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RESEARCH OF THE POSSIBILITY OF REMOVING ORGANIC POLLUTANTS FROM WATER BY MEMBRANE METHODS AND PURIFICATION OF THE OBTAINED CONCENTRATE BY CHEMICAL METHODS

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ABSTRACT: Reverse osmosis (RO) and nanofiltration (NF) stand out for their versatility in terms of applicability to a wide range of contaminants, including personal care product (PCP) residues and endocrine disrupting chemicals (EDCs). The aim of the research was to analyse the efficiency of the removal of dosed PCPs and EDCs from model water with the use of RO and NF membranes. A study was also undertaken on the oxidation of the resulting concentrate after the membrane process – the efficiency of oxidation with ultraviolet-activated peracetic acid – UV/PAA system was analysed. Analysis of samples after each stage of the study showed that reverse osmosis was more effective in purifying water of EDCs than nanofiltration, while the UV/PAA oxidation system has a high potential for neutralising the concentrate formed after membrane processes, and points in the right direction for further in-depth research.

KEYWORDS: nanofiltration, reverse osmosis, retentate, oxidation

Introduction

Reverse osmosis (RO) and nanofiltration (NF) are among the most popular and, at the same time, the most effective in terms of contaminant removal (Othman et al., 2021; Monachan et al., 2021). Other factors contributing to the mechanics of the membrane's operation include the pressure under which the membrane process is carried out, the temperature, the concentration of the substance in the test sample and the octanol-water partition ratio (Hidalgo et al., 2021). Reverse osmosis, thanks to its operation under high pressure and small pore diameters, allows the removal of dissolved organic substances with small particle sizes in solution, unlike other membrane processes, such as ultrafiltration, which is usually treated as a method for pretreating water from suspended solids and microorganisms. In addition, reverse osmosis is more effective at picking up dissolved substances than absorption methods, such as the popular activated carbon filtration, on whose porous structure larger pollutant particles can be deposited (Aziz et al., 2024).

When treating water containing highly hazardous substances, such as endocrine disrupting chemicals (EDCs), the concentrate, often harmful to the natural environment (Belhout et al., 2022), becomes a problematic waste that must be managed, and it is the management or treatment that becomes the challenge (Nowak & Włodarczyk-Makuła, 2020). To date, concentrate has most often been fed into the sewer system to dilute municipal wastewater (Scholes et al., 2021), which means that currently, there is no simple method developed for practical application in technology.

It should also be taken into account that water is a limited resource that is depleting faster and faster as the economy and population continue to expand (Barbier & Burgess, 2024). Moreover, the problem is not just an impending water shortage but the very existence of polluted watercourses. Studies have shown that river pollution has a direct impact on the economic development of the cities that use them – in the middle income countries with moderately polluted rivers, growth downstream is reduced by 1.8%, and with heavily polluted rivers, growth impacts are up to 2.5% (Russ et al., 2022). Environmental and economic analyses emphasise that the problem of pollution should be solved at its root, i.e. when the wastewater is properly treated – it makes economic sense to build a wastewater treatment plant for both the investor and the community using it. This allows for the achievement of further sustainable development goals, and the focus should be on membrane-based drinking water production systems (López-Serrano et al., 2023).

The purpose of the research presented in this article was to evaluate the possibility of treating water from selected contaminants and purifying the resulting concentrate using nanofiltration (NF) and reverse osmosis (RO) membrane processes. Achieving a high removal effect while introducing a significant amount of chemicals into the environment is not the right direction for technological development, as this equals the secondary pollution of the environment with compounds of a different, by no means less harmful, nature. Therefore, the research conducted was directed towards simple methods with an emphasis on minimising the chemical reagents used, which at the same time will be easily degradable in the aqueous environment. The obtained results will contribute to defining the direction of further research to improve the efficiency of EDC removal by membrane methods and concentrate oxidation, simultaneously minimising the use of chemical reagents (reaction optimum), which has a direct impact on reducing the potential cost of the whole process.

Research methods

Technological research methodology

The technological tests were carried out on a semi-technical pilot plant for membrane processes. The plant was designed by Stadtwerke Düsseldorf (SWD) and implemented by Cornelsen Umwelttechnologie GmbH, Essen, Germany. Grünbeck Wasseraufbereitung GmbH, Höchstädt a.d. Donau, Germany developed a low-pressure reverse osmosis (LPRO) unit (GENO-Nano RKF1800 S). The research was conducted using a spiral module.

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The study began with a nanofiltration membrane, pre-working it with distilled water. Then, a water sample with the analysed contaminants was passed through the membrane, and the actual nanofiltration process was carried out, after which permeate and concentrate samples were taken for analysis. The same test procedure was carried out on the reverse osmosis membrane.

The recovery rate for nanofiltration and reverse osmosis was about 75%. The membrane-forming material for reverse osmosis and for nanofiltration was a semi-crystalline polymer (polyamide), and the membrane area was 4 m^2. The transmembrane pressures were 0.8 MPa for nanofiltration and 1.3 MPa for reverse osmosis.

The study included hard-to-solubilize organic compounds from personal care products (PCPs), which also included active substances belonging to the group of EDCs. The chemicals comprising the mixture of dosed contaminants are shown in Table 1. Since the tests were conducted on distilled water with dosed contaminants (shown in Table 2 and Table 3), it was not necessary to dose antiscalants or pre-filter the analysed water before membrane processes to protect the membranes from scaling and fouling.

Table 1. Chemical compounds used in membrane studies

Analysed compound	Shortcut	Application
N,N-Diethyl-m-toluamid	DEET	a repelent
Benzophenone ($C_{13}H_{10}O$)	BPh	an UV filter
4-octylphenol	4OP	a stabilizer, non-ionic surfactant
3-(4-methylbenzylidene)camphor	4MBC	an UV filter
Triclosan	TRC	an antibacterial substance
Carbamazepine (Tegretol)	CBZ	a psychotropic and antiepileptic drug
Diethylstilbestrol $C_{18}H_{20}O_2$	DES	a syntetic oestrogen
Estrone $C_{18}H_{20}O_2$	E1	a steroid oestrogen
Estradiol $C_{18}H_{24}O_2$	E2	a natural oestrogen
Clotrimazole $C_{22}H_{17}ClN_2$	CTM	an antifungal substance

The efficiency of the membrane processes was determined by calculating the removal effect – the retention ratio (R) expressed in %.

The obtained retentates were then treated with UV/PAA oxidation. UV radiation with a wavelength of 254 nm in the presence of the oxidant peracetic acid (PAA) was used. Oxidation was carried out for two times (5 min and 10 min) with the nanofiltration-derived concentrate, as well as for four times (5 min, 10 min, 30 min and 60 min) with the reverse osmosis-derived concentrate. To samples of 29,858 mL, 0.1425 mL of PAA and 150 µL of 1M NaOH solution were added to bring the reaction conditions to alkaline pH. UV irradiation was carried out on a stirring device, with the samples and UV lamp completely covered, to avoid the influence of external light on the reaction. After the designated times, the reaction was stopped by adding 720 µL of sodium thiosulfate to the sample.

Analythical research methodology

Analytical determinations and gas chromatograph (GC-MS) tests were carried out based on current standards or based on analytical methodologies widely recognised and recommended in the scientific literature.

Extraction procedure methodology

To analyse the collected water samples, isolation and derivatisation of the mentioned compounds were carried out. This was followed by ultrasonic-assisted emulsification microextraction (USAME).

To the collected sample of 25 mL was added 0.75 g of disodium hydrogen phosphate (4%). To the prepared samples, 100 µL of solvent and 250 µL derivatisation reagent were added. Extractions were

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carried out at 42 kHz ultrasonic frequency and 230 W power for 5 min at ambient temperature. To obtain emulsions, the prepared samples were subjected to centrifugation at 4000 RPM for 5 minutes in a laboratory centrifuge.

The organic phase accumulated at the bottom of the conical tubes was extracted with a 100 µL Hamilton (USA) syringe and transferred to a 150 µL microfilm with an integrated insert.

GC-MS analysis

The extracted samples were subjected to gas chromatography-mass spectrometry (GC-MS) analysis. For this purpose, an HP 7890B gas chromatograph with an electronic pressure control device was used. The analyser was connected to an MSD 5977A mass detector (Agilent Technologies, USA), using a 30m HP-5MS column (5% phenylmethylsiloxane) and an inner diameter of 0.25 mm. The injection was carried out using a splitless injector at 250°C. The device used helium carrier gas with a constant flow rate of 1.0 mL/min.

The oven temperature was programmed from 110 °C, increased at 5 °C/min, to 300 °C. The total run time was 38 min. The MS detector worked in the selected ion monitoring (SIM) mode. The electron impact source temperature was 230 °C with an electron energy of 70 eV. The quadrupole temperature was 150 °C, and the GC interface temperature was 280 °C. The retention times of the individual analytes were as follows DEET 11.41 min, BPh 12.46 min, 4OP 17.15 min, 4MBC 20.89 min, TRC 23.08 min, CBZ 26.01 min, DES 28.28 min, E1 31.85 min, E2 32.12 min, CTM 33.20 min. The calibration curve method was used to carry out the quantitative determinations. To record calibration curves, a series of solutions containing a mixture of tested compounds with concentrations 1 µg/L, 5 µg/L, 10 µg/L, 50 µg/L, 100 µg/L and 500 µg/L were subjected to an extraction procedure, and the obtained extracts were analysed by GC-MS. The calibration curves showed linearity in the entire analysed concentration range, the values of the determination coefficients R^2 for the obtained dependencies were in the range from 0.984 (E2) to 0.999 (DEET, BPh and 4MBC). The limits of detection (LOD) were between 0.005 µg/L (DEET) and 0.2 µg/L (E2) while the limits of quantification (LOQ) were between 0.015 µg/L and 0.5 µg/L. The repeatability expressed as the coefficient of variation (CV) value ranged from 5 to 17% and the accuracy of the determinations expressed as the recovery value ranged from 80 to 124%.

Results of the research and discussion

Nanofiltration allowed water purification rates to reach above 90% for most contaminants (Table 2). The best removal effect was obtained for estradiol, equal to 99.42%, and for triclosan, 99.33%, and estrone, 99.28%. Clearly weaker effects were shown for benzophenone removal (94.14%), and the weakest result was obtained for DEET (79.34%) In a study by Acero et al. (2016), DEET also proved to be a problematic compound for removal. The retention rate of DEET was different for the three types of nanofiltration membranes used – for example, the DK membrane and the HL membrane allowed about 70% removal of DEET, and the CK membrane only less than 40%. The main difference in the membranes used in this study was that the DK and HL membranes were made of a thin-film composite and were hydrophilic, while the CK membrane was a cellulose acetate hydrophobic membrane. In addition, the DK membrane was dedicated to a larger pH range than HL (Acero et al., 2016).

Reverse osmosis contributed to better removal effects than nanofiltration for almost all chemicals analysed (Table 3). As with nanofiltration, the best removal was for estradiol (99.57%), triclosan (99.44%) and estrone (99.34%). A slightly weaker removal effect was obtained for carbamazepine, equal to 99.27%, and diethylstilbestrol, equal to 99.26%. The weakest removal effect was shown for DEET, equal to 98.16%, but despite this, a high decrease in its concentration in the permeate after RO compared to its concentration in the permeate after NF was observed: with nanofiltration, the concentration in the permeate was 22.74 µg/L, and with reverse osmosis, the concentration dropped to 2.02 µg/L, which means that RO gave a more than eleven times better removal effect – a similar phenomenon confirmed by the study performed by Krzeminski et al. (2017).

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Table 2. Contaminant removal effect with the use of nanofiltration

Analysed compound	Concentration [µg/L]		Application
	Raw water	Permeate	
DEET	110.07	22.74	79.34%
BPh	110.13	6.45	94.14%
4OP	57.60	0.81	98.59%
4MBC	72.30	0.94	98.70%
TRC	69.75	0.47	99.33%
CBZ	128.10	13.68	89.32%
DES	43.73	0.86	98.03%
E1	125.68	0.91	99.28%
E2	182.76	1.06	99.42%
CTM	43.84	0.88	97.99%

An important part of analysing the obtained results is to understand the nature of the tested compounds and their chemical nature, which is helped by the octanol-water partition coefficient (Log Kow). The value of this coefficient indicates whether compounds and impurities tend to combine with water, which is directly related to their solubility in a given medium. The range of Log Kow from -3 to 10 is usually used for evaluation, with a value of -3 indicating that the substance is highly hydrophilic and 10 indicating that the substance is highly hydrophobic (Cumming & Rücker, 2017).

Table 3. Contaminant removal effect with the use of reverse osmosis

Analysed compound	Concentration [µg/L]		Application
	Raw water	Permeate	
DEET	110.07	2.02	98.16%
BPh	110.13	0.84	99.24%
4OP	57.60	0.81	98.59%
4MBC	72.30	0.93	98.71%
TRC	69.75	0.39	99.44%
CBZ	128.10	0.94	99.27%
DES	43.73	0.76	98.26%
E1	125.68	0.83	99.34%
E2	182.76	0.79	99.57%
CTM	43.84	0.86	98.04%

All of the compounds analysed in this study have Log Kow greater than 2 (National Library of Medicine, 2024; Chemical Book, 2024; DrugBank, 2024). This means that the compounds are mostly hydrophobic in nature. This often results in the phenomenon of retention of contaminants on the surface of the membrane, if the membrane is also hydrophobic in nature, which facilitates water purification – substances will leave the aqueous environment more easily.

The second main part of the conducted studies was the oxidation of the resulting concentrates (Table 4) in a UV/PAA system. The greatest advantages of this system are its simplicity and the minimisation of the chemical reagents introduced into the samples since the main role in the oxidation was played by ultraviolet radiation (Zhu & Liu, 2022).

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Table 4. Contaminant removal effect with the use of reverse osmosis

Analysed compound	Retentate [µg/L]		Degree of concentration of raw water	
	RO	NF	RO	RO
DEET	491.52	701.29	447%	637%
BPh	78.57	270.14	71%	245%
4OP	8.38	74.99	15%	130%
4MBC	72.20	142.75	100%	197%
TRC	10.90	152.67	16%	219%
CBZ	772.96	1037.85	603%	810%
DES	120.00	278.22	274%	636%
E1	319.00	650.06	254%	517%
E2	203.52	659.91	111%	361%
CTM	119.00	27.33	271%	-

Reverse osmosis allowed a significantly higher concentration of the retentate than nanofiltration (Table 4). The exception is the result obtained for clotrimazole, where its concentration in the nanofiltration retentate was 119 µg/L, and in the osmotic retentate – 27.33 µg/L. This phenomenon is most likely due to the fact that clotrimazole is a lipophilic substance (Balata et al., 2011). It is likely that very strong adsorption of the pollutant on the osmotic membrane occurred here, which is stronger the smaller the pores are. Previous results indicate that the resulting permeate in terms of CTM content was much cleaner for RO than for NF, so these contaminants had to stay on the membrane, causing partial membrane collation (also called fouling) (Farhat et al., 2012). For the remaining compounds, RO allowed the concentrate to be concentrated from 1.3 times to more than 8 times, as shown in Table 4. The phenomenon of better concentration of the concentrate by RO than by NF is confirmed by the studies of Ali and Qaisrani (2024).

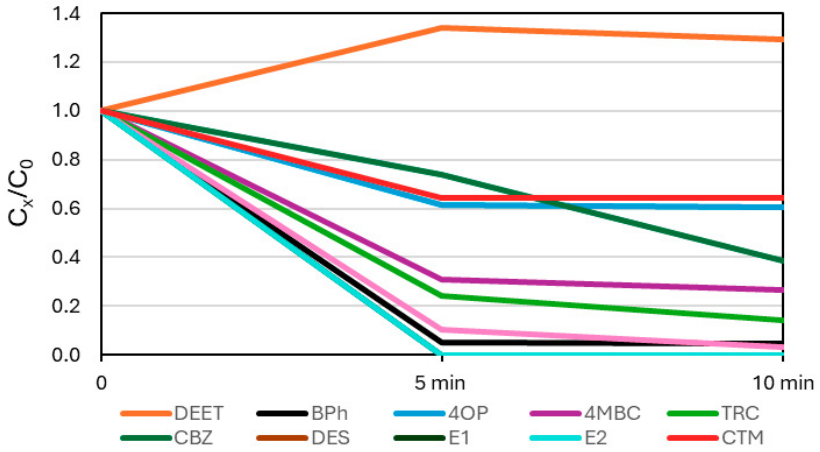

Figure 1. Time-dependent oxidation effect of NF concentrate

The concentrate obtained after nanofiltration was subjected to UV/PAA oxidation at two times: for 5 and 10 minutes (Figure 1). To determine the effect of removing impurities, the C_x/C_0 ratio was determined, with C_0 being the base concentration of impurities in the concentrate and C_x being the concentration of impurities after a given oxidation time.

The results showed that most of the compounds were oxidised with the exception of DEET, for which an increase of more than 30% was observed in the sample. It is possible that some of the remaining contaminants were not oxidised completely to CO_2 and H_2O but were partially broken

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down. It is possible that chemical reactions occurred in the concentrate, resulting in the formation of compounds like DEET, which would confirm its unexpected increase in concentration in the concentrate. More laboratory tests are needed to conclusively confirm or refute this.

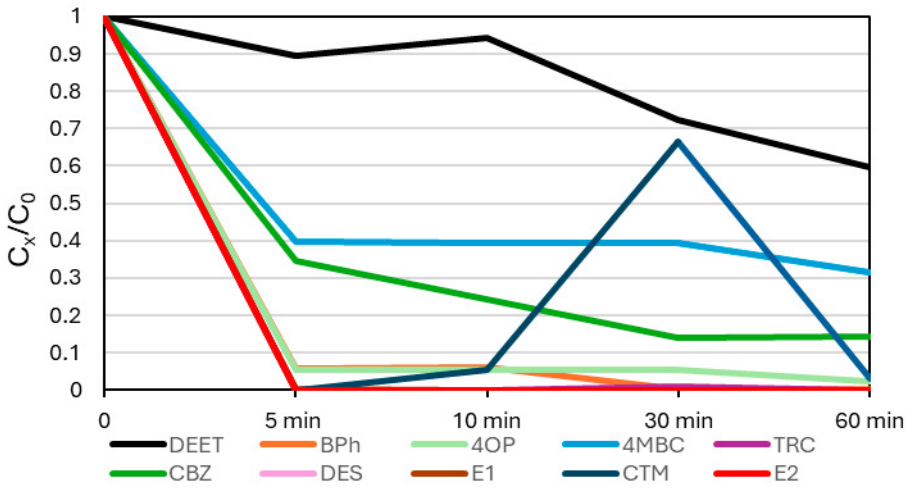

Figure 2. Time-dependent oxidation effect of RO concentrate

Carbamazepine and clotrimazole proved to be resistant compounds in oxidation. The removal effect was achieved at 50%. Compared to the other contaminants, where the oxidation effect ranged from 60% to as high as 100% for DES and E2, this is unsatisfactory and requires further in-depth laboratory analysis. The concentrate formed after reverse osmosis was subjected to UV/PAA oxidation at four times: for 5, 10, 30 and 60 minutes (Figure 2). Studies on the oxidation of reverse osmosis concentrate using UV were carried out by Mangalgiri et al. (2021), and analysed the removal of dissolved organic matter (DOM) in general. They showed that UV is able to remove organic compounds from the concentrate, but the removal efficiency depends on the oxidation enhancers used (Mangalgiri et al., 2021).

The obtained oxidation effects are overall more effective than with nanofiltration, as not only DES and E2, but also E1 and benzophenone were completely removed. In the case of DEET, again an anomaly was observed with oxidation – there was also an increase in DEET in the concentrate, however, not after a time of 5 minutes, but after 10 minutes, and also the increase was not greater than the original concentration of C_0. A similar situation was observed for clotrimazole. In its case, there was a small increment in the concentrate after 10 minutes, which increased sharply after 30 minutes of oxidation, to fall just as sharply after an hour, and finally amounted to 0.83 µg/L. Clotrimazole was resistant to removal from the concentrate with NF, and here, at higher concentrations in the RO concentrate, it likely proved unstable. For the other contaminants, their final removal rate ranged from nearly 70% to 100%.

Figures 1 and 2 clearly show that the purification effect of the concentrate is better the longer the oxidation process lasts. For some of the EDCs and PCP compounds, it allowed almost complete removal from the concentrate. Further research is needed for DEET and CTM, where complex processes of transition of partially broken down impurities into other compounds – their breaking down and re-synthesis – are most likely taking place. Oxidation effects are clearly visible but with lower efficiency than assumed. Nevertheless, the study shows that the UV/PAA system analysed is quite effective and has the potential to completely utilise the concentrate.

Conclusions

Based on the conducted research and the obtained results, the following conclusions were drawn:

- Reverse osmosis is a more effective method for removing PCP and EDC compounds from water than nanofiltration.

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- UV/PAA oxidation of the concentrate achieves quite high removal of PCP and EDC contaminants from the concentrate after nanofiltration and reverse osmosis.
- More in-depth research and consideration should be given to the use of other oxidation systems for the compounds analysed, including DEET and CTM.

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The contribution of the authors

Conceptualisation, J.P., M.I.B. and U.K.; methodology, J.P. and U.K.; validation, J.P., M.I.B. and U.K.; formal analysis, J.P. and U.K.; investigation, J.P. and M.I.B.; resources, J.P. and M.I.B.; data curation, J.P., M.I.B. and U.K.; writing – original draft preparation, M.I.B.; writing – review and editing, J.P. and U.K.; visualisation, M.I.B.; supervision, J.P., U.K. and P.K.; funding acquisition, J.P. and U.K.

The authors have read and agreed to the published version of the manuscript.

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MOŻLIWOŚCI USUWANIA ZANIECZYSZCZEŃ ORGANICZNYCH Z WODY METODAMI MEMBRANOWYMI I OCZYSZCZANIA OTRZYMANEGO KONCENTRATU METODAMI CHEMICZNYMI

STRESZCZENIE: Odwrócona osmoza (RO) i nanofiltracja (NF) wyróżniają się wszechstronnością pod względem możliwości zastosowania do szerokiego zakresu zanieczyszczeń, w tym pozostałości środków higieny osobistej (PCP) i związków endokrynnie czynnych (EDC). Celem badań była analiza skuteczności usuwania dozowanych PCP i EDC do wody modelowej przy użyciu membran RO i NF. Podjęto również badania nad utlenianiem powstałego koncentratu po procesie membranowym – analizowano skuteczność utleniania za pomocą aktywowanego promieniowaniem ultrafioletowym kwasu nadoctowego – układ UV/PAA. Analiza próbek po każdym etapie badań wykazała, że odwrócona osmoza była bardziej skuteczna w oczyszczaniu wody z EDC niż nanofiltracja, natomiast system utleniania UV/PAA ma duży potencjał do neutralizacji koncentratu powstałego po procesach membranowych i wskazuje właściwy kierunek dalszych pogłębionych badań.

SŁOWA KLUCZOWE: nanofiltracja, odwrócona osmoza, koncentrat, utlenianie

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