Forward osmosis as an effective concentration method for cationic surfactant solutions

Osmoza prosta jako skuteczna metoda zatężania roztworów kationowych substancji powierzchniowo czynnych

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Keywords: circular economy, quaternary-ammonium compound, membrane separation process, surfactant

Abstract

The paper presents experimental results on the effectiveness of forward osmosis (FO) for the recovery and concentration of cationic surfactants (quaternary ammonium salts) from solutions with a wide range of concentrations (50-1000 mg/L). The membrane process ensured an almost 100% separation of cationic surfactants and an effective concentration of the solution components without a leakage into the receiving solution. It has been shown that FO is a membrane technique that allows achieving the goals of a circular economy through an effective concentration and recovery of the compounds and water from the purified solutions.

Słowa kluczowe: gospodarka o obiegu zamkniętym, czwartorzędowy związek amoniowy, proces membranowy, substancja powierzchniowo czynna

Streszczenie

W pracy przedstawiono wyniki badań dotyczących skuteczności osmozy prostej (FO) do odzyskiwania i zatężania kationowych substancji powierzchniowo czynnych (czwartorzędowych soli amoniowych) z roztworów o szerokim zakresie stężeń (50-1000 mg/L). Proces membranowy zapewniał niemal 100% separację kationowych substancji powierzchniowo czynnych oraz skuteczne zatężenie składników roztworu bez ich przecieku do roztworu odbierającego. Wykazano, że FO jest techniką membranową pozwalająca na osiągnięcie celów gospodarki o obiegu zamkniętym, poprzez skuteczne zatężanie oraz odzyskiwanie składników oczyszczanego roztworu i wody.

Introduction

Industrial production in the 21st century is increasingly focused on implementing a circular economy. The goal is not only to treat wastewater but also to reuse it [22]. Post-process streams contain water and valuable components that can be recovered and utilized as substrates or intermediate products in subsequent technological processes.

Surface active agents (surfactants) represent a particular type of anthropogenic pollutants. They find applications in a wide range of industrial sectors, as well as consumer markets. Surfactants are being applied in detergents, cosmetics, drugs, pesticides, and others [30, 38]. The wide range of applications results from the specific chemical structure of surfactant molecules, which consists of a hydrophilic and a hydrophobic part which enables surfactants to solubilize both in polar and nonpolar liquids. Depending on the concentration, surfactants can exist in monomeric and aggregated forms in solution. The concentration at which monomers begin to form micellar aggregates is known as the critical micelle concentration (CMC). The diameter of the micelle is usually twice the length of the monomer, and the aggregated form usually consists of 50-200 molecules [20, 29, 35]. Cationic surfactants (CS) are commonly used as disinfectants. The primary group of cationic surfactants comprises quaternary ammonium compounds (QAC). The most common QAC disinfectants are benzalkonium choride (BAC), cetylpyridinium chloride (CPC), and didodecyldimethylammonium chloride/bromide [24]. BAC is utilized in hand sanitizers, soaps, cleaning wipes, hospital sanitation kits, surface disinfectants, etc. Moreover, BAC is used to protect against the SARS-CoV-2 virus (the deactivation effect is achieved at a concentration of 0.2% BAC and an exposure time of 15 seconds) [34].

The physical properties of surfactants resulting from their surface activity cause a potentially negative impact on the ecosystem (natural waters or soil). Cationic surfactants, specifically QACs, induce a disruption of the nitrification process at a concentration of 2 mg/L [43] and partially inhibit heterotrophic bacteria responsible for denitrification at concentrations greater than 50 mg/L [16]. Therefore, the treatment of concentrated wastewater containing QACs is a challenge.

The removal of surfactants from aqueous solutions can be achieved in processes such as coagulation [26, 33], adsorption [6, 8], ion exchange [13], and chemical oxidation [3, 32]. Treatment of

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Table 1. Retention of cationic surfactants from water solutions in pressure-driven membrane processes

Tabela 1. Skuteczność separacji kationowych substancji powierzchniowo czynnych w ciśnieniowych procesach membranowych

CS name	CS concentration in the feed as CMC ¹⁾ , mg/L	Membrane material	Membrane type	MWCO ²⁾ , kDa	CS retention, %	Reference				
Nanofiltration										
BAC	0.2	polyamide	AFC40	0.3	9	[28]				
	0.7				62					
	3				55					
СТАВ	0.2				84					
	0.7				60					
	3				85					
Cetrimide	0.03	polyamide	Desal 51HL	0.19	97	[10]				
	0.03	polyamide	NF270	0.155	89					
	0.03	polyethersulfone	NFPES10	0.12	21	[10]				
	0.03	modified polyethersulfone	NTR 7450	0.31	17					
Ultrafiltration										
ODA	3	polyethersulfone	UP004	4	83	[15]				
	3	cellulose	UC005	5	82					
CPC	3	polyethersulfone	UP004	4	80					
	3	cellulose	UC005	5	80					
BAC	0.2	polyethersulfone	ESP04	4	98	[28]				
	0.7				84					
	3				75					
	3				85					

¹⁾ CMC values: BAC = 350 mg/L, CTAB = 369 mg/L, Cetrimide = 1320 mg/L, ODA = 297 mg/L, CPC = 322 mg/L

²⁾ Molecular weight cut-off – defined as the minimum molecular weight of a solute that is 90% retained by the membrane

surfactant solutions can also be performed in membrane processes [5, 25], which not only provide efficient pollutant separation, but also allow the concentration and simultaneous recovery of both water and purified solution components. Pressure-driven membrane processes (PDMP) have gained increasing popularity in the water and wastewater treatment area, as well as many other industrial applications [21]. A review of the literature has shown that the efficiency of CS removal in these processes depends on the type and concentration of the surfactant present in the solution, as well as the parameters of the applied membranes [10, 15, 28]. The separation of surfactants in PMDPs, although high, remains incomplete (Table 1). Even the use of dense nanofiltration membranes did not allow 100% surfactant removal.

An alternative membrane technology to PDMPs for wastewater treatment and water recovery is forward osmosis (FO). In FO, the movement of the solvent through a semipermeable membrane is forced by an osmotic pressure gradient without the need for any external force [2]. Elimination of hydraulic pressure leads to less energy consumption during the process compared to PDMP [4]. Due to the nature of the process, a highly concentrated solution 'draws' water from a slightly concentrated solution, which 'feeds' water into the process [31, 46]. During the FO process, the draw solution is being diluted. Mass transport continues until the osmotic pressures of solutions on both sides of the membrane equalizes (Fig. 1).

According to Bhinder et al. [7], the FO process is not only more economically feasible but also less prone to fouling compared to pressure-driven nanofiltration (NF) and reverse osmosis (RO) processes. However, the FO process can result in an unfavourable phenomenon known as internal concentration polarization (ICP), which occurs because of enhanced concentration polarization inside the support layer, where the solvent (usually water) permeates through the support and dilutes the draw concentration at the inner side of



Fig. 1. Schematic diagram of forward osmosis Rys. 1. Schemat działania osmozy prostej

the active layer. This can lead to low water permeation, poor salt rejection, and membrane hydrolysis [39].

Although the FO process has been extensively discussed in the context of industrial wastewater treatment [18, 19, 44], the concentration of landfill leachate [23], food processing in the food industry [17, 37], and the desalination of saline and brackish water [1, 9, 11, 14], the data on surfactant removal are very limited.

The removal of surface active agents in the FO process using a thin-film composite membrane (TFC) was described in [45]. Model solutions containing sodium dodecylbenzenesulfonate (SDBS) at concentrations of 1, 5, and 10 mg/L and sodium chloride (NaCl) at a concentration of 10 mM were used in the study. By employing a DS solution with a chloride concentration comparable to that of seawater, that is, 35,000 mg/L, retention coefficients of SDBS above 99.75% were achieved across the entire range of investigated concentrations.

Laboratory set-up

The laboratory cross-flow filtration set-up (Fig. 2) was equipped with the SEPA module (Sterlitech, USA), two process solutions tanks (4 L volumes of each), pumps, and other equipment. Flatsheet cellulose triacetate (CTA) membranes (effective area 0.014 m^2) FTS H₂O (Sterlitech, USA) with an active layer towards the feed solution were used in the experiments. Forward osmosis was performed at atmospheric pressure and linear flow velocity at the membrane surface v=0.2 m/s. Cole-Palmer peristaltic pumps (USA) were used to force the flow of process solutions. To monitor changes in the mass of the process solutions, the system was equipped with two digital scales (Isolab, Archem, Poland).



Fig. 2. Laboratory set-up: 1 – feed solution (FS) tank; 2 – FS pump; 3, 10 – manometer; 4, 11 – valve; 5, 12 – rotameter; 6 – scale; 7 – SEPA FO module; 8 – draw solution (DS) tank; 9 – DS pump

Rys. 2. Instalacja laboratoryjna: 1 – zbiornik roztworu zasilającego (FS); 2 – pompa roztwory zasilającego; 3, 10 – manometr; 4, 11 – zawór; 5, 12 – rotametr; 6 – stanowisko wagowe; 7 – moduł SEPA FO; 8 – zbiornik roztworu odbierającego (DS); 9 – pompa roztworu odbierającego

Table 2. Characteristics of cationic surfactants [27, 40, 41]

Tabela 2. Charakterystyka kationowych substancji powierzchniowo czynnych [27, 40, 41]

Benzalkonium chloride (BAC) Cetrimonium bromide (CTAB) Name Molecular weight, Da 339.5-396.5 364.5 Туре Cationic, quaternary ammonium compound 80 96 Purity. % a mixture of homologues C12-C16 Carbon atoms in chain 10 appr. ratios: C12 (40%), C14 (50%), C16 (10%) CH₂ C_nH_{2n+1} Structural formula CH Critical micelle concentration (CMC), 368 7+131 4 350+5ma/L Micelle size distribution, nm 11.8±1.0 6.5±0.5

Feed solutions (FS)

Two cationic surfactants (quaternary ammonium compounds) were used in the tests: benzalkonium chloride (BAC, purity 80%, MP Biomedicals, USA) and cetrimonium bromide (CTAB, purity 96%, Sigma-Aldrich, USA). The basis for the selection of these compounds was the widespread use in commercially available disinfectants, biocides, and household products, which is related to their large production, and thus with significant negative impact of industrial effluents on the natural environment. The physical properties of the surfactants (critical micelle concentration and micelle size distribution) were determined by dynamic light scattering (Zetasizer Nano ZS, wavelength 532 nm, Malvern, UK). A summary of the characteristics of the surfactants is presented in Table 2. The concentration of surfactants in the FS was 50, 100, 250, 500 and 1000 mg/L to ensure the presence of both surfactant monomers and micelles. The temperature of the FS was 22°C.

Draw solutions (DS)

An initial volume of a draw solution was 3 L, and its temperature was 22°C. Sodium chloride solutions (NaCl, 99,9% purity, 58,44 g/mol, POCH, Poland) with a concentration of 0.5, 1 and 3 mol/L were used in the tests. The osmotic pressure of the draw solutions was equal to 24.5, 48.9 and 146.8 atm, respectively.

Analysis

During the tests, FS and DS samples (20 mL) were taken for surfactant and chloride concentration and conductivity analyses. BAC concentration was determined using spectrophotometric measurements (UV Mini 1240 spectrophotometer, Shimadzu, Japan). CTAB concentration was analysed by potentiometric titration (785 DMP Titrino, Metrohm, Switzerland). A conductometer (CC-411, Elmetron, Poland) was used for conductivity measurements and the chloride concentration was analysed according to the Mohr method.

Experiments

In the first part of the experiments, the initial volume of the feed solution was 3 L and its temperature was 22°C. The tests involved 180 min membrane filtration runs with the use of FS of a wide range surfactant concentration (50, 100, 250, 500 and 1,000 mg/L) and DS with a concentration of 1-3 mol NaCl/L. In the second stage of the

study, the tests were carried out until the maximum concentration factor was obtained, i.e. until it was impossible to carry out further experiments due to the small volume of the feed solution. The concentration mode was performed for FS with a volume of 4L and 500 mg/L of surfactant, and for DS with a concentration of 3 and 1 mol NaCl/L for the BAC and CTAB tests, respectively.

The CS concentration increase factor was calculated using the following formula:

$$CIF = (1 - \frac{c_0}{c_f}) \cdot 100,\%$$
 (1)

where C_0 , C_f , – concentration of CS (mg/L) in the feed solution at the beginning and during the process, respectively.

The volumetric water flux transported from the feed solution to the draw solution was calculated by the following equation:

$$J = \frac{\Delta V}{t \cdot A} , \frac{L}{m^2 h}$$
 (2)

where V – volume of water sample passing through the membrane, L; t – time, h; and A – effective membrane surface area, m^2 .

The solution concentration factor was calculated by the following formula:

$$CF = \frac{v_0}{v_t} \cdot 100, \% \tag{3}$$

where V_0 – initial volume of the feed solution, L; V_t – volume of the feed solution at time t, L.

To calculate the reverse diffusion of the draw solute, the following equation was used:

$$J_S = \frac{C_t v_t - C_z v_z}{At}, \frac{g}{m^2 h}$$
(4)

where C_F , V_F – salt concentration in FS (mol/L) and volume of FS (L) at the beginning of the process, C_t , V_t – salt concentration in FS (mol/L) and volume of FS (L) at time t (h), A – effective membrane surface area, m².

RESULTS AND DISCUSSION

Effect of surfactant concentration in FS

The findings of the preliminary experiments using BAC and CTAB with concentrations ranging from 50 to 1000 mg/L as the feed solution (FS) and a 1 mol NaCl/L as the draw solution (DS) are presented in Figure 3. The results indicated that an increase in the surfactant concentration in the feed solution (compared to the con-

centration before the experiment) was observed. Specifically, CIF were 9-18% for BAC (for the entire concentration range) and 12-16% for CTAB (for solutions with concentrations of 500 and 1,000 mg/L). However, no increase in CTAB concentration was observed in FS for solutions with initial concentrations of 50, 100, and 250 mg CTAB/L. This lack of increase in CTAB concentration is due to the surfactant foaming process that occurs in the experimental set-up. In particular, intensive foaming occurred in the feeding tank, which was the sampling point for CTAB concentration analyses. According to Pugh et al. [36], the foaming intensity of surfactant solutions below the critical micelle concentration (CMC) increases linearly with its concentration, with the maximum foamability typically achieved at a concentration near the CMC. After the CMC has exceeded, the foaming intensity often does not change, which may explain the significant differences in the results obtained for monomeric and micellar solutions as feed for forward osmosis. In the experiments with BAC, no foaming was observed as a result of the low foaming nature of the surfactant.

Unlike pressure-driven membrane processes, FO membranes proved to be 100% effective in the separation of surfactant particles. In our previous research [27] the application of PDMPs allowed the separation of 85 and 96% CTAB from the solution with the initial concentration of 1000 mg/L with the use of nanofiltration (NF) and ultrafiltration (UF) membranes, respectively. When BAC solutions (concentration of 1,000 mg/L) were treated, the retention coefficient was 56 and 75% for the NF and UF membranes, respectively. The very high separation of CS in the FO process can be attributed to the pore size of the membrane (the pore size of the CTA membranes applied in the FO has pores of 0,29-0,3 nm [42]) which is significantly smaller than the size of the surfactant particles used in the tests (Table 2). Because both the monomers and micelles are larger than the membrane pore sizes, surfactant retention as a result of the sieve mechanism is possible and very effective.

The hydraulic performance of FO was analysed in terms of the volumetric flux of water transported from the feed solution (FS) to the draw solution (DS). The results of a 180 min cycle are presented in Figure 4. The findings indicated that the hydraulic performance of the process was enhanced in the presence of both CSs compared to that of deionised water.

The average water flux during the deionised water test was 7.5 L/m^2h . In the presence of surfactants, this parameter increased to 10.5 and 9.5 L/m²h for BAC and CTAB, respectively. Similarly, Zhao et al. [45] observed an improvement in the hydraulic performance of the process in the presence of anionic surfactant (SDS). This improvement in the transport properties of the membrane may be attributed to an increase in the hydrophilicity of its surface resulting from contact with the CS particles present in the solution.



Fig. 3. Concentration increase factor of cationic surfactant in FS depending on its initial concentration and the process duration (DS 1 mol NaCl/L)

Rys. 3. Zależność wzrostu stężenia kationowej substancji powierzchniowo czynnej w roztworze zasilającym (FS) od jej stężenia początkowego i czasu trwania procesu (DS 1 mol NaCl/L)





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Fig. 4. Volumetric water flux transported from FS to DS depending on the initial concentration of CS in FS and the process duration (DS 1 mol NaCl/L)

Rys. 4. Zależność objętościowego strumienia wody przechodzącego z roztworu zasilającego (FS) do odbierającego (DS) od stężenia początkowego kationowej substancji powierzchniowo czynnej i czasu trwania procesu (DS 1 mol

Fig. 5. Volumetric water flux transported from FS to DS depending on the driving force (NaCl concentration in DS) and the process duration (FS 500 mg CSs/L)

Rys. 5. Zależność objętościowego strumienia wody przechodzącego z roztworu zasilającego (FS) do odbierającego (DS) od stężenia NaCl w roztworze odbierającym i czasu trwania procesu (stężenie kationowej substancji powierzchniowo czynnej w FS 500 mg/L)

Fig. 6. Changes in conductivity and chloride concentration in DS depending on the driving force (NaCl concentration in DS) and the process duration (FS 500 mg CSs/L) Rys. 6. Zależność przewodności elektrolitycznej i stężenia chlorków w roztworze odbierającym (DS) od stężenia NaCl w roztworze odbierającym (DS) i czasu trwania procesu (stężenie kationowej substancji powierzchniowo czynnej w FS 500 mg/L)

Effect of driving force

To evaluate the effect of salt concentration in DS (osmotic pressure) on FO performance, sodium chloride solutions with concentrations of 0.5, 1, and 3 M were used. As FS, BAC and CTAB solutions with a concentration of 500 mg/L were applied. The effect of NaCl concentration in the draw solution on the volumetric flux of water transported from the feed solution to the draw solution is evident (Fig. 5). The increase in the driving force resulted in more intensive water transport through the membrane: the average flux of water was 7.5, 11, 17.5 L/m²h for BAC and 7, 10.5, 17.5 L/m²h for CTAB when the salt concentration in DS was 0.5, 1, and 3 mol/L respectively.

At the highest concentration of NaCl in DS (3 mol/L), a significant decrease in water flux was observed during the process duration (Fig. 5). At the beginning of the filtration run, the volumetric water flux was 20.5 L/m²h for BAC and 18.5 L/m²h for CTAB, and after 180 min, the value of this parameter decreased to 16.5 and 16 L/m²h, respectively. The use of a significant driving force was associated with intensive water permeation through the membrane, which caused its significant dilution during the process time. Moreover, salt particles from DS penetrated into FS, which affected the dilution of DS. As a result of both phenomena described, there was a decrease in the driving force of the osmosis during its run. As shown in Figure 6, for all NaCl concentrations tested in DS (i.e. 0.5, 1, and 3 mol/L) a decrease in chloride concentration and conductivity was observed during the process.

One of the limitations of the FO process is the back-diffusion of salts from the draw solution to the feed solution, known as "salt leakage". This phenomenon can affect the properties of the feed solution, such as ionic strength, pH, and the concentration of individual ions [12]. The increase in chloride concentration and conductivity in the feed solutions (Fig. 7) was proportional to the duration of the process. The back-diffusion of salts was dependent on the concentration of NaCl in DS, as the intensity of this phenomenon resulted from the concentration gradient on both sides of the membrane. For example, in the BAC test, the average value of the reverse salt diffusion was 0.09, 0.12 and 0.20 mol NaCl/m²h for draw solutions of 0.5, 1, and 3 mol NaCl/L, respectively.

Concentration mode

In this part of the experiments the FO processes were run until the concentration factor amounted to 2. In Table 3 the parameters of the feed and draw solutions, before and after the FO process, are presented. Data analysis allows us to conclude that FO proved to be an effective method for concentrating micellar CS solutions.



Fig. 7. Changes in conductivity and chloride concentration in ES depending on the driving force (NaCl concentration in DS) (FS 500 mg CSs/L) Rys. 7. Zależność przewodności elektrolitvcznei i steżenia chlorków w roz tworach zasilających od steżenia NaCl w roztworze odbierającym (DS) i czasu trwania procesu (stężenie kationowej substancji powierzchniowo czynnej w FS 500 mg/L)

Table 3. Quality of FS and DS solutions before and after FO concentration Tabela 3. Parametry roztworów procesowych przed i po procesie osmozy prostej

BAC									
Devemeter	Feed solution (FS)		Draw solution (DS)						
Falametei	Prior FO	After FO	Prior FO	After FO					
BAC, mg/L	508	974	0	0					
Chlorides, mg/L	70	794	104,000	72,000					
Conductivity, µS/cm	124	2,000	235,000	152,000					
СТАВ									
Derometer	Feed solution (FS)		Draw solution (DS)						
Falametei	Prior FO	After FO	Prior FO	After FO					
CTAB, mg/L	488	980	0	2,2					
Chlorides, mg/L	68	1,216	34,380	23,820					
Conductivity, µS/cm	104	2,230	82,000	50,300					

The concentration of BAC and CTAB in the feed solutions increased from 508 and 488 mg/L to 974 and 980 mg/L, respectively, with an approximately twofold reduction in the volume of the feed solutions (CF at the level of two). In the case of BAC, there was no contamination of DS with CS particles, while in CTAB tests the penetration of surfactant molecules was insignificant (the concentration of surfactant in the draw solution at the end of the experiment was 2.2 mg/L, retention 99.8%).

An increase in the salinity of the feed solutions subjected to concentration was observed during the FO process. The concentration of chlorides increased about 10 times in the BAC tests and approximately 18 times in the CTAB tests at the end of the process. The higher degree of salinity of the CTAB solutions was most likely due to the longer process time compared to that of the BAC solution. In both tests, an increase in the conductivity of FS was also observed after membrane filtration, and this phenomenon can be attributed to both salt back-diffusion and surfactant concentration in FS.

Conclusions

Based on the experiments conducted, it was found that:

The foamability of cationic surfactants determines the possibility of applying FO as a concentration process. After a 180 min filtration cycle, the concentration increase was 9-18% (for the entire concentration range, that is, 50-1,000 mg/L) and 12-16% (for solutions of concentration exceeding the CMC value, that is, 500-1,000 mg/L) for low foaming BAC and foaming CTAB, respectively.

Strong hydrophilic forward osmosis membranes allowed 100% separation of cationic surfactants over the entire concentration range (50-1,000 mg/L) during short-term tests. There was no contamination of the drawn solutions with BAC particles (100% retention) in any of the experiments performed in long-term concentration modes; while in the case of CTAB, the penetration of surfactant molecules through the membrane was low (99.8% retention).

The increase in the driving force (salt concentration in the DS) affected two crucial process parameters: the water flux transported from the feed solution to the draw solution and the reverse diffusion of salt from the draw solution to the feed solution. Short-term tests with sodium chloride concentrations of 0.5, 1, and 3 mol/L improved the average water flux from 5.4, 7.5 and 17.3 L/m²h (for distilled water as FS) to 7.5, 11, 17.5 L/m²h for 500 mg of BAC/L and 7, 10.5, 17.5 L/m²h for 500 mg of CTAB/L, respectively. At the same time, the reverse salt diffusion increased to an average level of 0.09, 0.12 and 0.20 mol NaCl/m²h for 500 mg of CTAB/L.

Forward osmosis allowed for an effective concentration of surfactant solutions with a concentration of 500 mg/L. With a concentration factor of two, approximately a twofold increase in the CS concentration was obtained.

The use of FO allowed for the recovery of water from CS solutions, which after desalination can be reused for production purposes. Because of the lack of contamination with CS particles, regeneration of the draw solution can be easily achieved (e.g. by using reverse osmosis). An alternative way to manage DS could be its discharge into the environment (e.g. seas and oceans) if the aim of the process was to concentrate CS solutions.

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