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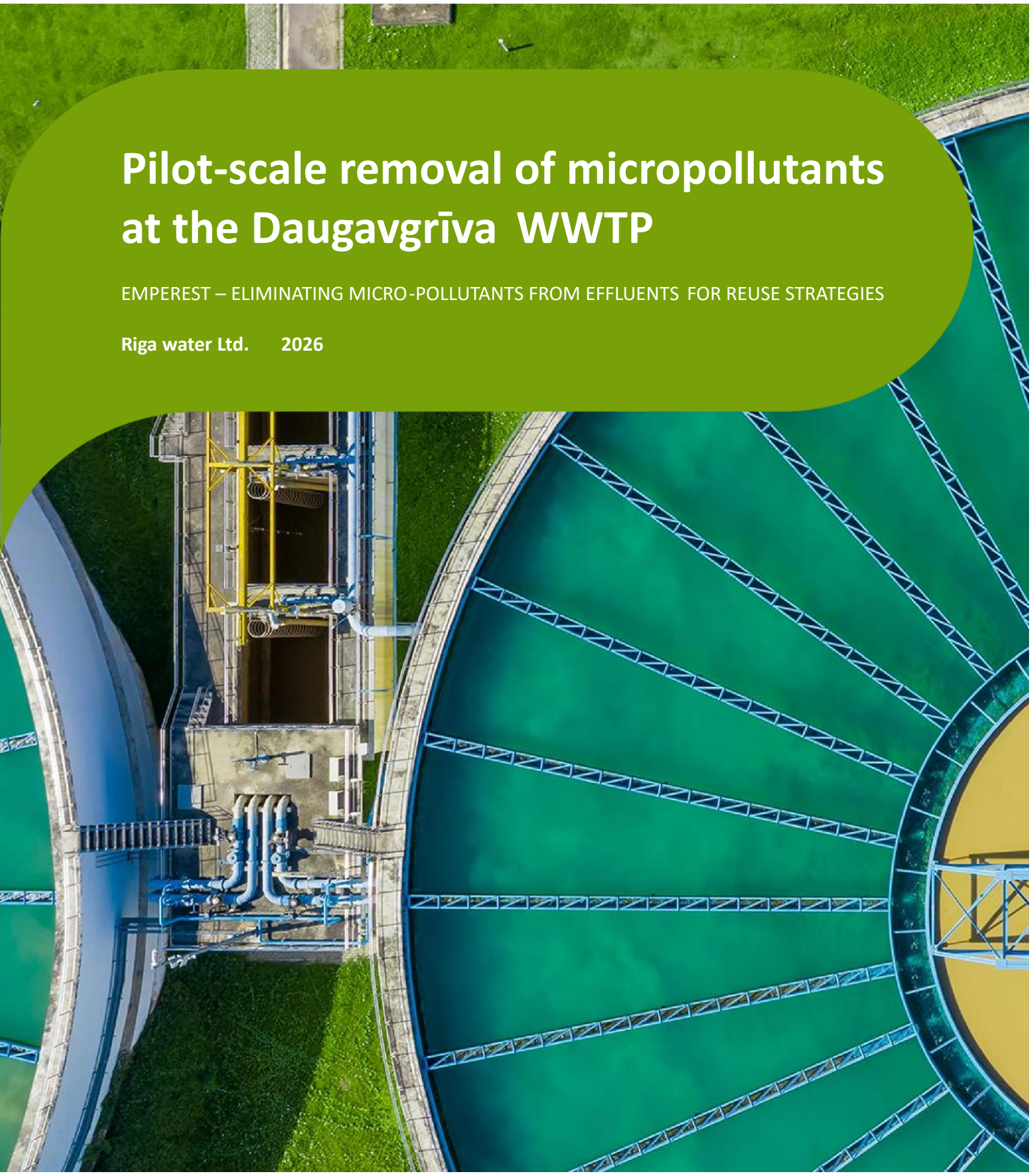
SUSTAINABLE WATERS
EMPEREST

Pilot-scale removal of micropollutants at the Daugavgrīva WWTP

EMPEREST – ELIMINATING MICRO-POLLUTANTS FROM EFFLUENTS FOR REUSE STRATEGIES

Riga water Ltd. **2026**



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.

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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). The sole responsibility for the content of this publication lies with the authors. It does not necessarily reflect the opinion of the European Union.

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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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 exceptionally 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 recognised as primary contributors of OMPs to aquatic environments. While wastewater treatment plants are meant to eliminate organic matter and nutrients, they frequently fall short when it comes to removing micropollutants like pharmaceuticals, personal care items, 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. To address this increasing risk, researchers are investigating solutions like advanced oxidation methods, membrane filtration, and activated carbon adsorption, aiming to safeguard human health and aquatic environments.

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 requires extra quaternary treatment at WWTPs serving 10,000–100,000 PE in areas sensitive to micropollutants, unless a risk assessment shows no environmental or public health risks. This report presents the results of pilot testing on the removal of organic micropollutants using of a mobile pilot plant, which enables the evaluation of the efficiency of advanced wastewater treatment processes.

2. Materials and methods

2.1. Study site

The test took place at the Daugavgrīva Wastewater Treatment Plant, Latvia's largest facility of its kind. The average dry weather influent flow rate to the plant is approx. 130,000 m^3/d and the pollutant load corresponds to 1000,000 population equivalents (PE). The primary treatment line comprises perforated screens, aerated grit chambers and six primary settling tanks. The secondary treatment includes aeration basins with activated sludge process and ten secondary clarifiers.

2.2. Pilot description

The pilot test was conducted using a semi-technical mobile pilot plant, designed in detail by Gdańsk Water Utilities and constructed by the Probiko-Aqua company (see Figure 1).

The pilot installation consists of the following main units:

Column1	Column2	Column3	Column4	Column5
Equipment	Parameter	Value	Unit	Notes
MITA TF2 VM cloth drum filter (DF)	Filter type	POLSTOFF "PILE"	-	thickness 4–5 mm
	Filtration surface	2	m2	-
	Max hydraulic capacity	20	m3/h	-
ProO2 Max 795MC PSA oxygen concentrator (OC)	Max gas flow	10	l/min	-
	Oxygen content at 2 l/min	>93	%	±3%
	Oxygen content at 10 l/min	87–93	%	-
Ozonia CFS-1 2G ozone generator (OG)	Nominal ozone concentration	10	wt%	-
	Nominal ozone production	55	g/h	-
Ozone contact tank	Diameter	0.705	m	-
	Height	2.30	m	-
	Working volume	0.200–0.800	m3	-
Gravity filters (2 units)	Diameter	0.35	m	-
	Surface area	0.962	m2	-

	Filter nozzles	Vertical slots	-	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	m3	-
Probiko-Aqua Protec 1200 EW UV lamp	Maximum flow	0.70	m3/h	-
	UV dose	400	J/m2	at 60% UV transmit-tance
Air compressor (Airpress LMO 25-250)	Free air delivery	150	L/min	-
	Maximum pressure	8	bar	-

The pilot unit includes these online measurement instruments for monitoring the process:

Equipment	Parameter	Value	Unit	Notes
IFM SM9100 magnetic-inductive water flow meter	Quantity	1	unit	-
IFM SM7120 magnetic-inductive water flow meter	Quantity	6	units	-
IFM SA5020 air flow sensor	Quantity	1	unit	-
IFM PN2098 water level sensor	Quantity	8	units	-
WTW UV 700 IQ SAC UV absorbance sensor (with ultrasound cleaning)	Quantity	3	units	-
WTW VisoTurb 700 IQ turbidity sensor (with ultrasound cleaning)	Quantity	2	units	-

Fig. 1 View of the pilot plant located at the Daugavgrīva WWTP.

Online UV absorbance (UVA) and turbidity sensors allow to continuously monitor removal performance of suspended solids an organic compound. 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 shipping container (Figure 2).

Fig. 2 View of mounted devices.

The pilot plant container design allows it to be quickly transported and installed at any drinking water treatment plant or wastewater treatment plant. **Figures 3** to **4** show a process flow diagram displayed on the touch panel of HMI.

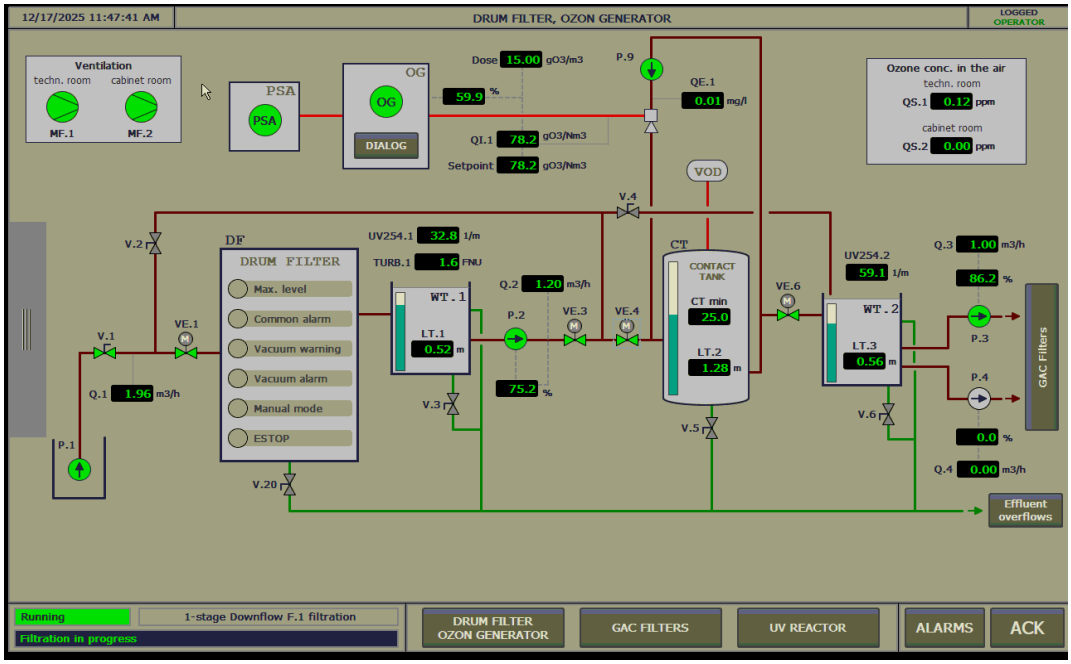

Fig. 3 Flow diagram displayed on screen 1 of HMI panel. Labelling of main components of the process train: DF - cloth drum filter, PSA - oxygen concentrator, OG - ozone generator, WT – intermediate process tank, P - process pump, UVA254 – UV absorbance sensor, TURB – turbidity sensor.

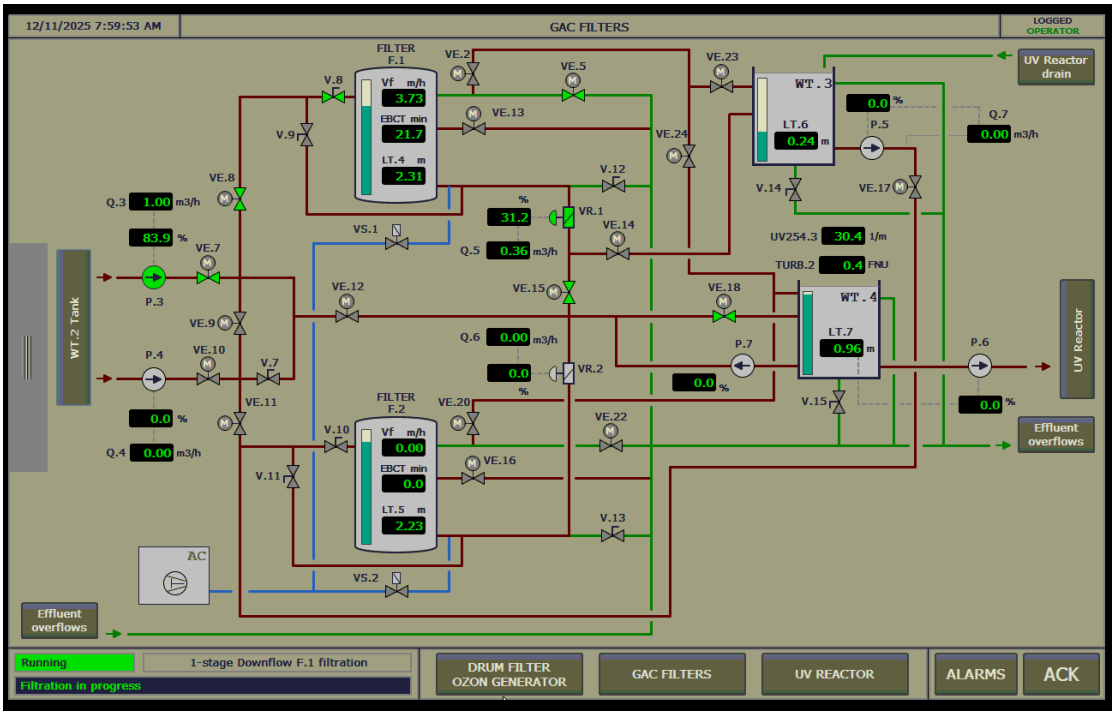

Fig. 4 Flow diagram displayed on screen 2 of HMI panel. Main components of the process: F – gravity filter, WT.3 - intermediate process tank, WT.4 – backwash water tank, P - process pump, AC - air compressor, UVA254 – UV absorbance sensor, TURB – turbidity sensor.

Remote monitoring and control of the process allows for easy and reliable tracking at any time.

2.3. Analytical methods

Online sensors were used to track how well the treatment system performed. Because there was only a short time available for sampling, samples were taken between 03 December 2025 and 17 December 2025, all under dry weather flow conditions. The samples were tested for wastewater quality, focusing on suspended solids, organic compounds, and contaminants of emerging concern (OMPs). The measured OMPs cover those listed in the UWWTD Directive, which mandates at least 80% removal efficiency (shown in bold).

The list of measured wastewater quality parameters is as follows:

- Dissolved organic carbon (DOC)
- COD
- UV absorbance
- Turbidity
- PFASs (PFAS 43)
- Pharmaceuticals
- One hormone: beta-estradiol
- Benzotriazole and sum of 4-methylbenzotriazole and 6-methylbenzotriazole

List of PFASs, evaluated in pilot plant:

No.	PFAS Group	Abbreviation	Name
1	**Fluorotelomer Sulfonates (FTS)**	4:2 FTS	4:2 Fluorotelomer Sulfonate
2		6:2 FTS	6:2 Fluorotelomer Sulfonate
3		8:2 FTS	8:2 Fluorotelomer Sulfonate
4		10:2 FTS	10:2 Fluorotelomer Sulfonate
5	**Fluorotelomer Carboxylic Acids (FTCA)**	3:3 FTCA	3:3 Fluorotelomer Carboxylic Acid
6		5:3 FTCA	5:3 Fluorotelomer Carboxylic Acid
7		7:3 FTCA	7:3 Fluorotelomer Carboxylic Acid
8	**Chlorinated Polyfluoroether Sulfonates**	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonate
9		11Cl-PF3OUdS	11-Chloro-eicosafluoro-3-oxaundecane-1-sulfonate
10	**Ether / Replacement PFAS**	DONA	Dodecafluoro-3H-4,8-dioxanonanoate
11		HFPO-DA (Gen X)	Hexafluoropropylene Oxide Dimer Acid (GenX)
12		PFEESA	Perfluoro(2-ethoxyethane)sulfonic Acid
13	**Sulfonamides and Precursors**	FOSA	Perfluorooctane Sulfonamide
14		MeFOSA	N-Methyl Perfluorooctane Sulfonamide
15		EtFOSA	N-Ethyl Perfluorooctane Sulfonamide
16		MeFOSE	N-Methyl Perfluorooctane Sulfonamidoethanol
17		EtFOSE	N-Ethyl Perfluorooctane Sulfonamidoethanol
18		MeFOSAA	N-Methyl Perfluorooctane Sulfonamidoacetic Acid
19		EtFOSAA	N-Ethyl Perfluorooctane Sulfonamidoacetic Acid
20		FBSA	Perfluorobutane Sulfonamide
21		FHxSA	Perfluorohexane Sulfonamide
22	**Perfluoroalkyl Carboxylic Acids (PFCAs)**	PFBA	Perfluorobutanoic Acid
23		PFPeA	Perfluoropentanoic Acid
24		PFHxA	Perfluorohexanoic Acid
25		PFHpA	Perfluoroheptanoic Acid
26		PFOA	Perfluorooctanoic Acid
27		PFNA	Perfluorononanoic Acid
28		PFDA	Perfluorodecanoic Acid
29		PFUnDA	Perfluoroundecanoic Acid
30		PFDoA	Perfluorododecanoic Acid
31		PFTrDA	Perfluorotridecanoic Acid
32		PFTeA	Perfluorotetradecanoic Acid
33		PFHxDA	Perfluorohexadecanoic Acid
34		PFODA	Perfluorooctadecanoic Acid
35	**Perfluoroalkane Sulfonic Acids (PFSAs)**	PFBS	Perfluorobutanesulfonic Acid
36		PFPeS	Perfluoropentanesulfonic Acid
37		PFHxS	Perfluorohexanesulfonic Acid
38		PFHpS	Perfluoroheptanesulfonic Acid
39		PFOS	Perfluorooctanesulfonic Acid
40		PFNS	Perfluorononanesulfonic Acid
41		PFDS	Perfluorodecanesulfonic Acid
42		PFUnDS	Perfluoroundecanesulfonic Acid
43		PFDoS	Perfluorododecanesulfonic Acid

List of Pharmaceuticals and one hormone (*Beta-estradiol)*, evaluated in pilot plant:

No.	Substance	Category	Use	UWWD (revised)	Monitor-ing
1	Amisulpride	Antipsychotic	Schizophrenia	⚠ Included in emerging pollutant screening	⚠ Yes
2	Citalopram	SSRI antidepres-sant	Depression/anxi-ety	⚠ Likely monitored (priority screening)	⚠ Yes
3	Venlafaxine	SNRI antidepres-sant	Depression/anxi-ety	⚠ Likely monitored	⚠ Yes
4	Diclofenac	NSAID	Pain/inflamma-tion	✔ Target compound (high priority re-moval)	✔ Yes
5	Irbesartan	ARB	Hypertension	⚠ Possible inclusion depending on risk	⚠ Partial
6	Candesartan	ARB	Hypertension	⚠ Possible inclusion depending on risk	⚠ Partial
7	Telmisartan	ARB	Hypertension	⚠ Possible inclusion depending on risk	⚠ Partial
8	Metoprolol	Beta-blocker	Heart disease	⚠ Included in screening lists	⚠ Yes
9	Clarithromy-cin	Antibiotic	Infection	⚠ Monitored (AMR relevance)	⚠ Yes
10	Carbamaze-pine	Antiepileptic	Epilepsy	⚠ Marker compound (persistent)	✔ Yes
11	*Beta-estra-diol*[1]	*Hormone*	*HRT*	✔ *High-priority endocrine disruptor*	✔ *Yes*

No.	Substance	Category	Use	UWWD (revised)	Monitoring
1	Benzotriazole	Corrosion inhibitor	Industry	⚠ Potential emerging pollutant	⚠ Yes
2	4-methylbenzotriazole	Corrosion inhibitor	Industry	⚠ Likely monitored in studies	⚠ Yes
3	6-methylbenzotriazole	Corrosion inhibitor	Industry	⚠ Likely monitored in studies	⚠ Yes

Standard procedures were used to measure COD, DOC, and TSS. Ultraviolet absorbance at a wavelength of 254 nanometers (UVA_{254}) was measured with WTW UV 700 IQ SAC sensors. Turbidity was measured according to the nephelometric principle with WTW VisoTurb 700 IQ sensors.

Beta-estradiol was measured by GC-MS (gas chromatography-mass spectrometry), while pharmaceuticals and benzotriazoles were analysed using LC-MS (liquid chromatography-mass spectrometry). PFASs were measured using LC-MS/MS (liquid chromatography-tandem mass spectrometry).

All analyses of OPMs were performed in an external accredited laboratory.

[1] It IS considered under EU water monitoring frameworks: EU Water Framework Directive, included in priority endocrine-disrupting substances lists / watch lists in monitoring programs Estradiol is widely documented in environmental science literature: Endocrine disruption in aquatic systems: Rosenfeld & Cooke (2019), *Endocrine Reviews*; Presence in wastewater effluents: Johnson et al. (2008), *Environmental Science & Technology*; Fish feminization effects at ng/L levels: Kidd et al. (2007), *PNAS*

3. Results

The pilot unit received effluent from the secondary clarifiers at the Daugavgrīva WWTP. The treatment process chain consisted of cloth filtration, ozone oxidation followed by granular activated carbon (GAC) filtration. Granular activated carbon was used to fill GAC filter No.2. This carbon was produced by steam activation of selected low-ash coal, specifically Kemira KemSorbTM 2300 PWG 830, featuring an effective particle mesh size of 8x30 (0.6–2.36 mm).
The height of filter bed equalled to 1,35 m.
The pilot test took place from 03 December to 17 December 2025, exclusively during dry weather flow conditions.
The studies were focused on determining the UV_{254} absorbance with various ozone dose, constant ozone contact time, evaluating the efficiency of organic micropollutant removal with ozonation, followed by GAC filtration.

3.1. Determining the UV_{254} absorbance reduction

UVA_{254} measurements serve as an indirect indicator of dissolved organic matter (DOM), which absorbs light at specific wavelength. Researchers continue to study how lowering organic micropollutants (OMPs) relates to decreasing UVA_{254} absorbance in water treatment. Both measurements play a key role in evaluating water quality and the success of ozone-based treatment methods.

UVA_{254} absorbance, ozone CT, GAC EBCT and UV reduction during the pilot unit testing period and sampling are shown below:

	Date	Dose, g O_3/m3	CT O_3, min	EBCT GAC, min	UV 254.1	UV 254.2	UV 254.3	UV reduction after O_3 tank, %	UV reduction after GAC filter, %
1.	03.12.2025.	8	25	32	50,9	37,9	27,9	25,5	45,2
2.	11.12.2025.	8	25	25	37,8	34,1	30,4	9,8	19,6
3.	12.12.2025.	10	25	25	37,2	32,1	16,4	13,7	55,9
4.	15.12.2025.	10	25	25	41,1	37,1	21,9	9,7	46,7
5.	16.12.2025.	12	25	25	45,1	28,2	21,4	37,5	52,5
6.	17.12.2025.	12	25	25	59,1	37,3	21	36,9	64,5

	Sensor	Description
1.	UV254.1	*After DRUM filter*
2.	UV254.2	*After ozonation*
3.	UV254.3	*After GAC filter*
4.	WT. 2	*Water tank 4 (UV254.2)*
5.	WT. 4	*Water tank 4 (UV254.3*

Tests were carried out under dry weather flow conditions only, in order to avoid fluctuations in the results. Based on these measurements, the UV_{254} absorbance reduction was calculated.

Increasing ozone dose significantly improved NOM (natural organic matter) removal efficiency, particularly when combined with downstream GAC filtration.

While ozonation alone achieved moderate UV254 reduction (up to 37.5%), the combined O_3/GAC process achieved up to 64.5% reduction. The highest reduction efficiency was observed at an ozone dose of 12 g O_3/m^3, indicating that higher oxidation intensity enhanced subsequent adsorption and/or biodegradation in the GAC filter. Variations in EBCT between 25 and 32 minutes showed less influence than ozone dose.

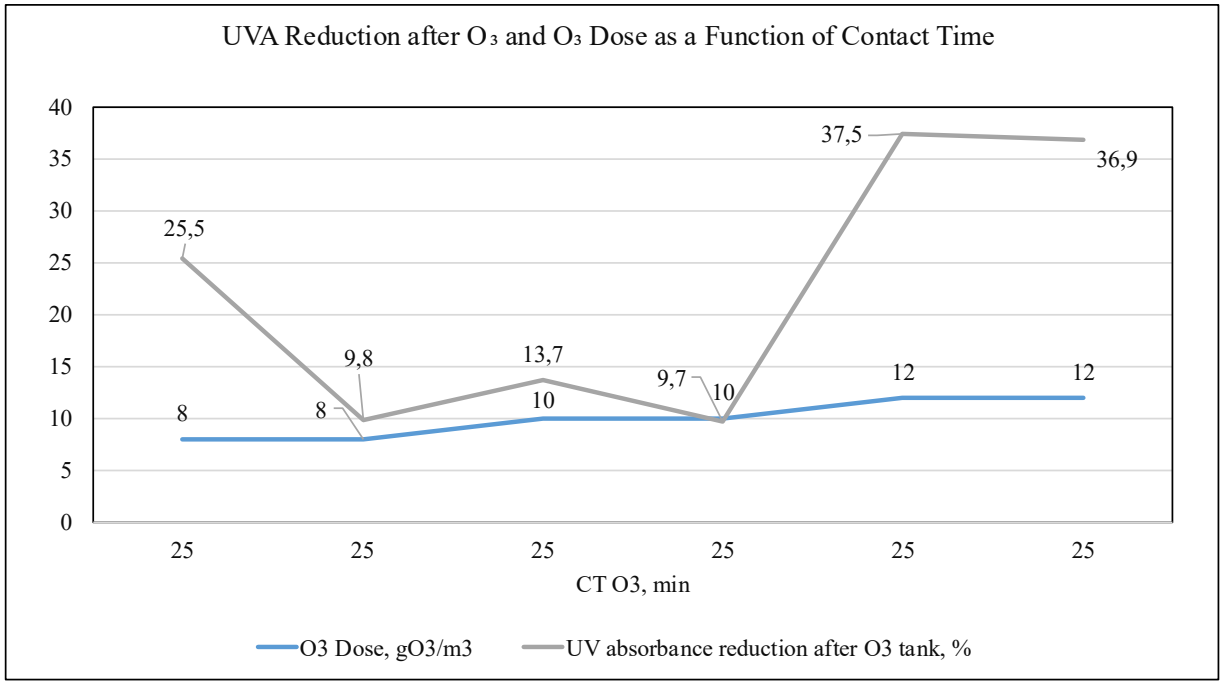

Fig. 5. UV_{254} Absorbance Reduction at varying ozone doses and contact times

3.2. Evaluating the efficiency of organic compounds removal

To evaluate the efficiency of micropollutant removal in the piloting container, 6 series of wastewater samples were collected after cloth filtration, ozonation, and GAC filtration. The ozone concentration was adjusted after every two-sample series. Additionally, samples of the WWTP influent were collected to assess the overall reduction of pharmaceuticals and PFAS across the entire treatment train, including biological treatment.

Piloting tests were conducted using one GAC filter column at a time, although the pilot plant configuration allows testing of two GAC filters in a lead–lag configuration. To ensure stable operation of the ozone generator, the cleaning cycles of the cloth filter were reduced during sampling hours. The backwash cycle of the GAC filter was set to 12 hours. Samples were collected using borosilicate 1 L glass jars (Pharmaceuticals) and HDPE (high-density polyethylene) jars for PFASs.

3.2.1. Removal of pharmaceuticals

This study evaluated the removal efficiency of pharmaceutical micropollutants in a combined wastewater treatment system consisting of biological treatment, ozonation (8–12 g O_3/m^3), and granular activated carbon (GAC) filtration. The behavior of various pharmaceutical compounds was assessed at different treatment stages, while the influence of ozone dose and dissolved organic matter on the overall treatment efficiency was also investigated. The dissolved organic carbon (DOC) concentration in the treated wastewater was 14.2 ± 2.6 mg C/L, corresponding to a specific ozone dose ranging from approximately 0.56 to 0.85 mg O_3/mg C.

The detected pharmaceutical concentrations in wastewater varied considerably, ranging from tens to several thousand ng/L. The highest concentrations were observed for Amisulpride (up to 8280 ng/L), as well as Diclofenac, Clarithromycin, Metoprolol, and Carbamazepine. Such contamination profiles are consistent with previous studies describing municipal wastewater matrices dominated by central nervous system drugs, antibiotics, and cardiovascular pharmaceuticals (Ternes, 1998; Verlicchi et al., 2012).

The biological treatment stage alone achieved only partial and highly variable pharmaceutical removal. Comparison between untreated wastewater and biologically treated wastewater showed minimal reduction for several compounds. For example, Amisulpride concentrations decreased only slightly from 8280 to 8090 ng/L (~2%), while Clarithromycin remained essentially unchanged (770 → 770 ng/L). In the case of Diclofenac, concentrations even increased after biological treatment (1140 → 1510 ng/L), which may be related to

deconjugation of metabolites or matrix-related analytical effects. Overall, the biological process alone was insufficient for reliable micropollutant removal, which agrees with findings from conventional wastewater treatment studies reporting limited pharmaceutical degradation during activated sludge treatment (Ternes, 1998; Onesios et al., 2009).

A substantial increase in treatment efficiency was observed after ozonation combined with GAC filtration. At the lowest ozone dose (8 g O_3/m^3, corresponding to ~0.56 mg O_3/mg C), the average pharmaceutical removal after O_3 + GAC treatment was approximately 65% when considering only pharmaceutical compounds. In this regime, some compounds showed very high removal efficiencies, including Diclofenac (~98%) and Carbamazepine (~98%), while others such as Candesartan exhibited considerably lower removal. These results suggest that at lower ozone doses, competition between pharmaceuticals and dissolved organic matter strongly affects oxidation efficiency. Similar observations have been reported by Hollender et al. (2009), who demonstrated that ozone consumption by background organic matter significantly reduces micropollutant oxidation selectivity.

Increasing the ozone dose to 10 g O_3/m^3 (~0.70 mg O_3/mg C) improved the average pharmaceutical removal to approximately 66%. Under these conditions, compounds such as Clarithromycin (~85%), Telmisartan (~94%), and Metoprolol (~72%) showed significantly improved degradation. However, certain compounds, including Candesartan, remained relatively persistent even at elevated ozone doses, indicating compound-specific oxidation resistance.

The highest treatment performance was achieved at 12 g O_3/m^3 (~0.85 mg O_3/mg C). At this ozone dose, several compounds were either below the analytical detection limit or no longer detectable after ozonation and GAC filtration, indicating nearly complete removal. Recalculated overall pharmaceutical removal reached approximately 94–97%. Particularly high removal efficiencies were observed for Amisulpride (>99%), Clarithromycin (~97%), and Telmisartan (~96%). These findings are consistent with previous reports showing that ozone doses above ~0.8 mg O_3/mg DOC are often sufficient to achieve extensive micropollutant oxidation in secondary effluents (Bourgin et al., 2018).

UV254 reduction further confirmed extensive oxidation of dissolved organic matter. At the highest ozone doses, UV254 reduction after ozonation reached approximately 37%, while after GAC filtration it increased up to 64.5%. These results demonstrate the strong synergy between ozonation and GAC filtration: ozone oxidizes dissolved organic matter and transforms pharmaceutical compounds, whereas GAC adsorbs residual micropollutants and oxidation by-products. Similar synergistic effects between ozonation and activated carbon treatment have been widely reported in advanced wastewater treatment studies (Altmann et al., 2014; Mailler et al., 2016).

Nevertheless, some findings differed from literature expectations. For example, Carbamazepine is commonly described as ozone-reactive and frequently used as an indicator compound for ozonation efficiency (Huber et al., 2005). In the present study, however, its removal behaviour was variable, suggesting possible matrix-related interferences, transient transformation products, or adsorption/desorption dynamics within the GAC stage. Similarly, the unstable behaviour of Diclofenac may indicate formation of transformation products or analytical fluctuations associated with complex wastewater matrices.

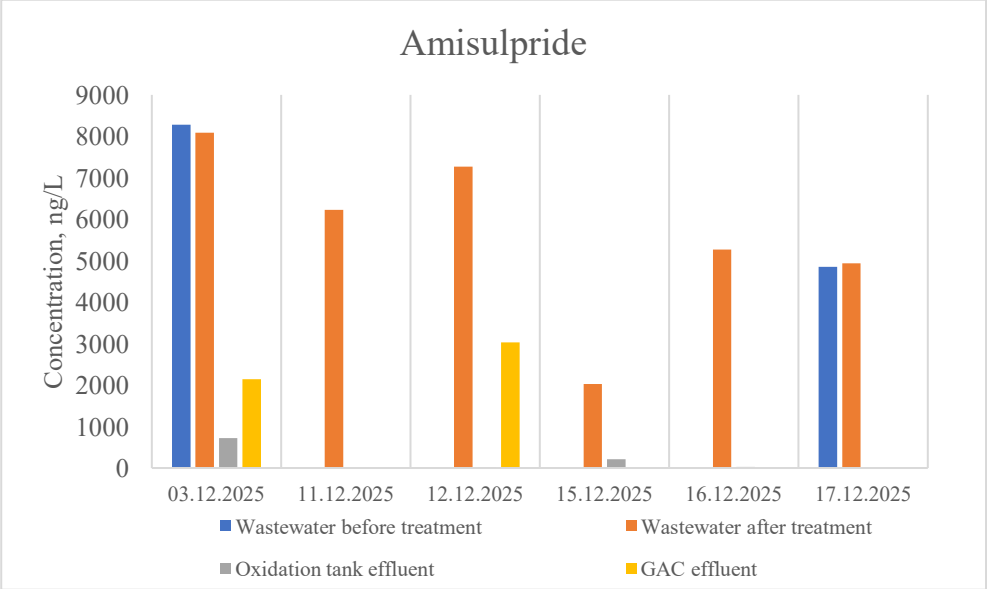

Fig. 6 Changes in amisulpride levels at different stages of wastewater treatment

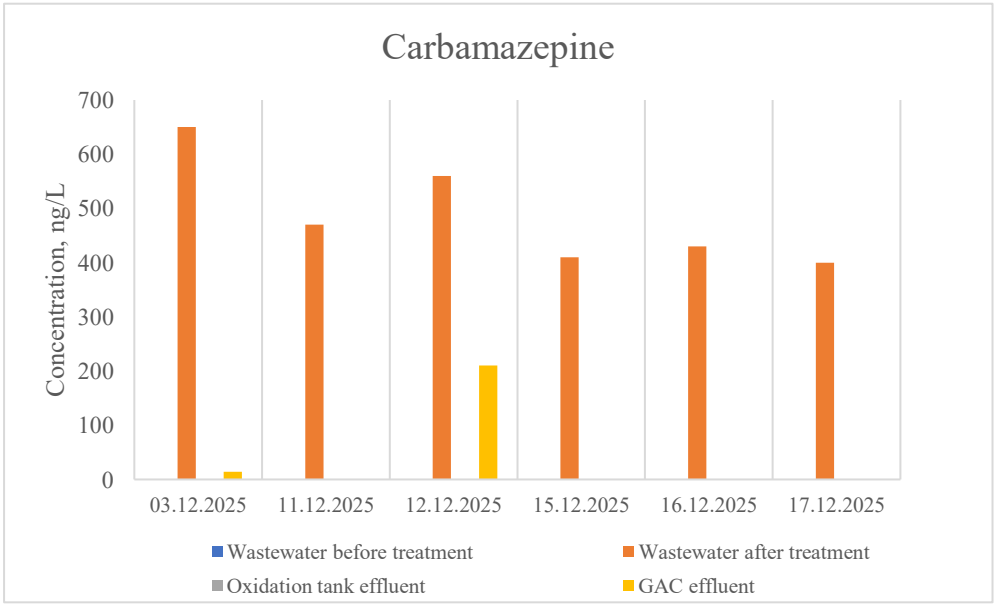

Fig.7 Changes in carbamazepine levels at different stages of wastewater treatment

Overall, the results clearly demonstrate that biological treatment alone provides insufficient pharmaceutical removal, whereas the combined O_3 + GAC process substantially enhances treatment performance. The system efficiency strongly depended on the O_3/DOC ratio, with optimal performance achieved at approximately 0.85 mg O_3/mg C. Under these conditions, the combined advanced treatment system enabled nearly complete removal of most pharmaceutical micropollutants from biologically treated wastewater.

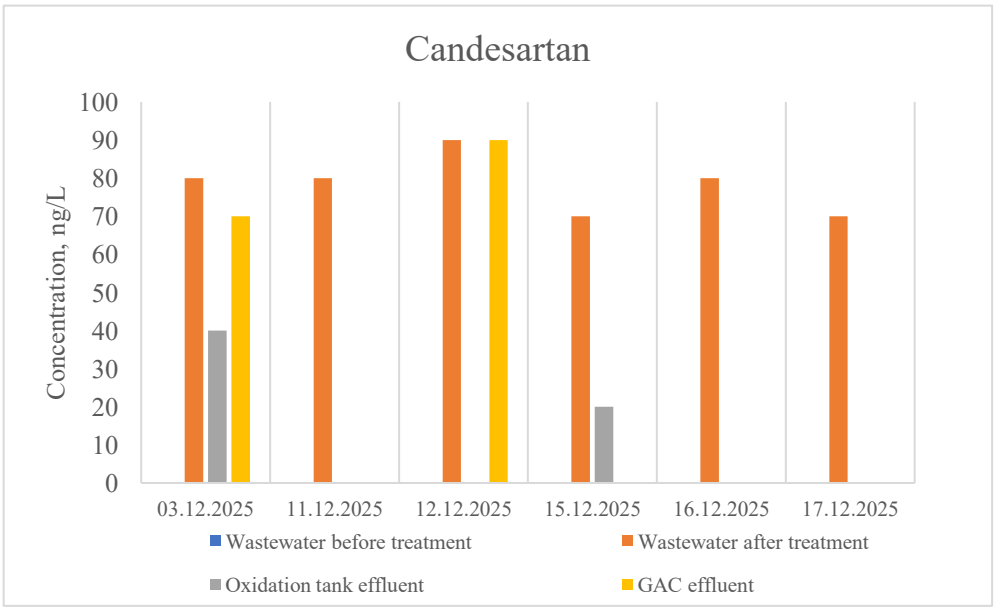

Fig. 8 Changes in candesartan levels at different stages of wastewater treatment

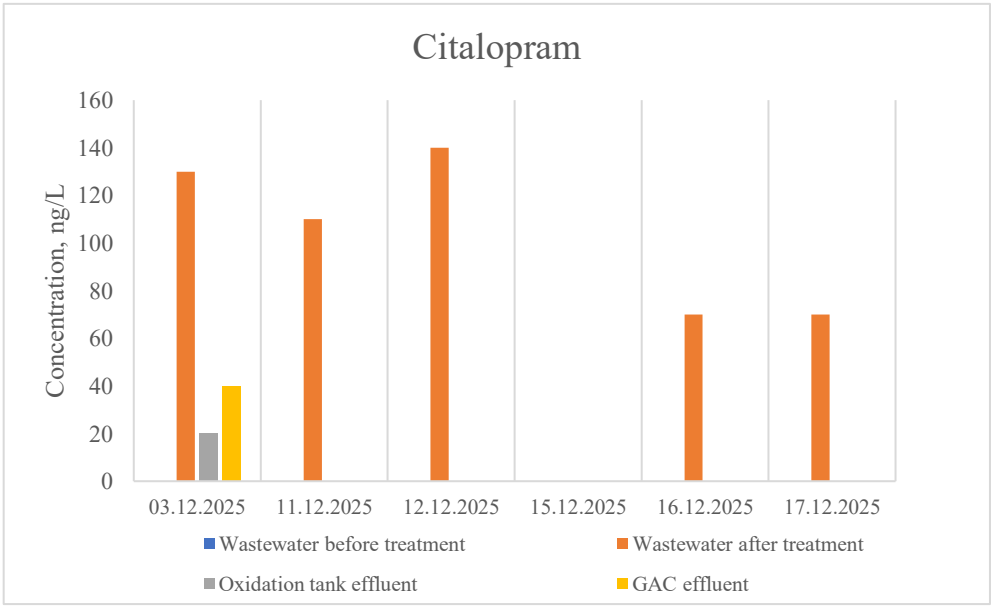

Fig. 9 Changes in citalopram levels at different stages of wastewater treatment

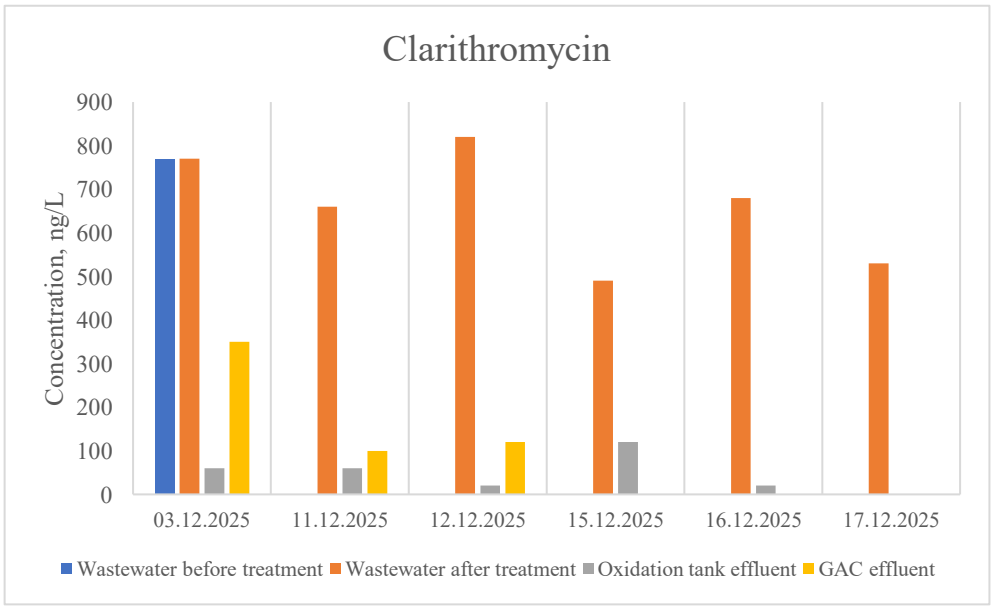

Fig. 10 Changes in clarithromycin levels at different stages of wastewater treatment

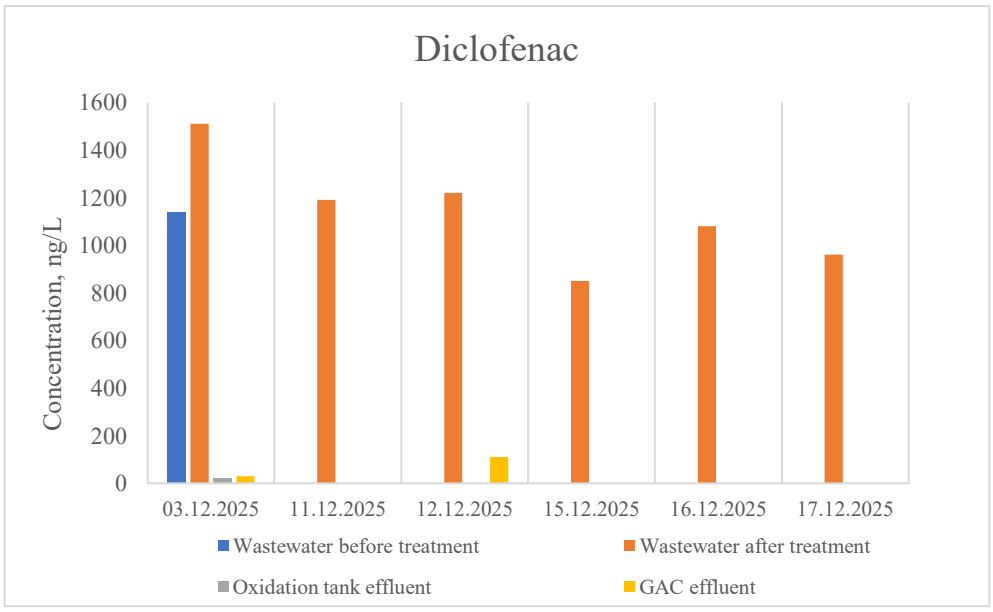

Fig. 11 Changes in diclofenac levels at different stages of wastewater treatment

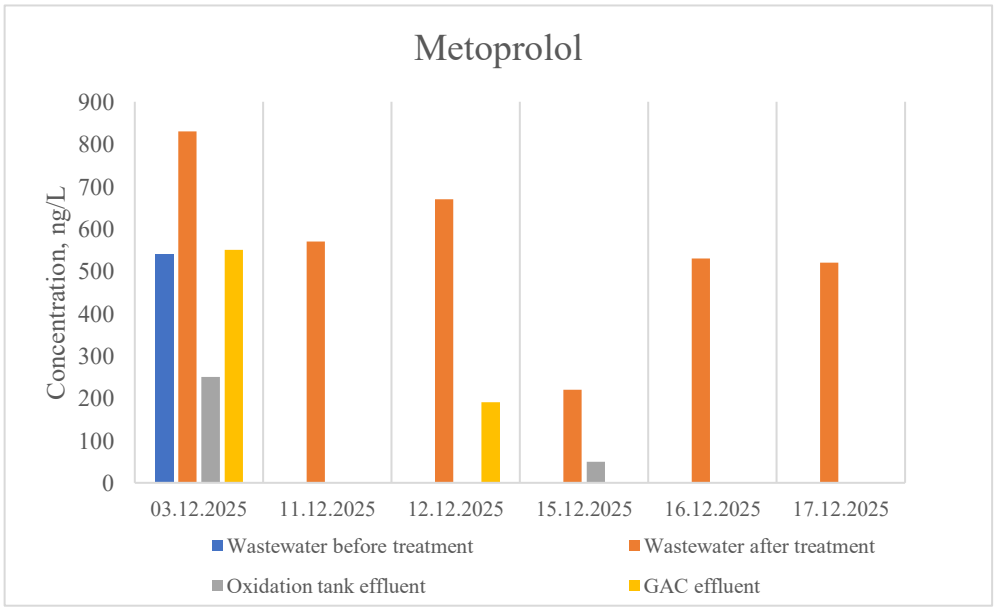

Fig. 12 Changes in metoprolol levels at different stages of wastewater treatment

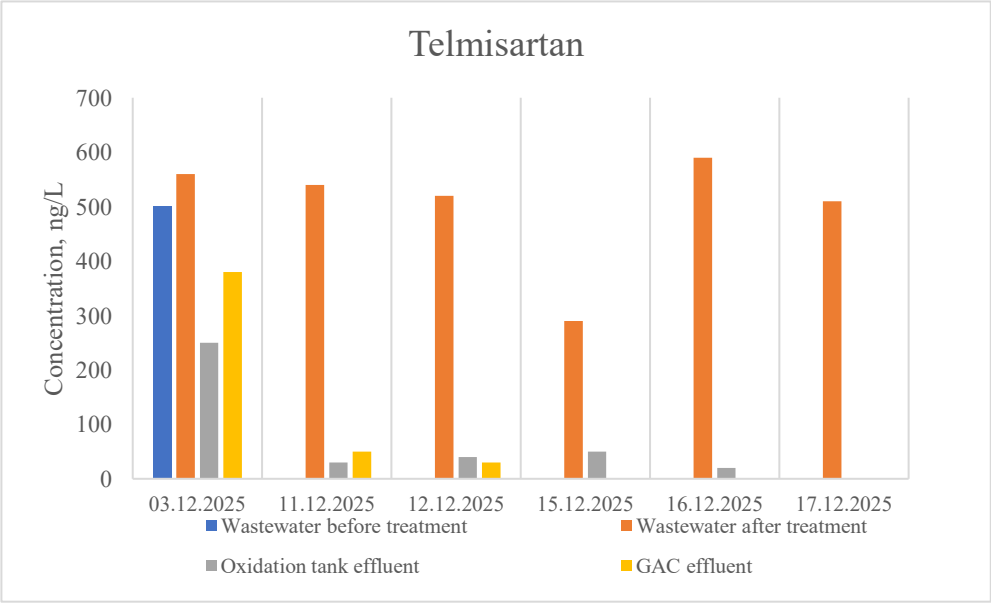

Fig. 13 Changes in telmisartan levels at different stages of wastewater treatment

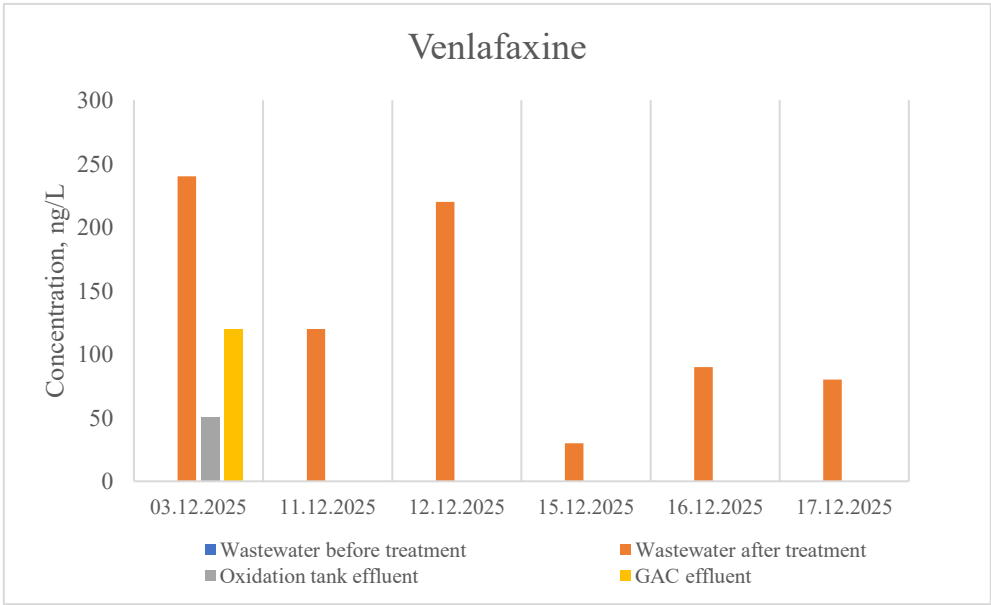

Fig. 14 Changes in venlafaxine levels at different stages of wastewater treatment

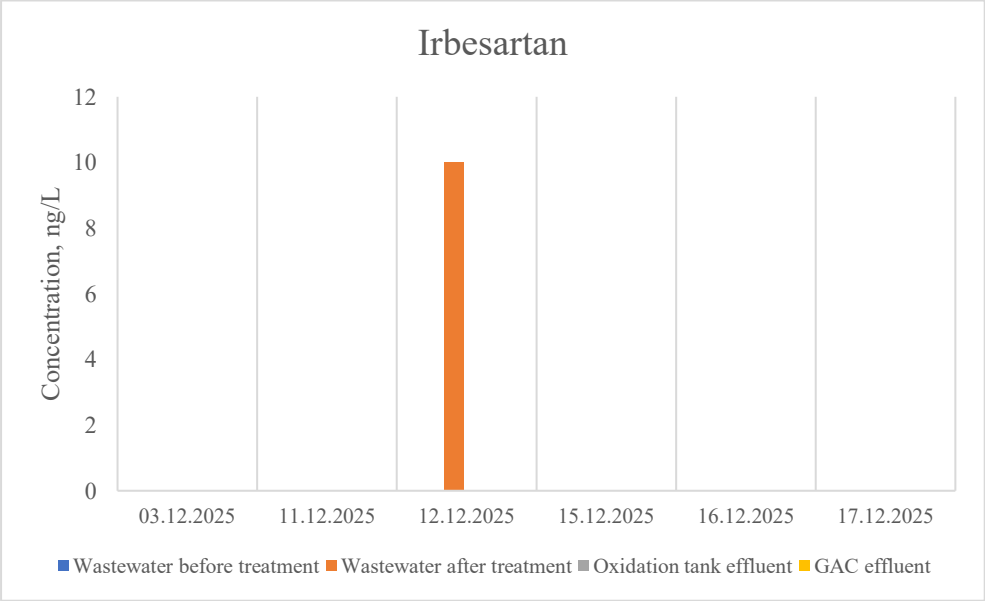

Fig. 15 Changes in irbesartan levels at different stages of wastewater treatment

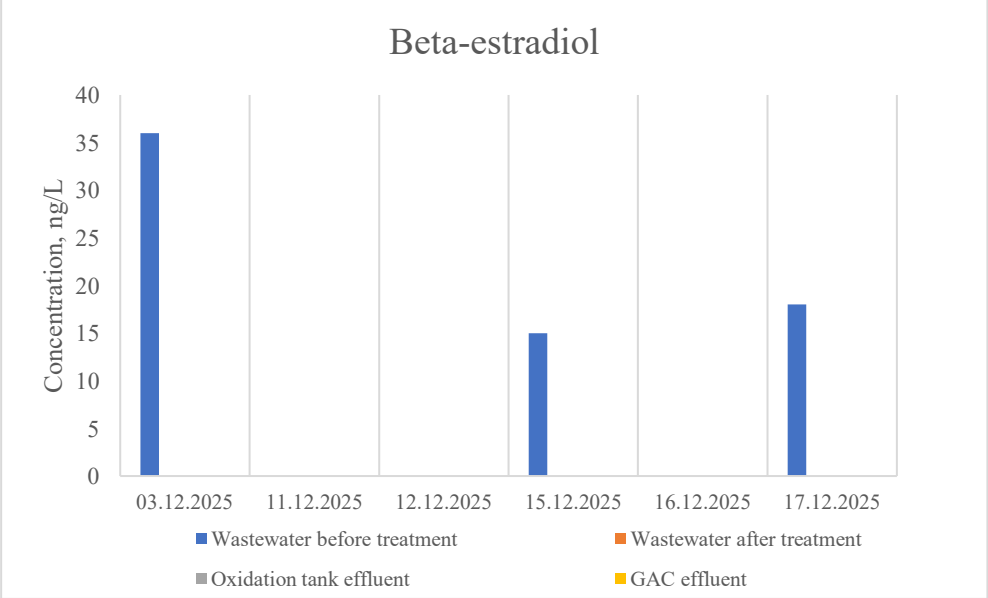

Fig. 16 Changes in β-estradiol levels at different stages of wastewater treatment

The observed disappearance of 17β-estradiol (E2) after biological wastewater treatment can be explained by several well-documented mechanisms reported in peer-reviewed literature.

3.2.2. 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 (4MBT 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.

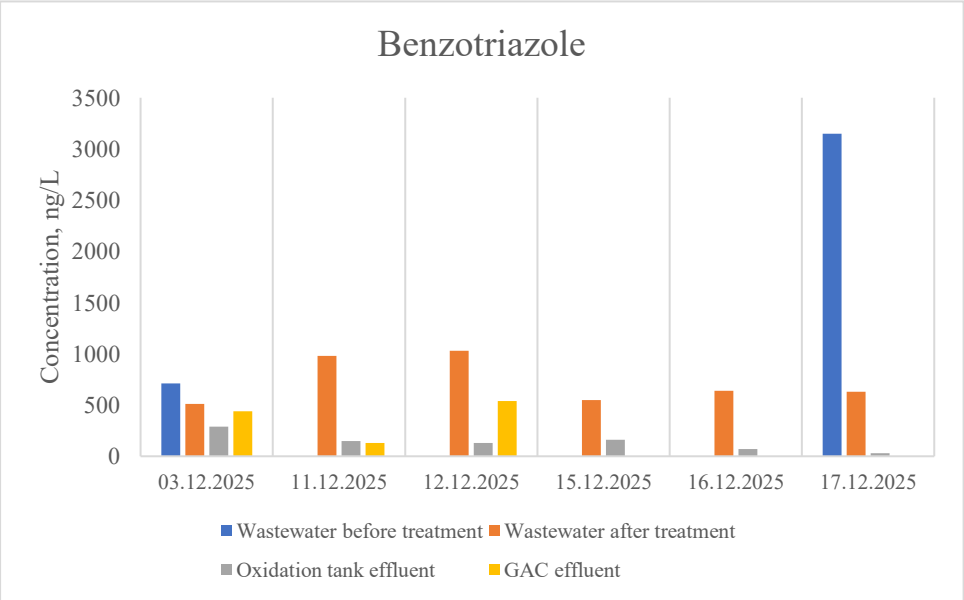

Fig. 17 Changes in Benzotriazole levels at different stages of wastewater treatment

The behaviour of Benzotriazole in the investigated wastewater treatment system showed considerable variability across treatment stages and ozone doses. Benzotriazole is not a pharmaceutical compound, but an industrial corrosion inhibitor widely used in dishwasher detergents, cooling systems, aircraft de-icing fluids, and metal protection applications. Due to its high polarity and persistence, benzotriazole is frequently detected in municipal wastewater and is considered relatively resistant to conventional biological degradation.

The measured benzotriazole concentrations ranged from 510 to 3150 ng/L in biologically treated wastewater. The highest influent concentration was observed on 17.12.2025 (3150 ng/L before treatment), indicating substantial fluctuations in source loading. Such variability is commonly reported in municipal wastewater due to industrial discharges and seasonal use patterns.

Comparison between untreated and biologically treated wastewater demonstrated only limited removal of benzotriazole. On 03.12.2025, the concentration decreased from 710 to 510 ng/L, corresponding to approximately 28% removal. However, on 17.12.2025, the concentration remained relatively high even after biological treatment (3150 → 630 ng/L), suggesting incomplete biodegradation and strong persistence within the activated sludge process.

These findings agree with previous studies describing benzotriazole as biologically recalcitrant under conventional wastewater treatment conditions. Literature reports typical biological removal efficiencies ranging from 0–40%, depending on sludge retention time and redox conditions.

A substantial improvement in removal efficiency was observed after ozonation.

At lower ozone doses (8 g O_3/m^3), benzotriazole concentrations decreased from:

- 510 → 290 ng/L (43% removal)
- 980 → 150 ng/L (85% removal)

At 10 g O_3/m^3:

- 1030 → 130 ng/L (87% removal)

At the highest ozone dose (12 gO_3/m^3):

- 640 → 70 ng/L (89% removal)
- 630 → 30 ng/L (95% removal)

These results clearly demonstrate that benzotriazole oxidation efficiency increased with increasing ozone dose and O_3/DOC ratio. At approximately 0.85 mg O_3/mg C, nearly complete oxidation was achieved.

The observed trend is consistent with literature describing benzotriazole as moderately ozone-reactive. Although the compound is relatively stable during biological treatment, it can be effectively degraded during advanced oxidation processes due to electrophilic ozone attack on the aromatic triazole structure.

GAC filtration performance

The effect of GAC filtration on benzotriazole removal was more variable. At lower ozone doses:

- 510 → 440 ng/L (14% total removal after O_3 + GAC)

- 980 → 130 ng/L (87% total removal)
- 1030 → 540 ng/L (48% total removal)

These results suggest that GAC performance depended strongly on the preceding ozonation efficiency and possibly on competition with dissolved organic matter. In some cases, GAC substantially improved overall removal, while in other cases adsorption appeared limited, likely due to saturation effects or competition from natural organic matter.

The discrepancy between oxidation tank effluent and GAC effluent concentrations may also indicate:

- desorption phenomena,
- analytical variability,
- transformation product formation,
- or heterogeneous adsorption behaviour within the activated carbon bed.

Results indicate that benzotriazole was relatively resistant to biological treatment but highly susceptible to advanced oxidation at elevated ozone doses. The most effective removal was achieved at 12 g O_3/m^3 (~0.85 mg O_3/mg C), where concentrations decreased to near-detection-limit levels.

The study further demonstrates that ozonation played the dominant role in benzotriazole removal, whereas the contribution of GAC filtration was secondary and more variable. This behaviour differs from many hydrophobic pharmaceutical compounds that are more effectively removed by adsorption processes.

The findings confirm that advanced oxidation processes are necessary for efficient benzotriazole removal from municipal wastewater and support previous research showing that ozone-based tertiary treatment can significantly reduce persistent industrial micropollutants in treated effluents.

Published studies consistently show that benzotriazole is a persistent and poorly biodegradable micropollutant, with conventional activated sludge systems typically achieving only 0–45% removal (Herzog et al., 2014; Ternes, 1998; Verlicchi et al., 2012). This matches widely reported observations that wastewater treatment plants are a continuous emission source for this compound.

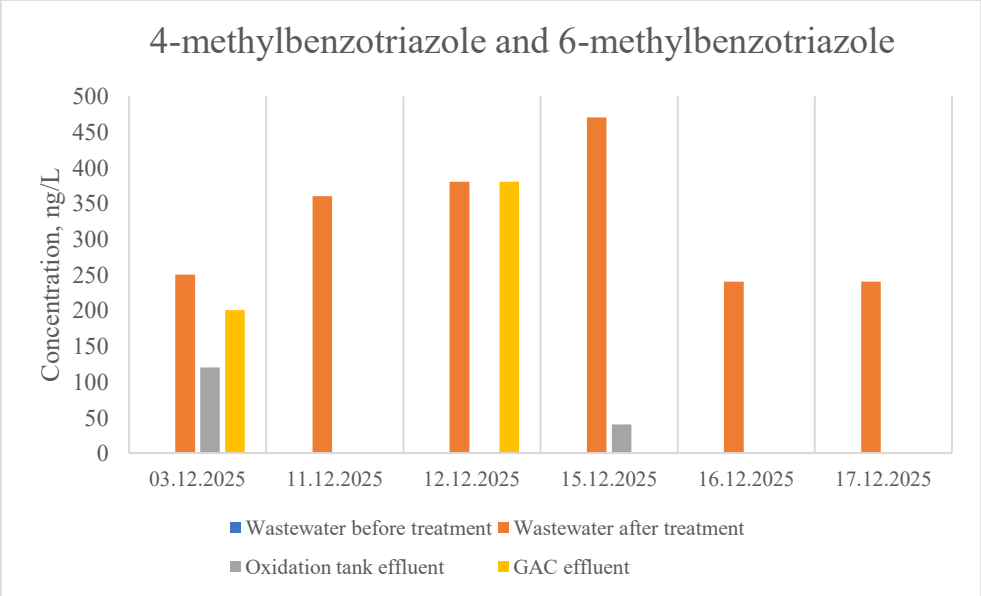

Fig. 18 Changes in 4-MBT and 6-MBT levels at different stages of wastewater treatment

The methylated benzotriazole derivatives 4-methylbenzotriazole and 6-methylbenzotriazole were consistently detected in the wastewater system at concentrations ranging from 240 to 470 ng/L in biologically treated effluent, indicating continuous presence and moderate temporal variability of these compounds in the influent matrix. The highest concentration was observed on 15.12.2025 (470 ng/L), while lower values (~240 ng/L) occurred on 16–17.12.2025, suggesting fluctuating loading rather than stable emission patterns.

Biological treatment performance:

The biological treatment stage showed low and inconsistent removal efficiency. Where both influent and effluent data are available, the calculated biological removal varied significantly. For example, on 03.12.2025 concentrations decreased from 250 ng/L to 120 ng/L, corresponding to approximately 52% removal under

favourable conditions. However, this level of removal was not stable across all sampling points, and in several cases no clear reduction could be confirmed, indicating strong variability in biodegradation performance.

Overall, biological treatment performance for these compounds can be summarized as ~0–52% removal, with an average behaviour closer to the lower end of this range when considering all sampling dates. This confirms that methylated benzotriazoles are poorly and inconsistently biodegradable, consistent with their aromatic stability and resistance to microbial transformation.

Ozonation performance:

A clear improvement in removal efficiency was observed after ozonation, with a strong dependence on ozone dose. At 8 g O_3/m^3, removal was limited (e.g., 250 → 200 ng/L, ~20%), while intermediate conditions showed inconsistent behaviour. At higher ozone intensity (12 g O_3/m^3), removal became highly effective, with concentrations decreasing from 470 to 40 ng/L (~91% removal).

This demonstrates a strong dose–response relationship, where sufficient ozone exposure is required to overcome competition from dissolved organic matter and achieve effective oxidation of these compounds.

GAC filtration performance:

Granular activated carbon (GAC) showed variable and system-dependent performance. In some cases, GAC contributed to additional removal after ozonation, while in others no significant improvement was observed or even fluctuations occurred. This suggests that adsorption capacity was influenced by:

- competition with natural organic matter,
- variability in influent concentration,
- and the degree of prior ozonation.

Thus, GAC functioned mainly as a polishing step rather than a primary removal mechanism, with inconsistent efficiency depending on operational conditions.

The most removal was achieved at the highest ozone dose (12 g O_3/m^3), confirming that advanced oxidation is the key mechanism controlling the fate of these compounds in wastewater treatment systems. Biological processes alone are insufficient, while combined ozonation and GAC treatment provides reliable removal only under sufficiently high ozone exposure conditions.

3.2.3. Removal of PFAS

Per- and polyfluoroalkyl substances (PFAS) are man-made chemicals with very strong carbon–fluorine bonds, giving them high thermal stability and resistance to degradation. Because they are extremely persistent—often called “forever chemicals”—they are widely used in products such as firefighting foams, non-stick coatings, waterproof textiles, food packaging, and electrical insulation. Consequently, PFAS are continuously discharged into wastewater from domestic, commercial, and industrial sources.

Conventional wastewater treatment processes remove PFAS poorly. Most compounds pass through primary and secondary biological treatment, with only limited accumulation in sewage sludge, which creates additional disposal challenges. Their persistence, potential for bioaccumulation, and possible health impacts (including endocrine disruption and carcinogenicity) have led to increasing regulatory concern.

As a result, regulations are becoming stricter. The revised EU Urban Wastewater Treatment Directive (UWWTD) now requires large treatment plants (>100,000 population equivalent) to monitor PFAS in influent and effluent, improving environmental control and risk management.

PFAS removal therefore relies on advanced technologies. Adsorption methods such as granular activated carbon (GAC) and ion exchange resins (IXR) are widely used; GAC is more effective for long-chain PFAS, while IXR often performs better overall, including for short-chain compounds.

Membrane processes like nanofiltration and reverse osmosis achieve high removal efficiency but generate concentrated waste streams requiring further handling. Emerging destructive methods—such as electrochemical

oxidation, plasma treatment, and sonochemical processes—show promise but are still mostly at laboratory or pilot scale.

3.2.3.1. 6:2 FTS

In the influent wastewater (before biological treatment), 6:2 FTS concentrations showed an increasing trend over time. This indicates a rising load of 6:2 FTS entering the system, likely driven by fluctuating upstream emissions or variable hydraulic conditions.

In the oxidation tank effluent, concentrations fluctuated considerably over the monitoring period. Results show that the oxidation step did not consistently reduce 6:2 FTS concentrations. In several cases, concentrations in the oxidation effluent exceeded those in the influent, suggesting possible formation pathways such as precursor transformation into 6:2 FTS during ozonation, incomplete oxidation, or matrix-related effects reducing treatment efficiency.

Despite increased oxidation intensity, no consistent improvement in 6:2 FTS removal was observed.

This indicates that higher ozone doses did not enhance degradation of 6:2 FTS.

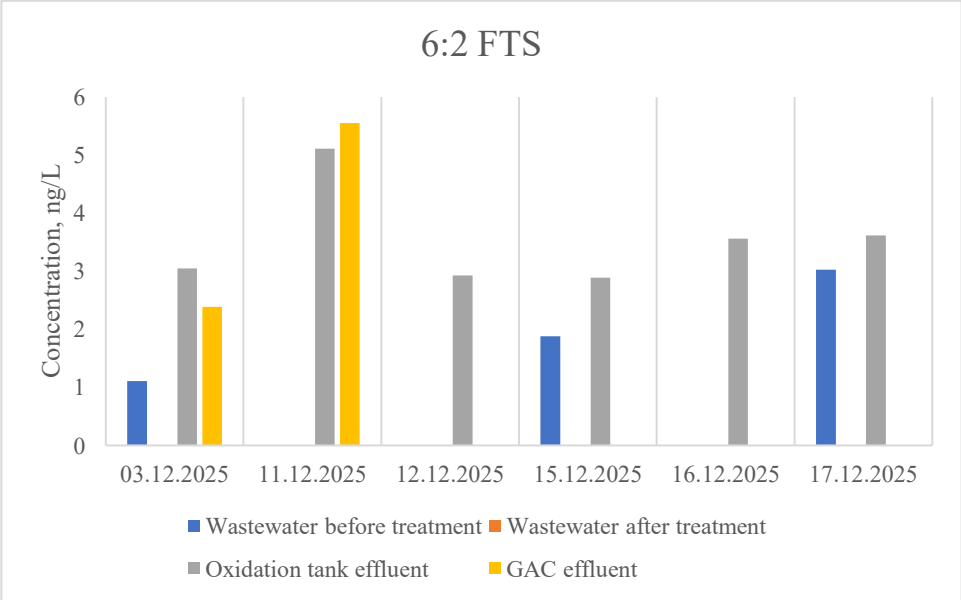

Fig.19 6:2 FTS Behaviour during the sampling period

3.2.3.2. 8:2 FTS

In the influent wastewater (before biological treatment), 8:2 FTS was detected at low levels, with a concentration of 1.12 ng/l on 03.12.2025 and 1.71 ng/l on 15.12.2025, indicating a slight increase in incoming load over time.

After biological treatment, 8:2 FTS concentrations were below the detection limit, which limits direct evaluation of removal performance in piloting stages. However, based on system behaviour and comparison with structurally related fluorotelomer compounds, overall transformation and retention processes can still be inferred.

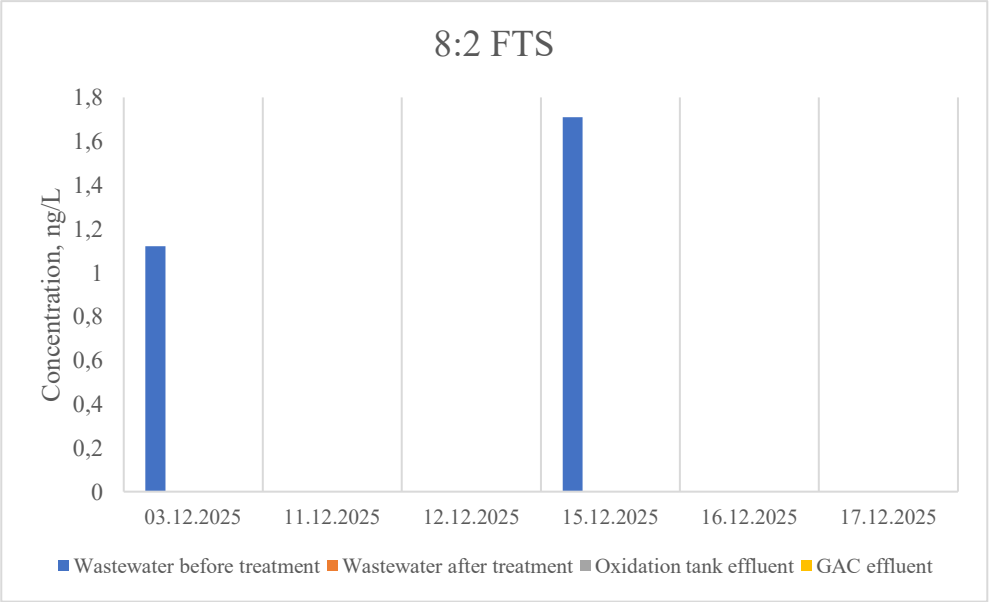

Fig.20 8:2 FTS Behaviour during the sampling period

3.2.3.3. PFBA

The behaviour of PFBA (perfluorobutanoic acid) across the treatment system indicates a compound that is largely removed during biological treatment and shows limited persistence in downstream advanced treatment stages.

In the influent wastewater (before biological treatment), PFBA was detected at 7.93 ng/l on 03.12.2025 and 2.97 ng/l on 17.12.2025, showing a decreasing trend in the incoming load over time. This suggests variability in upstream emissions or reduced input of PFBA into the system during the monitoring period.

A key observation is that PFBA was only detected in the influent stage, while after biological treatment it was below the detection limit throughout the study period. This indicates that the biological treatment step was highly effective for PFBA removal or transformation to concentrations that are no longer quantifiable with the applied analytical method.

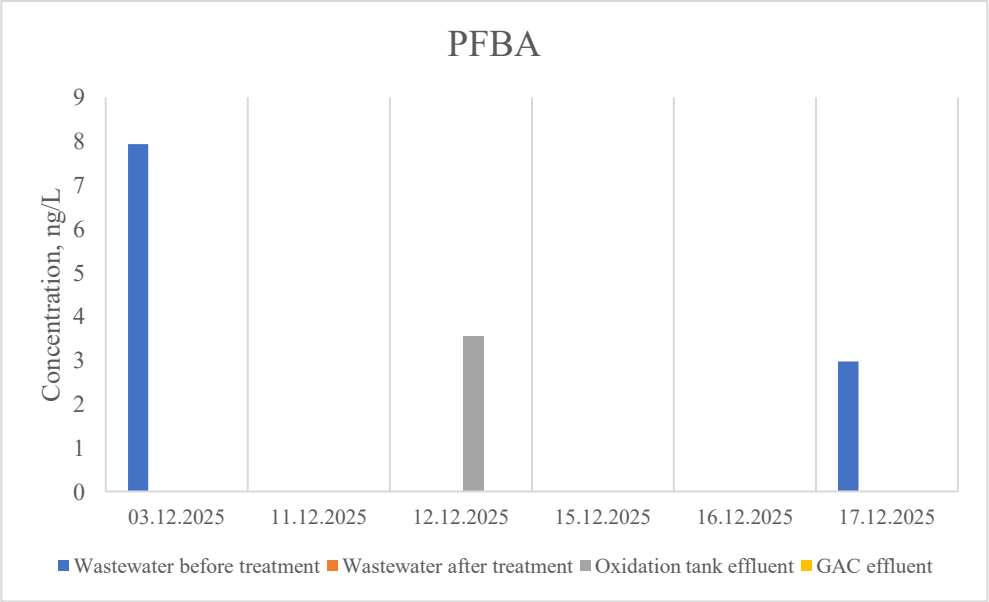

Fig.21 8:2 PFBA Behaviour during the sampling period

3.2.3.4. FBSA

The behaviour of FBSA (fluorobutane sulfonamide) across the treatment shows a more complex distribution pattern compared to other monitored PFAS, with partial removal in biological treatment, reappearance in later stages, and indications of limited but variable persistence through the process. In the influent wastewater (before treatment), FBSA was not consistently detected, suggesting either low and intermittent upstream input or

concentrations near the analytical detection limits. In the biological treatment stage (wastewater after treatment), FBSA was clearly detected on multiple dates, with different concentrations, ranging from 1,00 to 2,25 ng/l This indicates that FBSA is present in the system and partially persists through biological treatment, although a decreasing trend is observed over time, suggesting some level of removal or transformation within this stage. In the oxidation tank effluent, FBSA was detected on twice: 1.21 ng/l and 1.08 ng/l. These values are lower than or comparable to post-biological concentrations, indicating that oxidation does not significantly increase or degrade FBSA. Instead, it suggests relatively stable behaviour through this process step, with no strong evidence of formation or complete degradation.

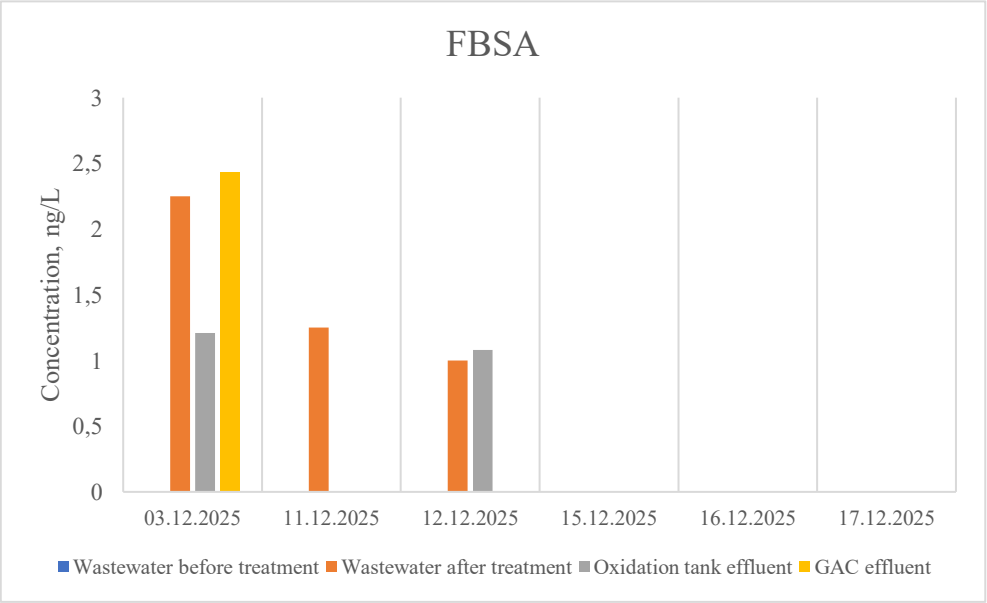

Fig.22 FBSA Behaviour during the sampling period

3.2.3.5. PFBS

The behaviour of PFBS (perfluorobutane sulfonic acid) across the removal showed a consistently high presence throughout all treatment stages, with limited and variable removal performance and indications of persistence and partial transformation under changing operational conditions. These results show that PFBS is not effectively removed by biological treatment, and in some cases concentrations in the effluent are even higher than in the influent. This suggests possible desorption from sludge, transformation of precursor compounds, or accumulation effects within the biological system.

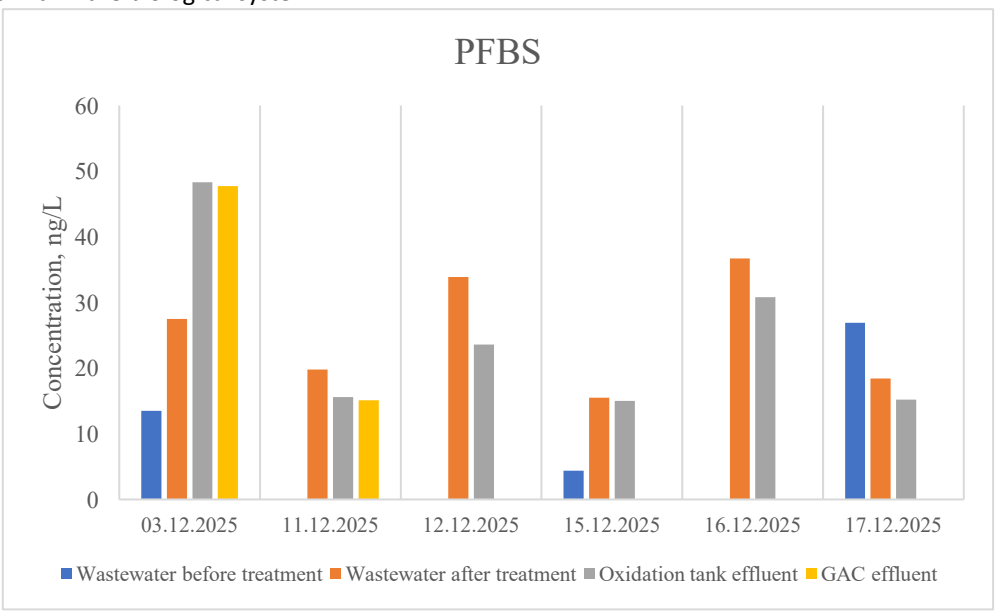

Fig.23 PFBS Behaviour during the sampling period

3.2.3.6. PFHqA

Biological treatment does not effectively remove PFHpA. The elevated concentrations compared to influent may indicate release from sludge phases, transformation of precursor compounds, or redistribution processes within the biological reactor.

These results indicate that oxidation does not significantly degrade PFHpA. The relatively stable concentrations suggest that PFHpA is resistant to ozonation under the applied conditions (8–12 gO_3/m^3), with only minor fluctuations likely linked to upstream variability rather than treatment-driven removal. This indicates limited adsorption onto activated carbon. The similarity between oxidation and GAC effluent concentrations suggests that GAC provides little additional removal for PFHpA under the applied EBCT conditions.

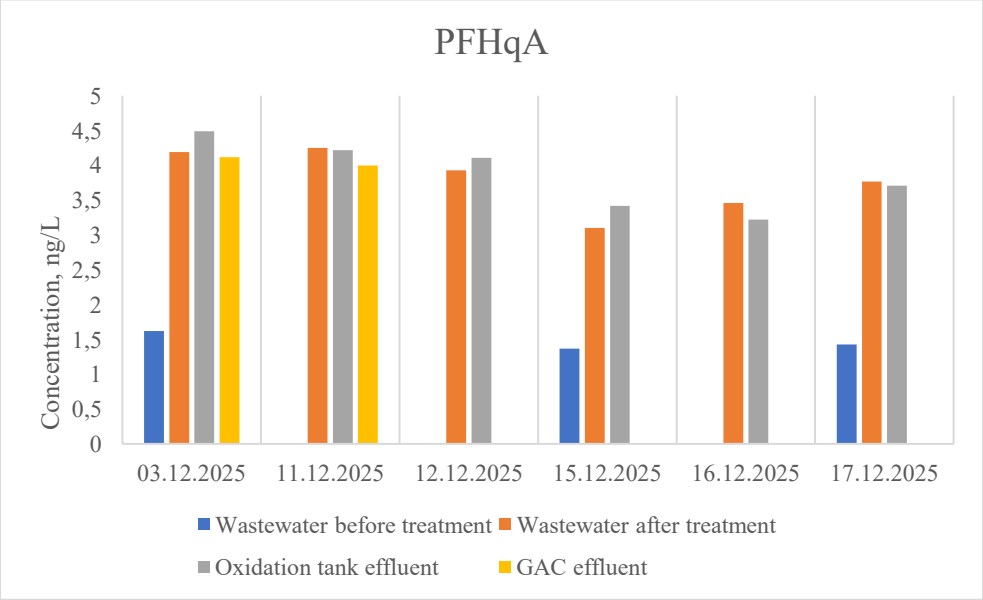

Fig.24 PFHqA Behaviour during the sampling period

3.2.3.7. PFHxA

The behaviour of PFHxA (perfluorohexanoic acid) across the treatment demonstrated consistent presence in all monitored stages.PFHxA was only sporadically reported, with a single detected value of: 1,32 ng/l. After biological treatment, PFHxA was consistently detected across all monitoring dates. These results indicate that PFHxA is not effectively removed during biological treatment. Concentrations are consistently higher than in the influent (where available), suggesting possible release from sludge phases, transformation of precursor compounds, or redistribution within the biological reactor. In the oxidation tank effluent, PFHxA remained relatively stable.

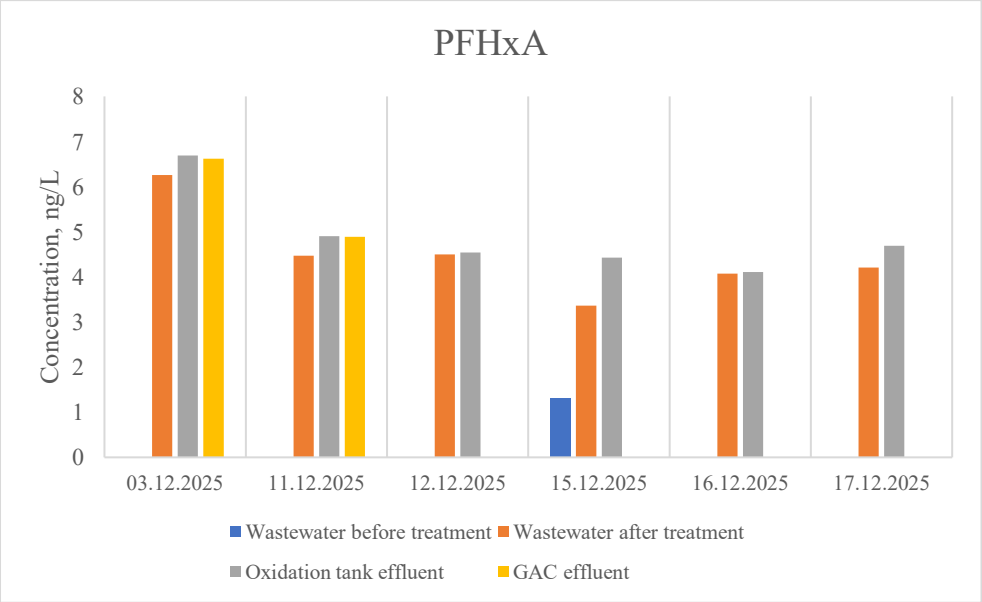

Fig.25 PFHxA Behaviour during the sampling period

3.2.3.8. PFNA

PFNA shows a consistent presence across all monitoring dates and a clear pattern of partial removal through the treatment stages.

In WWTP influent PFNA was detected at relatively low concentrations in the raw wastewater, ranging from approximately 1.01–1.13 ng/L, depending on the sampling date.

After biological treatment, PFNA concentrations increased to ~2.08–2.57 ng/L, which is consistently higher than the influent and indicates that PFNA is not removed in biological treatment and may even be released from transformation processes or sludge-associated desorption. After GAC treatment, PFNA concentrations drop further to around ~1.66–2.00 ng/L, showing additional but limited removal

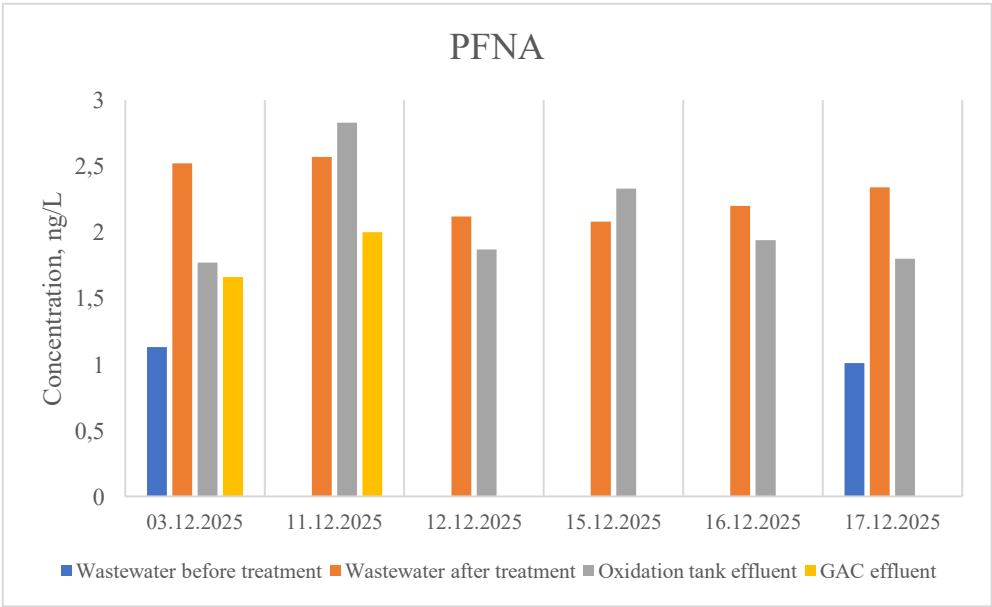

Fig.25 PFNA Behaviour during the sampling period

3.2.3.9. PFOA

PFOA was presented in all sampling series, with concentrations ranging from approximately 1.26–1.53 ng/L. This indicates a stable low-level input into WWTP. After biological treatment, PFOA concentrations increase to ~1.98–3.39 µg/L, which is consistently higher than in the influent. After ozonation, PFOA shows variable behavior. In several cases, concentrations are equal to or higher than post-biological levels. GAC provided the most consistent reduction step, but not complete elimination.

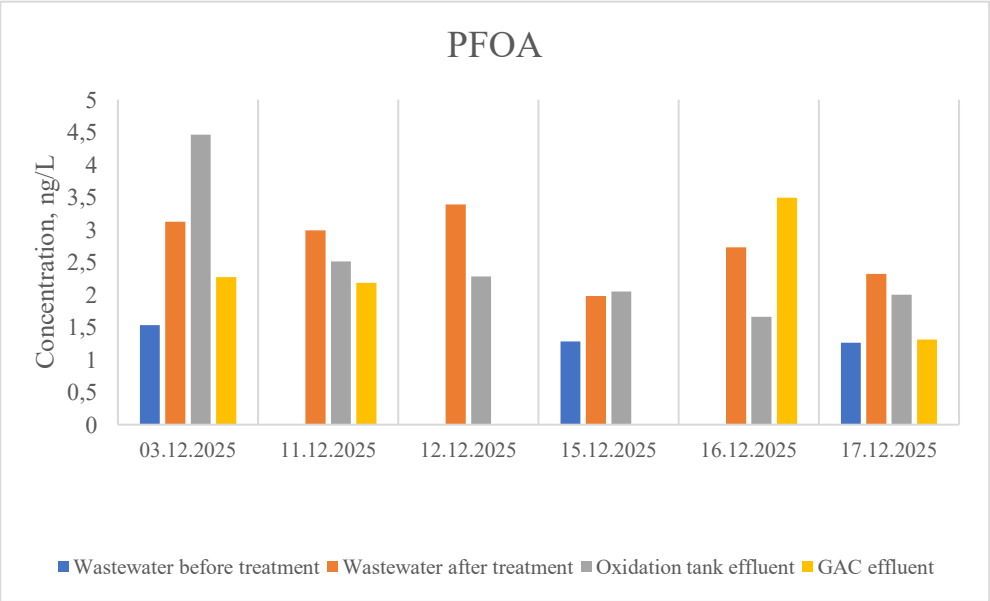

Fig.26 PFOA Behaviour during the sampling period

3.2.3.10. PFPeA

PFPeA is a short-chain PFAS that is generally more mobile and less strongly sorbed than longer-chain compounds. PFPeA is mostly not detected or below reporting levels in the influent during most sampling dates. PFPeA appeared more clearly after biological treatment in some samples (e.g., ~1.44 ng/L on 12.12.2025), even when not present in the influent. After ozonation, PFPeA was detected at slightly higher or comparable levels (~1.82–2.23 ng/L. GAC showed imited adsorption. PFPeA is poorly retained by GAC compared to long-chain PFAS.

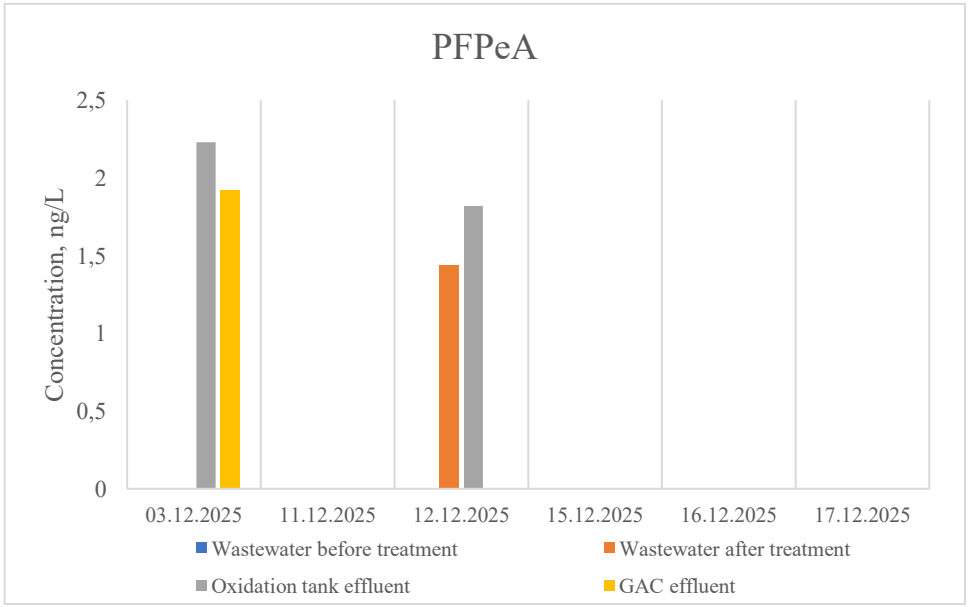

Fig.27 PFPeA Behaviour during the sampling period

CONCLUSIONS

PHARMACY

The present study demonstrated that conventional biological wastewater treatment alone was insufficient for effective removal of pharmaceutical micropollutants. Most compounds showed low or highly variable removal efficiencies during the biological treatment stage, while some substances, such as Diclofenac, even exhibited increased concentrations after treatment, likely due to metabolite deconjugation or matrix-related effects. These findings confirm that conventional activated sludge systems are not capable of consistently eliminating pharmaceutical residues from municipal wastewater.

The implementation of ozonation combined with granular activated carbon (GAC) filtration substantially improved pharmaceutical removal efficiency. The treatment performance strongly depended on the applied ozone dose and the corresponding O_3/DOC ratio. At 8 g O_3/m^3 (~0.56 mg O_3/mg C), the average pharmaceutical removal after O_3 + GAC treatment reached approximately 65%, whereas increasing the ozone dose to 10 g O_3/m^3 (~0.70 mg O_3/mg C) improved the average removal to approximately 66%.

The highest treatment efficiency was achieved at 12 g O_3/m^3 (~0.85 mg O_3/mg C), where the average pharmaceutical removal reached approximately 94–97%. Several compounds, including Amisulpride, Clarithromycin, and Telmisartan, were reduced to concentrations below the analytical detection limit, indicating nearly complete removal under high ozone exposure conditions.

The results also demonstrated a strong synergistic effect between ozonation and GAC filtration. Ozonation effectively oxidized dissolved organic matter and transformed pharmaceutical compounds, while GAC filtration further removed residual contaminants and oxidation by-products. This was additionally supported by significant UV254 reduction, which reached up to 64.5% after GAC filtration at the highest ozone doses.

Pharmaceutical removal efficiency was highly compound-specific. Certain compounds showed excellent degradation during advanced treatment, while others, such as Candesartan and partially Carbamazepine, exhibited greater resistance to oxidation and adsorption processes. These differences highlight the importance of considering chemical structure and wastewater matrix interactions when optimizing advanced oxidation processes.

Overall, the study confirms that the combination of ozonation and GAC filtration represents an effective advanced treatment strategy for the removal of pharmaceutical micropollutants from biologically treated municipal wastewater. The optimal treatment performance in this study was achieved at approximately 0.85 mg O_3/mg C, where nearly complete removal of most target pharmaceuticals was observed.

BETA-ESTRADIOL

First, the primary removal pathway is biodegradation, where microbial communities in activated sludge rapidly transform E2 into less active compounds, mainly estrone (E1), and subsequently into further degradation products with reduced or negligible estrogenic activity. High removal efficiencies (>90%) have been reported in conventional activated sludge systems depending on operational parameters such as sludge retention time and oxygen conditions (Joss et al., 2004; Andersen et al., 2003; Kirk et al., 2002).

Second, E2 is extensively subject to biotransformation rather than complete disappearance, with rapid oxidation to E1 occurring within hours under aerobic conditions. This means that monitoring only E2 can lead to apparent “non-detection” despite the presence of transformation products that may still contribute to overall estrogenicity (Czajka & Londry, 2006; Baronti et al., 2000).

Third, a considerable fraction of E2 is removed via sorption onto activated sludge flocs and organic matter, reducing its dissolved concentration in the aqueous phase and contributing to apparent elimination from effluent (Ternes et al., 1999; Ying et al., 2002).

Fourth, the absence of detectable E2 may also result from analytical limitations, as post-treatment concentrations frequently fall below method detection limits (LOD/LOQ), meaning that trace amounts may still be present but not quantifiable (Belfroid et al., 1999).

Typically, the result of a combination of biodegradation, transformation, sorption, and dilution effects, all of which act simultaneously in biological wastewater treatment systems, leading to very high apparent removal efficiencies (Clara et al., 2005).

Benzotriazoles (BTA) and methylbenzotriazoles (4-MBT, 6-MBT)

Benzotriazoles (BTA) and methylbenzotriazoles (4-MBT, 6-MBT) are persistent industrial contaminants that are widely present in municipal wastewater due to domestic and industrial usage. Their concentrations in the influent showed strong temporal variability, with values reaching up to 3150 ng/L for BTA and 470 ng/L for methylbenzotriazoles, indicating fluctuating source loads.

Biological treatment was largely ineffective for both compound groups. BTA removal ranged from 0–28% under typical conditions, while methylbenzotriazoles showed variable and generally low removal efficiency (0–52%). In several cases, no clear reduction was observed, confirming their resistance to biodegradation and their role as persistent micropollutants in activated sludge systems.

Ozonation proved to be the dominant and most effective treatment step. For BTA, removal increased with ozone dose, reaching up to 95% at 12 g O_3/m^3, while methylbenzotriazoles achieved up to ~91% removal under the same conditions. This demonstrates a clear dose-dependent oxidation effect and confirms the suitability of advanced oxidation processes for their degradation.

GAC filtration showed variable and secondary performance. In some cases, it enhanced overall removal, while in others its efficiency was limited, likely due to competition with dissolved organic matter, saturation effects, or dependence on prior ozonation performance.

Overall, the results show that both benzotriazoles and methylbenzotriazoles are poorly removed by biological treatment, while ozonation is the key process controlling their elimination in the pilot system. GAC acts mainly as a polishing step with inconsistent contribution. The study confirms that effective removal of these compounds requires sufficiently high ozone doses combined with advanced treatment stages.

PFAS

The study of PFAS and fluorotelomer compounds in the pilot wastewater treatment system shows compound-specific behaviour across biological treatment, ozonation, and granular activated carbon (GAC) filtration. Overall, the results indicate limited and highly variable removal efficiency, with frequent evidence of transformation processes, desorption from sludge, and incomplete oxidation.

- **6:2 FTS** in influent wastewater showed an increasing trend over time, indicating rising loads entering the system. In the oxidation tank effluent, concentrations fluctuated significantly, and in several cases were higher than in the influent. This suggests formation from precursor transformation during ozonation or incomplete oxidation. No improvement was observed with increased ozone doses (8–12 gO_3/m^3), indicating that higher oxidation intensity did not enhance removal.
- **8:2 FTS** was detected at low levels in influent (1.12–1.71 ng/L), showing a slight increase over time. After biological treatment it was below detection limits, indicating effective removal or transformation during this stage, although full process quantification was not possible.
- **PFBA** decreased in influent from 7.93 to 2.97 ng/L and was not detected after biological treatment, indicating very effective removal in the biological stage or transformation below detection limits.
- **FBSA** was not consistently present in influent but was detected after biological treatment (1.00–2.25 ng/L). In the oxidation effluent it remained at similar or slightly lower levels (1.08–1.21 ng/L), indicating limited removal and no significant formation or degradation during oxidation.
- **PFBS** showed persistent occurrence throughout all stages, with evidence of limited removal and occasional concentration increases after biological treatment, suggesting desorption from sludge or precursor transformation. Overall, biological and advanced treatments did not effectively reduce PFBS.
- **PFHpA** was not effectively removed biologically and even showed elevated concentrations compared to influent in some cases. Ozonation (8–12 g O_3/m^3) did not significantly degrade PFHpA, and GAC showed limited additional removal, with similar effluent concentrations observed.
- **PFHxA** was detected consistently across all stages, with only one influent measurement (1.32 ng/L). After biological treatment, concentrations were higher than influent, indicating release or transformation processes. Oxidation did not significantly reduce concentrations, showing stability through advanced treatment.
- **PFNA** increased from influent levels (~1.01–1.13 ng/L) to 2.08–2.57 ng/L after biological treatment, indicating release or transformation. GAC reduced concentrations slightly to ~1.66–2.00 ng/L, but removal remained limited.
- **PFOA** was consistently detected in influent (~1.26–1.53 ng/L), but increased significantly after biological treatment (1.98–3.39 ng/L). Ozonation showed variable effects with no consistent reduction, while GAC provided the most stable but incomplete removal.

- **PFPeA**, a short-chain PFAS, was mostly absent or below detection in influent but appeared after biological treatment (~1.44 ng/L) and increased slightly after ozonation (1.82–2.23 ng/L). GAC showed limited adsorption capacity for PFPeA.
- **Overall:** Biological treatment often led to increases in several PFAS concentrations, likely due to desorption from sludge or transformation of precursors. Ozonation (8–12 g O_3/m^3) did not consistently degrade PFAS and in some cases contributed to formation or redistribution effects. GAC provided partial and compound-dependent removal, with better performance for some long-chain compounds but limited effectiveness for short-chain PFAS. Overall, the system achieved only partial and inconsistent PFAS removal, strongly dependent on compound properties and treatment stage.

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