

Characterization of Polymeric Nanofiltration Membranes

KUI – 31/2007
Received February 15, 2007
Accepted August 30, 2007

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As membrane processes are increasingly used in industrial applications, there is a growing interest in methods of membrane characterization. Traditional membrane characteristics, such as cut-off value and pore size distribution, are being supplemented by membrane surface characteristics, such as charge density or zeta potential and hydrophobicity. This study, therefore, characterizes the three different polymeric membranes used (NFT-50, DL and DK). The molecular mass cut-off (MMCO) value was determined using a set of reference solutes within the molecular range 150–600 Da, whereas streaming potential measurements enabled quantification of the surface charge characteristics. Hydrophobicity was studied using contact angle measurements. The results indicated that even though all three membranes had very similar layer compositions which consisted of poly(piperazneamide), as top layers they showed different values of measured quantitative. The NFT-50 membrane had the lowest MMCO value and the most hydrophilic membrane surface, followed by DK and DL. Membrane fouling as measured by flux reduction was determined by streaming potential measurements and accompanied by a positive change in zeta potential.

Key words: *Characterization, nanofiltration membrane, MMCO, streaming potential, fouling, contact angle*

Introduction

Nanofiltration (NF) is a relatively new process among pressure driven membrane separation processes and it offers higher fluxes than reverse osmosis and significantly better retentions than ultrafiltration for lower molar mass molecules such as sugars, natural organic matter (NOM) and even ions. The most attractive applications for NF membranes concern the treatment of different industrial effluents for reuse, especially, when the removal of organics is desired but retentions of monovalent ions are not needed.¹

Nanofiltration membranes have special characteristics: high retention coefficients for neutral molecules with a molecular mass (M_m) \sim 300 Da, which is equivalent to a pore diameter of app. $d_p = 1$ nm. They also have a surface electrostatic charge, which gives them great selectivity with respect to ions or charged molecules. Due to their low MMCO, nanofiltration membranes are used to treat textile

industry effluent when removing colouring agents, in the pharmaceutical industry to treat fermentation effluent, and in the paper industry to limit the consumption of clean water by recycling wastewater.² Fouling is still a major problem in NF operations, resulting in product water flux decline and larger operating costs. The final cause of fouling is the interaction of the molecules in the solution with the membrane surface groups. It has been shown that if the membrane is hydrophilic and charged both as molecules in the solution, it is less apt to be fouled.³ High charge density keeps the molecules away from each other and from the membrane, then retention increases normally.

Fouling due to organic matter has previously been observed to strongly correlate with membrane hydrophobicity, and charge effect and electrokinetic phenomena.^{4–5} Streaming potential measurements are often used for the electrokinetic characterization of membrane surfaces, and it is one of the more convenient techniques.⁶ Membrane hydrophobicity is usually expressed in terms of a contact angle (θ), which is a common and relatively easy method of characterizing the surface properties of membranes.⁷

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In this study, membrane characteristics (membrane size, permeability, hydrophobicity and charge) were studied thoroughly for three NF membranes. The measurements of zeta potential were carried out on new and fouled NF membranes at different pH.

Separation mechanisms of nanofiltration

Transport mechanism

In order to utilize the possibilities of nanofiltration properly when developing a filtration process, it is important to understand the separation mechanism of nanofiltration. A membrane is considered a porous medium that is selective to certain species. The transport mechanism in NF by nature lies somewhere between reverse osmosis and ultrafiltration. With looser NF membranes, the mechanism is closer to sieving (steric hindrance), as in ultrafiltration, and with the tighter NF membranes it is closer to a solution-diffusion process. In most cases, however, both mechanisms must be taken into account.⁸ As a majority of NF membranes are negatively charged or amphoteric, the negatively charged solutes are effectively rejected, whereas the rejection on neutral solutes is less effective.⁹ Generally, co-ions will be repelled due to the electric repulsive forces, whereas the counter-ions will be attracted by membrane surface.^{10,11} Therefore, the separation mechanism, in the case of a charged NF, is not based on sieving and diffusion only, but also on the charge effects.¹²

Charge effect

When brought into contact with an aqueous electrolyte solution, NF membranes acquire an electric charge in several possible ways, e. g. by dissociation of functional groups and by the adsorption of ions, polyelectrolytes or ionic surfactant from the solution.

The general chemistry of the selective layer of polymeric composite commercial NF membranes is well represented by one of its large families, poly(piperazineamide)-based membranes that are normally produced by means of interfacial polymerization.¹³ Fig. 1 shows a hypothetical polymeric NF membrane with carboxylic groups attached to the surface of the membrane, when the membrane surface is brought into contact with an aqueous solution of an electrolyte. The presence of the dissociated carboxylic groups on the membrane surface is the source of membrane charge. The surface charge repels for instance large SO_4^{2-} ions, allowing at the same time, the passage of smaller Cl^- ions through the membrane.

Membrane surface charge determination

In practice, experiments are required to determine the sign and the density of the membrane charge.¹⁴ Experimental determination of the membrane's effective charge density can be carried out analytically by equilibrating the membrane sequentially in acid and salt solution or indirectly by electrokinetic methods, e. g. streaming potential measurements, electrophoresis or electro-osmosis. Electrokinetic phenomena occur whenever an electrically charged phase moves with respect to the adjoining phase. When an electrolyte is pressure-driven through a thin slit, the mobile charges are dragged towards the slit end (streaming cur-

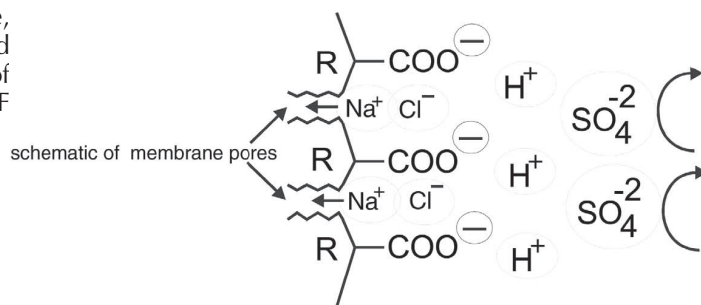


Fig. 1 – Hypothetical nanofiltration membrane containing carboxylic groups. Dissociated carboxylic groups on the membrane surface create a charge on the NF membrane surface.¹³

Slika 1 – Nanofiltracijska membrana koja sadrži karboksilne skupine. Disocijacija karboksilnih skupina membrane generira naboj na površini.¹³

rent); thereupon the charges accumulated generate an electrical potential difference between the slit edges. At steady state, a steady potential difference (streaming potential) is established along the slit and back flow current equals the streaming current.¹⁵ In general, the streaming potential is the potential difference at zero current caused by the convective flow of charge due to a pressure gradient through a charged capillary, plug, diaphragm or membrane.¹⁶ The streaming potential quantifies an electrokinetic effect reflecting the properties of the surface, the flow characteristics and the chemistry and thermodynamics of the electrolyte solution.¹⁷

The idea behind streaming potential measurements relates to the double-layer theory. According to the theory, ions in the electrolyte solution that are in contact with a charged surface will not be distributed randomly. The concentration of counter-ions near the membrane will be higher than that in the bulk solution. The charges at (and adjacent to) the surface will cause a potential difference between the region near the surface and the bulk of the solution. The potential decreases within the solution as a function of the distance from the charged surface. At a certain distance from the charged surface there is an imaginary border between mobile ions in the bulk of solution and the immobile ions attracted by the surface, the so-called slip plane. The potential at that plane is known as electrokinetic or zeta potential.

The relationship between the measurable streaming potential and the zeta potential was calculated using the method described by Fairbrother and Mastin for flat sheet samples with insignificant surface conductivity. It is based on the Helmholtz-Smoluchowski equation, eq.1:

$$\zeta = \frac{V_p}{\Delta p} \cdot \frac{\eta}{\varepsilon \cdot \varepsilon_0} \cdot \frac{L}{A} \cdot \frac{1}{R} \quad (1)$$

where: ζ is zeta potential (mV), $V_p/\Delta p$ the changes in streaming potential with pressure (mV Pa^{-1}), η the dynamic viscosity of solution (Pas), L the channel length (m), ε_0 the permittivity of free space ($8.854 \cdot 10^{-12} \text{ m}^{-1} \text{ } \Omega^{-1}$), ε the dielectric constant of the solution, A cross-section area of the channel (m^2) and R is the ohmic resistance of the electrolyte and channel (Ω).

Using the Fairbrother and Mastin approach, the ratio $L A^{-1}$ can be replaced by $\kappa_s R_s$ where R_s is the electrical resistance of the channel when the measurement cell is filled with a standard solution whose conductivity κ_s is known. The $c = 0.1 \text{ mol L}^{-1}$ KCl solution is commonly used to avoid problems associated with surface conductance.¹⁸

$$\zeta = \frac{V_p}{\Delta p} \cdot \frac{\eta}{\varepsilon \cdot \varepsilon_0} \cdot \frac{R_p \cdot \chi_p}{R} \quad (2)$$

Zeta potential data calculated from the streaming potential for the studied membranes over the pH range, indicated the change of charge sign and the isoelectric point (IEP). The isoelectric point of the membrane is the pH where the zeta potential is zero. If the zeta potential is positive, the membrane surface is positively charged and when zeta potential goes sub-zero, the charge is negative. As seen in Fig. 2, NF membranes are usually negatively charged under alkaline or neutral conditions, but the charges change to positive as the solution becomes more acidic. Usually, the isoelectric point of the NF membranes can be found around a pH of 3–6.

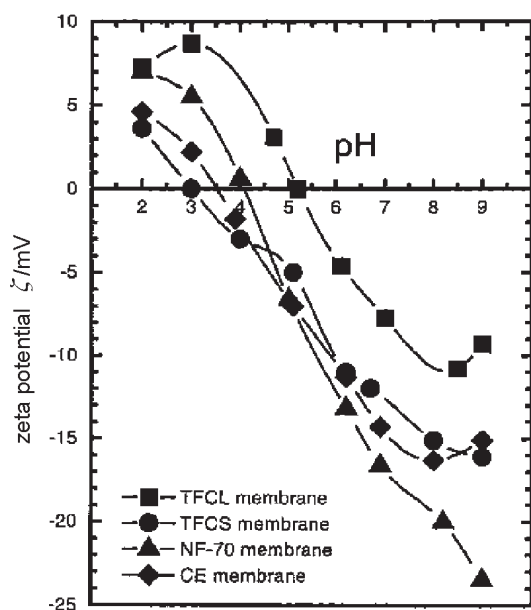


Fig. 2 – Comparison of zeta potentials of four different NF membranes over the pH range 2–9. The experiments were carried out with a background electrolyte concentration of 0.01 mol L^{-1} NaCl.¹⁹

Slika 2 – Usporedba zeta-potencijala četiri različite NF-membrane u pH području 2–9. Mjerenja su izvršena u $0,01 \text{ mol L}^{-1}$ NaCl.¹⁹

Contact angle and hydrophobicity

When a drop of liquid is placed on a dry membrane surface, the contact angle is the angle that develops between the membrane surface and the air/liquid interface. The contact angle is formed at the junction of the three phases i.e. the solid, liquid and gas phases.

Contact angle is based on the theory contained in the equation developed by Young in 1805.²⁰ For systems with at least three bulk phases there can be, in addition to an area of

contact, a line of contact. The figure below shows a liquid drop resting on a solid surface in a vapour environment. This system consists of three bulk phases; the solid bulk phase, the liquid bulk phase and the saturated liquid-vapour bulk phase.

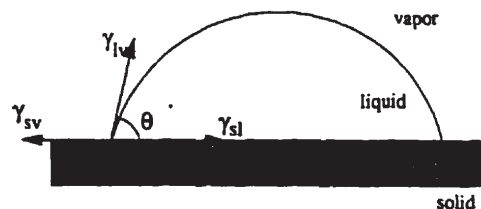


Fig. 3 – Contact angle of a liquid drop on a solid surface

Slika 3 – Kontaktni kut kapljice na krutoj površini

Young's equation, one of the basic equations to characterize wettability, describes the equilibrium contact angle (θ) of a liquid (l) on top of a solid (s):

$$\gamma_{lv}^{TOT} \cos \theta = \gamma_{sv}^{TOT} - \gamma_{sl}^{TOT} \quad (3)$$

$$\gamma_s^{TOT} = \gamma_{sl}^{TOT} + \gamma_l^{TOT} \cos \theta \quad (4)$$

where:

- γ_{lv}^{TOT} (mN m^{-1}) is the surface tension of the liquid-air interface
- γ_{sv}^{TOT} (mN m^{-1}) is the solid-air surface tension
- γ_{sl}^{TOT} (mN m^{-1}) is the solid-liquid interface surface tension

Young's equation illustrates the relationship between the adhesion of the liquid to the solid surface and cohesion of the liquid itself. When the self-cohesion of the liquid is greater than the adhesion of the liquid to the solid, a large contact angle is formed. As the self-cohesion of the liquid decreases, the contact angle formed by the liquid on the solid decreases. When the adhesion of the liquid to the solid is much greater than the self-cohesion of the liquid, a small contact angle is formed.²¹

Materials and methods

Membranes used

Commercially available thin-film composite NF membranes were used. The three NF membranes selected for investigation are given in Table 1.

Membrane characterization

The membranes were thoroughly soaked by circulating deionized water at $p = 10 \text{ bar}$ for $t = 6 \text{ h}$, so that the excess of chemicals attached to the membrane sheets could be released, and also to prevent their compaction throughout the permeation experiments.

The hydraulic permeability of the membrane was calculated according to eq. (5): it is the slope of J_w ($\text{Lh}^{-1}\text{m}^{-2}$) vs. p (bar) in experiments with the solvent (deionized water) at $\vartheta = 20 \text{ }^\circ\text{C}$.

Table 1 – Overview of the used NF membranes

Tablica 1 – Pregled primijenjenih NF membrana

Membrane Membrana	NFT-50	DL	DK
Manufacturer Proizvođač	Alfa Laval ^a	Osmonics Desal ^b	Osmonics Desal ^b
Retention (MgSO ₄)/%	99	96	96
Zadržavanje (MgSO ₄)/%			
p /bar	1–55	0.5–28	0.5–28
ϑ_{\max} /°C	50	50	50
pH	2–10	2–11	2–11
Morphology Morfologija	TFC ^c	TFC	TFC
Support Nosač	polyester	polyester	polyester
Top layer composition Sastav gornjeg sloja	poly(piperazineamide)	poly(piperazineamide)	poly(piperazineamide)

^a Denmark, ^b GE Osmonics, USA ^c thin film composite

$$L_p = \frac{J_w}{\Delta p} \quad (5)$$

The sieving properties of the NF membranes depend on their molecular mass cut-off (MMCO), which is defined as the molecular mass of an uncharged solute with a rejection of 90%. A set of reference solutes in the molecular range 150 – 600 Da is chosen, namely, xylose, glucose, saharoze and rafinose. These characterization experiments were carried out in total recirculation mode under the same conditions (20 °C, 8 bar, mass concentration $\gamma = 0.2 \text{ mg}^{-1}$).

The contact angles of the liquids on the membranes were measured using a Dynamic contact angle, Fibro DAT 500/1100 (Fibro, Sweden). This DAT instrument is an automated contact-angle tester working on the principle of the goniometer-sessile drop technique. It enables optical evaluation of the contact-angle θ of a drop of liquid on a solid surface by measuring drop height h and base diameter d , as follows:

$$\theta = 2 \cdot \arctan\left(\frac{2h}{d}\right) \quad (5)$$

The zeta potential of the membranes was determined by streaming potential measurements. The streaming potential developed in the electrolyte flowing along a thin rectangular slit formed by a 'sandwich' of identical parallel membranes was measured using an electrokinetic Brookhaven-Paar analyzer (EKA). In the experiment, the measuring cell contained $c = 1 \text{ mmol L}^{-1}$ KCl as a background electrolyte at pH range of 2–10 (pH was changed using HCl). First, the pH was set to 11 with $c = 0.1 \text{ mmol L}^{-1}$ NaOH and, further on, it was automatically changed by dosing 0.1 mol L^{-1} HCl to electrolyte. The analyzer also measured the pH, conductivity and electrical resistance, pressure and voltage and automatically calculated the zeta potential using the eq. 2.

Charge reversal concentration (CRC) was determined by cationic surfactant addition to the electrolyte at pH 10 where the zeta potential reached its maximal and constant value. The CRC is determined at pH 10 where the zeta potential reached its maximal and constant value.

Results and discussion

Determination of the MMCO and the membranes' hydraulic permeability

The rejection characteristic of a specific NF membrane is often quantified by the MMCO. The MMCO is a measure used for the pore diameter and is defined as the molecular mass of a component that is 90% retained by the membrane.²² A retention curve is obtained by plotting the observed retention for each compound against its molecular mass. The experimental MMCO values for all three membranes, as calculated from best-fit polynomial relationship, are presented in Fig. 4. Fig. 5 shows the permeate flux of pure water vs. applied pressure for all three membranes.

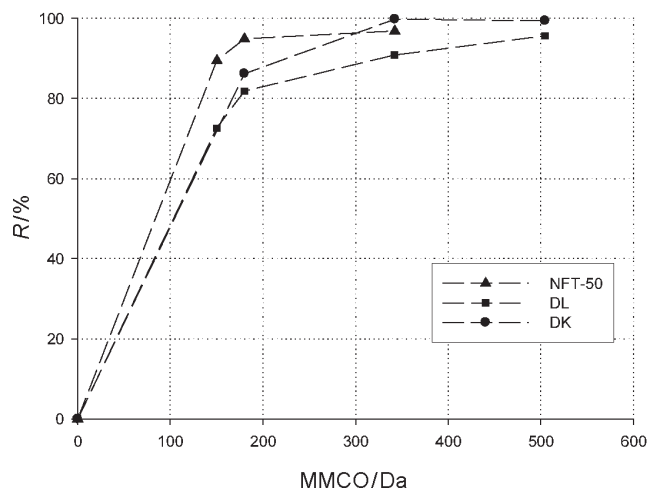


Fig. 4 – Determining the MMCO of the membranes
Slika 4 – Određivanje MMCO membrana

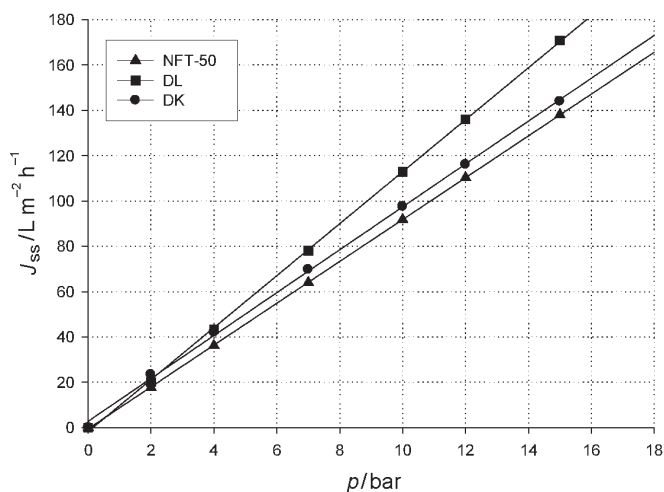


Fig. 5 – Pure water flux versus applied pressure of the NF membranes

Slika 5 – Protok čiste vode ovisno o tlaku NF-membrane

Determination of hydrophobicity

Contact angle determination is a well-known method of studying the membrane hydrophobicity. Hydrophilic membrane gives rise to small contact angles.²³ Fig. 6 shows the results of contact angles for a water droplet on NFT-50, DL and DK membranes. The contact angle equilibrium is reached in the measured time, so the static contact angles are obtained and presented in Table 2.

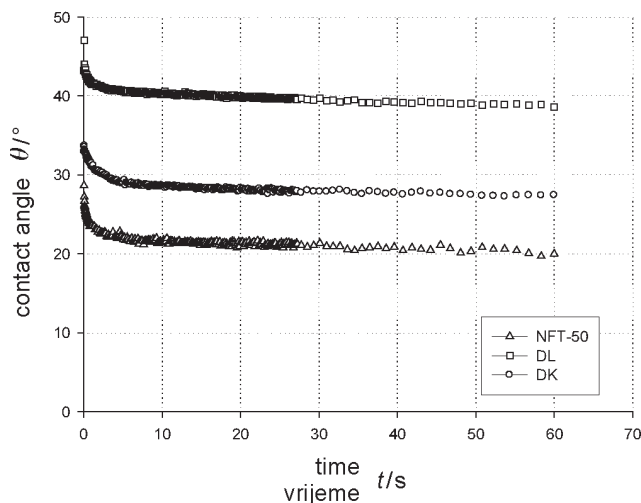


Fig. 6 – Time dependence of contact angles of water on membranes

Slika 6 – Ovisnost kontaktnog kuta kapljice vode na membrana o vremenu

Determination of the surface charge

Zeta potential of unfouled membranes

Zeta potential versus pH data for experiments performed in 0.001 mol L⁻¹ KCl is shown in Fig. 7. From these results, a few generalizations may be made regarding thin-film NF membranes used. The DK and DL membrane are charged more negatively than the NFT-50 membrane. More specifically, DK and DL have lower isoelectric points, 2.08 and 2.71, respectively, and they acquire more negative charge at the highest pH tested. Compared to the other membranes, NFT-50 membrane has the highest IEP and is less negatively charged at the pH > IEP and more positively charged at lower pH. For all the membranes, the surface charge is positive at lower pH range, it passes through the isoelectric point (between pH 3 and 4) and becomes negative in the higher pH range. The shape of the zeta potential curve is indicative of amphoteric surfaces.

In the literature, an IEP around 4 was determined for membrane DK with NaCl solutions (2 mmol L⁻¹)²⁴ and an IEP of 4.2 was determined for membrane NFT-50 in a KCl solution (0.001 mol L⁻¹).²⁵ Concerning membrane DL it is reported that the charge is always negative.⁹

The results of ζ -potential in dependence on the addition of cetyl pyridinium chloride (N-CPC) in electrolyte 1 mmol L⁻¹ KCl at pH 10, are shown in Fig. 8. The quantity of CPC for obtaining of CRC is relatively low, indicating the slight hydrophobic nature of these membranes. CRC of NFT-50 and DL membranes are very close.

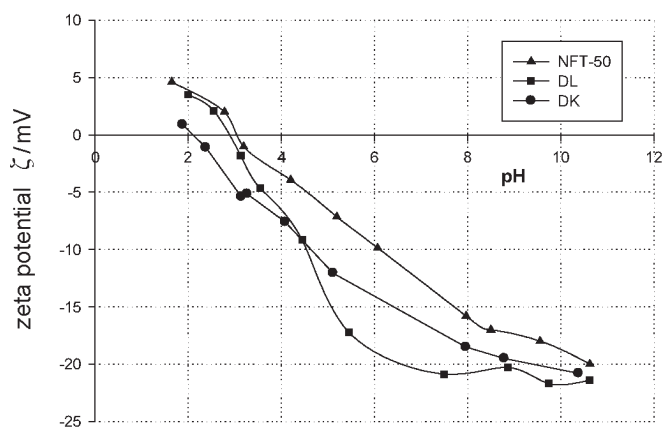


Fig. 7 – Zeta potential of membranes versus pH of 0.001 mol L⁻¹ KCl

Slika 7 – Zeta-potencijal membrana u ovisnosti o pH 0,001 mol L⁻¹ KCl

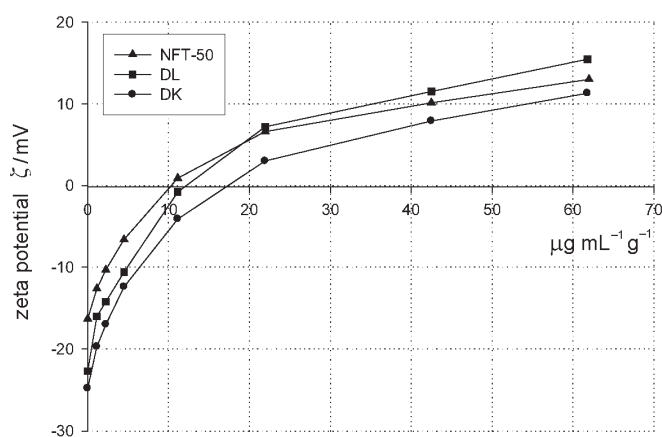


Fig. 8 – Zeta potential of the membranes vs. the addition of N-CPC in electrolyte 0.001 mol L⁻¹ KCl at pH 10

Slika 8 – Zeta-potencijal membrane u ovisnosti o dodatku N-CPC u 0,001 mol L⁻¹ KCl pri pH 10

Zeta potential of fouled membranes

The permeate flux reduction over NFT-50 membrane occurred during filtration of the wastewater after performing reactive dyeing and rinsing of cotton. Even after thorough cleaning, the membrane did not recover its permeability and the flux did not increase. The fouled membranes (all three) were removed from the filtration unit and placed in the streaming potential cell without rinsing, in order not to disturb any surface deposits on the membrane. Fig. 9 demonstrates the zeta potential values of the clean and fouled membranes as a function of pH. It can be observed that the zeta potential values of all the fouled membranes are less negative when compared to the cleaned one. The shape and slope of curves is different especially in alkaline region indicating contamination of membrane surface due to fouling.

Membrane characteristic values obtained are presented in Table 2.

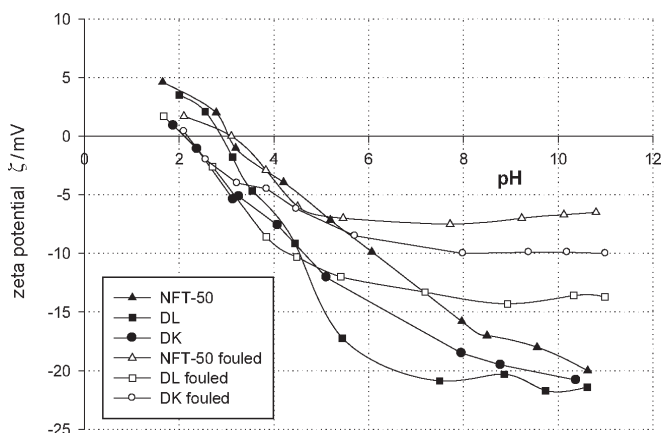


Fig. 9 – Zeta potential of clean and fouled membranes in function of pH

Slika 9 – Zeta-potencijal čistih i zaprljanih membrana u ovisnosti o pH

Table 2 – Measured quantities of the NF membranes

Tablica 2 – Mjerne veličine NF-membrana

	NFT-50	DL	DK
MMCO/Da	159	340	230
Contact angle/ $^{\circ}$	21.2 ± 0.9	39.7 ± 6.3	28.1 ± 4.8
ζ – potential/mV at pH 10	-20.15	-21.5	-17.5
IEP clean/pH	2.95	2.71	2.08
$\Delta\zeta$ – potential/mV at pH 10	13.5	7.7	10
CRC/ $\mu\text{g ml}^{-1}$	10.35	12.23	17.38

There is a clear correlation between MMCO and $\Delta\zeta$ ($\Delta\zeta = \zeta$ (fouled) – ζ (clean)) at pH 10. The highest $\Delta\zeta$ is obtained for NFT-50, DK and DL membrane, respectively. It can be concluded that the membrane which had the lowest MMCO had the highest $\Delta\zeta$, pointing to permeate flux reduction during filtration due to small pores.

Conclusions

All three membranes have very similar layer compositions which consist of poly(piperazneamide) as top layers, and polyester as a support. The carboxylic groups ($-\text{COO}^-$) which may be present on polyamide membranes are weakly acidic and cannot be dissociated at low pH. A poly(piperazneamide) membrane can also contain amino groups which can be positively charged (NH_3^+ is only positively charged in acid medium, while $-\text{R}_3\text{N}^+$ (R different than H) is positively charged over the entire pH range), and, therefore, can exhibit negative and positive surface charge depending on pH. High negative values of zeta potential under alkaline conditions and comparatively low positive values at low pH ($\text{pH} < \text{IEP}$) support the conclusion that, in this case, the carboxylic groups dominate over the amino groups at the membrane surface. Also shown was that NFT-50 membrane had the lowest MMCO value and the most hydrophilic membrane surface, followed by DK and DL.

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List of symbols Popis simbola

- MMCO – molecular mass cut-off
– 90 % molekularne mase čestica koje membrana može zadržati
- IEP – iso-electric point
– izoelektrična točka
- NF – nanofiltration
– nanofiltracija
- NOM – natural organic matter
– prirodne organske tvari

θ	– contact angle, ° – kontaktni kut, °	TFC	– thin film composite – tanka kompozitna membrana
ζ	– zeta potential, mV – zeta-potencijal, mV	J_w	– permeate flux, L h ⁻¹ m ⁻² – gustoća protoka permeata, L h ⁻¹ m ⁻²
V_p	– streaming potential, mV – potencijal strujanja, mV	L_p	– hydraulic permeability, L h ⁻¹ m ⁻² bar ⁻¹ – hidraulična permeabilnost, L h ⁻¹ m ⁻² bar ⁻¹
Δp	– pressure difference, Pa ⁻¹ – razlika tlakova, Pa ⁻¹	h	– drop height, m – visina kapi, m
η	– dynamic viscosity, Pa s – dinamička viskoznost, Pa s	d	– drop diameter, m – promjer kapi, m
L	– capillary length, m – duljina kapilare, m	EKA	– electrokinetic analyzer – elektrokinetički uređaj
ϵ_0	– permittivity of free space, m ⁻¹ Ω ⁻¹ – permitivnost vakuuma, m ⁻¹ Ω ⁻¹	CRC	– critical reverse charge – kritični suprotni naboj
ϵ	– dielectric constant – dielektrična konstanta	R	– retention, % – zadržavanje, %
A	– cross-section area of the capillary, m ² – površina presjeka kapilare, m ²	J_{ss}	– steady state permeate flux, L h ⁻¹ m ⁻² – jednakomjerna gustoća protoka, L h ⁻¹ m ⁻²
R	– ohmic resistance of the electrolyte and channel – omski otpor otopine elektrolita i kapilare	ϑ	– temperature, °C – temperatura, °C
κ_s	– conductivity, mS m ⁻¹ – provodnost, mS m ⁻¹	M_m	– molecular mass, Da – masa molekule, Da
γ_{lv}^{TOT}	– surface tension of the liquid/vapour interface, mN m ⁻¹ – granična površinska napetost tekuće/zrak, mN m ⁻¹	d_p	– pore diameter, nm – promjer pore, nm
γ_{sv}^{TOT}	– the solid–vapour surface tension, mN m ⁻¹ – granična površinska napetost kruto/zrak, mN m ⁻¹	c	– concentration, mol L ⁻¹ – koncentracija, mol L ⁻¹
γ_{sl}^{TOT}	– solid–liquid interface surface tension, mN m ⁻¹ – granična površinska napetost kruto/tekuće, mN m ⁻¹		

SAŽETAK

Karakterizacija polimernih nanofiltracijskih membrana

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Znanstvena istraživanja metoda za karakterizaciju membrana za pročišćavanje otpadnih voda u stalnom su porastu. Tradicionalne metode karakterizacije veličinom i raspodjelom pora dopunjuju se metodama karakterizacije određivanjem zeta-potencijala i hidrofilno/hidrofobnih svojstva. Stoga su u ovom radu karakterizirane tri različite polimerne nanofiltracijske membrane NFT-50, DL i DK karakterizirane veličinom MMCO odabirom tri referentne otopine molekulske mase 150–600 Da. Elektrokinetičke karakteristike tih membrana vrednovane su pomoću potencijala strujanja a hidrofobnost pomoću kontaktnog kuta. Rezultati istraživanja ukazuju na to da su, unatoč gotovo jednakom gornjem sloju kompozitnih membrana, mjerne veličine različite. Najnižu MMCO vrijednost i najvišu hidrofilitnost ima NFT-50 membrana kojoj slijede DK i DL. Smanjivanje protoka permeata ukazuje na začepijavanje membrana, koje je izmjereno pomoću potencijala strujanja te rezultiralo smanjivanjem zeta-potencijala.

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Prispjelo 15. veljače 2007.
Prihvaćeno 30. kolovoza 2007.

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