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Comparison of phenol removal in synthetic wastewater by NF or RO membranes

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ABSTRACT

The performance of NF membranes (NF90, NF97, NF99, NF99HF) and RO membranes (RO98pHt, RO99, SW) to remove phenol in phenolics-containing synthetic wastewater was compared. In terms of both rejection and permeate flux, NF97 and RO98pHt showed superior performance over other membranes. Water and phenol permeability constants were obtained according to solution-diffusion model and influences of cross flow (0.39–0.96 m/s), temperature (20–40 °C), pressure (5–30 bar), phenol concentration (10–1,000 ppm), pH (3–11) salt concentration (1,000–3,000 ppm) on the phenol rejection were investigated in this work. Results indicate that cross flow, phenol concentration and salt concentration have little influence on phenol rejection, while phenol rejection decreases with temperature increase, and it increases with pressure increase. The pH showed significant effect on phenol rejection.

Keywords: Nanofiltration membrane; Reverse osmosis membrane; Phenol rejection

1. Introduction

Phenol and their derivatives are widely used as raw materials in chemical industry, and they are also generated from some industry process. Therefore, phenolic compounds are frequently present in wastewaters deriving from many industries such as chemical and pharmaceutical industries, pulp and paper mills, tannery and foundries (washing of gas influents), phenolic resins industrials, steel plant, ceramic plant, especially petrochemical industries, petroleum refineries (washing and conditioning of the alkaline or acid products), and coal conversion processes [1]. Phenolic compounds have environmental hazards due to their high degree of toxicity even at low concentration. It is said that phenolics' toxic effects include permeabilisation of cellular membranes and cytoplasmic coagulation, damaging sensitive cells and thus causing profound health and environmental problems. Fatal doses can be absorbed through the human skin. Gastrointestinal disturbances, kidney malfunction, circulatory system failure, lung edema and convulsions can be caused by acute phenolics poisoning. Key organs damaged by chronic phenol exposure include spleen, pancreas and kidneys [2].

Many methods have been developed for treating phenolics-containing wastewater such as adsorption, solvent extraction, oxidation, distillation, chemical precipitation, steam stripping, enzymatic treatment and so on. Membrane technologies have been preferred for treatment of wastewater in recent years. Some technologies have been investigated for treating phenolics-containing wastewater by using emulsion liquid membrane [3], pervaporation [4], membrane-based solvent extraction (MBE), membrane aromatic recovery system (MARS) [5], membrane bioreactor (MBR) [6], and micellarer-enhanced ultrafiltration [7], but all the above methods

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have disadvantages to a certain degree. For example, emulsion liquid membrane process that has higher removal is very complex including emulsion and rupture emulsion process; the flux of pervaporation, MBE and MARS is low; Fouling and the toxicity of phenol toward microorganisms are big problems in MBR. In micellarer enhanced ultrafiltration process, new pollutant that is a surfactant used for solubilize the phenolics will be introduced, and how to reuse the surfactant is still a question.

The application of NF or RO process to remove low molecular weight organic pollutants including phenolics has been analyzed in several recent publications [8-10]. Factors such as molecular weight, molecular width, pKa, logP on the removal efficiency were studied. It is clear that the removal efficiency depends on membrane type, solute and the mutual interaction between them. Temperature, pH, pressure, concentration also have influence on rejection [2,11]. Whether NF or RO process could be used in the treatment of phenolics-containing wastewater depends on rejection and flux. In the present work, performance of different NF and RO membranes on phenol removal in synthetic wastewater was examined. The focus of this work is on the comparison of performance of the membranes. Influence of cross flow, temperature, pressure, concentration, pH and salt concentration on phenol rejection was investigated. The results may be used as reference for applying NF or RO process in the treatment of phenolics-containing wastewater.

2. Theory

The performance of a given membrane is determined by two parameters, the selectivity and the permeability through the membrane [2,12]. The selectivity of a membrane towards the mixture is usually expressed in terms of the solute rejection coefficient which is calculated as

$$R = 1 - \frac{C_p}{C_f} \tag{1}$$

where C_p and C_f are the permeate and feed concentrations respectively.

The flux J is defined as the volume flowing through the membrane per unit area and time according to solution-diffusion model,

$$J_w = A_w(\Delta P - \Delta\pi) \tag{2}$$

$$J_s = B_s\,(C_f - C_p) \tag{3}$$

where J_w is pure water flux, J_s is solute flux, ΔP called transmembrane pressure, which is the average pressure of inlet and outlet, $\Delta\pi$ the osmotic pressures of the solutions, for distilled water $\Delta\pi = 0$, A_w is the water permeability constant, A_w depends on the membrane and temperature, and it can be obtained by operating the experimental system at different pressures and using distilled water as feed. Eq. (2) can be written: $J_w = A_w \Delta P$. Therefore the slope of the plot of J_w versus ΔP gives A_w. B_s is the solute permeability constant, and B_s can be determined by operating the experimental system at different feed concentrations, at constant pressure. Bearing in mind that $J_s = \frac{J_w C_p}{C_w}$, where C_w is solvent concentration which can be seen $C_w \approx 1{,}000$ g/L in this study Eq. (3) can be written $J_w\,C_p = B_s\,(C_f - C_p)$ and the slope of the plot of $J_w\,C_p$ versus $(C_f - C_p)$ gives B_s.

3. Experimental

Cross-flow filtration experiments were performed using a plate and frame module (Alfa Laval Lab unit M20). Schematic diagram of the filtration system is shown in Fig. 1. Transmembrane pressure recorded in this study is the average pressure of inlet and outlet pressure. Four pieces of flat sheet membranes with effective area of 0.072 m^2 were used and permeate flux and rejection were evaluated. Membranes used in the study and their main characteristics are described in Table 1. The stable experiments results can be obtained after pre-treatment of NF and RO membrane [13]. The following steps of pretreatment were carried out according to membrane manufacturer's instruction.

1. A 0.2 wt% ECOLAB Ultrasil 10 solution was used for recirculation for 30 min at 50 °C, 4 bar for getting rid of the remaining chemicals in membranes.
2. Flush with distilled water.
3. Recirculate with distilled water at 25 °C, 20 bar for 3 h.

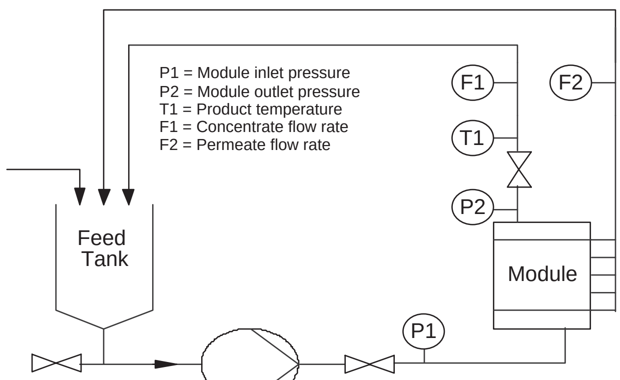

Fig. 1. Schematic diagram of Alfa Laval M20 filtration system.

Table 1
Main characteristics of membranes used in the study

Membrane	NF90	NF97	NF99	NF99HF	RO98pHt	RO99	SW
Type	NF	NF	NF	NF	RO	RO	RO
Manufacturer	Dow FilmTec	Alfa Laval	Alfa Laval	Alfa Laval	Alfa Laval	Alfa Laval	Dow FilmTec
pH range	2–11	3–10	3–10	3–10	2–10	3–10	2–11
Temperature/°C	Max. 45	Max.50	Max. 50	Max. 50	Max. 50	Max. 50	Max.45
Pressure/bar	Max.41	Max. 55	Max. 55	Max. 55	Max. 55	Max. 55	Max. 69

The feed tank contained 10 L synthetic phenol-containing wastewater (concentration <1,000 ppm) prepared from phenol and distilled water. Permeate and concentrate were recycled to the feed tank to keep a constant concentration of feed solution. Permeate samples were taken and flux was measured after recirculation for 20–30 min. The effect of sample volume which is 20 ml on the feed concentration was neglected. The permeate flux was determined by measuring the volume of the permeate collected at the given intervals. Rejection value was determined by measuring the concentration of feed and permeate. The salt concentration of solutions was measured by a Radiometer CDM92 conductivity meter. The phenol concentration of solution was measured by UVIKON-860 spectrometer at 269 nm when pH of sample is below 10 and at 287 nm when pH of sample is above 10.

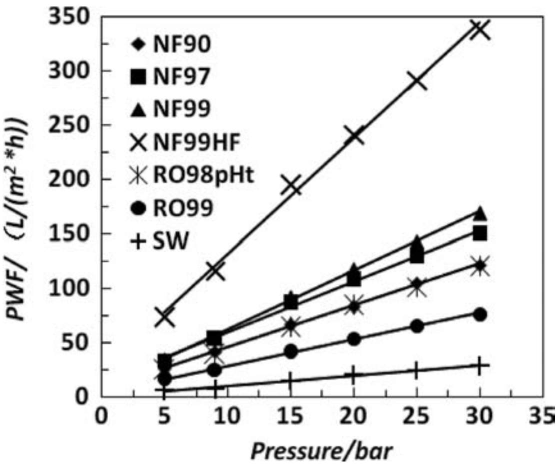

Fig. 2. Influence of pressure on the PWF.

4. Results and discussion

4.1. Membrane performance test

Pure water flux (PWF) and salt rejection are two parameters often used for the characterization of membrane performance. They were determined at the same operating conditions in this study.

4.1.1. PWF test

PWF can reflect the pore structure of membrane indirectly, it is necessary to determine PWF for different membranes at the same operating conditions.

A. Influence of pressure on the PWF

Fig. 2 displays PWF data against transmembrane pressure ranged from 5 bar to 30 bar at temperature 25 °C at cross flow of 0.58 m/s. As shown in Fig. 2, PWF increases linearly with transmembrane pressure. Water permeability constant A_w was obtained by fitting PWF over transmembrane pressure of these membranes and the A_w values are shown in Table 4. Fig. 2 shows good linear relation between PWF and pressure, which indicates that there is no compaction of the membrane due to pressure. The A_w values of the membranes used in this work decrease in the following order: NF99HF>NF99>NF97 > NF90 > RO98pHt > RO99 > SW. It should be noted that RO98pHt almost has the same A_w as NF90.

B. Influence of temperature on the PWF

Fig. 3 displays PWF data against temperature at transmembrane pressure 15 bar, cross flow 0.58 m/s. The figure shows that PWF increases with temperature lineally, and the slopes of fitting lines decrease in the following order: NF99HF > NF99 > NF97 > NF90 > RO98pHt > RO99 > SW. It was reported that temperature may reorient the polymer chains in an opened membrane [13, 14]. The slope differences among these membranes could be due to the different dense degree of the thin film layer.

C. Influence of pH on the PWF

Table 2 displays PWF data against pH at transmembrane pressure 15 bar, crossflow 0.58 m/s, temperature 25 °C. It can be seen that pH of feed has little influence on PWF of RO98pHt, RO99 and SW, but PWF of NF

Table 2
Influence of pH on the PWF (25 °C, 15 bar)

Membrane	NF90	NF97	NF99	NF99HF	RO98pHt	RO99	SW
PWF/ ($L^*m^{2}*h^{-1}$) at pH 3	40.8	80.0	113.3	212.5	63.3	40.0	15.0
PWF/ ($L^*m^{2}*h^{-1}$) at pH 5	43.8	84.2	105.8	208.3	63.3	41.3	15.3
PWF/ ($L^*m^{2}*h^{-1}$) at pH 7	45.8	86.7	93.3	204.2	64.2	41.3	15.1
PWF/ ($L^*m^{2}*h^{-1}$) at pH 9	40.8	81.7	84.2	179.2	63.3	40.8	15.1
PWF/ ($L^*m^{2}*h^{-1}$) at pH 11	40.0	79.2	80.0	166.7	63.3	40.8	15.0

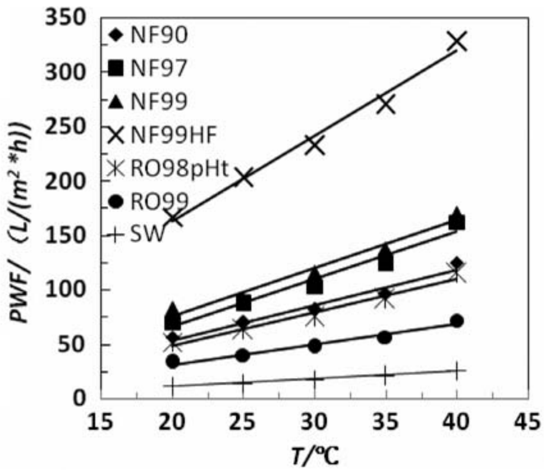

Fig. 3. Influence of temperature on the PWF.

membranes changes over the pH. The PWF of NF99HF and NF99 decreases with pH, and PWF of the NF97 and NF90 have the highest value at pH about 7. This behavior may be attributed to the charges of membranes. The increment of hydrophilic sites in the membrane material is caused by acidic hydrolysis, which is responsible for the crosslinking reduction of the polymer matrix through the disruption of chemical bonds in the polymer material [10,15]. Therefore, it is expected that decreasing in the membrane rigidity facilitates the swelling of membrane material, which causes an increment of the permeate fluxes. At higher pH values, the increase in negative zeta potential promotes stronger electrostatic interaction between dissociated functional groups of membrane material causing a pore constriction (shrinking membrane material) [10]. Thus, water flux decreases for NF99HF and NF99.

4.1.2. Salt rejection test

Salt rejection of NF and RO membrane is expressed as the rejection of $MgSO_4$ and NaCl respectively. Both NaCl and $MgSO_4$ rejection were measured at temperature 25°C, cross flow 0.58 m/s in this work (NaCl, 2 g/L, at 15 bar and $MgSO_4$, 4 g/L, at 9 bar). As shown in Table 3, NF97 and NF90 have higher salt rejection among NF membranes. All RO membranes showed higher salt rejection than NF membranes. The salt rejection difference between RO98pHt and RO99 is very small with NaCl rejection of 98.67% and 98.75%, respectively. SW has the highest salt rejection among all membranes in this work with NaCl rejection up to 99.12% and $MgSO_4$ rejection up to 99.51%.

4.2. Influences of operating conditions on the phenol rejection

4.2.1. Cross flow

Cross flow is a very important parameter in filtration which affects Reynolds number of fluid in the module. Concentration polarization can be avoided at a higher cross flow for some solutions. Experiments were carried out at pressure 15 bar, temperature 25 °C, cross flow ranged from 0.39 to 0.96 m/s with phenol concentration of 500 ppm. As displayed in Fig. 4, influence of cross flow on the rejection of phenol is small. Phenol rejection keeps the similar value at cross flow ranged from 0.39 to 0.96 m/s. Hence, effect of concentration polarization can probably be neglected in this work. Membranes that show higher salt rejection also have higher phenol rejection, but it is not the case for RO98pHt and RO99 although the difference is small. This could be explained by Solution-diffusion model [12,17]. Formula $R = \dfrac{A_w(\Delta P - \Delta\pi)}{A_w(\Delta P - \Delta\pi) + B_s}$ can be written

Table 3
Salt rejection of the membranes (NaCl, 2 g/L, at 15 bar and $MgSO_4$, 4 g/L, at 9 bar)

Membrane	NF90 (%)	NF97 (%)	NF99 (%)	NF99HF (%)	RO98pHt (%)	RO99 (%)	SW (%)
Rejection of NaCl	92.78	97.88	74.70	56.66	98.67	98.75	99.12
Rejection of $MgSO_4$	98.79	99.41	98.07	96.18	99.15	99.37	99.51

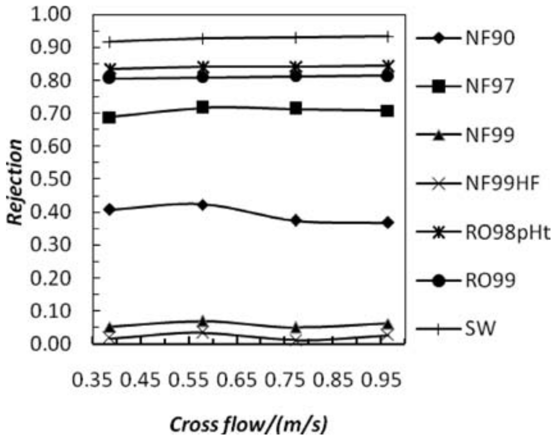

Fig. 4. Influence of cross flow on the phenol rejection.

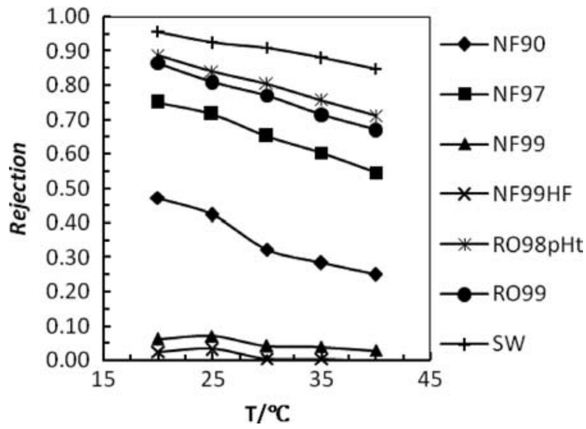

Fig. 5. Influence of temperature on the phenol rejection.

as $R = \dfrac{1}{1 + \dfrac{B_s}{A_w(\Delta P - \Delta\pi)}}$. Namely solute rejection depends on solute permeate constants B_s and water permeability constant A_w. $\dfrac{B_s}{A_w}$ of each membrane is different for different solutions. $\dfrac{B_s}{A_w}$ of RO98pHt is smaller than RO99, which is confirmed in Section 4.3.2, leading to higher rejection of RO98pHt than RO99.

4.2.2. Temperature

Fig. 5 displays phenol rejection against temperature at transmembrane pressure 15 bar, cross flow 0.58 m/s, phenol concentration 500 ppm. It can be seen that phenol rejection declines remarkably with temperature. For example, phenol rejection of RO98pHt decreases from 90% to 71%, and NF97 decreases from 70% to 55% with temperature increasing from 20 °C to 40°C. As described in Section 4.1.1, PWF increases by temperature. It seems that phenol rejection should increase according solute-diffusion model [12,17], but it is not the case in the study. The phenomenon can be explained that organic solute diffusion and absorption by membrane are enhanced by elevating temperature [13,16]. Higher temperature tends to distribute phenol more evenly between the solution and membrane phases, which means less selective partitioning, and as a result of lower phenol rejection. Phenol transport through membranes can be described as activated process which is usually expressed as Arrhenius type of equation $P = P_0 e^{(-Ea/RT)}$ (where P is permeability constant, P_0 is pre-exponential permeability coefficient, E_a is activation energy and R is gas constant). Higher permeability constant of phenol which is obtained by elevating temperature results in lower rejection. Another reason could be considered is that membrane becomes more open due to the swelling of polymer chain at higher temperature.

4.2.3. Pressure

Fig. 6 displays rejection against transmembrane pressure at 25 °C, cross flow 0.58 m/s, phenol concentration 500 ppm. It can be observed that phenol rejection (SW > RO98pHt > RO99 > NF97 > NF90 > NF99 > NF99HF) increases with pressure. Effect of pressure on phenol rejection ranged from 5 bar to 20 bar is more significant than from 20 bar to 30 bar. This can be explained by solution-diffusion model [12,17] except NF99HF. Rejection depends on concentration of feed near the surface of membrane and permeate; higher flux attained at higher transmembrane pressure results in lower

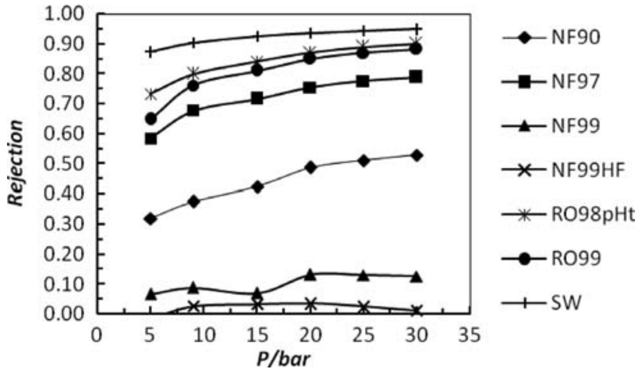

Fig. 6. Influence of pressure on phenol rejection.

permeate concentration, leading to higher phenol rejection. It is reported that phenol rejection of some membranes decreased with pressure, which may be caused by phenol adsorption into membrane [2] and phenol rejection of some membranes increased first and then decreased [18]. Therefore, phenol transport mechanism is complex, and it is not the same for different membranes. In this work, phenol rejection does not increase significantly when pressure increases from 20 bar to 30 bar especially for RO membranes.

4.3. Influences of wastewater properties on phenol rejection

The influences of the characteristics of phenol-containing wastewater such as pH, phenol concentration, and salt concentration on the phenol rejection were investigated.

4.3.1. pH

Fig. 7 displays phenol rejection against pH at the temperature 25 °C, pressure 15 bar, phenol concentration 500 ppm. It can be observed that phenol rejection (SW > RO98pHt > RO99 > NF97 > NF90 > NF99 > NF99HF) of NF membranes increases with pH. It is interesting to note that phenol rejection increases suddenly between pH 9 and pH 11. This phenomenon can be explained that pH affects dissociation of phenol and the surface charges of membrane. Degree of phenol dissociation against pH was plotted in the Fig. 8. It can be observed that the change of phenol dissociation degree and the change of phenol rejection over pH have similar pattern (compare Figs. 7 and 8). The surface charges of NF membrane are affected by pH [19]. Generally most polymeric NF membranes have a positive surface charge in very acidic conditions (pH < 3), and isoelectric points (IEP) are normally between pH 3 and 6 [20,21]. Zeta potential is an indicator of the surface charge of NF membranes. It

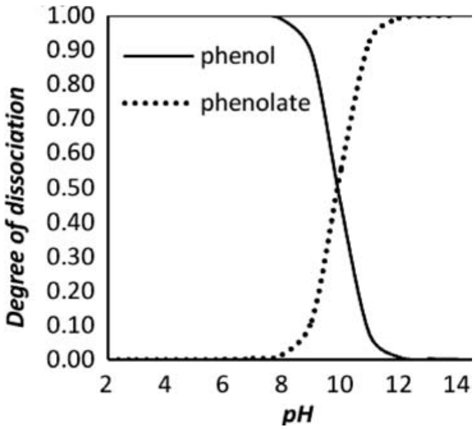

Fig. 8. Influence of pH on phenol dissociation.

is reported that Zeta potential of NF90 membrane shifts from about 5 mV to –20 mV when pH is from 3 to 12 and its isoeletric point is about pH 3.5, which is determined from streaming potential measurements using 0.001 M KCl solution [19]. Namely in pH range 3.5–12, higher pH means higher charge density. The higher phenol rejection in pH range 9–11 can be explained by the electrostatic repulsion between negative charges of membrane surface and the phenolate ions. RO membranes display almost the same trend but not as significant as NF membranes. This may be attributed to lower charges density on RO membrane surface than NF membranes. There is limited potential for RO membranes to increase the phenol rejection by varying pH, because phenol rejection of all RO membranes is more than 80%.

4.3.2. Phenol concentration

Fig. 9 shows phenol rejection against the phenol concentration in synthetic wastewater at temperature 25 °C, pressure 15 bar, cross flow 0.58 m/s. As can be seen from

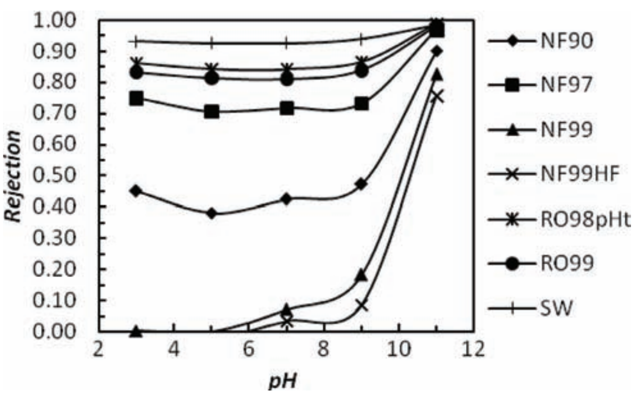

Fig. 7. Influence of pH on phenol rejection.

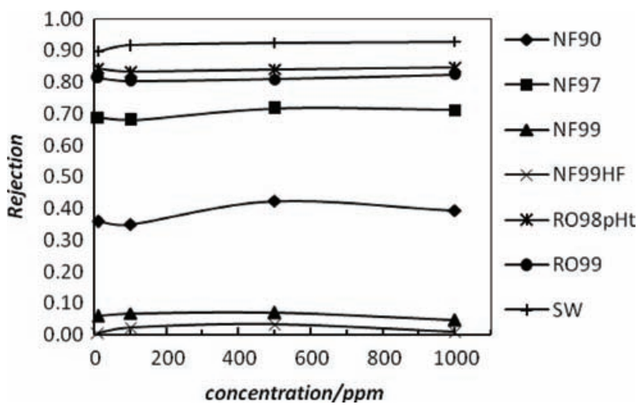

Fig. 9. Influence of feed concentration on phenol rejection.

Table 4
Water permeability and phenol permeate constants of membranes at 25 °C

Membrane	NF90	NF97	NF99	NF99HF	RO98pHt	RO99	SW
A_w/(L*m^{2}*h^{-1}*bar^{-1})	3.8536	4.6823	5.4118	10.6610	3.8370	2.4525	0.9776
B_s /(m*h^{-1})	99.906	34.499	1709.2	5357.9	11.3894	8.712	1.155

Table 5
The relation between B_s/A_w and phenol rejection

Membrane	NF90	NF97	NF99	NF99HF	RO98pHt	RO99	SW
A_w/(L*m^{2}*h^{-1}*bar^{-1})	3.8536	4.6823	5.4118	10.6610	3.8370	2.4525	0.9776
B_s/(m*h^{-1})	99.906	34.499	1709.200	5357.900	11.389	8.7120	1.1550
B_s/A_w (L^{-1}*m^{-1}*h^{-1}*bar^{-1})	25.925	7.368	315.828	535.790	2.968	3.552	1.182
R	0.41	0.72	0.07	0.03	0.84	0.81	0.93

the figure, concentration of synthetic wastewater has little influence on the phenol rejection in the range of this work. Phenol permeability constant B_s with the following order: NF99HF > NF99 > NF90 > NF97 > RO98pHt > RO99 > SW can be obtained by plotting of $J_w C_p$ versus $(C_f - C_p)$, shown in Table 4. It seems that membranes which have larger B_s also have lower phenol rejection, but it is not the case for RO98pHt and RO99, because solute rejection depends on solute permeability constants B_s and water permeability constant A_w according to formula $R = \dfrac{A_w(\Delta P - \Delta\pi)}{A_w(\Delta P - \Delta\pi) + B_s}$. Membranes with lower $\dfrac{B_s}{A_w}$ value have higher rejection. The relationship between A_w, B_s and phenol rejection at 15 bar, 25 °C of membranes used in this study is given in Table 5. For phenol solution, $\dfrac{B_s}{A_w}$ of RO98pHt is smaller than $\dfrac{B_s}{A_w}$ of RO99. Therefore, phenol rejection of RO98pHt is larger than RO99 although RO99's B_s is smaller.

4.3.3. Salt concentration

Influence of salt concentration from 1,000 to 3,000 ppm on the phenol rejection and flux was investigated. Filtration of synthetic wastewater with phenol concentration of 500 ppm was carried out at temperature 25 °C, cross flow 0.58 m/s, pressure 15 bar. NaCl was chosen as a model salt in this work. Fig. 10 displays the influence of NaCl concentration on phenol rejection. It seems that phenol rejection is not influenced by NaCl concentration in the range of this work. All the membrane used in this work keep the same phenol rejection at different NaCl concentrations from 1,000 to 3,000 ppm. But NaCl concentration in the feed showed pronounced influence on flux as seen from Fig. 11. Flux of each membrane decreases significantly when NaCl concentration increases from 0 to 3,000 ppm. This phenomenon is due to osmotic pressure. Feed pressure can be offset when increasing salt concentration in the feed. On the other hand, phenol concentration also influences the flux. Decrease in flux was observed for the membranes used in this study at phenol concentration between 0 and

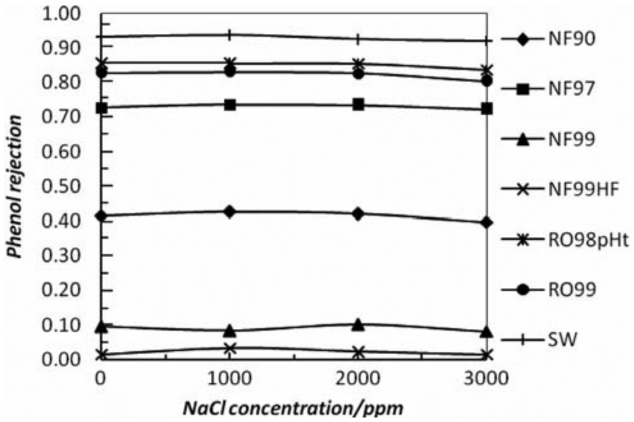

Fig. 10. Influence of NaCl concentration on phenol rejection.

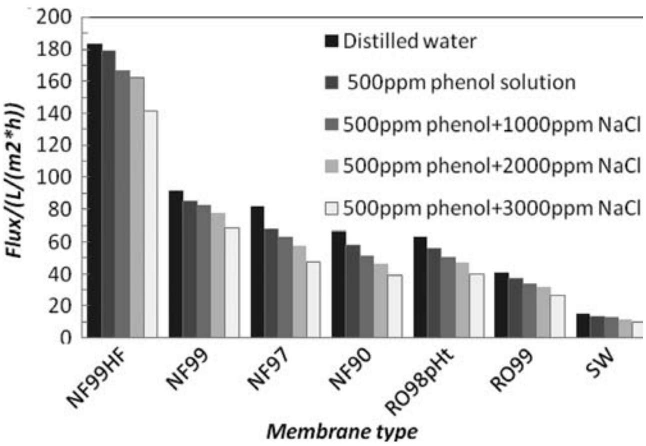

Fig. 11. Influence of NaCl concentration on flux.

500 ppm. But phenol rejection is not affected as shown in Fig. 9. In reality, all wastewater contains salts with low or high concentrations. Salt concentration probably only affects the permeate flux, but not phenol rejection.

5. Conclusions

In the present work, NF membranes (NF90, NF97, NF99, NF99HF) and RO membranes (RO98pHt, RO99, SW) were chosen to reject phenol in a model low concentration phenol-containing wastewater with concentration lower than 1,000 ppm. Water and phenol permeability constants have been obtained according solution–diffusion model. Cross flow rate ranged from 0.39 to 0.96 m/s and phenol concentration ranged from 10 to 1,000 ppm have small effect on phenol rejection. Phenol rejection decreases with temperature as diffusion and sorption are enhanced at elevating temperature. Phenol rejection increases with pressure. The pH value has significant influence on phenol rejection, which can be explained by the change of phenol dissociation degree and the surface charges of membrane. The NaCl concentration in the range of 1,000 to 3,000 ppm has no influence on phenol rejection but has significant negative influence on flux. All NF membranes used in this work show lower phenol rejection than RO membranes, but higher flux, which is expected. NF97 showed the best performance among NF membranes considering both flux and phenol rejection. RO membrane SW has the highest phenol rejection, while the flux is the lowest. RO98pHt has second highest phenol rejection among all membranes and shows relatively high flux, almost the same as NF90. Therefore, NF97 and RO98pHt are the preferred membranes to be used for treating low concentration phenolics-containing wastewater especially alkali phenolics-containing wastewater.

Nowadays, methods of treating phenolics-containing wastewater focus on how to reuse phenolics and meet the standard of wastewater discharge using combined techniques. Conventional extraction has many advantages such as easy to operate, higher removal of phenolics, economically attractive, but it is only suitable for high concentration phenolics-containing wastewater. So combining conventional extraction with membrane filtration would be a good choice. Multistage process of membrane filtration might be feasible to concentrate low concentration phenolics-containing wastewater (<1,000 ppm).

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Symbols

R — solute rejection coefficient
C — solute concentration, g/L
J_w — pure water flux, L/(m^2*h)
J_s — solute flux, g/(m^2*h)
ΔP — transmembrane pressure, bar
$\Delta\pi$ — osmotic pressures of the solutions, for distilled water $\Delta\pi = 0$, bar
A_w — water permeability constant, L/(m^2*h*bar)
B_s — solute permeability constant, m/h

Subscripts

f — feed
P — permeate
w — water

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