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Nanofiltration treatment of swimming pool water in the aspect of the phenolic micropollutants elimination

Anna Lempart*, Edyta Kudlek, Mariusz Dudziak

Institute of Water and Wastewater Engineering, Faculty of Energy and Environmental Engineering, Silesian University of Technology, Konarskiego 18, 44-100 Gliwice, Poland, emails: Anna.Lempart@polsl.pl (A. Lempart), Edyta.Kudlek@polsl.pl (E. Kudlek), Mariusz.Dudziak@polsl.pl (M. Dudziak)

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ABSTRACT

Guaranteeing the safety and comfort of swimmers is done by providing swimming pool water properties that will not pose a threat to the health of users. Meanwhile, recently published publications more and more often pay attention to the growing number of organic micropollutants classified as endocrine-disrupting chemicals (EDCs) identified in the swimming pool water environment. The removal of phenols, as an example of compounds from EDCs group, from swimming pool waters by nanofiltration was investigated in this study. Commercial semi-aromatic polyamide membrane NF-270 was used. It has been documented that the occurrence of phenolic micropollutants in pool water is common, and the nanofiltration process can be used as effective method to eliminate them. The degree of phenol removal in the nanofiltration process exceeded 51%, depending on the water matrix, the type and parameters of pool water, the concentration of removed compounds and the duration of the filtration process. It was also shown that membrane techniques can help and improve the overall quality of pool water. The membrane is an additional barrier for viruses, bacteria and protozoa. Furthermore, nanofiltration has been shown as an effective method for removing water-soluble organic compounds, including precursors of water disinfection by-products.

Keywords: Nanofiltration; Phenolic micropollutants; Swimming pools; Membrane techniques; Endocrine-disrupting chemicals (EDCs)

1. Introduction

The occurrence of many hazardous biologically active substances in various water streams, for example, municipal wastewater, surface water, and even in tap water intended for consumption, is becoming an increasingly common problem for scientists [1–4]. Examples of such compounds are phenols, for example, octylphenol (OP), nonylphenol (NP), bisphenol A (BPA) and pentachlorophenol (PCP). As they belong to the group of endocrine disrupting chemicals (EDCs), they arouse public interest due to their potential genotoxicity and carcinogenicity [5]. The specificity of their action is based on the dysfunction of the endocrine system, which regulates the work of the entire human body. OP, NP and PCP have been classified by the European Parliament and the Council as Priority Substances in the field of water policy [6].

Worldwide monitoring studies have shown the presence of these compounds in the environment in a various range of concentrations [7–10]. Their frequent occurrence in the environment results from their widespread use. Nonylphenols are used to manufacture nonylphenol ethoxylates (NPEs) that are widely used in commercial and household cleaning products, industrial processing, and in many consumer products such as fabrics, shoes, paints and coatings, lawn care and crop protection products, personal care products such as lotions and liquid cosmetics [11,12]. Octylphenol is a chemical used to manufacture many products including octylphenol ethoxylates (OPEs), for example, paints,

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* Corresponding author.

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coatings, adhesives, inks and products containing rubber. OP is also present in detergents and surfactants used in some household, industrial and pesticide products, and in some personal care products, such as cosmetics, body lotions, soaps, face creams and hair products [13,14].

BPA is commonly applied in plastics, mainly polyesters and epoxy resins used in the optical and automotive industry, for the production of electronic and sports equipment, glass, medical devices and bottles and food packaging, as well as laminates, adhesives, paints and varnishes with a wide range of applications [15,16]. PCP is a manufactured chemical that was once widely used as a pesticide, preservative and disinfectant. Although the use and sale of PCP has been restricted to preserving wood products, such as utility poles, used in exterior settings, PCP is still present in the collected waste in landfills, where it can be released into the environment. It can get into the soil, air or water at places where it is still used to treat wood, or where it was spilled or discarded. Such sources of PCP may contaminate ground water or surface water and sediments. PCP-treated products might also release PCP into surrounding soil, air or water. Pentachlorophenol may also be formed as by-products in the chlorination of water [17,18].

The preliminary studies carried out recently by the authors of this work [19,20] have documented the occurrence of BPA, OP, NP and PCP also in the swimming pool water (SWP). It is a very specific environment in which there are many different chemical substances, both introduced with the source tap water, getting from the air together with dust and dirt, brought by swimmers on their skin, clothing and hair, as well as introduced with the products of human metabolic activity and body secretions. The occurrence of EDCs in the SWP is particularly worrying, because the body of the swimmer is directly exposed to the chemicals present in the water, as they can be both absorbed through the skin, swallowed and inhaled while breathing just above the surface of the water.

The use of a closed water circuit with an active overflow makes it possible to extend the total water exchange time in swimming pools up to 1 year. It favours the accumulation and concentration of micropollutants that are not removed in traditional water purification devices used in typical pool installations.

Pressure membrane processes, mainly nanofiltration (NF) is considered to be an effective method of removing organic micropollutants from water streams [21,22] and production of high-quality water [23]. There are many examples of its use in practice, especially in the drinking water industry [24,25]. The use of compact membranes allows for the retention of substances with a molecular weight of 150–500 g/mol in the NF process. In paper [26] it was shown that in terms of removing micropollutants and process efficiency, membrane filtration is most preferably carried out in a cross-flow mode. In this system, unfavourable phenomena occurring on the surface of the membrane, which usually cause a reduction in the retention of compounds, are much less intense than in the dead-end method. The forming boundary layer thickness is less, resulting in lower filtration resistance values and higher values of the volume flow of the permeate.

Effective use of NF to remove organic micropollutants in water requires knowledge about the mechanism of separation and factors affecting it [27]. The most important mechanisms of removing micropollutants in NF are the sieve effect, adsorption and hydrophobic interaction between the small-molecule organic compound, the membrane surface and the electrostatic interaction. These mechanisms may be related to both the physicochemical properties of the removed compounds and the membrane characteristics.

The main aim of this research is to assess the effectiveness of removing phenols from SWP in NF processes. The application of this modern innovative technique for water treatment may improve the quality of SWP and reduce the health risk of swimmers caused by exposure to biologically active micropollutants.

2. Materials and methods

2.1. Research methodology

Research work was carried out in three stages:

(1) Preliminary study, which included the determination of phenols in pool water samples taken from 46 different pools in Poland, aimed at checking the occurrence of the selected micropollutants above the limit of detection (LOD) value in real pool water samples. The study included 10 sport swimming pools, 14 hot tubs, 8 recreational pools, 4 swimming pools for learning to swim (including 2 school ones), 8 paddling pools for children and 2 slide installations. Five of the examined swimming pools were outdoors and 39 were indoors. The 5 of tested pools are private pools, including 4 hotels.

At this stage of the research, the identification of phenolic micropollutants was performed on the basis of the mass spectra obtained during gas chromatography analysis, using the NIST 17 Mass Spectral Library and by comparing the response of the mass detector with the standards response. The probability of correct matching in all cases was over 70%. Table 1 summarizes the data necessary at this stage of work, that is, characteristic ions of the tested compounds, the retention time of their standards and the LOD for the applied methodology. The procedure of developing the analytical methodology and its validation is described in Ref. [28].

(2) NF study, which included the filtration of model waters prepared on the basis of deionized water (DW) with the addition of selected micropollutants standards at the concentration of individual compounds of 1.0 or 10 μg/L,

Table 1
Identification parameters of selected compounds

Compound	SIM ions	t_R ± SD	LOD (ng/L)
Octylphenol	107-108-206	26.55 ± 0.07	0.10
Nonylphenol	107-220-108	26.65 ± 0.02	0.08
Bisphenol A	213-228-119	28.81 ± 0.03	0.02
Pentachlorophenol	103-266-264	34.60 ± 0.04	0.06

SIM – selected ion monitoring, t_R – retention time (the time taken for a solute to pass through a chromatography column), SD – standard deviation, LOD – limit of detection (it is the lowest quantity of a substance that can be distinguished from the absence of that substance (a blank value) with a stated confidence level; determined by signal-to-noise approach).

conducted in a cross-flow system at a transmembrane pressure of 2.0 MPa.

(3) Main research, including the filtration of SWP with the addition of selected micropollutants standards at the concentration of individual compounds of 1 or 10 μg/L in a cross-flow system under a transmembrane pressure of 2.0 MPa.

2.2. Chemicals and materials

2.2.1. Standards

Four phenols were used in this study, including OP, NP, BPA and PCP. All of them were purchased from Sigma-Aldrich (Poznań, Poland) in analytical grades with >97% purity. Characteristics of compounds are summarized in Table 2. Two stock solutions containing mixtures of 1 μg/L (low concentration, LC) and 10 μg/L (high concentration, HC), respectively, of each phenol was prepared by dissolving the pure chemicals in DW by Elix® Water Purification System. Reagents used for quantification were of GC grades for solvents (methanol and acetonitrile), by Avantor Performance Materials Poland S.A.

2.2.2. Membrane

Commercial semi-aromatic polyamide membrane NF-270 (Dow Filmtec) was investigated in this study. The membrane has been characterized by Dudziak [29]. The molecular weight cut-off was 200 Da, water permeability determined at ΔP = 2.0 MPa was 43.86 × 10^{-6} m^3/m^2 s, the removal of salts determined in experiment during filtration of $MgSO_4$ or NaCl solution (1,000 mg/L) at ΔP = 2.0 MPa was, respectively, 92.1% for $MgSO_4$ and 41% for NaCl. The transport and separation parameters of the NF membranes were assessed using the equations given in Table 3. The determination of NF effectiveness was based on the measurements of both membrane efficiency (J_v and α) – Eqs. (1) and (2) and selectivity (R) – Eq. (3).

2.2.3. Swimming pool water

Two solutions, containing, respectively, mixtures of 1 μg/L (LC) and 10 μg/L (HC) of each phenol, were prepared. They were made by dissolving the phenolic standards in SPW. The characteristic of applied water is shown in Table 4. Conductivity and pH were measured with the ELMETRON multifunctional CX-461 meter. Total organic carbon (TOC) was gauged by TOC-L total organic carbon analyser of Shimadzu. Free and bound chlorine concentration was obtained by Merck NOVA 30 A spectrophotometer. The absorbance UV_{254} gauged at a universal wavelength of 254 nm using a CECIL CE1021 spectrophotometer corresponds to the total concentration of organic compounds. It enables the identification of the dissolved organic carbon (DOC) fraction characterized by a high content of aromatic components, and thus a large potential for disinfection by-products formation.

2.2.4. Solid phase extraction tubes

The isolation of micropollutants from the water matrix was carried out by solid phase extraction (SPE) in

Table 2
Characteristics of investigated pharmaceuticals

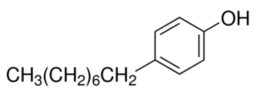
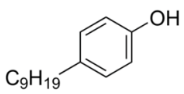
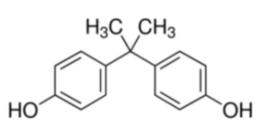
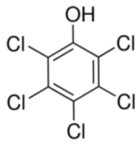
Compound	Octylphenol (OP)	Nonylphenol (NP)	Bisphenol A (BPA)	Pentachlorophenol (PCP)
Structural formula				
Molecular formula	$CH_3(CH_2)_7C_6H_4OH$	$C_{15}H_{24}O$	$(CH_3)_2C(C_6H_4OH)_2$	C_6Cl_5OH
Molecular weight (g/mol)	206.32	220.35	228.29	266.34
CAS number	1806-26-4	84852-15-3	80-05-7	87-86-5

Table 3
Parameters of the nanofiltration membranes

Parameter	Mark	Unit	Equation	Number
Volumetric permeate flux (deionized water)	J_v (J_w)	$m^3/m^2\cdot s$	$J_v(J_w) = \frac{V}{A \times t}$	(1)
Relative permeability of the membrane	α	–	$\alpha = \frac{J_v}{J_w}$	(2)
Retention coefficient	R	%	$R = \left(1 - \frac{C_p}{C_f}\right) \times 100$	(3)

V – volume (m^3), A – membrane area (m^2), t – filtration time (s), C – concentrations (mg/L), p – permeate, f – feed.

Table 4
Physicochemical parameters of the swimming pool water

Parameter	Swimming pool water (SPW)
Absorbance UV_{254} (cm^{-1})	0.043
Conductivity (mS/cm)	2.178
pH (–)	7.46
Free chlorine concentration (mg/L)	0.5
Bound chlorine concentration (mg/L)	0.34
Total organic carbon (mg/L)	14.05
Water hardness	6.32

Supelclean™ ENVI™-18 tubes filled with nonpolar adsorbent C-18 (octadecyl) bonding, polymerically bonded with volume – 6 cm^3 and phase – 1.0 g by Supelco (Poznań, Poland). The particle size is 45 µm, pore size – 60 Å and pore volume 0.8 cm^3/g. Surface area is 457 m^2/g. The matrix is silica gel base material (irregular shaped, acid washed).

2.3. Analytical procedure

The isolation of micropollutants from the water matrix was carried out by SPE. The bed was first conditioned in sequence with 5 mL of methanol and 5 mL of acetonitrile. Then it was washed with 5 cm^3 of DW. Afterwards 100 mL of water sample was applied. To ensure optimal retention, extraction was carried at a consistent and reduced flow rate of ~1–2 drop/s. After extraction the bed was dried for 5 min under vacuum. The extract was eluted, respectively, with 1.5 mL of acetonitrile and 1.5 mL of methanol.

The extracts were analysed using a gas chromatograph coupled to mass spectrometry (GC/MS) with electronic ionization, Model 7890B by Perlan Technologies (Warszawa, Poland). It combines two techniques to provide the identification of compounds with low detection limits. The extract was separated in a SLB™ – 5 ms capillary GC column of Supelco with an internal diameter of 0.25 mm, a length of 30 m and a layer thickness of 0.25 µm. The oven temperature program was as follows: 80°C (6 min), 5°C/min to 260°C and 20°C/min to 300°C. The support phase was helium with a flow of 1.1 mL/min. Sample injections of 1 µL were performed automatically. The mass detector worked in the ion recording mode in the range of 50–700 m/s. The schematic diagram of the GC/MS analysis details is presented in Fig. 1.The procedure for developing the analytical methodology and its validation is described in Ref. [28].

2.4. Membrane filtration system

The process was carried out at a transmembrane pressure of 2.0 MPa in the installation enabling filtration in the cross-flow system made by Z.A. Rotameter according to the Dudziak project (Fig. 2(a)), equipped with a membrane module from Osmonics Inc. type SEPA CF – HP in high pressure version with active membrane surface of 155 cm^2 (Fig. 2(b)). Considering the nature of the tests carried out, the installation was made entirely of stainless steel and equipped with a 33 L tank with a heat exchanger that allows maintaining a constant temperature of 5°C, a high pressure pump with a capacity of up to 15 L/min and the pump head up to 7 MPa by Grundfos (Denmark) and the control-measurement equipment. Pressure gauges were placed before and after the membrane module, and the flow meter was installed on the retentate line. The filtration was carried out to receive 50% of the feed volume. The time of process was 5 h.

3. Results and discussion

3.1. Determination of phenolic micropollutants in SWP

Fig. 3 shows the frequency of phenolic micropollutants detected in the samples from 46 swimming pools. The occurrence of these four phenolic micropollutants in swimming pools has not yet been reported in any other literature data. Table 5 illustrates the frequency of NP, OP, BPA and PCP occurrence in the pool water by venue type. Notably, the NP was present in almost 90% and PCP in 70% of pool water samples, regardless of season, location and venue type. Thus, there is a need for an effective method to eliminate this type of micropollutants from SWP.

3.2. Efficacy of the NF in the removal of phenolic micropollutants

In the next stage of the research, the effectiveness of phenolic micropollutants elimination from model SWP in the

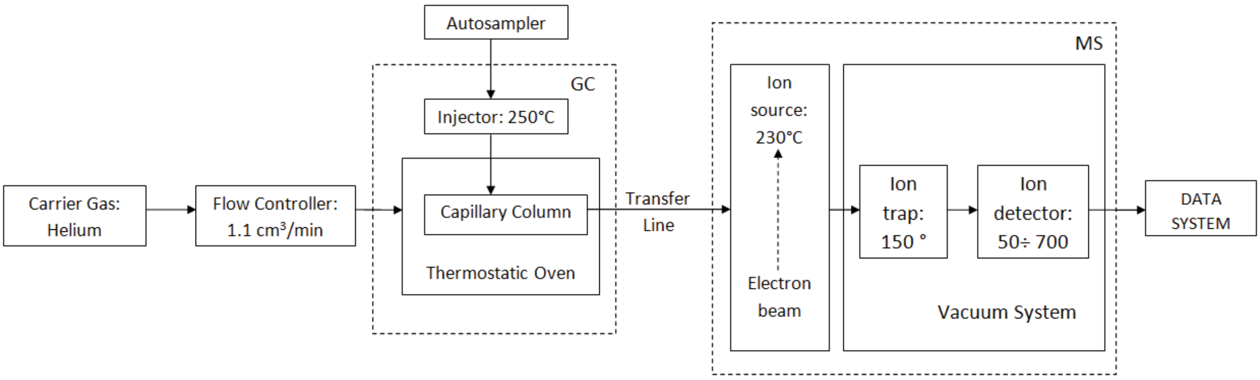


Fig. 1. The schematic diagram of the GC/MS analysis.

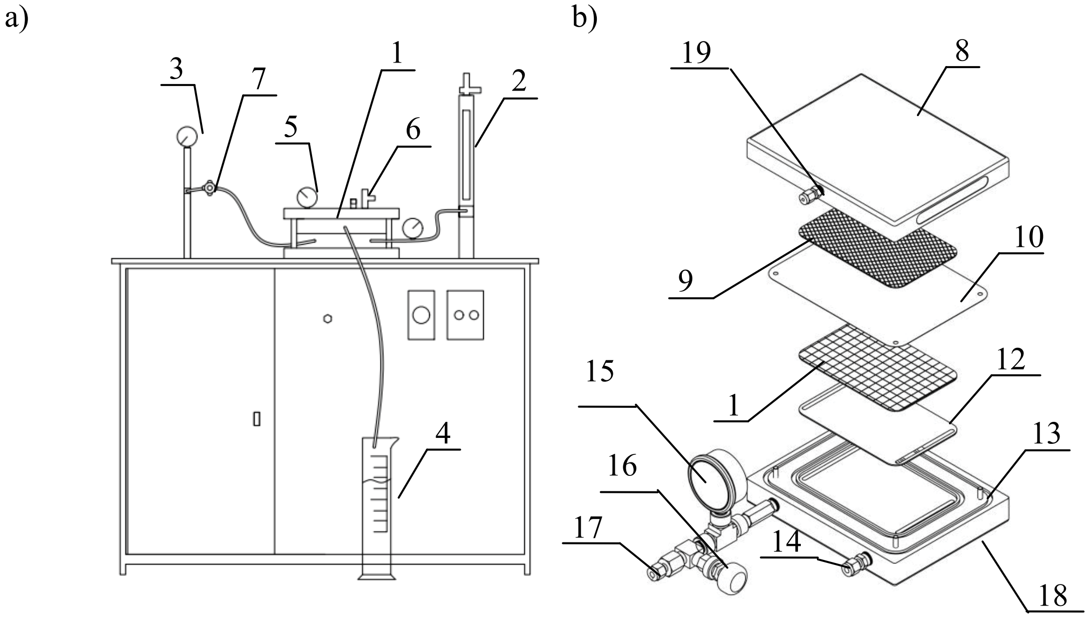


Fig. 2. (a) The installation for testing the membrane filtration process in cross-flow mode, (b) The construction of the membrane module, 1 – membrane module, 2 – rotameter, 3 – manometer, 4 – permeate reception, 5 – module pressure gauge, 6 – module pressure pump connection, 7 – concentrate feed flow control, 8 – cell body, 9 – permeate carrier, 10 – membrane NF-270, 11 – feed spacer, 12 – shim, 13 – guide post, 14 – concentrate feed inlet, 15 – concentrate pressure gauge, 16 – concentrate flow control, 17 – concentrate outlet, 18 – cell body bottom, 19 – permeate outlet.

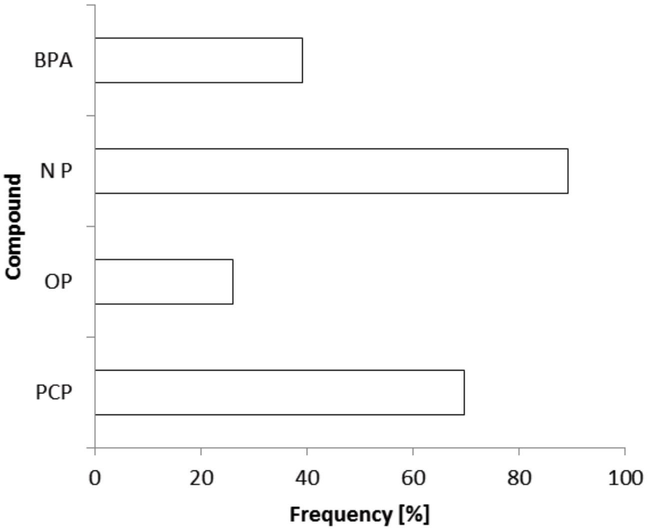


Fig. 3. The frequency of micropollutants detected in the samples from 46 swimming pools.

Table 5
The frequency of micropollutants occurrence in the pool water by venue type

Venue type (*n*)	PCP (%)	OP (%)	NP (%)	BPA (%)
Sports pool (10)	60	40	70	50
Paddling pool (8)	63	50	100	50
Recreation pool (8)	75	63	75	25
Hot tube (14)	79	64	93	36
Pool to learn to swim (4)	75	100	100	25
Hotel’s pool (4)	50	25	75	75
Outdoor pool (5)	80	100	80	40

n – the number of swimming pools classified in such venue type in these studies.

NF process has been evaluated. The retention coefficient of the investigated micropollutants depended on the type and concentration of the removed compound, the water matrix and the duration of the filtration process.

The efficiency of phenolic micropollutants removal in the NF process in the cross-flow system varied in the range of 50%–99%. The sieve mechanism had an influence on the high removal efficiency of the phenolic micropollutants tested. The molar mass of all removed compounds was greater than the molar mass limit of the used NF-270 membrane. The highest retention rate in DW, regardless of the concentration of removed compounds, was achieved for OP and not much lower for NP. Similarly for pool water, however, in this matrix, the efficiency of OP removal was slightly lower than NP. The lowest retention ratio in DW was characterized by BPA, while by PCP in SWP. For all tested compounds, the general dependence of the decrease in the retention factor in SPW was observed as compared with DW.

In order to determine the effect of the physicochemical composition of the treated water on the efficiency of phenolic micropollutants removal, according to formula (2), the relative volume permeate stream α was determined. The average values during water filtration were 0.72 for SPW and 0.63 for SPW with the addition of micropollutants, regardless of their concentration. The experimental data indicated by Rajca et al. [30] when the operating time was over 7 h, the NF-270 membrane permeability was, on the whole, stable. Relative permeate flux value obtained in paper [30] during the filtration of geothermal waters using the same, NF-270 membrane was a bit higher than obtained in this work. This shows the type of feed affects the permeate flux. Values of coefficient α below 1.0 indicate contamination of the membrane surface. There was also a gradual decrease in the hydraulic efficiency of the membrane over time, as shown in Fig. 4. At the base of this phenomenon is the formation of complexes between Ca^{2+} ions and the membrane surface, which leads to a decrease in the negative zeta potential of the membrane and at the same time limits the absorption of the compound on the surface of the membrane. This phenomenon could also have a negative effect on the removal of micropollutants during filtration.

The change in the retention coefficient of selected phenolic micropollutants on the NF-270 membrane depending on the filtration time in a 5-h cycle is presented in Fig. 5. During

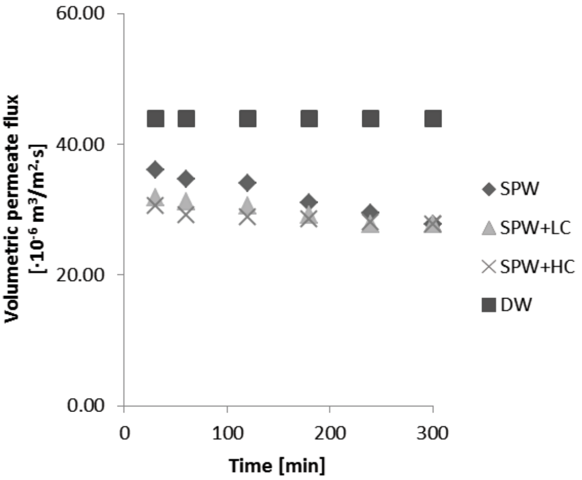


Fig. 4. The changes of the volumetric permeate flux during the nanofiltration process. SPW – swimming pool water, DW – deionized water, LC/HC – low/high concentration of micropollutants.

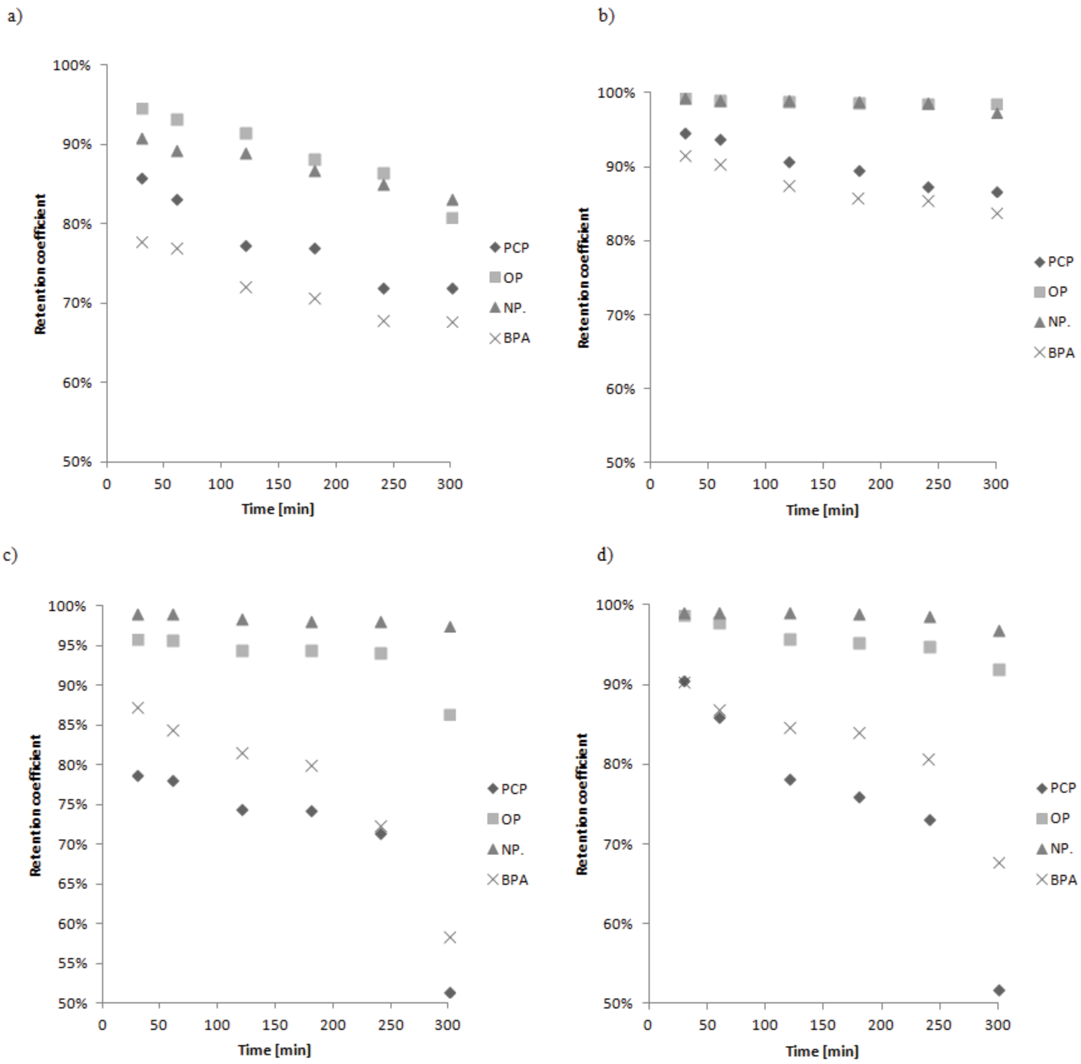


Fig. 5. The changes of the retention coefficients of PCP, OP, NP and BPA during nanofiltration: (a) LC in DW, (b) HC in DW, (c) LC in SPW and (d) HC in SPW. SPW – swimming pool water, DW – deionized water, LC/HC – low/high concentration of micropollutants.

the NF, the filtering efficiency was significantly reduced. During tests performed by Tomaszewska et al. [23], using the same, NF-270 membrane, the changes of membrane effectiveness with time were observed. The degree of the retention coefficient reduction depended on the type of compound, its concentration and matrix (Table 6). The molar mass of the removed compound had a significant influence on the observed dependence. The greatest reduction in retention occurred for PCP, which was characterized by the highest molar mass among the tested compounds. In works [31–33], it was shown that the reduction in the removal of micropollutants in the NF process results from changes in the transfer and transport mechanisms during the membrane process. Due to saturation of the surface charge of the membrane, the retention of the compound decreases, and the transport mechanism results from the sieve effect.

3.3. Improvement of general SWP quality indicators in NF

Membrane techniques can help and improve the overall quality of pool water. The membrane is an additional barrier for viruses, bacteria and protozoa. Furthermore, for example, NF is an effective method for removing water-soluble organic compounds, including precursors of water disinfection by-products. Table 7 summarizes the general quality parameters of the tested stream of SPW before and after the NF process.

Table 6
Degree of the retention coefficient reduction depending on the water matrix and the compound concentration

Compound	SPW (%)		DW (%)	
	LC	HC	LC	HC
PCP	34.6	42.7	16.15	8.30
OP	9.8	6.8	14.52	0.77
NP	1.5	2.2	8.51	1.90
BPA	33.1	24.9	12.91	8.36

SPW – swimming pool water, DW – deionized water, LC/HC – low/high concentration of micropollutants.

Table 7
Physicochemical parameters of the swimming pool water solution before and after NF

Parameter	SPW with addition of micropollutants standards	SPW after NF
Absorbance UV$_{254}$ (cm^{-1})	0.166	0.037
Conductivity (mS/cm)	2.167	1.305
pH (–)	7.55	7.43
Free chlorine concentration (mg/L)	0.5	0.17
Bound chlorine concentration (mg/L)	0.43	0.03
Total organic carbon (mg/L)	104.2	82.9
Water hardness	6.56	2.0

SPW – swimming pool water, NF – nanofiltration.

There was a significant decrease in the content of organic matter indicators, that is, the absorbance UV$_{254}$ and the TOC content. These parameters decreased by 77.7% and 20.44%, respectively. Peng in studies [34] also showed a positive effect of the NF on the reduction of the DOC and AOX (absorbable organic halogens) levels of the pool. The rejection of DOC by the membrane has been shown to be 84% ± 4%, while AOX were 95% ± 2%.

It is also worth paying attention to a significant reduction in the concentration of bound chlorine, which is a general quantitative indicator of the content of disinfection by-products in SWP. According to DIN 19643, the concentration of bound chlorine should not exceed 0.2 mg Cl_2/L [35]. Achieving this value is very troublesome for most swimming pools. Meanwhile, the applied NF process allowed to decrease this parameter by more than 93%, from 0.43 to 0.03 mg Cl_2 L, which is much below the requirements of DIN 19463. Furthermore, studies [34] showed the rejection of trihalomethane 70% ± 11%.

After the NF process, the indicators of inorganic substances in water also decreased: conductivity by 39.8% and water hardness by 70%.

4. Conclusions

- It has been documented that the occurrence of phenolic micropollutants in pool water is common, and the NF process can be used to eliminate them.
- The degree of phenol removal in the NF process exceeded 51%, depending on the water matrix, the type and parameters of pool water, the concentration of removed compounds and the duration of the filtration process.
- Research on the process of NF of pool water containing micropollutants in the cross-flow system confirms the possibility of joint removal of both organic and inorganic substances in the NF process. The conductivity of the permeate, which is an indicator of the content of inorganic substances in water, decreased by 39.8%, while the indicators of organic matter content, that is, absorbance and TOC, decreased by 77.7% and 20.44%, respectively.
- A significant decrease in the concentration of bound chlorine before and after the NF process was observed. It is worth noting that this parameter is currently the biggest problem in the exploitation of swimming pools. In many pools, it exceeds the permissible value of 0.2 mg/L. The tests have shown that the NF process can effectively reduce the concentration of bound chlorine.

Acknowledgements

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