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EFFECTIVNESS OF REMOVAL OF SELECTED BIOLOGICALLY ACTIVE MICROPOLLUTANTS IN NANOFILTRATION

OCENA SKUTECZNOŚCI USUWANIA WYBRANYCH SUBSTANCJI AKTYWNYCH BIOLOGICZNIE W PROCESIE NANOFILTRACJI

Abstract: This study addressed the removal efficiency of five different compounds classified as biologically active compounds *ie* benzo(a)pyrene (BaP), anthracene (ANT), diclofenac (DCL), pentachlorophenol (PCP), octylphenol (OP) in nanofiltration. They were removed from deionized water solution (500 $\mu g/dm^3$) and comparatively from synthetic and municipal effluent. It was found that the efficiency of the nanofiltration depends on significantly both on type of membrane and the environmental matrix and physic-chemical properties of the compounds contained in the treated feed. The highest retention was observed for benzo(a)pyrene removed from deionized water. In this case, the retention of BaP varied from 99.82% to 99.94%. For other compounds (excluding octylphenol) we observed an inverse trend, higher retention degrees were obtained when the synthetic or real effluent were filtered. This study documented a complex mechanism of separation of low molecular weight organic micropollutants in nanofiltration, which could be a result of intermolecular interactions, sieve effect and adsorption. In addition, in the last part we compare our experimental data with predicted retention coefficients, which were computed from models for predicting retention of micropollutants in nanofiltration.

Keywords: biologically active compounds, nanofiltration

Introduction

The group of biologically active substances include polycyclic aromatic hydrocarbons, pharmaceuticals, pesticides and the other substances used in industry *eg* bisphenol A and octylphenol. Their negative impact on living organisms has been repeatedly documented [1–5]. Among individuals exposed to toxic substances we can observe, aside the lethal effects, growth and development disruptions or hormonal irregularities [6–8].

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At the same time, many of these substances have been identified by European Parliament in Water Framework Directive as particularly dangerous and priority. Initially 33 compounds were designated as particularly dangerous, among which were octylphenol, pentachlorophenol, anthracene and many others [9]. Currently the list of priority substances or priority hazardous has been extended with 12 new compounds [10]. Moreover, to ensure good chemical state of surface waters, for some of these substances environmental quality standards were established, which should be achieved by the end of 2021, and for the 33 priority substances and by the end of 2027, for newly identified compounds [11].

In this light, it is advisable to conduct research to enhance cheap and effective methods of micropollutants removal from effluent waters. Process that guarantees effective separation of low molecular weight organic compounds is nanofiltration (NF) [12–15]. Thanks to using compact nanofiltration membranes with pore size in active layer usually not exceeding 2 nm, retained compounds have the molecular weight in the range of 150–500 Da [16]. In addition, nanofiltration membrane surface is additionally charged. Therefore, mechanism of separation and mass transport in NF is complex and results from occurring various effects and processes in filtration. Separation mechanism can therefore be based on both the molecular sieve effect, which is typical for ultrafiltration, as well as diffusion and dilution effects occurring mainly in reverse osmosis process or an electrostatic interaction and adsorption [15–16]. Physicochemical properties of membrane and separated pollutants decide which mechanism is dominant. They determine in direct way the type and strength of interactions between membrane surface and substances contained in feed [17].

Knowledge of micropollutants separation mechanisms in nanofiltration process has become a basis for developing retention models. They allow in very high accuracy predict the retention of particular feed ingredients. One of the simplest and earliest method of forecasting retention coefficients is diagram proposed in work [18]. Depending on the membrane properties and pollutants, authors presented a method of approximating retention coefficient of pollutants in high pressure membrane techniques. It shows that in the first place you should take into account the molecular weight of the compound and the molecular weight cut off (MWCO) of membrane, followed by the pKa of compound and pH of the feed. It should also be considered that the degree of removal of the compound is dependent on hydrophobic nature and the size ratio of diameter of retained particles into the membrane pore size. Newer micropollutants retention models are based on statistical analyses, allowing more accurately identify the most important factors affecting their retention. In work [19] derived equation that allows to calculate the retention of organic micropollutants in NF process according to value of the log D and geometry of the molecule, and the retention degree of the divalent ions. Similar results were shown by the research of the retention of estrogenic compounds. In this case, the variables in equation allowing to estimate the retention size were: molecular weight of the compound, the retention coefficient of NaCl and absorbance value of treated water [17].

The purpose of presented study was to determine the efficiency of nanofiltration process in removal of biologically active substances of various origin (PAHs, pesticides, EDCs). It dealt with the influence of membrane nanofiltration type and the aqueous matrix on the efficiency of their removal. In the second part, obtained results were used for validation of the micropollutants retention models available in the literature.

Materials and methods

Chemicals

Chemical standards of benzo(a)pyrene (BaP), anthracene (ANT), (2), diclofenac (DCL), pentachlorophenol (PCP), octylphenol (OP) were supplied by Sigma Aldrich. Stock solution of individual standards (1 mg/cm^3) were prepared in methanol for PCP, OP and DCL or acetone for BaP and ANT. The structural and physicochemical properties of selected micropollutants are shown in Table 1.

Table 1

Physicochemical properties of selected biologically active substances

Compound	Molecular weight[a] [g/mole]	Log K_{ow}[b] [-]	Length[b] [nm]	Width[b] [nm]	Depth[b] [nm]	Eqwidth[b] [nm]	Log D[d] [-]
Pentachlorophenol (PCP)	266.34	4.40	0.59	0.55	0.15	0.28	2.45
4-tert-octylphenol (OP)	206.32	4.12	0.87	0.79	0.40	0.56	5.47
Diclofenac (DCL)	296.15	4.51	0.96	0.90	0.26	0.48	1.37
Benzo(a)pyrene (BaP)	252.31	6.35	1.10	0.78	0.06	0.23	6.35
Anthracene (ANT)	178.22	4.45	0.90	0.51	0.39	0.49	4.68

[a] https://pubchem.ncbi.nlm.nih.gov/compound/2336; [b] calculated with ChemBio3D Ultra 12.0; [c] geometric mean of width and depth; [d] ACD/Labs Percepta Platform.

The concentration of selected biologically active compounds was determined using solid phase extraction (SPE) and HPLC analysis at a wavelength of λ: 220 nm (for PCP, DCL, OP), 254 nm for ANT and 250 nm for BaP. For SPE, glass columns filled with C18 phase (Supelco) were used.

Preparation of feeds

To investigate the retention of selected biologically active compounds from aquatic solutions, artificial solutions made of deionized water were prepared. Comparatively, synthetic and real effluents were used. Synthetic effluent was prepared by diluting in the tap water the following organic substances: (0.152 g/dm^3 of broth; 0.226 g/dm^3 of peptone) and inorganic substances (0.007 g/dm^3 of NH_4Cl; 0.0075 g/dm^3 of NaCl; 0.002 g/dm^3 of $CaCl \cdot 6H_2O$; 0.04 g/dm^3 of $MgSO_4 \cdot 7H_2O$; 0.016 g/dm^3 of K_2HPO_4; 0.04 g/dm^3 of KH_2PO_4) and then solution was vaccinated (1 cm^3/dm^3) with surface water containing natural bacteria. Finally, obtained solution was aerated for 5 days in order to guarantee a biodegradation of high molecular weight compounds. In all types of feed concentration of micropollutants was maintained at constant level of 500 μg/dm^3 by adding sufficient volume of stock solutions.

Membranes and filtration run

Nanofiltration was carried out in a membrane cell equipped with a magnetic stirrer (volume 0.4 dm^3, membrane filtration area 0.00385 m^2), operating in a dead-end mode at the transmembrane pressure 2 MPa. Prior to the first application, the membranes were conditioned by means of filtration of deionized water. Setup used in nanofiltration is illustrated in Fig. 1.

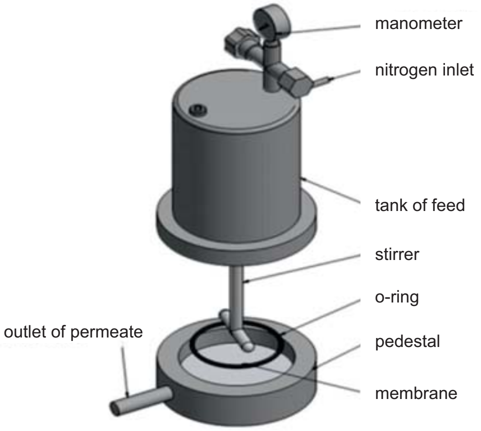

Fig. 1. Dead-end nanofiltration unit

Four types of commercial nanofiltration membranes were used in nanofiltration process. Their properties are shown in Table 2.

Table 2

Characteristics of nanofiltration membranes

Symbol	DK	HL	NF-90	NF-270
Manufacturer	GE	GE	Dow Filmtec	Dow Filmtec
Material[a]	Thin film			
MWCO[b] [Da]	150–300	150–300	150	200
Salt retention – NaCl[b] [%]	63	44	47	87
Salt retention – $MgSO_4$[2b] [%]	94	98	97	99
Contact angle[c] [°]	37	25	63	17
Volume deionized water flux[d], $J_v \cdot 10^6$ [$m^3 \cdot m^{-2} \cdot s^{-1}$]	16.47	42.42	47.03	33.28

[a] Data provided by manufacturer; [b] own measurements: NaCl and $MgSO_4$ of 1 g/dm^3 at ΔP = 2 MPa; [c] own measurements by means of goniometer; [d] own measurements at ΔP = 2 MPa.

Before nanofiltration, membranes were stored in deionized water for 24 h, then they were conditioned with deionized water. After that, initial deionized water flux (J_w) was measured. During nanofiltration, the volume of permeate was measured and then permeate flux (J_v) and volume reduction factor (*VRF*) were computed according to equation 1 and 2 respectively. Fouling behavior was described by means of relative permeate flux from equation 3.

$$J_{v/(w)} = \frac{V_p}{S \cdot t} \tag{1}$$

$$VRF = \frac{V_p}{V_n} \cdot 100\% \tag{2}$$

$$\alpha_v = \frac{J_v}{J_w} \tag{3}$$

where: $J_v/_{(w)}$ – permeate/deionized water flux;
VRF – volume reduction factor;
α_v – relative volume permeate flux;
S – membrane area;
t – time of permeate collection;
V_p; V_n – volume of permeate and feed respectively.

External validation of models for predicting retention of micropollutants by nanofiltration membranes

In the last stage of this study, the experimental data were used as data set for external validation of available in literature models for predicting retention of micropollutants by nanofiltration membranes. The first model, designated as M1, assumes that the retention of micropollutants should be predicted with molecular weight of compounds, NaCl retention coefficient and a certain indicators describing physicochemical properties of feed. The equation of this model was the following [16]:

$$R = 42.894 + 0.083\ M_w + 0.193\ SR_{\mathrm{NaCl}} + 74.120\ ABS \tag{4}$$

where: M_w – molecular mass;
SR_{NaCl} – sodium chloride retention;
ABS – absorbance (UV_{254}) was at the level of 0.0; 0.061 and 0.218 for deionized water, synthetic effluent and real effluent respectively.

In the second model (M2 symbol), retention was predicted by means of geometrical dimensions of molecule, hydrophobic-hydrophilic properties of micropollutants and membrane *MWCO*. The equation of M2 retention model is written as [20]:

$$R = 265.150\ \mathrm{eqwidth} - 117.356\ \mathrm{depth} + 81.662\ \mathrm{length} - 5.229\ \log D - 0.272\ MWCO - 62.565 \tag{5}$$

where: *MWCO* – molecular weight cut off of membrane – when *MWCO* is between 150–300 Da, proper value is average *ie* 225 Da [20].

Fitting of models to experimental data were determined by means of mean relative estimation error according to the equation:

$$MRE = \frac{1}{n}\sum_{t=1}^{n}\left|\frac{y_t - y_p}{y_t}\right| \cdot 100\% \qquad (6)$$

where: MRE – mean relative estimation error;
n – number of samples;
y_t – experimental value of retention;
y_p – estimated value of retention.

In addition, strength of relationship between a certain parameters used for prediction of retention and experimental retention coefficient was determined. This was done by calculation of correlation coefficient according to equation 7.

In that, we could explain the discrepancy between the existing experimental retention coefficients and computed from M1 and M2 models.

$$r = \frac{\sum_{t=1}^{n}(x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum_{i=1}^{n}(x_i - \bar{x})^2 \sum_{i=1}^{n}(y_i - \bar{y})^2}} \qquad (7)$$

where: x_i, y_i – values of variables X and Y respectively for i observations;
n – numer of observations;
$\bar{x}, \bar{y}$ – arithmetic mean for observed values of each variable.

Results and discussion

Effect of membrane type on micropollutants removal and nanofiltration performance

Effectiveness of nanofiltration in micropollutants removal from artificial solution of deionized water is presented in Fig. 2. Retention coefficients of anthracene and benzo(a)pyrene indicated almost complete their removal for all tested membranes. That can be explained by very hydrophobic properties of PAHs, normally described by log K_{ow}. Retention of PCP, DCL and OP was more dependent on membrane type. Their retention coefficients were in the range 75.9–92.3%; 89.9–98.9% and 21.4–96.9% for pentachlorophenol, octylphenol and diclofenac. The highest retention was obtained with HL and NF-90 membranes. Different separation properties of tested nanofiltration membranes are probably caused by their different hydrophilic-hydrophobic properties, which are determined by contact angle. The higher contact angle is, the more hydrophobic is membrane and more intensive adsorption of pollutants on membrane surface. In many studies, relationship between hydrophobicity of membranes and degree of adsorption of micropollutants during nanofiltration was proved [16, 21, 22].

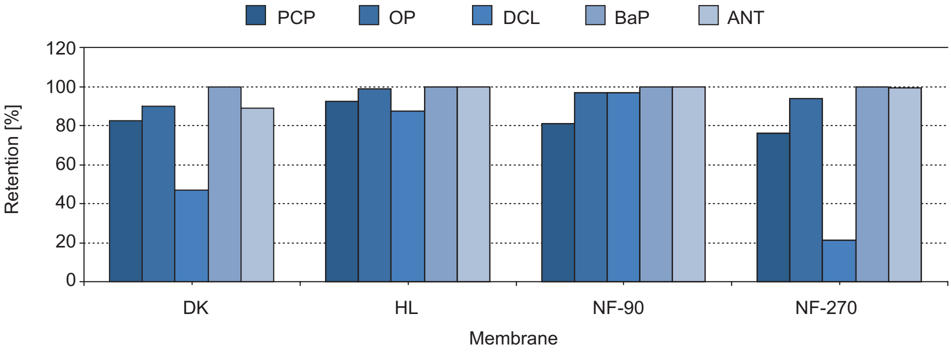

Fig. 2. Impact of membrane on retention of micropollutants (feed: deionized water)

Therefore, adsorption is considered as one of mechanisms of separation in high pressure membrane techniques [18, 23]. Take into account, effect of membranes type and their properties on effectiveness of micropollutants removal, normally we should consider also *MWCO* of membranes. However, due to similar value of *MWCO* of tested NF membranes we assume that sieve effect in separation mechanism of micropollutants was comparable in that case.

Figure 3 shows effect of volume reduction factor on relative permeate flux during nanofiltration of artificial solution of deionized water for all tested membranes. It was found, that relative permeate flux slightly decreased with increase in VRF. α_v was in the range from 0.89 to 1.05. It means that, solution of deionized water with micropollutants as a feed did not cause significant fouling of nanofiltration membranes.

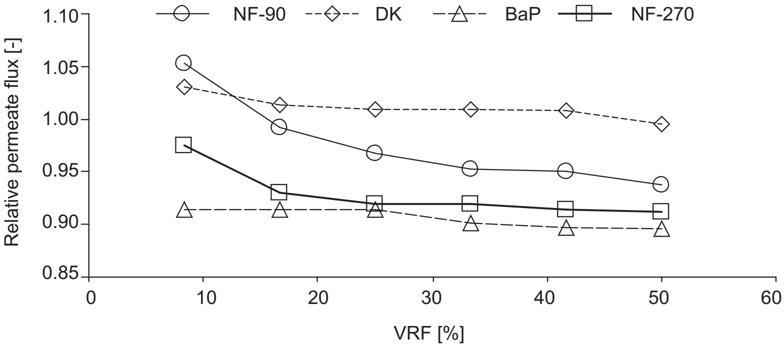

Fig. 3. Variation of relative permeate flux versus with VRF during the filtration of artificial solution of deionized water containing selected biologically active compounds

Effect of the water matrix on micropollutants removal and nanofiltration performance

Type of feed affected also the effectiveness of micropollutants removal in nanofiltration (Fig. 4). Retention coefficients of PCP for synthetic effluent were around 12%

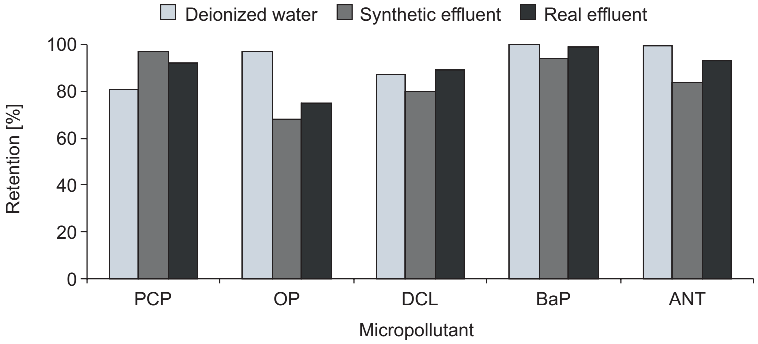

Fig. 4. Comparison of effectiveness of micropollutants removal from deionized water, synthetic and real effluents

and 17% higher than for artificial deionized water solution containing micropollutants. Opposite tendency was observed for OP, DCL, BaP and ANT. Effectiveness of removal of these micropollutants was the highest for artificial solution of deionized water and the lowest for synthetic effluent. Intervening effects were obtained while removal of micropollutants from synthetic effluent. These results indicate complex and dependent on many factors separation mechanisms of low molecular weight organic compounds in nanofiltration. Substances and pollutants contained in artificial and real effluent formed a filtration cake, that can be consider as additional separation layer – secondary membrane, enhancing removal of micropollutants. This effect was probably a reason of higher removal of pentachlorophenol from effluents than from artificial solution of deionized water. However, this effect did not affect the retention of OP, DCL, BaP and ANT. In case of the latter compounds, dominant mechanisms of separation could be adsorption. For effluent samples, adsorption of micropollutants was lower due to other organic and inorganic compounds normally present in wastewater. They could preferentially occupy active sorption sites on the membrane surface.

Taking onto consideration effect of feed type on nanofiltration performance (NF-90 membrane), it was found that a reduction of permeate flux versus increasing VRF was the lowest for filtration of deionized water solution and the highest for real effluent (Fig. 5a). In initial phase of nanofiltration of real effluent, permeate flux was around

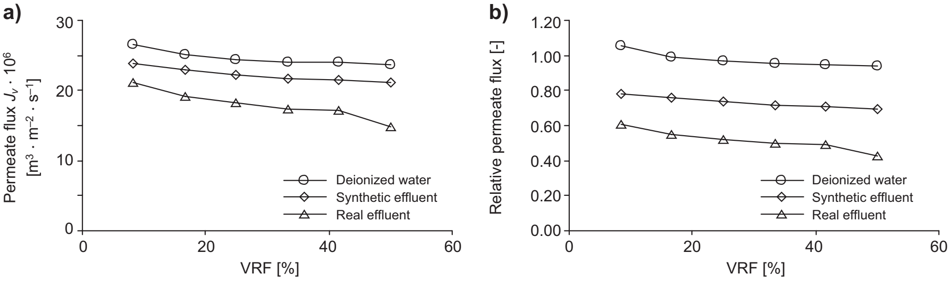

Fig. 5. Dependence of a) volume permeate flux and b) relative volume permeate flux versus VRF

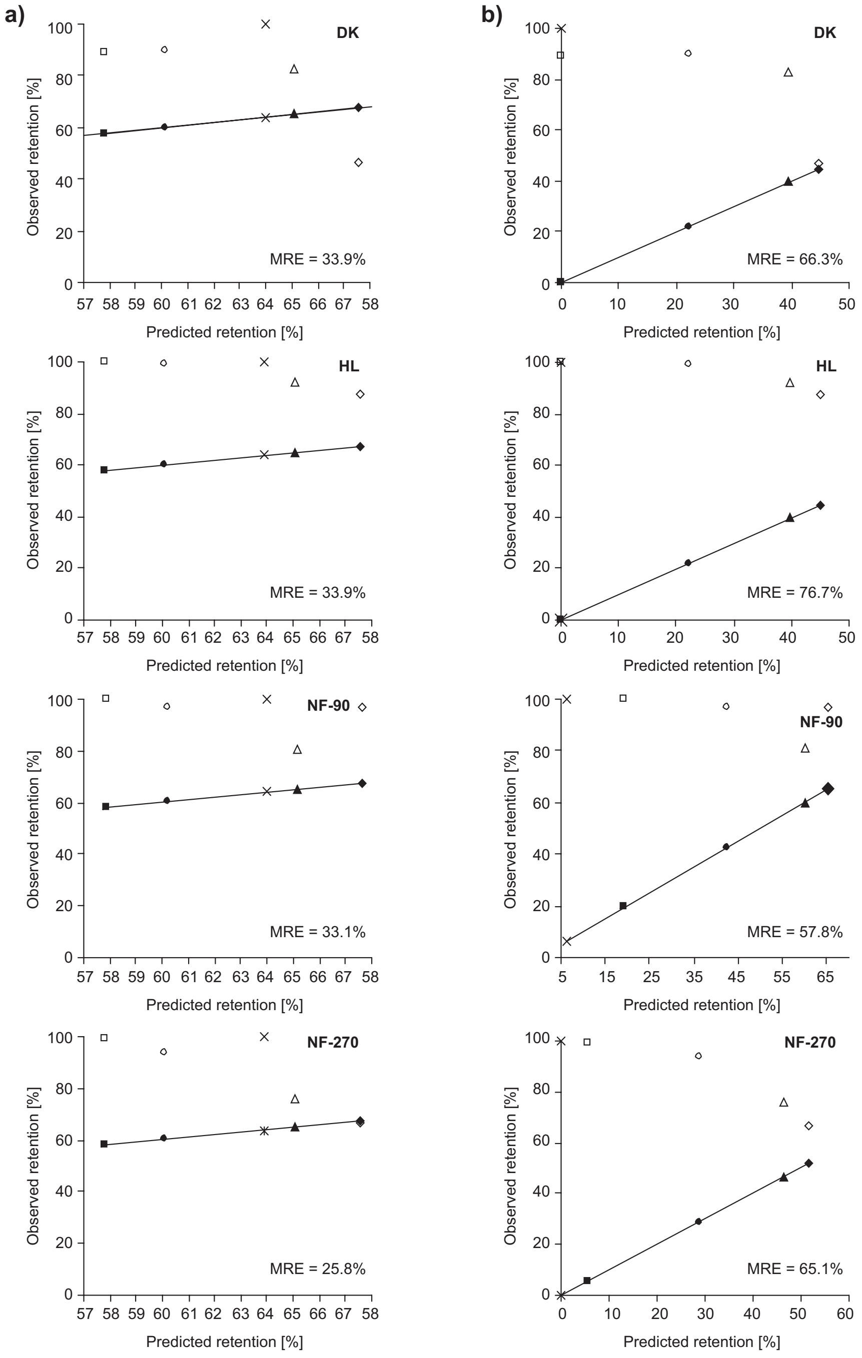

Fig. 6. Observed retention versus predicted retention computed from models: a) M1 and b) M2 (feed: deionized water with Δ – PCP, ○ – OP, ◇ – DCL, X – BaP, ☐ – ANT

20% lower in comparison to permeate flux for filtration of deionized water solution. Thus, organic and inorganic pollutants present in real and synthetic effluents caused more intensive fouling of NF-90 membrane. This is confirmed also by the values of relative permeate flux (Fig. 5b), which in case of more intensive coating the membrane surface with a layer of pollutants take significantly lower values.

Prediction of biologically active substances retention in nanofiltration based on mathematical models and statistical analysis

Relationship between observed and predicted retention coefficients of biologically active substances is presented in Fig. 6a and 6b. Based on MRE parameters, it was found that M1 model predicted the retention of micropollutants more precise than M2 model. More specifically, divergence between experimental and predicted retention coefficient computed from M1 model were in the range from 25% to 33%. While, computed from M2 model retention coefficients deviated from experimental data in the range of 57–76%.

Moreover, computed from M1 model retention coefficients were very similar to experimental data (observed retention coefficients) for synthetic and real effluents (Fig. 7). Precision of M1 model, described by MRE parameter reached 11 and 19% for synthetic and real effluent respectively. For comparison, for M2 model, MRE parameters were equaled 53 and 55% respectively.

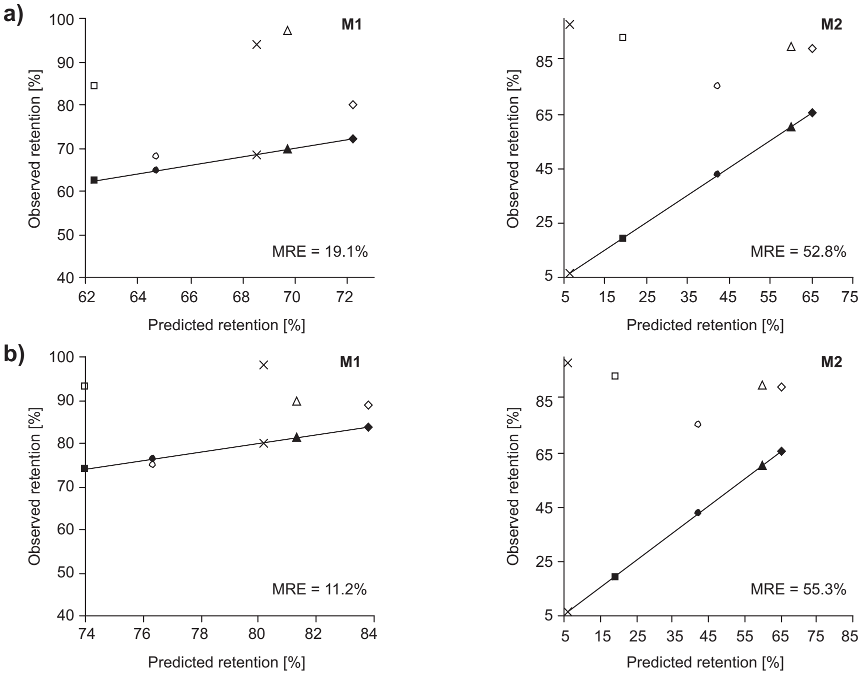

Fig. 7. Observed retention versus predicted retention for nanofiltration (membrane NF90) of a) synthetic and b) real effluents (with Δ – PCP, ○ – OP, ◇ – DCL, X – BaP, □ – ANT)

As was mentioned, computed from M1 model retention coefficients predicted retention better than M2 model for all considered feed types. Thus, retention of biologically active compounds was conditioned by molecular weight and also potential of membrane for monovalent ions separation and organics content in the feed. Mechanisms of micropollutants separation include not only sieve effect, dependent on size of micropollutants molecule and size of membrane pores, but also a few phenomena accompanying membrane filtration *eg* intermolecular interactions occurring between different feed ingredients. Potential of membrane for divalent ions retention (usually reached 96–99%) as well as parameters describing geometrical dimensions of compounds seem to be less important factors for predicting the micropollutants retention. Moreover, we did not observe positive correlation between parameters such as length, width and eqwidth of molecule and retention coefficients (Table 3).

Table 3

Correlation coefficient between retention and chosen structural and physicochemical parameters of micropollutants (deionized water)

Membrane	Length [nm]	Width [nm]	Eqwidth [nm]	log K_{ow} [-]
DK	0.085	–0.45	–0.37	0.40
HL	0.33	–0.33	0.51	0.32
NF90	0.91	0.40	0.33	0.34
NF270	0.42	–0.3	0.16	0.39

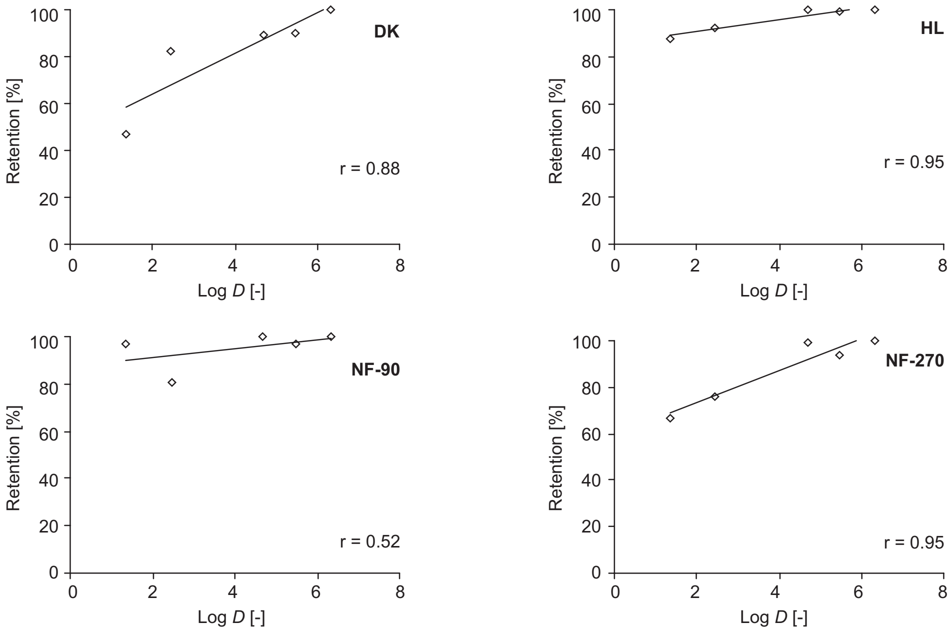

Fig. 8. Dependence of retention coefficients and log D of compounds (deionized water)

It was found that, the reason of slight divergences between computed from M1 model retention coefficients and experimental retention coefficients may be caused by omitted impact of adsorption of compounds on membrane surface during nanofiltration. It is confirmed by shown in Fig. 8 positive linear correlation between log D parameter (logarithm of the distribution coefficient (D) at a selected pH, assumed to be measured in octanol), and retention coefficients of biologically active substances.

Conclusions

Based on the carried out experiments can be concluded that:

– In nanofiltration of deionized water, retention of micropollutants was in the range from 21.5% to 99.82%. The removal efficiency of biologically active substances depends on the type of the nanofiltration membrane. The best results were obtained using the membrane of the symbols HL and NF-90.

– The highest removal efficiency was obtained for anthracene and benzo(a)pyrene, and the lowest for diclofenac. This effect could be due to a more hydrophobic nature of PAHs.

– Comparing the efficiency of removal of biologically active substances from deionized water and effluent – it has been found that the retention of all the compounds apart from PCP was higher during the filtration of deionized water than the effluent samples.

– The results confirm that the separation mechanism in the nanofiltration process is complex and dependent on both the properties of the membrane and separated material as well as the feed type.

– It was found that contaminants contained in synthetic and real wastewater caused significant fouling of the membrane NF-90. The observed reduction in the permeate flux ranged from 20% (initial phase of filtration) to 40% (the end of filtration) of the values obtained in the nanofiltration of deionized water.

– Model based on molecular weight of molecule and absorbance of feed as well as membrane potential for sodium chloride separation (M1 model) predicted well retention of biologically active substances with different properties. In comparison model based on potential of membrane for divalent ions retention and parameters describing geometrical dimensions of compounds (M2 model) was not applicable to predict retention selected in this study micropollutants.

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OCENA SKUTECZNOŚCI USUWANIA WYBRANYCH SUBSTANCJI AKTYWNYCH BIOLOGICZNIE W PROCESIE NANOFILTRACJI

Instytut Inżynierii Wody i Ścieków
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Abstrakt: W ramach pracy podjęto badania nad oceną skuteczności usuwania pięciu różnych związków należących do grupy substancji aktywnych biologicznie, tj. benzo(a)piren (BaP), antracen (ANT), diklofenak

(DCL), pentachlorofenol (PCP) i oktylofenol (OP) w procesie nanofiltracji. Przedmiotem badań były modelowe roztwory tych substancji o stężeniu 500 μg/dm^3 wykonane na bazie wody zdejonizowanej. Uzyskane wyniki badań porównano pod kątem skuteczności usuwania wybranych związków z syntetycznych i rzeczywistych odpływów z komunalnej oczyszczalni ścieków. Wykazano, że na skuteczność procesu nanofiltracji istotny wpływ ma rodzaj membrany nanofiltracyjnej, właściwości fizykochemiczne usuwanych związków, jak również rodzaj matrycy środowiskowej poddawanej oczyszczaniu. Najwyższą efektywność usuwania zaobserwowano dla benzopirenu w trakcie nanofiltracji wody zdejonizowanej. Współczynniki retencji wynosiły wówczas od 99,82% do 99,94%, co oznacza praktycznie jego całkowite usunięcie. Z kolei dla pozostałych związków z wyjątkiem oktylofenolu zaobserwowano odwrotną tendencję, wyższe współczynniki retencji uzyskano, gdy filtrowanym medium były ścieki syntetyczne lub rzeczywiste. Przeprowadzone badania udokumentowały złożony mechanizm separacji małocząsteczkowych mikrozanieczyszczeń organicznych w procesie nanofiltracji wynikający m.in. z oddziaływań międzycząsteczkowych, efektu sitowego, jak i adsorpcji. Dodatkowo, w ostatniej części pracy porównano uzyskane dane doświadczalne z przewidywanymi współczynnikami retencji, które zostały obliczone z modeli dotyczących przewidywania retencji mikrozanieczyszczeń w procesie nanofiltracji.

Słowa kluczowe: związki aktywne biologicznie, nanofiltracja