTD also imposes additional quaternary treatment in WWTPs serving agglomerations between 10,000 and 100,000 PE in areas identified as sensitive to micropollutant pollution, unless they demonstrate the absence of environmental or public health risks through risk assessment. This report presents the results of pilot testing on the removal of organic micropollutants using of a mobile pilot-scale plant, which enables the evaluation of the efficiency of advanced wastewater treatment processes. The pilot test aimed at assessing the potential of ozone oxidation, activated carbon adsorption to reduce micropollutant emissions with wastewater, as well as determining the process parameters that ensure the highest micropollutant removal efficiency.

2. Materials and methods

2.1. Study site

The pilot test was conducted at the Kaunas WWTP, which is second largest wastewater treatment plant in Lithuania. The average dry weather influent flow rate to the plant is approx. 65,000 m³/d and the pollutant load corresponds to 390,000 population equivalents (PE). The primary treatment line comprises perforated screens, aerated grit chambers and two primary settling tanks. The secondary treatment line includes four bioreactors and four circular secondary clarifiers operated in parallel. Configuration of the bioreactors is based on the simultaneous nitrification/denitrification (SN/DN) system. In 2024 average concentrations of pollutants in the treated wastewater were as follows:

– Biochemical oxygen demand (BOD ₇)	6.5 mg O ₂ /L
– Chemical oxygen demand (COD)	32 mg O ₂ /L
– Total suspended solids (TSS)	7.1 mg/L
– Total nitrogen (TN)	6.9 mg N/L
– Total phosphorus (TP)	0.63 mg P/L

2.2. Pilot plant description

The pilot test was carried out by means of a semi-technical mobile pilot plant constructed by the Probiko-Aqua company, based on a detailed design concept developed by Gdańsk Water Utilities (Figures 1 and 2). The pilot installation consists of the following main units:

- MITA TF2 VM cloth drum filter (DF)

- | | | |
|--|--|----------------------|
| | POLSTOFF "PILE" type filter cloth with a thickness of 4–5 mm | |
| | filtration surface | 2 m ² |
| | max hydraulic capacity | 20 m ³ /h |
- ProO2 Max 795MC pressure swing adsorption (PSA) oxygen concentrator (OC)

max gas flow	10 l/min
oxygen content at 2 l/min	> 93% ± 3%
oxygen content at 10 l/min	87 to 93%
 - Ozonia CFS-1 2G compact ozone generator (OG)

nominal ozone concentration	10 wt%
nominal ozone production	55 g O ₃ /h
 - Ozone contact tank

diameter	0,705 m
height	2,30 m
working volume	0,200 – 0,800 m ³
 - Gravity filters – 2 units

diameter	0,35 m
surface area	0,962 m ²
filter nozzles	vertical slots of the width 0,30 mm
 - Backwash water storage tank

length	0,120 m
width	0,069 m
height	0,99 m
maximum working volume	0,750 m ³
 - Probiko-Aqua Protec 1200 EW ultraviolet (UV) lamp

maximum flow	0,70 m ³ /h
UV dose	400 J/m ² at 60% UV transmittance
 - Air compressor (AC)

Airpress oil-free compressor LMO 25-250	
free air delivery	150 L/min
maximum pressure	8 bar

The treatment train is equipped with the following online measuring instruments for process monitoring:

- IFM SM9100 magnetic-inductive water flow meter – 1 unit
- IFM SM7120 magnetic-inductive water flow meter – 6 units
- IFM SA5020 air flow sensor – 1 unit
- IFM PN2098 water level sensors – 8 units
- WTW UV 700 IQ SAC UV absorbance sensors with ultrasound cleaning system – 3 units
- WTW VisoTurb 700 IQ turbidity sensors with ultrasound cleaning system – 2 units



Figure 1. View of the pilot plant located at the Kaunas WWTP.



Figure 2. View of the pilot plant located at the Kaunas WWTP.

Online UV absorbance (UVA) and turbidity sensors allow to continuously monitor removal performance of suspended solids and organic compounds. Programmable logic controller (PLC) based control system and Human-Machine Interface (HMI) enable monitoring and control of the devices and processes within the pilot plant. An on-site VPN router provides secure remote access to the pilot plant over cellular networks.

The devices that constitute the treatment train can be operated in various configurations depending on the research needs. All the devices are mounted in a 20-feet high cube shipping container (Figure 1). The way the pilot plant was designed and constructed allows for easy transport and quick commissioning at any water or wastewater treatment plant. Figures 3 – 5 show a process flow diagram displayed on the touch panel HMI.

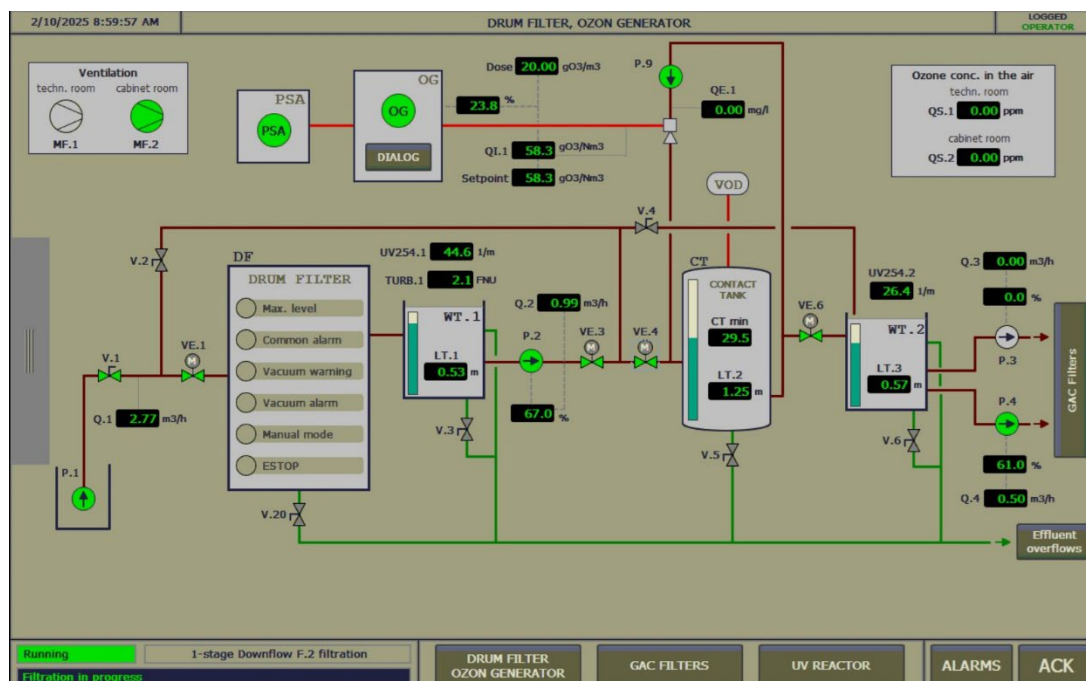


Figure 3. Flow diagram displayed on screen 1 of HMI panel. Labelling of main components of the process train: DF - cloth drum filter, PSA - oxygen concentrator, OG - ozone generator, WT – intermediate process tank, P - process pump, UVA254 – UV absorbance sensor, TURB – turbidity sensor.

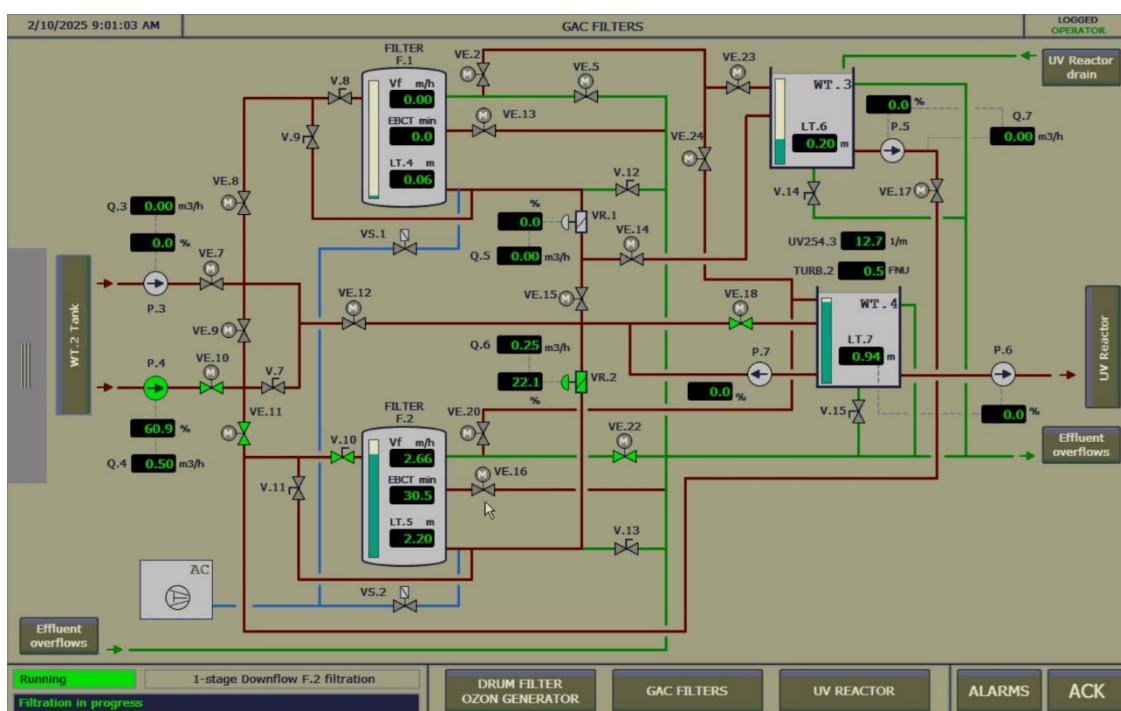


Figure 4. Flow diagram displayed on screen 2 of HMI panel. Labelling of main components of the process train: F – gravity filter, WT.3 - intermediate process tank, WT.4 – backwash water tank, P - process pump, AC - air compressor, UVA254 – UV absorbance sensor, TURB – turbidity sensor.

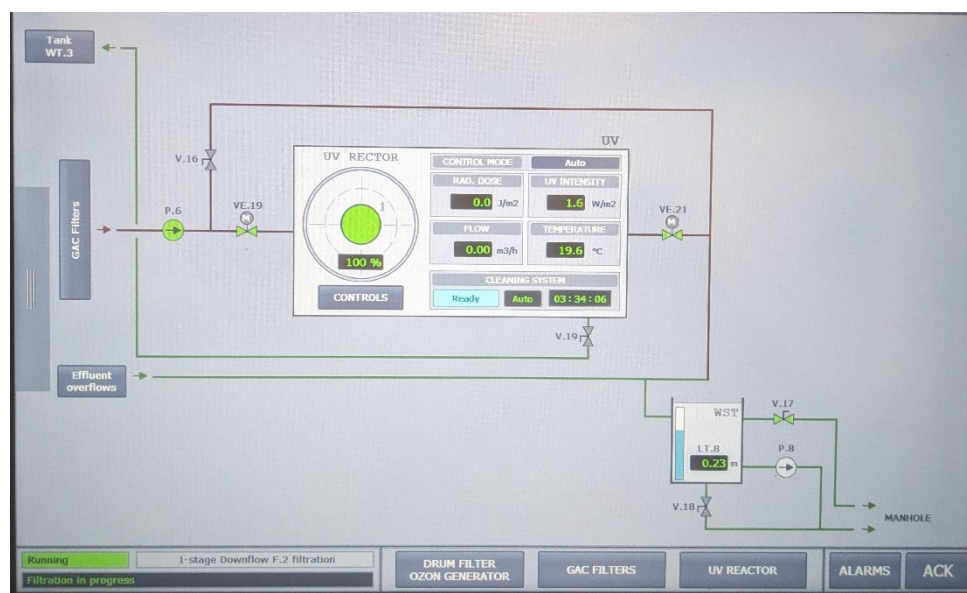


Figure 5. Flow diagram displayed on screen 3 of HMI panel. Labelling of main components of the process train: UV – ultraviolet lamp, WST - backwash and overflow tank.

2.3. Analytical methods

Performance of the examined treatment train was monitored using 12-hour and 24-hour composite sampling, as well as online sensors. Samples were collected once a week. The samples were examined for wastewater quality parameters indicating the content of suspended solids and organic compounds, including OMPs of emerging concern. The measured OMPs include those specified in the UWWTD for which a minimum removal of 80% is required (highlighted in bold). The list of measured wastewater quality parameters is as follows:

- COD
- Dissolved organic carbon (DOC)
- UVA
- TSS
- Turbidity
- Forty nine PFASs (PFAS 49):
 - 3:3 Fluorotelomer carboxylic acid (3:3 FTCA); 6:2 Fluorotelomer sulfonic acid (4:2 FTSA; 4:2 FTS); Perfluoroethylcyclohexane sulfonate (PFecHS); 5:3 Fluorotelomer carboxylic acid (5:3 FTCA); 6:2-Fluorotelomersulfonic acid (6:2 FTSA; 6:2 FTS); 7:3 Fluorotelomer carboxylic acid (7:3 FTCA); 8:2 Fluorotelomer sulfonate ammonium salt (8:2 FTSA; 8:2 FTS); Perfluoro(2-((6-chlorohexyl)oxy)ethanesulfonic acid (9Cl-PF3ONS); 10:2 Fluorotelomer sulfonic acid (10:2 FTS); Perfluoro-3-oxaundecane sulfonic acid (PF3OUdS); Hexafluoropropylene oxide dimer acid (HFPO-DA) ammonium salt (DONA; ADONA); 2-(N-Ethylperfluorooctane sulfonamido)ethanol (EtFOSE); Perfluorobutanesulfonamide (FBSA); Perfluorohexanesulfonamide (FHxSA); Perfluorooctanesulfonamidoacetic acid (FOSAA); Hexafluoropropylene oxide dimer acid (HFPO-DA (Gen X)); Hexafluoropropylene oxide trimer acid (HFPO-TA); N-Ethylperfluorooctanesulfonamide (EtFOSA; N-EtFOSA); N-Ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA; EtFOSAA); Nonafluoro-3,6-dioxaheptanoic acid (NFDHA); N-Methylperfluorooctanesulfonamide (MeFOSA; N-MeFOSA); N-

Methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA; MeFOSAA); 2-(N-Methylperfluorooctane sulfonamido)ethanol (MeFOSE); Perfluorobutanoic acid (PFBA); Perfluorobutanesulfonic acid (PFBS); Perfluorodecanoic acid (PFDA); Perfluorododecanoic acid (PFDoA); Perfluorododecanesulfonic acid (PFDoS); Perfluorodecanesulfonic acid (PFDS); Perfluoroethylethanesulfonic acid (PFEEESA); Perfluoroheptanoic acid (PFHpA); Perfluoroheptanesulfonic acid (PFHpS); Perfluorohexanoic acid (PFHxA); Perfluorohexadecanoic acid (PFHxDA); Linear Perfluorohexanesulfonic acid (PFHxS Linear); Branched Perfluorohexanesulfonic acid (PFHxS Branched); Total Perfluorohexanesulfonic acid (PFHxS Total); Perfluoro-2-methoxyacetic acid (PFMOBA); Perfluoro-2-methoxypropanoic acid (PFMOPrA); Perfluorononanoic acid (PFNA); Perfluoronananesulfonic acid (PFNS); Linear Perfluorooctanoic acid (PFOA Linear); Branched Perfluorooctanoic acid (PFOA Branched); Total Perfluorooctanoic acid (PFOA Total); Perfluorooctadecanoic acid (PFODA); Linear Perfluorooctanesulfonic acid (PFOS Linear); Branched Perfluorooctanesulfonic acid (PFOS Branched); Total Perfluorooctanesulfonic acid (PFOS Total); Perfluorooctanesulfonamide (FOSA (PFOSA)); Perfluoropentanoic acid (PFPeA); Perfluoropentanesulfonic acid (PFPeS); Perfluorotetradecanoic acid (PFTeA); Perfluorotridecanoic acid (PFTrDA); Perfluoroundecanoic acid (PFUDa; PFUnA); Perfluoroundecanesulfonic acid (PFUnDS)

- Eleven pharmaceuticals:
diclofenac, clarithromycin, candesartan, hydrochlorothiazide, metoprolol, irbesartan, venlafaxine, citalopram, amisulpride, carbamazepine, telmisartan.
- One estrogen - beta-estradiol
- Ten phthalates:
Benzyl butyl phthalate; Di-cyclo hexyl phthalate; Di-ethyl phthalate; Di-ethylhexyl phthalate; Di-isobutyl phthalate; Di-n-butyl phthalate; Di-n-octyl phthalate; Di-n-propyl phthalate; Di-pentyl phthalate; Dimethyl phthalate
- **Benzotriazole and sum of 4-methyl-benzotriazole (4-MTB) and 6-methyl-benzotriazole (6-MTB)**

COD, DOC and TSS were determined according to the standard methods. Ultraviolet absorbance at a wavelength of 254 nanometers (UVA₂₅₄) was measured with WTW UV 700 IQ SAC sensors. Turbidity was measured according to the nephelometric principle with WTW VisoTurb 700 IQ sensors.

The concentrations of estrogen beta-estradiol and phthalates were determined using a gas chromatography-mass spectrometry (GC-MS) method, while pharmaceuticals, benzotriazoles using a liquid chromatography-mass spectrometry (LC-MS) method. In order to measure PFASs, liquid chromatography-tandem mass spectrometry (LC-MS/MS) was employed. All analyses of OPMs were performed in an external accredited laboratory.

3. Results

The pilot plant was fed with effluent from secondary clarifiers of the Kaunas WWTP. The treatment process train consisted of cloth filtration, ozone oxidation followed by granular activated carbon (GAC) filtration. The GAC filter No.1 was filled with peat based granular activated carbon Kemira KemSorb™ 8385 with effective particle mesh size 30x60 (0.6 – 0.25mm). Filter No.2 was loaded with peat based granular activated carbon Kemira KemSorb™ 8380 with particle mesh size 8x30 (2.36 – 0.6mm). The height of both filter beds equalled to 1,35 m.

The pilot test was conducted from January 14, 2025 to April 30, 2025. The study duration was structured into two consecutive periods allowing for comparative analysis between different treatment configurations: **Period 1** focused on determining the UV₂₅₄ absorbance with various ozone dose, ozone contact time, and **Period 2** evaluated the efficiency of organic micropollutant removal with ozonation, GAC filtration.

3.1. Testing Period 1 - determining the UV₂₅₄ absorbance reduction

UVA₂₅₄ measurements serve as an indirect indicator of dissolved organic matter (DOM), which absorbs light at this specific wavelength. The relationship between the reduction of organic micropollutants (OMPs) and the

decline in UVA₂₅₄ absorbance is of ongoing interest in the field of water treatment, as both are commonly used to assess water quality and the effectiveness of treatment with ozone processes.

Tests were carried out under dry weather flow conditions only, in order to avoid fluctuations in the results. On each test day, a stable ozone contact time was manually set in the piloting equipment, ranging from 16 to 40 minutes. For each selected contact time, a series of different ozone doses was applied to the wastewater, varying from 4 mgO₃/L to 30 mgO₃/L. After each ozone dose adjustment, UV₂₅₄ absorbance was recorded both before ozone introduction and after the ozone contact tank. Based on these measurements, the UV₂₅₄ absorbance reduction was calculated.

Ozonation at doses of 4–5 mgO₃/L resulted in a UV₂₅₄ absorbance reduction of 14–15%. At such low doses, the contact time in the ozone reactor had no significant impact on the extent of UV₂₅₄ absorbance reduction. A dose of 10 mgO₃/L led to a UV₂₅₄ absorbance reduction of 21.5% with both 16 and 20 minutes of contact time. Increasing the contact time to 30 minutes at the same dose improved the reduction to 26.27%, while further extending it to 40 minutes did not yield a significant additional effect, with a reduction of 26.47% (Figure 6).

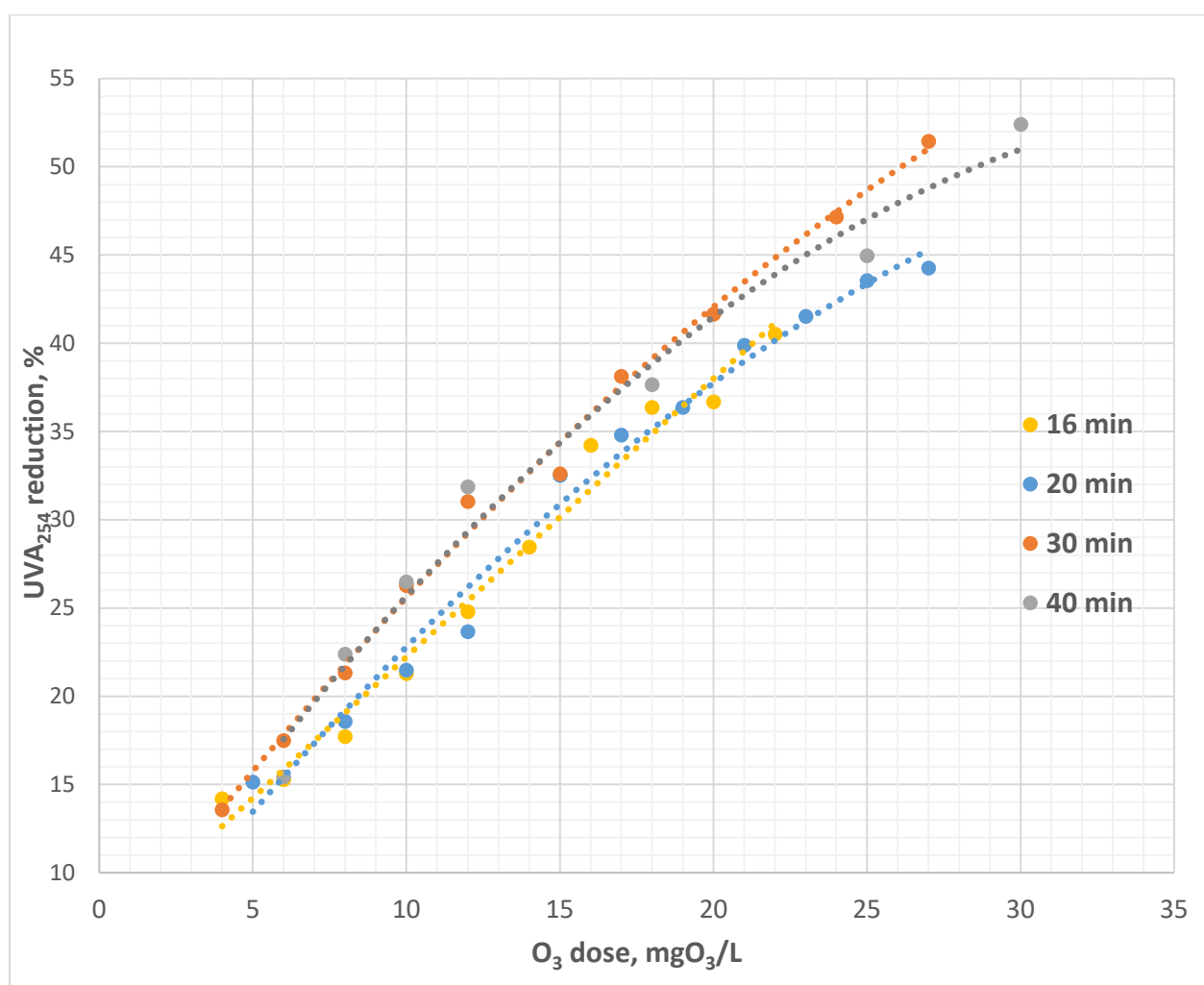


Figure 6. UV₂₅₄ Absorbance Reduction at varying ozone doses and contact times

3.2. Testing Period 2 - evaluating the efficiency of organic compounds removal

To evaluate the efficiency of micropollutant removal in the piloting container, ten series of wastewater samples were collected after cloth filtration, ozonation, and GAC filtration. Additionally, starting from series 3, samples of the WWTP influent were collected to assess the overall reduction of pharmaceuticals and PFAS across the entire treatment train, including biological treatment. The key operational parameters for all sampling series are presented in Table 1.

	Series 1	Series 2	Series 3	Series 4	Series 5	Series 6	Series 7	Series 8	Series 9	Series 10
O ₃ dose, mgO ₃ /L	6	10	20	8	15	15	8	5	5	10
O ₃ dose, mgO ₃ /mg DOC	0,24	0,46	1,06	0,40	1,20	1,40	0,37	0,25	0,31	0,56
CT O ₃	16 min	20 min	30 min	20 min	20 min	30 min	17 min	30min	20 min	17 min
EBCT GAC F.1					10 min	20 min	11 min	20 min		
EBCT GAC F.2	15 min	15 min	30 min	15 min					20 min	30 min

Table 1. Piloting process parameters during all testing series. (CT – wastewater contact time with O₃ in contact reservoir; EBCT – Empty bed contact time in granular activated carbon filter)

During the piloting tests one GAC filter column was used at a time, although pilot plant configuration allows to test two GAC filters in lead-lag configuration. In testing series 5, 6, 7, and 8, the F.1 filter filled with KemSorb™ 8385 GAC was used. In all other series, the F.2 filter with KemSorb™ 8380 GAC media was utilized.

To ensure stable operation of the ozone generator, the cloth filter's cleaning cycles were reduced during sampling hours. The backwash cycle of the GAC filters was set to 24 hours.

In general, sampling during pilot testing days was conducted from 7:00 a.m. to 7:00 p.m., with hourly collection of equal volumes of wastewater from each sampling point (excluding the WWTP inlet). Composite 12-hour samples were prepared using borosilicate glass jars and stainless steel containers. For raw, untreated wastewater at the WWTP inlet, an automated 24-hour sampler was used.

Details on the measured concentrations of micropollutants are discussed in Section 3.2.2 – 3.2.5.

3.2.1. Removal of DOC, COD, nitrogen and phosphorus

Figure 7 illustrates the reduction of dissolved organic carbon (DOC) during the advanced treatment of effluent from the Kaunas wastewater treatment plant. The combined treatment process—ozonation followed by granular activated carbon (GAC) filtration—achieved an average DOC removal efficiency of approximately 55%.

The majority of DOC reduction was attributed to the GAC filtration stage, while ozonation contributed only a modest decrease in DOC concentrations. This outcome was expected, as the applied ozone doses were not intended to fully mineralize organic matter to carbon dioxide. Instead, ozonation served primarily to partially oxidize complex organic molecules, increasing their biodegradability and facilitating their subsequent removal in the GAC filter. GAC filtration, known for its strong adsorptive capacity, effectively removed a wide range of partially oxidized and residual organic compounds, leading to the overall DOC reduction observed. Since many micropollutants are present in the dissolved phase, DOC is often a key indicator for evaluating the effectiveness of removal strategies

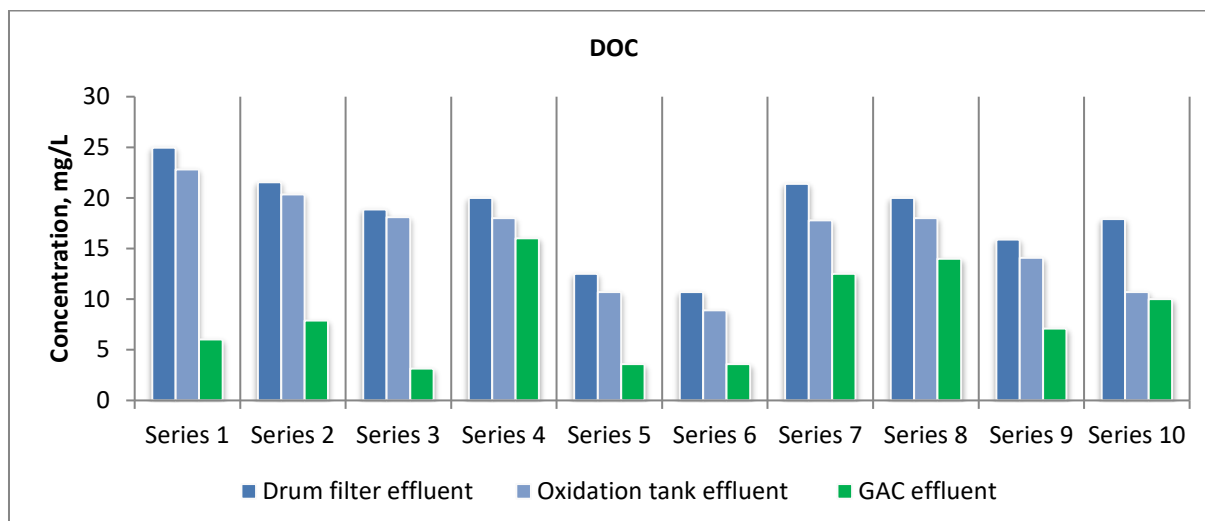


Figure 7. Concentrations of dissolved organic carbon in various treatment stages.

Figure 8 shows the reduction of chemical oxygen demand (COD) during the advanced treatment of Kaunas WWTP effluent. The COD removal pattern closely mirrors that of DOC, with an average reduction of approximately 51% achieved through the combined process of ozonation and GAC filtration. Similar to DOC removal, the greater share of COD reduction was attributed to the GAC filtration stage due to its strong sorption capacity for a broad range of organic contaminants.

Ozonation alone contributed minimally to COD reduction. This is because, during ozonation, complex organic compounds are only partially oxidized, forming intermediate oxidation products such as aldehydes, ketones, and short-chain acids. These by-products still exert oxygen demand and are not fully mineralized. As a result, they continue to contribute to COD values when measured using standard dichromate-based chemical oxidation methods. Therefore, while ozonation plays a critical role in transforming and breaking down persistent organic compounds, its full effect on COD is only realized when followed by an effective polishing step such as GAC filtration.

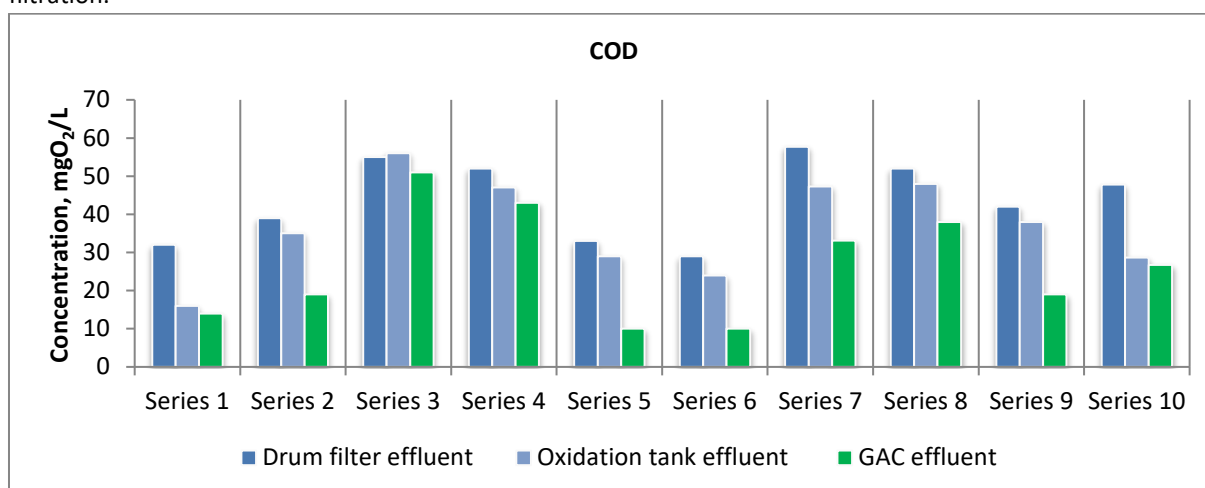


Figure 8. Chemical oxygen demand concentrations during various treatment stages.

During the pilot tests, in addition to micropollutants, conventional wastewater parameters such as nitrogen and phosphorus were also monitored to evaluate their behaviour in the advanced treatment stages. Overall, total nitrogen removal was relatively low, with an average reduction of approximately 13%. However, a significant reduction in nitrite nitrogen (NO₂-N) was observed after ozonation, averaging around 85%.

This is consistent with the well-known reactivity of ozone with nitrite. Ozone reacts with nitrite at a much faster rate than with many organic micropollutants. As a result, even moderate concentrations of nitrite can rapidly consume ozone, limiting its availability for the oxidation of more complex and persistent organic compounds. This competitive ozone demand must be taken into account when optimizing ozonation processes, especially in cases where elevated $\text{NO}_2\text{-N}$ levels are present in the influent.

GAC filtration, on the other hand, contributed little to nitrogen removal. This is expected, as GAC is primarily effective for the adsorption of organic molecules and has limited capacity for retaining inorganic nitrogen species such as nitrite or nitrate. These compounds are highly soluble, do not readily adsorb to activated carbon surfaces, and typically require biological processes for efficient removal.

Therefore, although nitrite is effectively removed through ozonation, its presence may inadvertently reduce the efficiency of ozonation for organic micropollutants removal. Figures 9 - 11 illustrate various nitrogen forms concentrations measured after each treatment stage.

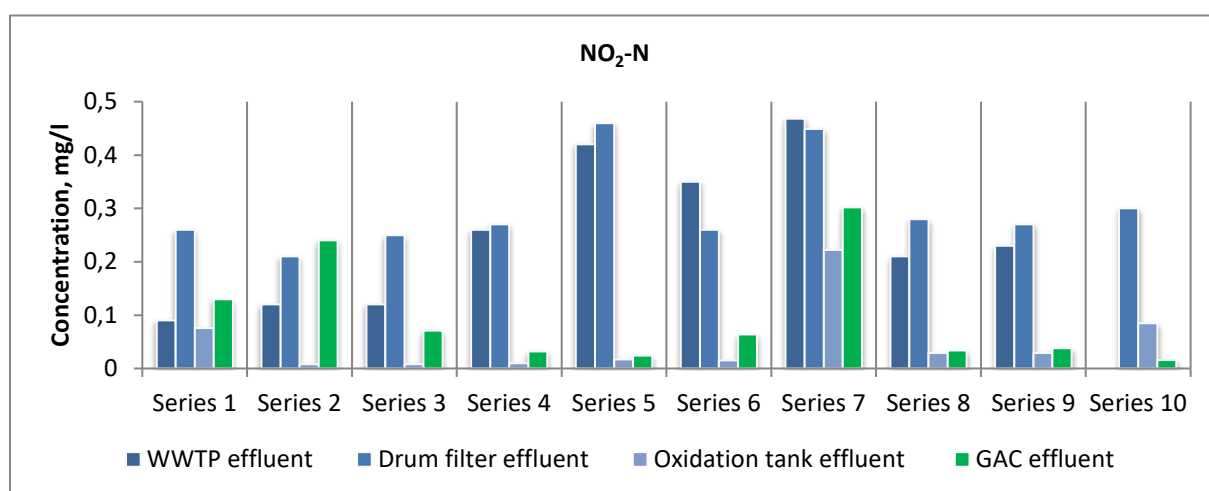


Figure 9. Nitrite nitrogen concentrations after each treatment stage.

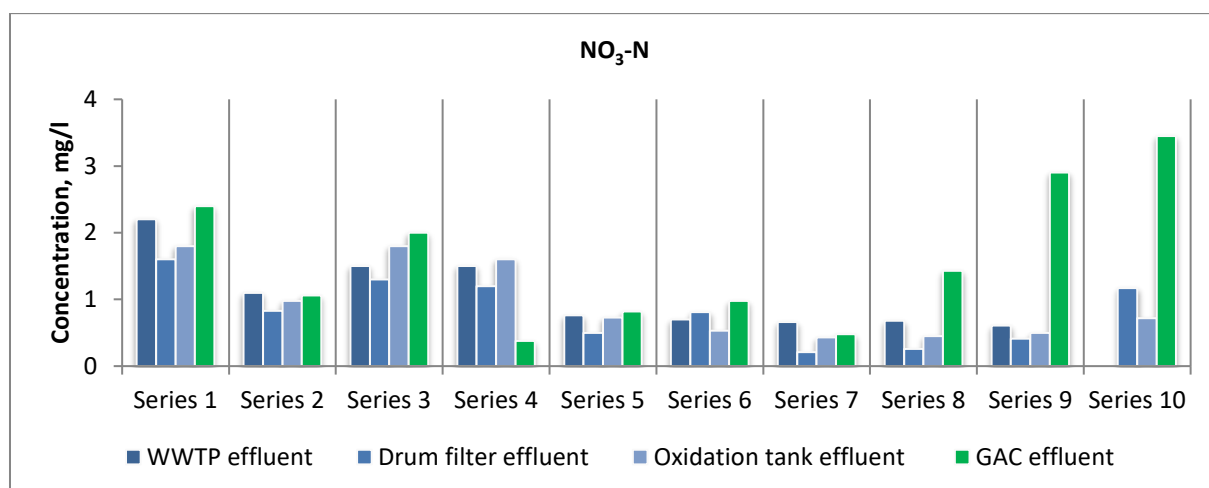


Figure 10. Nitrate nitrogen concentrations after each treatment stage.

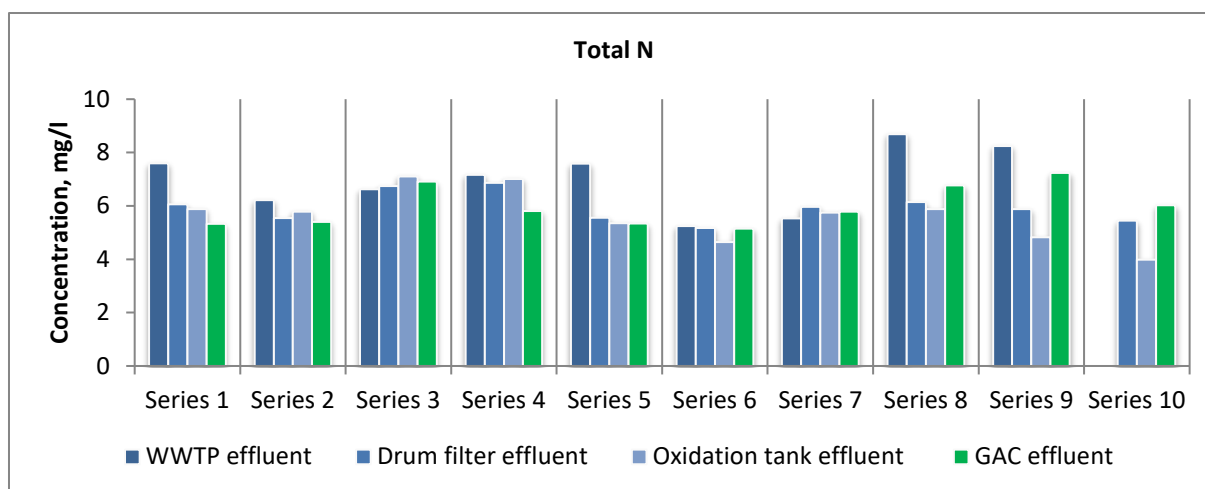


Figure 11. Total nitrogen concentrations after each treatment stage.

During the advanced treatment of Kaunas WWTP effluent, an average total phosphorus reduction of approximately 24% was observed. This reduction is primarily attributed to the physical removal of suspended particles—mainly residual activated sludge flocs—that still contain bound or particulate phosphorus.

Since neither ozonation nor granular activated carbon (GAC) filtration is specifically designed to target dissolved phosphate, their impact on soluble phosphorus is minimal. However, the filtration processes, particularly through the cloth filter and GAC media, effectively remove fine particulate matter from the effluent. These particulates often include biomass residues and inorganic precipitates that contain phosphorus in adsorbed or incorporated forms.

Therefore, the observed phosphorus reduction is largely a result of solid-liquid separation, rather than chemical or adsorptive transformation.

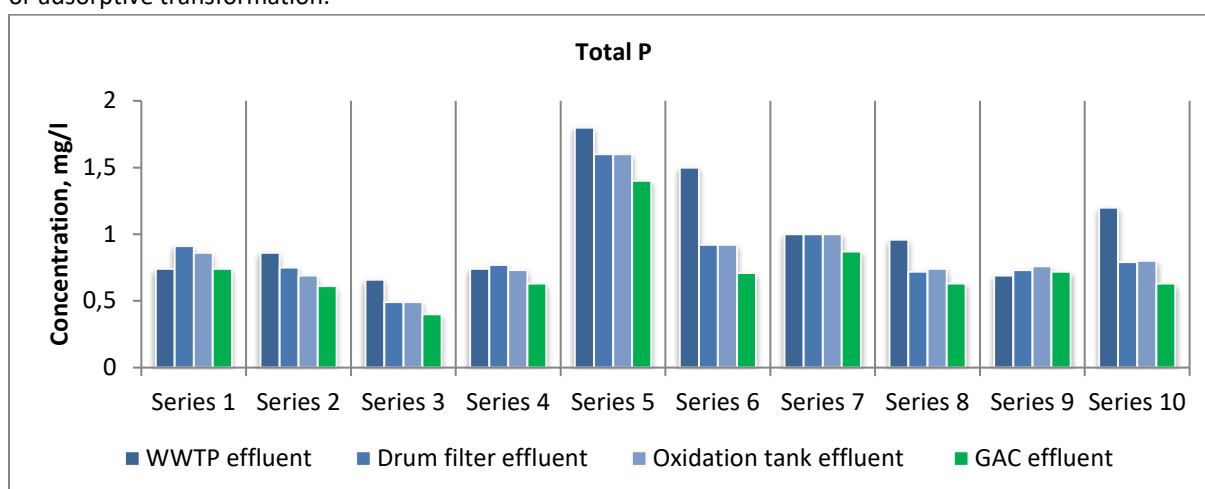


Figure 12. Concentrations of total phosphorus after each treatment stage.

3.2.2 Removal of pharmaceuticals

The pharmaceuticals analyzed during the pilot tests belonged to several therapeutic categories: antihypertensives (candesartan, irbesartan, metoprolol, hydrochlorothiazide), antidepressants (citalopram, venlafaxine), antipsychotics and mood stabilizers (amisulpride, carbamazepine), antibiotics (clarithromycin), and anti-inflammatory/pain relief agents (diclofenac). Analytical results confirmed the presence of ten out of the eleven tested compounds in the wastewater, with irbesartan being the only substance not detected.

As all of these organic micropollutants are present in dissolved form, samples were collected after the cloth filtration stage. The purpose of the drum filter is to remove suspended solids and it does not significantly affect the concentration of soluble OMPs. Therefore, in the context of this study, the drum filter effluent is considered representative of the WWTP final effluent. The results presented in the subsequent figures should be interpreted accordingly. Starting with series 3, samples of inlet wastewater to the WWTP were taken as well.

The concentrations of individual pharmaceuticals after each stage of wastewater treatment are presented in Figures 13 – 22. The detection limit of all substances was 10ng/L. In drum filter effluent, diclofenac was found at the highest concentrations among all the pharmaceuticals, ranging from 2370 ng/L to 6830 ng/L. In some sampling series, concentrations exceeding 2000 ng/L were measured for clarithromycin (1130–2670 ng/L) and metoprolol (1070–2470 ng/L). For the remaining pharmaceuticals, concentrations ranged from 21 ng/L to 1300 ng/L.

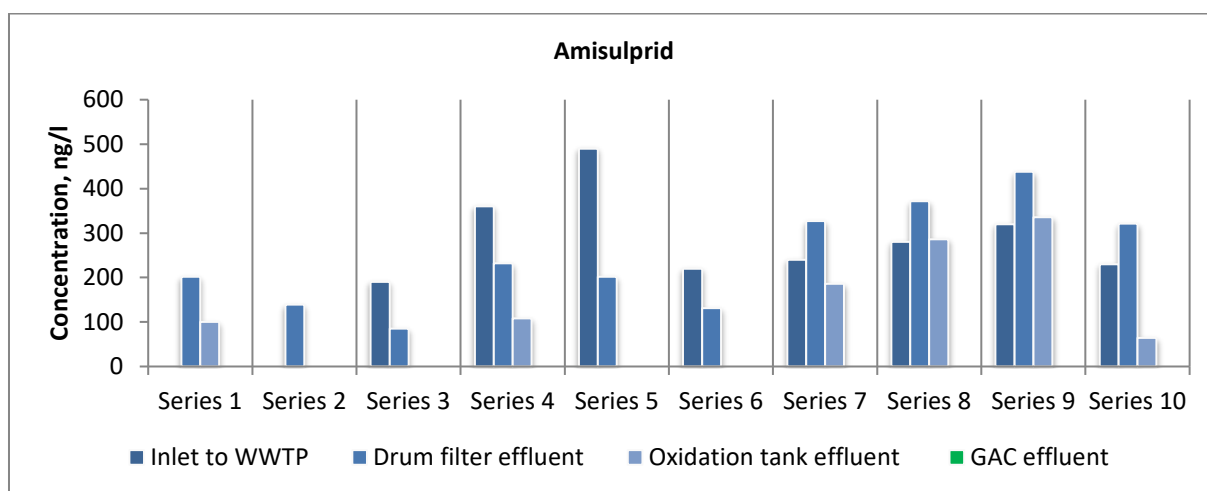


Figure 13. Concentrations of amisulprid at different stages of wastewater treatment.

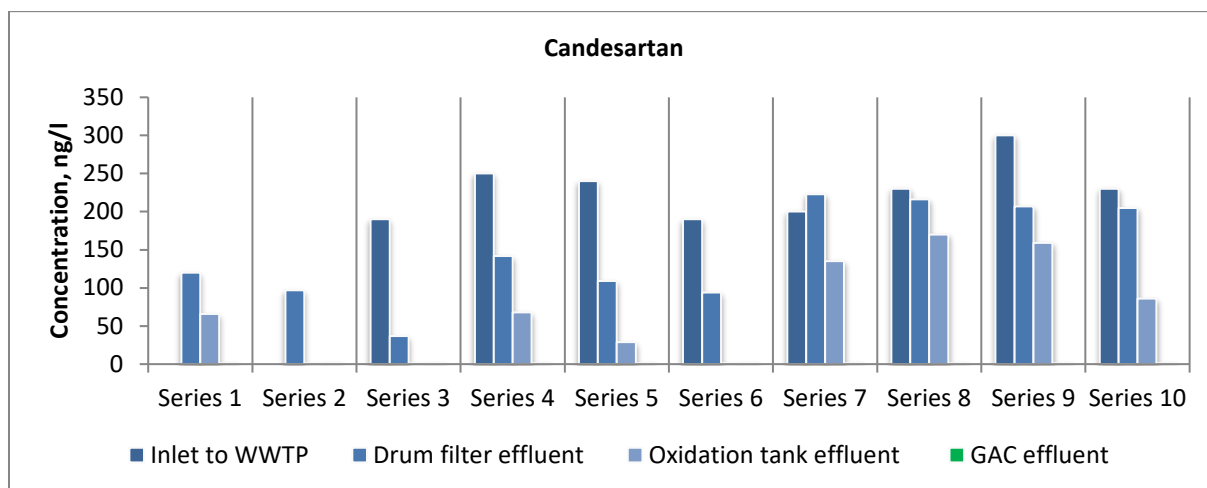


Figure 14. Concentrations of candesartan at different stages of wastewater treatment

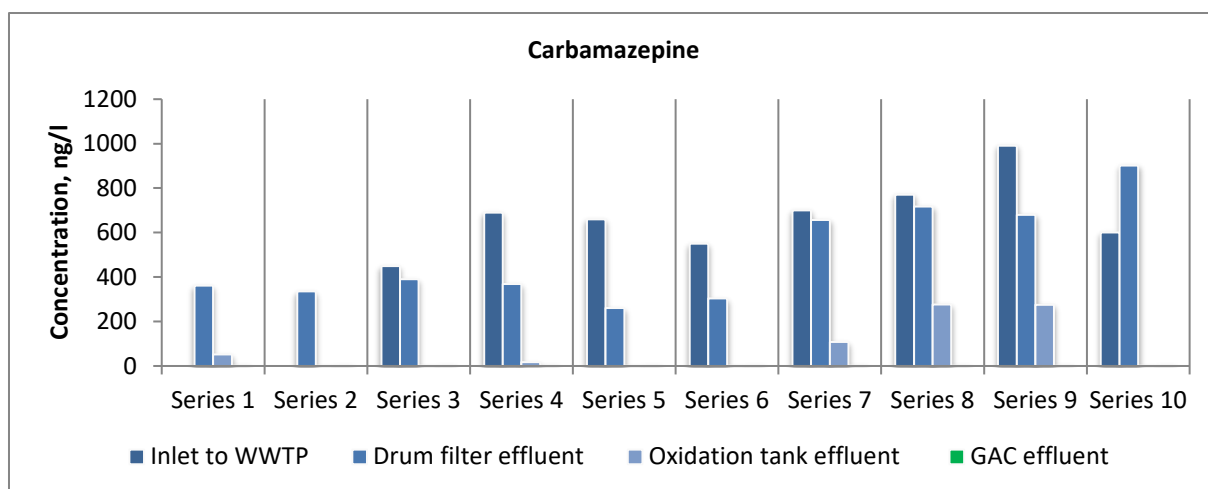


Figure 15. Concentrations of carbamazepine at different stages of wastewater treatment

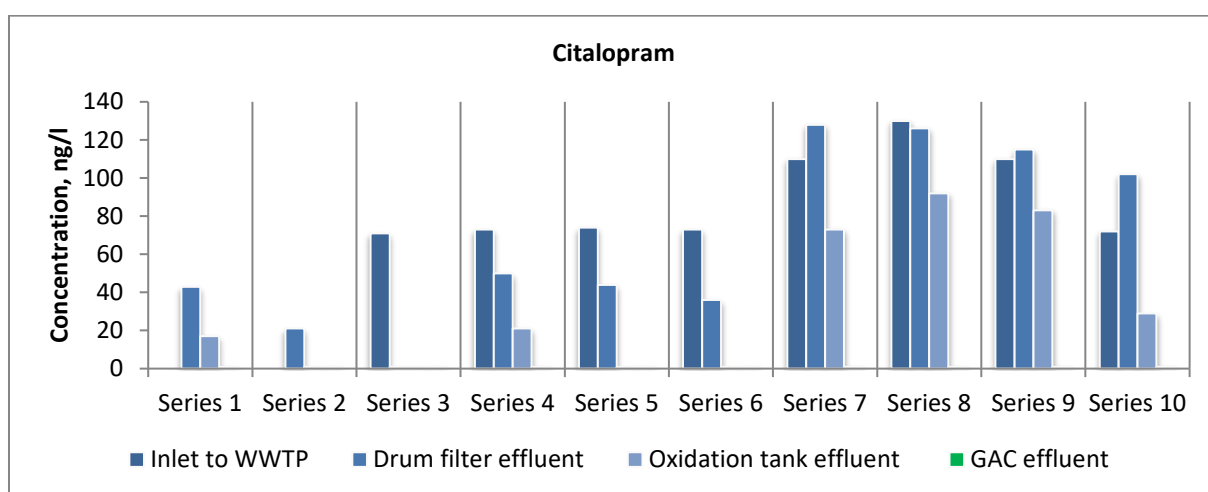


Figure 16. Concentrations of citalopram at different stages of wastewater treatment.

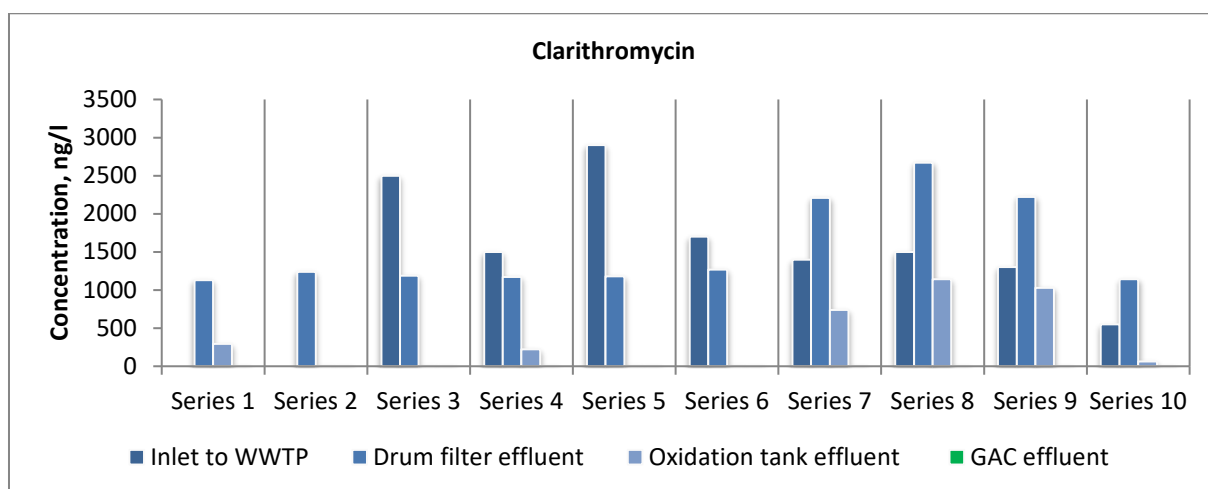


Figure 17. Concentrations of clarithromycin at different stages of wastewater treatment.

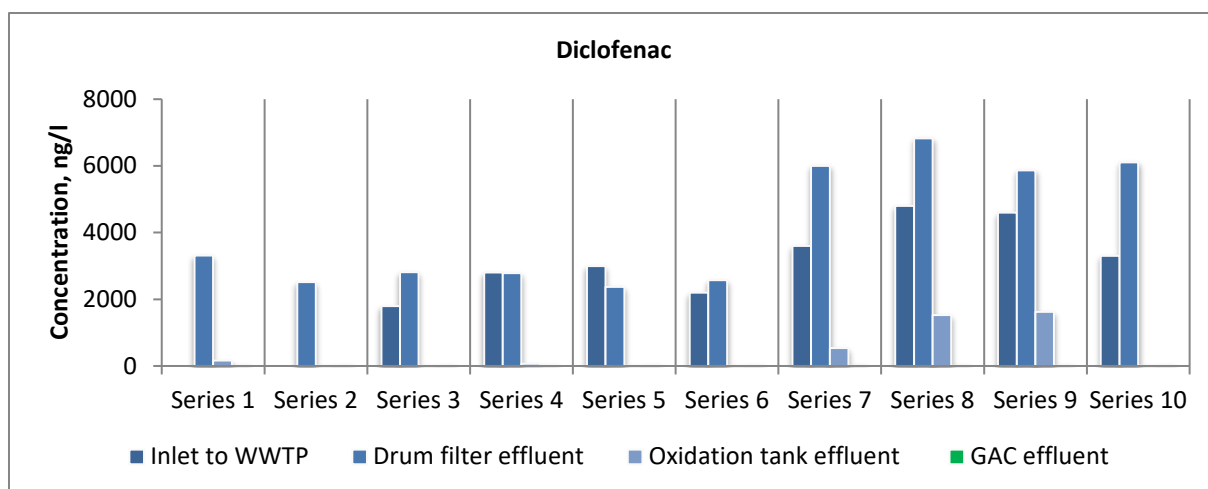


Figure 18. Concentrations of diclofenac at different stages of wastewater treatment.

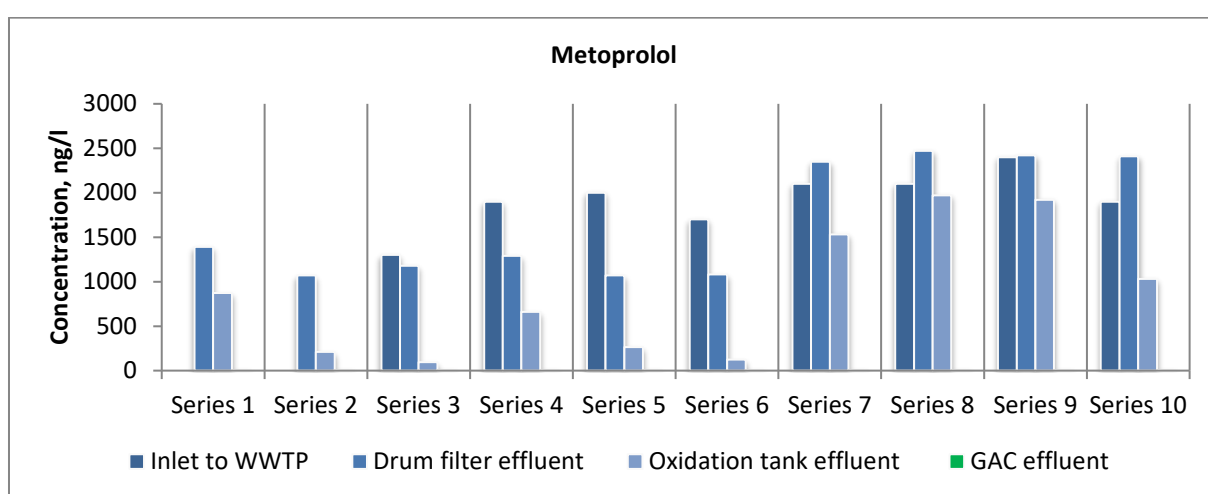


Figure 19. Concentrations of metoprolol at different stages of wastewater treatment.

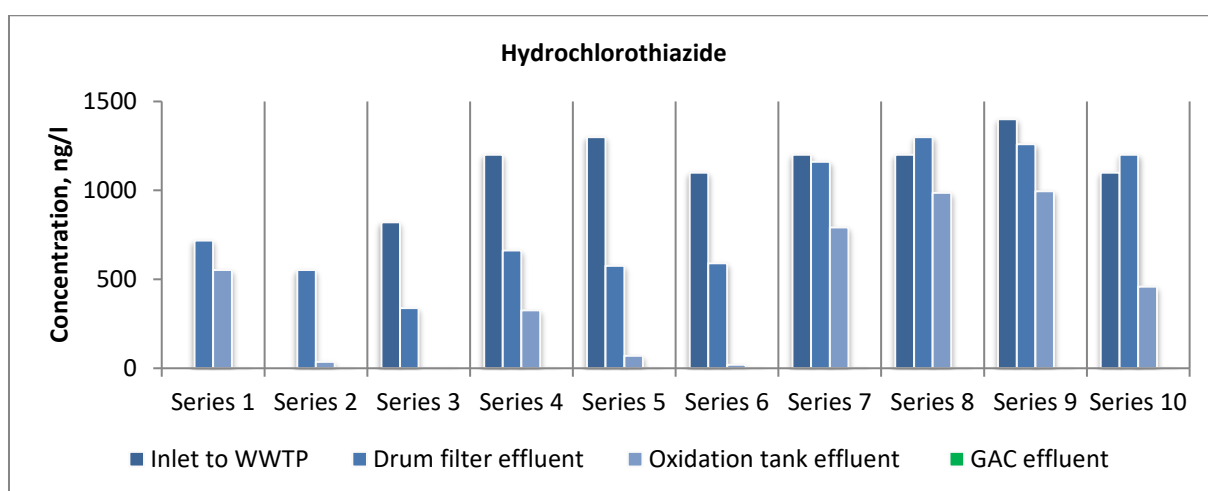


Figure 20. Concentrations of hydrochlorothiazide at different stages of wastewater treatment.

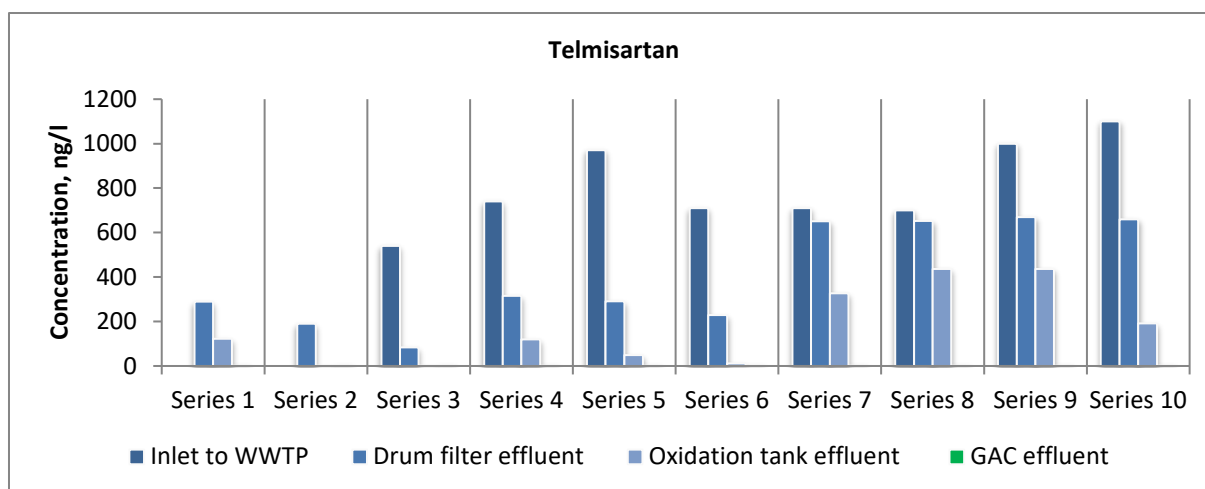


Figure 21. Concentrations of telmisartan at different stages of wastewater treatment.

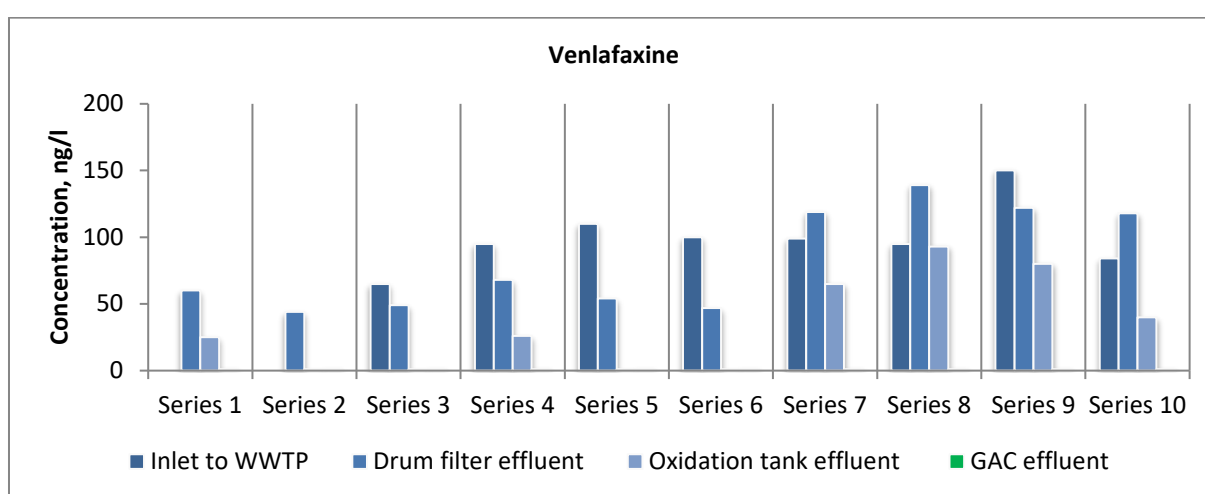


Figure 22. Concentrations of venlafaxine at different stages of wastewater treatment.

Based on the compiled results from all pilot testing series, it can be confirmed that ozonation alone is effective in reducing the concentration of pharmaceuticals in biologically treated wastewater. The efficiency of removal strongly depended on the applied ozone dose. In the test series with the lowest ozone dose (0.25 mg O₃/mg DOC, Series 8), the average reduction of pharmaceutical concentrations was 32%. In contrast, the highest removal efficiency of 97% was achieved in Series 6, where a significantly higher ozone dose of 1.4 mg O₃/mg DOC was applied.

Furthermore, in all tested series, no pharmaceuticals were detected in the effluent after granular activated carbon (GAC) filtration. This confirms the effectiveness of the GAC polishing step in ensuring complete removal of residual micropollutants, regardless of the preceding ozone dose. These results demonstrate that a combination of ozonation followed by GAC filtration is a robust and reliable treatment approach for the advanced removal of pharmaceuticals from wastewater effluents.

In some testing series, the results for diclofenac and clarithromycin showed higher concentrations in the biologically treated wastewater compared to the corresponding influent samples at the plant inlet. This apparent negative removal efficiency can be explained by compound-specific transformation mechanisms during biological treatment processes.

For diclofenac, this effect is primarily attributed to the deconjugation of human metabolites. In municipal wastewater, diclofenac is often excreted in conjugated forms (e.g., glucuronidated or sulfated), which are not readily detected by standard analytical methods. Microbial enzymatic activity during biological treatment can cleave these conjugates, releasing the parent compound—diclofenac—into the aqueous phase. As a result, measurable concentrations of diclofenac in the effluent may increase, even if the total diclofenac-related load

(including conjugated forms) is reduced. This phenomenon has been documented in full-scale WWTPs across Europe, where effluent concentrations have been observed to exceed influent levels, resulting in removal rates as low as –154 % to +33 % (Ek Henning et al. (2020)). Vieno & Sillanpää (2014) report mean diclofenac removal of only ~36 %, with deconjugation being a key transformation pathway.

Similarly, clarithromycin, a macrolide antibiotic, is subject to back-transformation during biological treatment. It is excreted partly as metabolites, including 14-hydroxy-clarithromycin, which may degrade or revert to the parent compound under activated sludge conditions. Additionally, clarithromycin's high sorption affinity for sludge and biofilms can lead initially to adsorption, followed by desorption or transformation back into the aqueous phase. Several studies from Finnish WWTPs reported negative removal efficiencies for clarithromycin—effluent concentrations were higher than influent—linking this behavior to both sampling/analytical variability and microbial transformation of conjugates (Kortesmäki et al. (2020)).

3.2.3 Removal of phthalates

Phthalates, or phthalate esters, are a group of synthetic organic chemicals commonly used as plasticizers to enhance the flexibility and durability of plastics, particularly in polyvinyl chloride (PVC) materials. They are also found in a wide range of consumer products, including personal care items, detergents, packaging, and medical devices. Due to their widespread use and weak chemical bonding to polymer matrices, phthalates can easily leach into the environment during manufacturing, product use, or disposal. Wastewater treatment plants (WWTPs) receive significant loads of phthalates from domestic, industrial, and stormwater sources.

The phthalates detected in wastewater during the pilot testing were di(2-ethylhexyl) phthalate (DEHP), di-n-butyl phthalate, di-n-octyl phthalate, di-isobutyl phthalate and diethyl phthalate, which are among the most commonly detected in municipal wastewater and known for their endocrine-disrupting potential. These compounds are hydrophobic and tend to adsorb onto suspended solids and sludge, but their persistence and potential for bioaccumulation raise concerns for aquatic life and human health. DEHP, in particular, is listed as a priority hazardous substance under the EU Water Framework Directive due to its toxicity and environmental stability. Figure 23 presents the total concentrations of these compounds.

Effective removal of phthalates from wastewater requires a combination of physical, chemical, and biological treatment steps. Conventional activated sludge processes may only partially reduce phthalate concentrations. Advanced treatment technologies such as granular activated carbon (GAC) adsorption, ozonation, and membrane filtration have demonstrated higher removal efficiencies for these compounds (Net et al., 2015; Gao & Wen, 2016). Continued monitoring and improved removal strategies are necessary to minimize the discharge of phthalates into receiving water bodies and to protect ecological and public health.

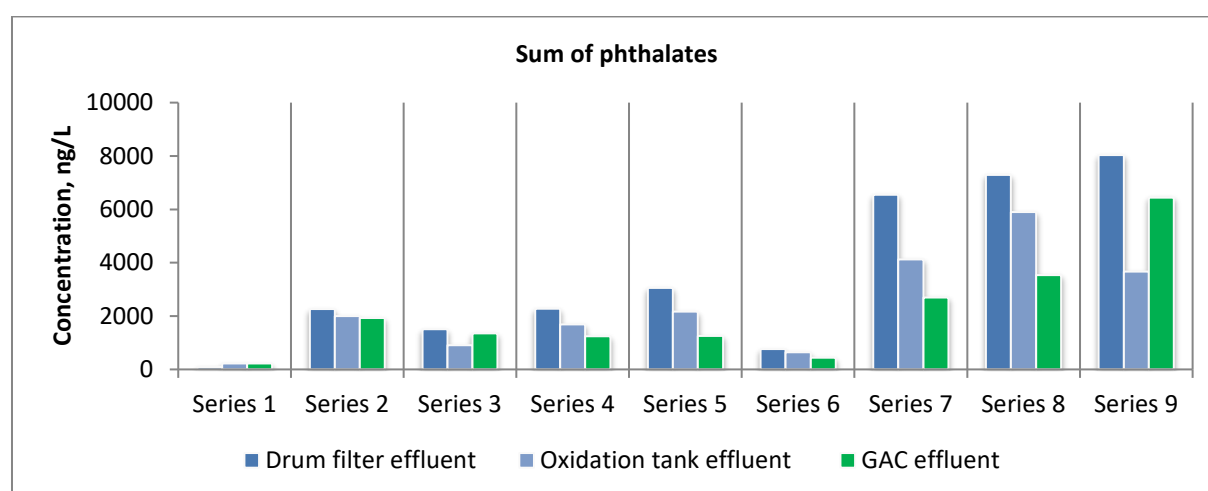


Figure 23. Total concentrations of all phthalates compounds at different stages of wastewater treatment.

Phthalate concentrations were measured in 9 out of the 10 piloting test series. Due to the complex matrix of untreated municipal wastewater and the limitations of standard GC-MS analytical methods in achieving appropriate limits of quantification (LOQ), only samples collected after biological treatment and cloth filtration were analysed, along with subsequent treatment stages—ozonation and GAC (granular activated carbon) effluents.

Measured concentrations of phthalates ranged from 89 ng/L for di-isobutyl phthalate in Series 1 to 4980 ng/L for the same compound in Series 8. Di-isobutyl phthalate was the only phthalate detected consistently in all 9 testing series. Other compounds, such as di(2-ethylhexyl) phthalate (DEHP), were detected in 5 out of 9 series, while di-n-octyl phthalate appeared in only 2 series. Figure 23 presents the total phthalate concentrations measured in the drum filter effluent, oxidation tank effluent, and GAC effluent for each series.

The average removal efficiency of phthalates following ozonation was approximately 29%, with an additional average 41% reduction observed after GAC filtration. These removal rates are notably lower than those observed for most pharmaceuticals tested under similar conditions.

This comparatively lower efficiency can be attributed to the physicochemical properties of phthalates. Being less polar and more hydrophobic, phthalates are less reactive with ozone, which primarily targets electron-rich, polar functional groups typical in many pharmaceuticals. As a result, phthalates exhibit significantly lower reactivity and oxidation rates during ozonation processes (Lee & von Gunten, 2010; Net et al., 2015).

Additionally, their strong sorption to suspended solids and organic matter may limit their availability for oxidation. In the GAC filtration stage, competition with other organic micropollutants and natural organic matter reduces the adsorption efficiency for phthalates (Gao & Wen, 2016). Moreover, the surface chemistry and pore structure of standard activated carbon media are not specifically optimized for phthalate compounds, contributing to their lower removal performance compared to more hydrophilic pharmaceuticals.

3.2.4 Removal of benzotriazoles

Benzotriazoles are synthetic corrosion inhibitors commonly used in industrial applications, metalworking fluids, automotive antifreeze, and household products such as dishwasher detergents. Their widespread use leads to their frequent detection in municipal wastewater, primarily due to domestic and industrial discharges. The compounds analysed during the pilot test—benzotriazole (BTA) and the sum of 4- and 6-methylbenzotriazole (4-MBT and 6-MBT)—are widely used due to their ability to form stable complexes with metal ions and prevent corrosion on copper and its alloys.

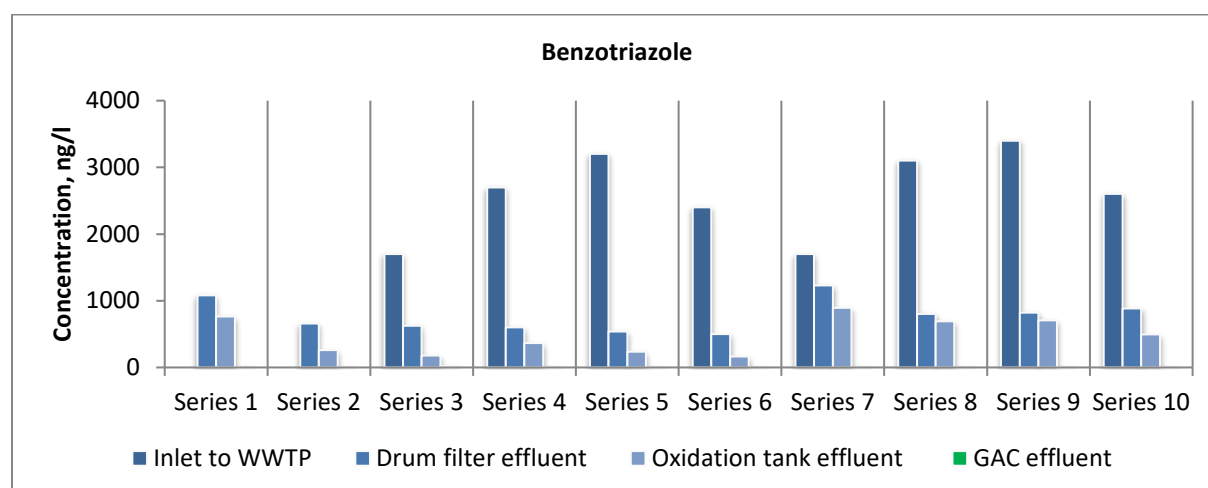


Figure 24. Benzotriazole concentrations at different stages of treatment.

Benzotriazole concentrations in the secondary (biologically treated) effluent were measured in all 10 pilot test series and ranged from 498 ng/L to 1230 ng/L. Starting from Series 3, additional samples were also taken from

the untreated wastewater at the WWTP inlet, where benzotriazole concentrations ranged from 1700 ng/L to 3400 ng/L.

Surprisingly, the results indicated that conventional biological wastewater treatment achieved a higher reduction of benzotriazole than typically expected, with an average removal efficiency of 68%. This is considerably higher than values reported in the literature, where full-scale activated sludge systems typically achieve removal rates of only 10–30% (Weiss & Reemtsma, 2005).

The ozonation process alone further enhanced benzotriazole removal, with efficiency strongly dependent on the applied ozone dose (as shown in Table 1). Removal rates across the pilot test series ranged from 14% at lower ozone doses to 66% under higher doses. These results are in line with the known oxidative behavior of benzotriazole, which, despite its chemical stability, can be effectively degraded by advanced oxidation processes such as ozonation—particularly when sufficient ozone exposure is ensured.

Following the ozonation step, granular activated carbon (GAC) filtration was applied, and the combined treatment consistently achieved complete removal of benzotriazole, with concentrations falling below the analytical detection limit in all test series. This demonstrates the strong complementarity of ozonation and GAC adsorption: while ozonation partially breaks down the parent compound, GAC effectively captures both residual benzotriazole and its oxidation byproducts, ensuring a high level of treatment performance.

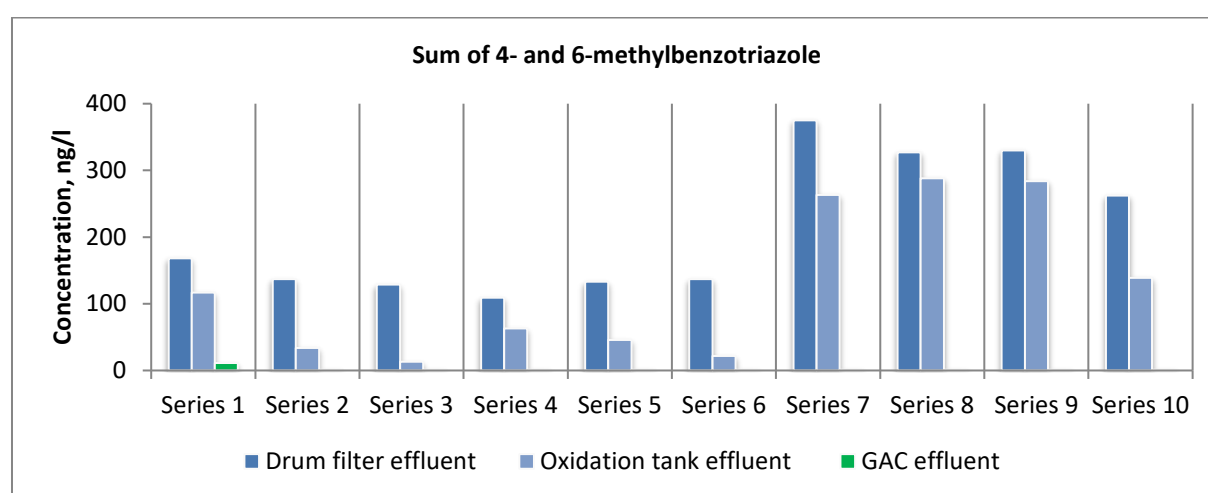


Figure 25. Sum of 4-MBT and 6-MBT concentrations at different stages of treatment.

The sum concentration of 4-methylbenzotriazole (4-MBT) and 6-methylbenzotriazole (6-MBT) prior to the advanced treatment stage ranged from 109 ng/L to 375 ng/L across the pilot test series. These methylated derivatives of benzotriazole are commonly used in similar applications, such as corrosion inhibition, and exhibit comparable environmental persistence and resistance to biodegradation. 4-MBT serves also as an essential component within the mixture of aircraft de-icing fluid.

The removal efficiency during the subsequent ozonation process followed a similar trend to that observed for benzotriazole, with performance depending on the applied ozone dose. Removal rates varied from 12% at low ozone exposure to 89% at higher doses, reflecting the oxidative transformation potential of these compounds under advanced treatment conditions.

In GAC filter effluent concentrations of both 4-MBT and 6-MBT fell below the detection limit in all test series, except for Series 1, where trace amounts remained.

3.2.5 Removal of PFAS

Per- and polyfluoroalkyl substances (PFAS) are a large group of synthetic chemicals defined by their robust carbon–fluorine bonds, giving them exceptional thermal and chemical stability. These “forever chemicals” are found in numerous industrial and consumer products, including firefighting foams, non-stick cookware,

waterproof textiles, food packaging, and electrical insulation due to their oil- and water-repellent properties. They frequently enter wastewater systems through domestic, commercial, and industrial channels.

PFAS are highly resistant to conventional biological degradation, passing through primary and secondary treatment processes with minimal attenuation. Additionally, some PFAS compounds may partition into sewage sludge, complicating its disposal. This persistence, combined with documented bioaccumulation and health risks (e.g., endocrine disruption and carcinogenicity), has prompted increased regulatory scrutiny.

Under the revised EU Urban Wastewater Treatment Directive (UWWTD), large wastewater treatment plants (>100,000 population equivalent) are now required to monitor PFAS in both influent and effluent streams—marking a significant regulatory advancement.

To address PFAS removal, advanced treatment technologies are essential. Adsorption methods, such as granular activated carbon (GAC) and ion exchange resins (IXR), are among the most effective non-destructive options. GAC is effective for long-chain PFAS like PFOA and PFOS, while IXR shows high efficiency—often outperforming GAC—for both long- and short-chain PFAS.

Membrane processes (e.g., nanofiltration and reverse osmosis) also provide robust PFAS removal, though they generate a concentrated waste stream that requires further handling. Emerging destructive technologies such as electrochemical oxidation and advanced plasma or ultrasound treatments show potential but remain largely in the research stage.

Out of the 49 different PFAS compounds analysed during the piloting series, only eight—6:2 FTSA, PFBA, PFBS, PFHpA, PFHxA, PFOA, PFPeA, and PFOS—were detected in the wastewater samples. In testing series 1, 2, and 3, samples for PFAS analysis were collected after cloth filtration, ozone oxidation, and granular activated carbon (GAC) filtration stages.

Starting from Series 4, additional samples were also collected from the WWTP influent, enabling a more complete assessment of PFAS removal efficiency across the treatment train. Beginning with Series 5, samples from the ozone oxidation effluent were no longer collected, as initial results indicated that ozonation had limited effectiveness in PFAS removal, particularly under the conditions used in the pilot setup.

The concentrations of individual PFAS at different stages of wastewater treatment are presented in Figures 26 – 33.

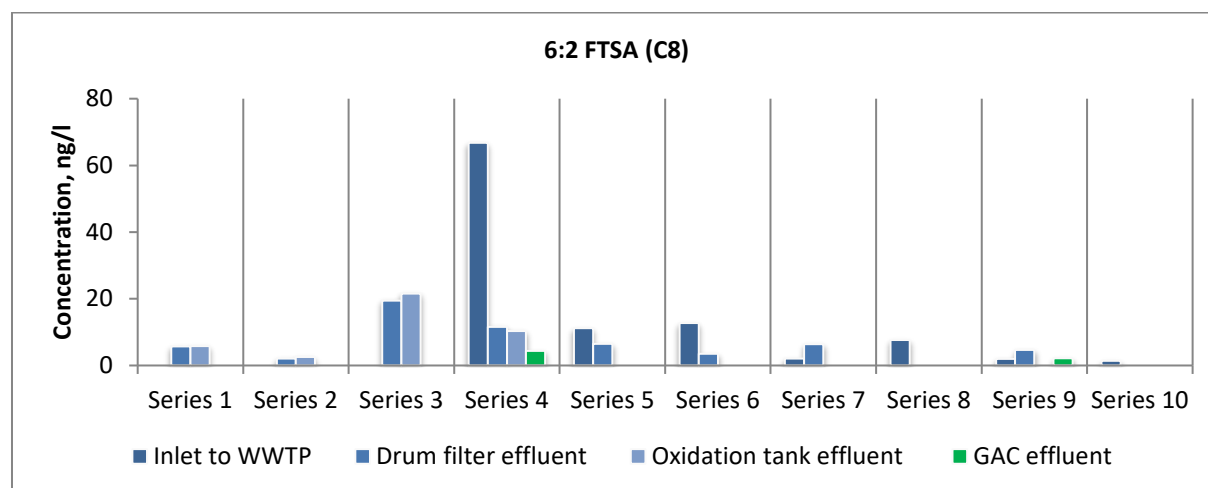


Figure 26. Concentration of 6:2 Fluorotelomer sulfonic acid at different stages of wastewater treatment.

Concentrations of 6:2 FTSA in the drum filter effluent varied between pilot test series, ranging from 2.1 ng/L to 19.4 ng/L. In Series 1 to 4, the application of ozonation did not result in a substantial decrease in 6:2 FTSA concentrations. However, granular activated carbon (GAC) filtration contributed significantly to the final removal, with residual concentrations frequently falling below the detection limit. These results confirm that

adsorption onto activated carbon is an effective method for controlling 6:2 FTSA in wastewater treatment.

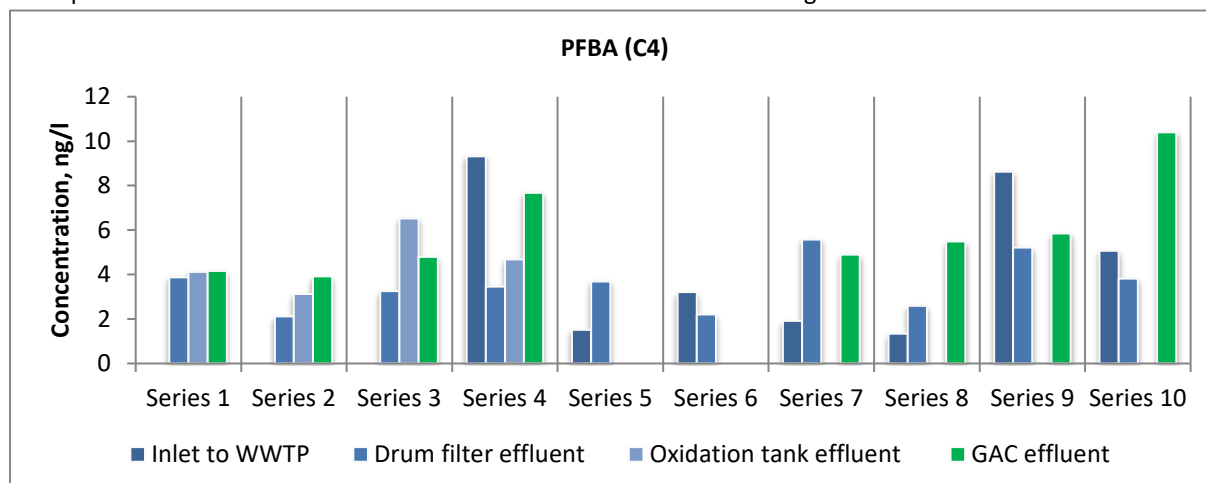


Figure 27. Concentration of Perfluorobutanoic acid at different stages of wastewater treatment.

PFBA was detected in the drum filter effluent at relatively low concentrations (2.1–5.6 ng/L), and its levels remained relatively stable throughout the pilot test series. Neither ozonation nor GAC filtration achieved meaningful removal of PFBA; in some cases, slightly higher concentrations were observed after treatment.

It should be noted that these results must be interpreted with caution, as the detected concentrations were only slightly above the limit of detection (LOD) of the analytical method. Therefore, no definitive conclusions regarding PFBA removal efficiency can be drawn from these measurements.

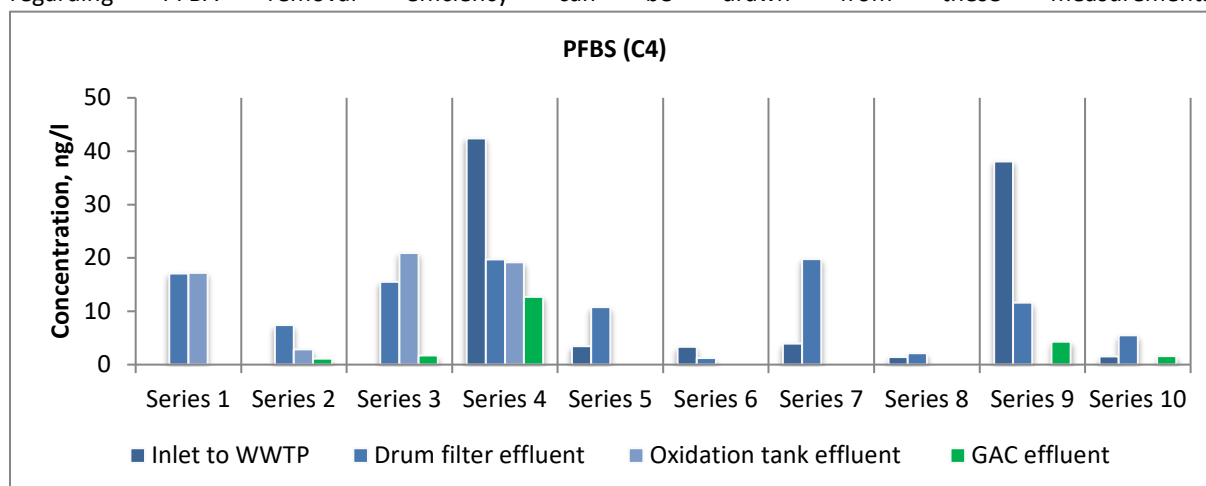


Figure 28. Concentration of Perfluorobutanesulfonic acid at different stages of wastewater treatment.

PFBS concentrations in the drum filter effluent varied between 1.2 ng/L and 19.8 ng/L across the test series. Similar to PFBA, PFBS exhibited minimal removal through ozonation, demonstrating its high persistence and low reactivity with ozone. In Series 3, where the highest ozone dose of 20 mg/L was applied, an increase in PFBS concentration by 25% was observed. This aligns with findings from Pranjali et al. (2021), who reported an average 31% increase in PFBS levels after ozonation, likely due to the transformation of precursor compounds.

However, GAC treatment led to a measurable reduction in PFBS concentrations, with removal efficiencies ranging from 35% to 60% in several test series. These results suggest that while PFBS is relatively resistant to oxidation, adsorption onto activated carbon offers a partially effective removal pathway under the tested conditions.

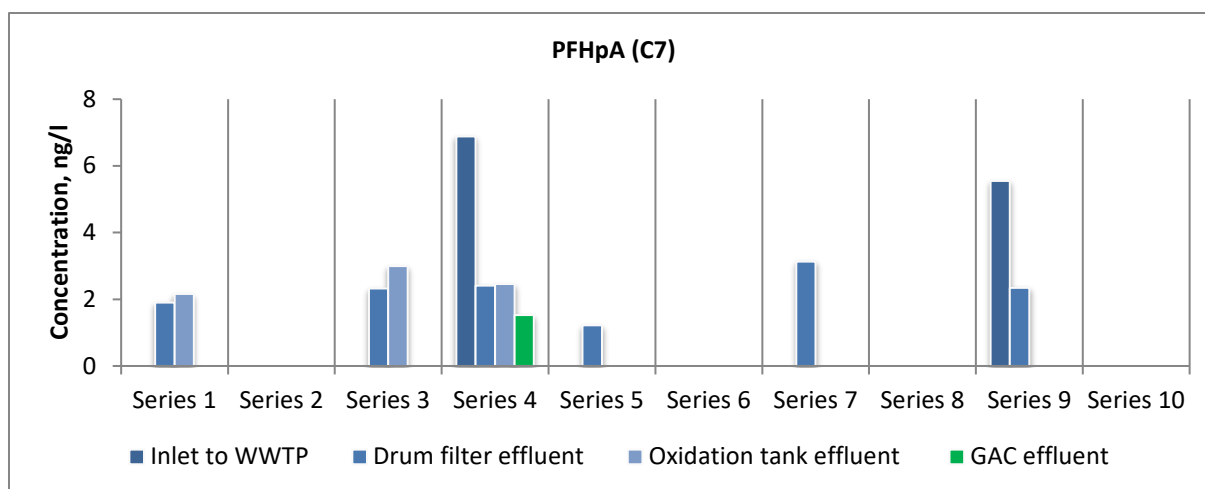


Figure 29. Concentration of Perfluoroheptanoic acid at different stages of wastewater treatment.

PFHpA was not consistently detected in all samples, and when present, it appeared at very low concentrations—ranging from 1.2 ng/L to 3.13 ng/L in the biologically treated wastewater. Ozonation produced inconsistent removal results, which is expected given the limited reactivity of perfluoroalkyl carboxylic acids with ozone under typical treatment conditions.

Granular activated carbon (GAC) filtration proved to be more reliable, as PFHpA was not detected in the GAC effluent in most cases. An exception was observed in Series 4, where a concentration of 1.53 ng/L was measured in the GAC effluent—only slightly above the method’s 1 ng/L limit of detection (LOD).

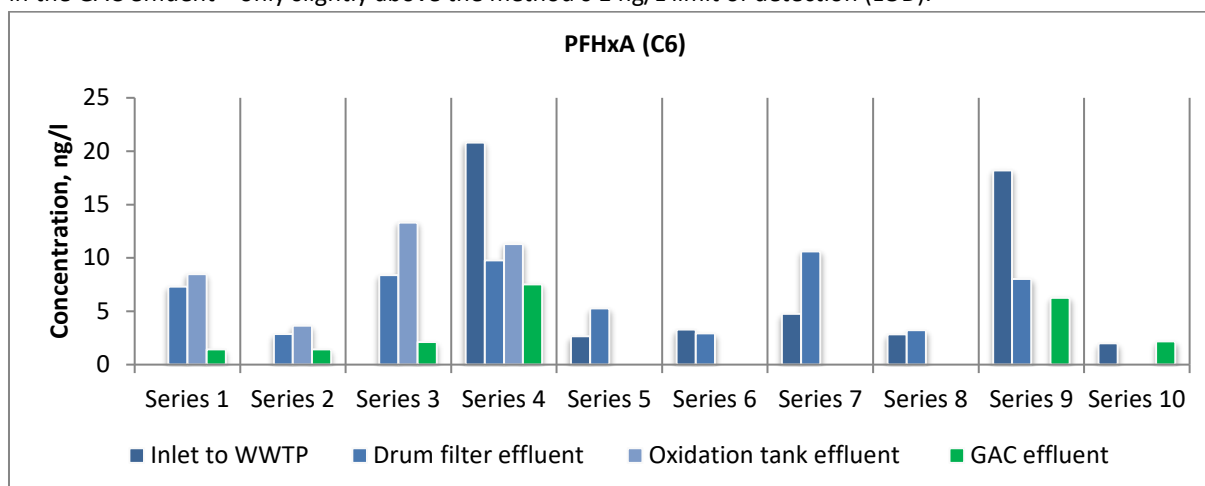


Figure 30. Concentration of Perfluorohexanoic acid at different stages of wastewater treatment.

PFHxA concentrations in the drum filter effluent ranged from 2.8 ng/L to 10.6 ng/L across the pilot test series. In testing Series 1 through 4—where ozonation effluent samples were collected—ozonation appeared to have a negative removal efficiency, as PFHxA concentrations were found to be slightly higher after treatment.

This phenomenon is supported by literature, where ozonation has been shown to increase PFHxA concentrations, likely due to the oxidative transformation of precursor compounds. For example, Pranjali et al. (2021) reported average PFHxA increases of 11% at lower ozone doses (below 6.5 mg/L) and 18% at higher doses (above 7 mg/L). Similar findings were presented by Dickenson et al. (2015) and Sari et al. (2020), who also attributed such increases to precursor oxidation processes during ozonation.

The observed results in this study align with these findings:

Series 1: PFHxA increased by 13.7% after ozonation at 6 mg/L ozone dose.

Series 2: Increase of 20.8% at 10 mg/L ozone dose.

Series 3: Increase of 36.8% at a high dose of 20 mg/L.

Series 4: Increase of 13.6% at an 8 mg/L dose.

FHxA concentrations in the GAC filter effluent during Series 5 to 8 were below the analytical detection limit. In these test series, GAC filter F.1 was used, which was filled with a different type of activated carbon than in the

other series (see Table 1)—specifically, KemSorb™ 8385, featuring an effective particle size of 0.6–0.25 mm. The use of this finer-grained carbon media may have contributed to the improved removal efficiency, as smaller particle sizes generally provide greater surface area and improved contact time, enhancing sorption of PFAS compounds such as PFHxA.

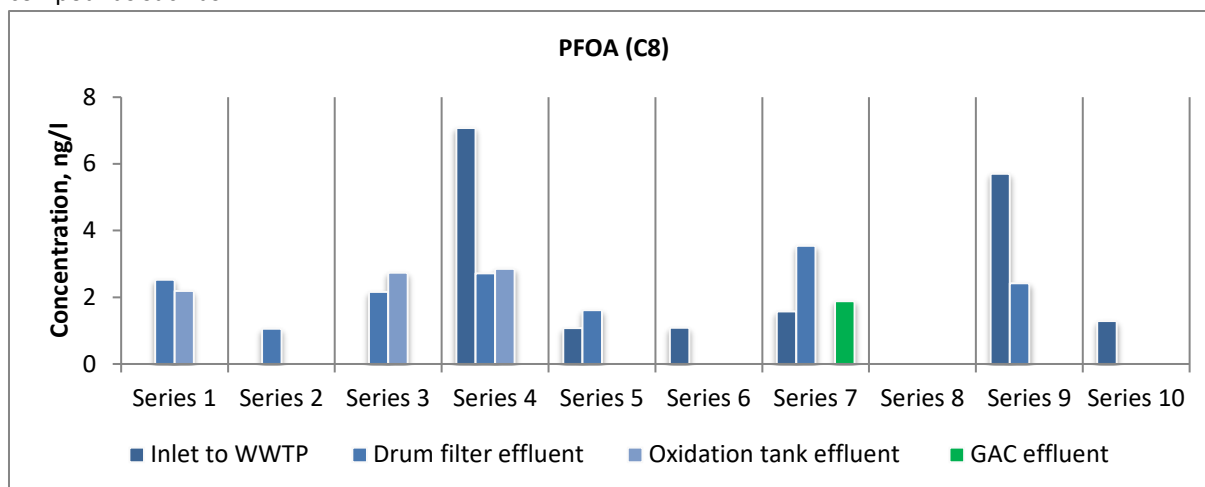


Figure 31. Concentration of Perfluorooctanoic acid at different stages of wastewater treatment.

PFOA concentrations in the drum filter effluent were consistently low across the majority of test series, ranging from 1.06 ng/L to 3.56 ng/L. As expected, granular activated carbon (GAC) filtration proved effective at removing this long-chain PFAS compound. In all test series, PFOA concentrations in the GAC effluent were below the detection limit, except in Series 7, where it was detected at 1.88 ng/L.

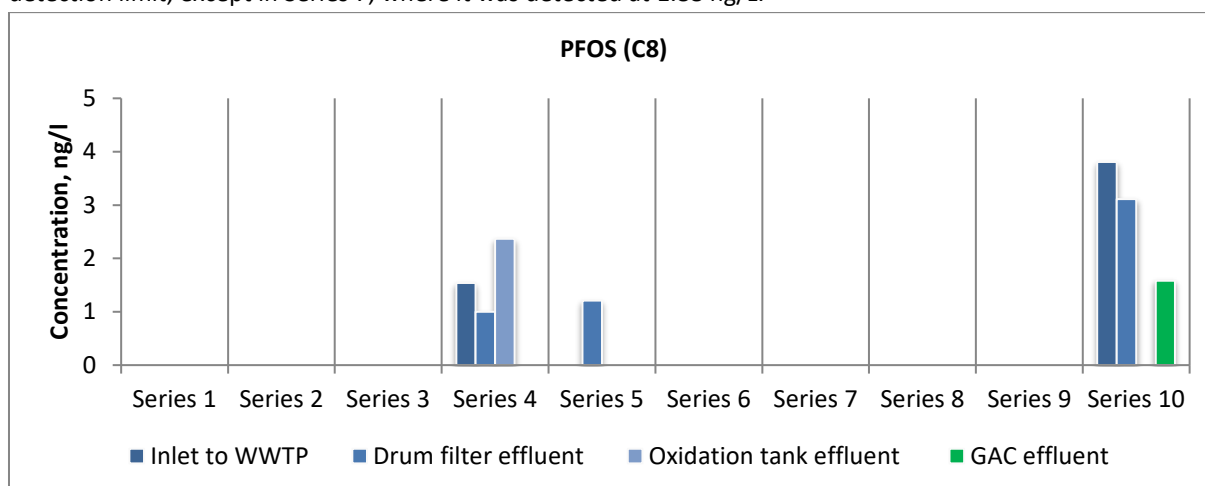


Figure 32. Concentration of Perfluorooctanesulfonic acid at different stages of wastewater treatment.

PFOS was detected in three out of ten testing series, at very low concentrations ranging from 1.0 ng/L to 3.8 ng/L. Due to this infrequent occurrence and the low concentration levels, no definitive conclusions regarding PFOS removal efficiency can be drawn from these measurements.

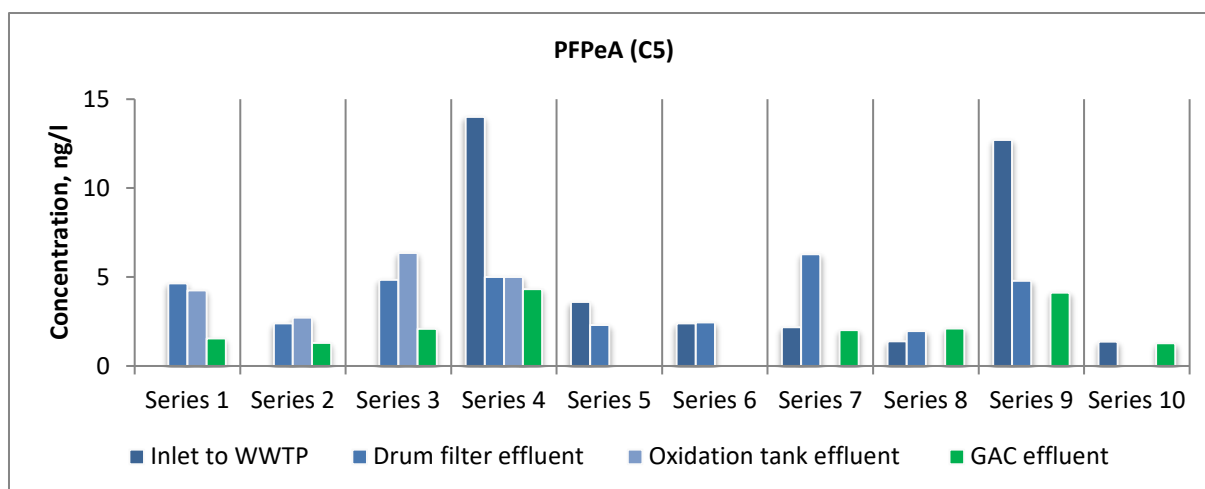


Figure 33. Concentration of Perfluoro-n-pentanoic acid at different stages of wastewater treatment.

PFPeA was detected in all testing series, with concentrations in the drum filter effluent ranging from 1.96 ng/L to 6.29 ng/L. Similar to PFHxA, scientific studies have reported that PFPeA concentrations may increase following ozonation, likely due to the oxidation of precursor compounds. For example, Pranjali et al. (2021) observed an average increase of 9% in PFPeA concentrations at ozone doses above 7 mg/L.

In the present study, a 12.1% increase in PFPeA was observed in Series 2, where the applied ozone dose was 10 mg/L. In Series 3, a more substantial increase of 23.9% occurred at an ozone dose of 20 mg/L. In contrast, no increase in PFPeA concentration was observed in the remaining test series, where lower ozone doses were applied.

These results support existing evidence that ozonation may lead to the formation of short-chain PFAS such as PFPeA, particularly when treating wastewater containing PFAS precursors and when higher ozone doses are applied.

CONCLUSIONS

This pilot-scale study at the Kaunas WWTP comprehensively demonstrated the feasibility and limitations of advanced wastewater treatment technologies—specifically ozonation and granular activated carbon (GAC) filtration—for the removal of organic micropollutants (OMPs), including pharmaceuticals, phthalates, benzotriazoles, and per- and polyfluoroalkyl substances (PFAS).

The pilot plant showed some effectiveness in removal of DOC and COD, achieving ~55% and ~51% reduction, respectively. Total nitrogen and phosphorus removal remained low (13% and 24%, respectively), highlighting the limited role of these technologies for nutrient elimination.

Pharmaceuticals were effectively eliminated during the pilot tests, with ozonation achieving up to 97% removal at higher doses, and GAC filtration consistently reducing residual concentrations to below detection limits. However, in some cases, negative removal values were observed, primarily due to biotransformation or deconjugation of pharmaceutical metabolites during the biological treatment stage—particularly for diclofenac and clarithromycin.

These findings are important to consider in the context of full-scale implementation of quaternary treatment, especially under the requirements of the revised Urban Waste Water Treatment Directive (UWWTD), which mandates an average micropollutant removal efficiency of 80%, calculated from inlet and outlet concentrations. It is important to note that lower concentrations of diclofenac and clarithromycin measured at the WWTP inlet may not accurately reflect the total load, as these compounds are often excreted in conjugated forms (e.g., glucuronides or sulfates) that may not be detected by standard analytical methods. During biological treatment, these conjugates can be cleaved, releasing the parent compounds into the water phase and lowering the apparent removal efficiency when comparing influent and effluent concentrations.

Phthalate removal was moderate—ozonation averaged 29% and GAC an additional 41%—due to the hydrophobic nature and lower ozone reactivity of these compounds. Benzotriazoles, in contrast, responded well to the combined treatment: biological treatment alone removed up to 68%, ozonation further improved it, and GAC ensured complete removal.

PFAS proved the most challenging. Out of 49 tested compounds, only 8 were consistently detected. Ozonation was ineffective and, in some cases, even increased concentrations of short-chain PFAS, likely due to precursor oxidation. GAC demonstrated selective efficacy—removing long-chain PFAS (like PFOA and PFOS) more effectively than short-chain variants.

Overall, the results validate the synergistic role of ozonation and GAC as a robust treatment combination for many OMPs, aligning with the updated EU Urban Wastewater Treatment Directive's requirement for quaternary treatment. However, PFAS removal remains a significant challenge requiring further technological advancements.

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