Retention of sulfate and chloride ions in commercially available tubular membranes

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Abstract. Performance evaluation of four commercially available tubular membranes (AFC 80, AFC 30, PU 608, ES 404) was accomplished in self-assembled membrane testing unit. Effects of varying transmembrane pressure, feed concentration and anion type were investigated. Aqueous solutions of salts such as calcium chloride, calcium sulfate, tin chloride and tin sulfate were prepared for this study. It was noted that the investigated parameters e.g., pressure and concentration had significant effects on membrane's performance. Nevertheless, anion type effectively played its role in the rejection of salts since salt having SO4-2 anions had a better rejection than the salts containing Cl-1. It is observed that rejection was dominated by Donnon exclusion for strongly charged nanofiltration membranes whereas for weakly charged ultrafiltration membranes, size exclusion was the key mechanism to reject the ions.

Keywords: tubular membranes; nanofiltration; ultrafiltration; salt analysis; tin salts; performance analysis

1. Introduction

Tubular membrane is one of the membrane types that comes in tube and long shape and is operated in their own module. This type of membrane can be individually used or arranged in bundle which depends on the type of module (Ji *et al.* 2015). Generally, the feed for tubular membrane is entered in cross-flow direction which means the feed is pumped along the membrane tubes and permeate pass through the surface of the membranes. The rigid structure of the tubular membrane enables the membranes to be operated at high transmembrane pressure and in turbulence mode of operation (by creating turbulence through inserting turbulence spacer or other medium) (Ji *et al.* 2016). Industry with high mass production prefers this type of membrane due to its durability. Tubular membranes are preferred for longer period of operation due to their less fouling tendency when operated under crossflow feed stream. Hence, low fouling in this type of membranes reduces the overall operational cost (Damak *et al.* 2005, Motin *et al.* 2015).

Tubular membranes have been widely used for various industrial applications such as heavy metal removal (Gherasim *et al.* 2013, Gherasim and Mikulášek 2014, Gherasim *et al.* 2015), metal complexes removal (Lastra *et al.* 2004), dye removal (Zahrim *et al.* 2013), concentrating the

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juices(Warczok *et al.* 2004), etc. Nevertheless, nanofiltration and ultrafiltration type tubular membranes have been excessively employed for water and wastewater treatment purposes (especially for removal of divalent ions) (Efligenir *et al.* 2014, Jie 2014) because of their distinctive performance in comparison to conventional technologies i.e., ion exchange, liquid-liquid extraction, precipitation and adsorption (Gherasim *et al.* 2013).

Performance of these membranes is a result of many factors like membrane properties and feed solution type. Membrane characteristics such as porosity, structure, pore size distribution, thickness; surface charge and membrane material are among these characteristics. Apart from membrane characteristics, feed solution properties such as feed concentration, feed pH, and feed composition are also of vital importance (Bartels *et al.* 2005, Hu *et al.* 2013, Luo and Wan 2013).

Separation of solutes in pressure driven membranes (such as reverse osmosis, nanofiltration, ultrafiltration and microfiltration) is either a result of combination of mechanisms such as diffusion, convection and electrostatic repulsion or it is dominated by any single mechanism (Bruggen et al. 2003, Agarwal and Goswami 2016). Rejection of solutes in nanofiltration is a complex process since diffusion, convection and strong surface charge, all of them play critical role. In the presence of ionic feed, surface charge of the membrane causes attraction for counter ions while co-ions are repelled by membrane charge, such separation is called Donnon exclusion as reported by many in literature (Lalia et al. 2013, Mancinelli and Hallé 2015). Donnon exclusion is reported to be affected adversely in case of higher feed solutions since excessive cations attach themselves to membrane surface (negative) and subsequently diminish the membrane surface charge, which results in passage of anions through membrane causing a decrease in rejection. It is important to note that the separation through ultrafiltration membranes is mainly due to sieving mechanism or separation on solute size basis. These membranes have weak surface charges, hence role of Donnon exclusion is limited or insignificant in case of higher feed concentrations (Mancinelli and Hallé 2015, Mohammad et al. 2015). A common problem with such membranes is concentration polarization which occurs when rejected solute gets accumulated on the surface of the membrane and it causes a reduction in overall flux of the membrane. Such observations have been previously reported by many researchers in their work (Damak et al. 2005, Luo and Wan 2013, Gherasim et al. 2015). Concentration polarization can be controlled by varying the crossflow rate of the feed streams as shown by (Sablani et al. 2001, Koyuncu and Topacik 2004).

In this work, four commercialized tubular membranes have been used in order to study the effects of feed parameters i.e., types of salts, feed concentration, transmembrane pressure on salt retention. The experiment has been conducted using four types of salts i.e., calcium chloride (CaCl₂), calcium sulphate (CaSO₄), tin chloride (SnCl₂) and tin sulphate (SnSO₄). The study of commercialized tubular membranes is important to assess the capability of commercially available membranes to reject the tin salts, operational issues as well as to generate a data for tin salts rejection. For this investigation, experiments were run at constant crossflow rate of 1.5 litres per minute and transmembrane pressure from 2 to 10 bar, while feed concentration was gradually raised from 2500 ppm to 10,000 ppm. The findings of this study will be a significant addition to tin based metal salts removal from wastewater since no extensive studies have been found in literature.

2. Experimental

The performance of commercialized tubular membranes was studied in term of pure water flux,

Table 1 Summary of tubular membranes information obtained from PCI membrane manual

Membrane Name	Material	Apparent Retention Character	Type of Membrane	Pure Water Permeability (L_p) $(L.m-^2.h-1.Bar^{-1})$ *This study
ES404	Polyethersulphone	4,000 MW	UF	15.75
PU608	Polysulphone	8,000 MW	UF	21.1
AFC80	Polyamide Film	80% NaCl	NF	2.91
AFC30	Polyamide Film	75% CaCl ₂	NF	6.32

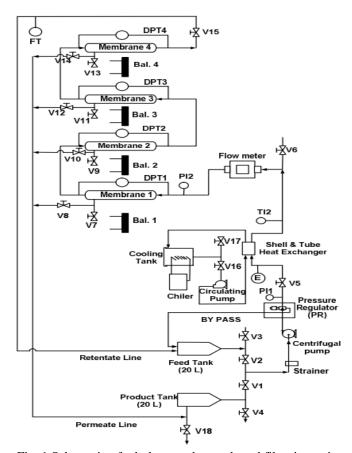


Fig. 1 Schematic of tubular membranes based filtration unit

permeate flux and anions type. This unit was constructed with four membrane housing tubes, arranged in parallel housing. Feed parameters such as feed concentration and operating pressure were varied for both membranes to observe their effects on flux and salt rejection.

2.1 Materials

Deionized water of 18 m Ω was used during the experiments to study pure water flux and also

to prepare the salt solutions. Four types of salts were used; calcium chloride (CaCl₂), calcium sulphate (CaSO₄), tin chloride (SnCl₂) and tin sulphate (SnSO₄). The salts were purchased from Merck, Acros. and R&M. All four membranes were single-tube tubular membranes and were purchased from PCI Limited (United Kingdom) with length of each tube is 1.2 m, the outer diameter of the tube is 12.5 mm and the thickness of each tube is 0.5 mm. The summary for the details of the commercialized membranes was shown as in Table 1.

2.2 Equipment

In this work, a lab scale membrane test unit SOLTEQ model TR08 was used throughout the experiments shown in Fig. 1. The cross flow feed was pumped simultaneously to the parallel tubes. Permeate and retentate were fed back to feed tank to keep the bulk feed concentration constant. Feed and permeate samples were collected until steady state condition (permeate flux and permeate concentration) was achieved. A conductivity meter from Hanna Instrument model EDGE was used to measure the electrical conductivity of the salt solutions. Permeate was collected through gravimetric method where a weighing balance (GF3000) was attached to computer for continuous monitoring. Resolution of weighing balance GF3000 was 0.01 g whereas the resolution of HANNA EDGE meter was 0.01μ S/cm.

2.3 Pure water flux

The assembled membranes were allowed to run with deionized water for 30 minutes at maximum working pressure of 10 bar. The cross flow rate was maintained at 1.5 litres per minute (LPM) in order to remove the trace residue attached to the membranes and to allow the membranes to be compressed. Then operating pressure was varied from 2 bar to 10 bar. Samples for each pressure were noted after one minute interval for five minutes and mean value is reported here. The standard deviation was calculated to be ± 0.5 . The effective area of the membrane was calculated as 0.05 m^2 and the flux (J_{ν}) measured in unit L/h.m² was determined by using equation as below (Li *et al.* 2011)

$$J_{v} = \frac{V}{A.t} \tag{1}$$

Where "V" is the volume of permeate in unit litre (L), "A" represents the effective area of the membrane in unit meter square (m^2) and "t" is the time taken to collect permeate in unit hour (h).

2.4 Rejection studies

Rejection studies were accomplished by running the aqueous solutions of different feed salts at set conditions. Effect of transmembrane pressure on rejection was determined by introducing the aqueous solution of feed salts at 2500 ppm and crossflow rate of 1.5 (LPM) and then gradually varying the pressure. Only 100 ml sample of permeate was taken for rejection studies and effect of this volume on bulk concentration was neglected. Permeate samples were taken after steady state was achieved which usually took 15±1 minutes. Between the different feed concentration tests, filtration set up was rinsed and washed with the water until original pure water permeability was achieved. Moreover, effect of feed concentration on membrane's performance was evaluated by providing the system with tested feed concentrations of 2500 ppm, 5000 ppm, 10,000 ppm at 10

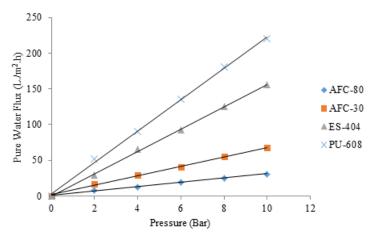


Fig. 2 Pure water flux (PWF) for tubular membranes at crossflow rate of 1.5 LPM

Bar and 1.5 LPM. Permeate was collected through gravimetric method, a weighing balance was attached to a computer for continuous data collection. Concentrations of feed and permeate were measured by EDGE conductivity meter (HANNA) and observed rejection ($\%R_{obs}$) was calculated by the formula as follows (Li *et al.* 2011)

$$\% R_{obs} = (1 - C_p/C_f) *100$$
 (2)

Where " C_p " denotes the permeate concentration (ppm) and " C_f " corresponds to the feed concentration (ppm).

3. Results and discussions

3.1 Pure water flux

Fig. 2 shows the relations of pure water flux of each tubular membrane with transmembrane pressure. It can be seen that pure water flux increases linearly with the increase of operating pressures as expected. It is noteworthy that among nanofiltration membranes used here, AFC30 had higher pure water flux (67.31 L.m⁻².hr⁻¹ @10bar) than AFC80. Moreover, among ultrafiltration membranes tested in this study, PU608 had higher pure water flux (220.6 L.m⁻².hr⁻¹ @10bar) than ES404. This pure water flux test provides a benchmark to the changes in permeate flux with pressure varying to see either the membrane was fouled or not when tested for the salt solution (Huang *et al.* 2014). When pressures were applied to these membranes, the flux responded to the applied pressure with linear correlation, showing absence of any further compaction effects. Since in case of compaction, flux increases linearly at low pressure but becomes almost constant or plateau at higher pressure and is often categorized as irreversible fouling (Liu *et al.* 2015, Padaki *et al.* 2015, Ray *et al.* 2015).

3.2 Effect of trans-membrane pressure on rejection

Figs. 3 and 4 show the rejection and permeate flux of four salts on above mentioned tubular

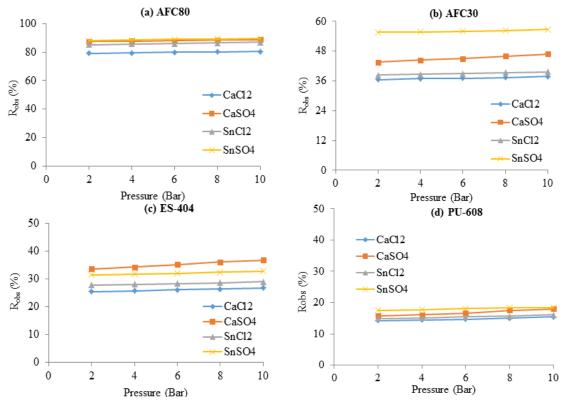


Fig. 3 Effect of feed pressure on salts rejection of tubular membranes at feed concentration of 2500 ppm and crossflow rate 1.5 LPM

membranes respectively. A linear relationship was observed between transmembrane pressure and rejection as slight increase in rejection was observed with an increase in transmembrane pressure. Also retention appears to be influenced by the Donnan Exclusion phenomenon in case of negatively charged membranes here. It is noticed that sulfate based ions had higher rejections than their counterparts e.g., chlorides of the same cations, as noted by (Huang *et al.* 2014, Mehdipour *et al.* 2015) in their study. These results are quite consistent with the theory, as increase in pressure results in excessive convective flow towards the membrane causing more solvent as well as ions to the membrane surface, hence resulting in more rejection and permeate flux (Gherasim *et al.* 2013, Mehdipour *et al.* 2015). It is noted that PU608 had higher rejection than the ES-404 membrane because of its tighter porous structure. On the other hand, in case of nanofiltration membranes, AFC80 had better rejection than AFC30 membrane.

It is found that between the two nanofiltration membranes, rejection of tin sulfate is higher than the rejection of calcium sulfate, though the difference of the rejection in these membranes is significantly higher in case of AFC 30 nanofiltration membrane. Likely reason for this could be the slightly porous structure of its membrane, which can be noted from apparent retention character shown in Table 1. The same effect can be seen for the ultrafiltration membrane tested in this study, where ES 404 showed better tin ion rejection than the PU608 ultrafiltration membrane since ES 404 had a tighter pores than the PU608. Hence, calcium ion being the smaller one than its competitive ion (i.e., tin ion) moved through the membrane pores easily resulting in lesser

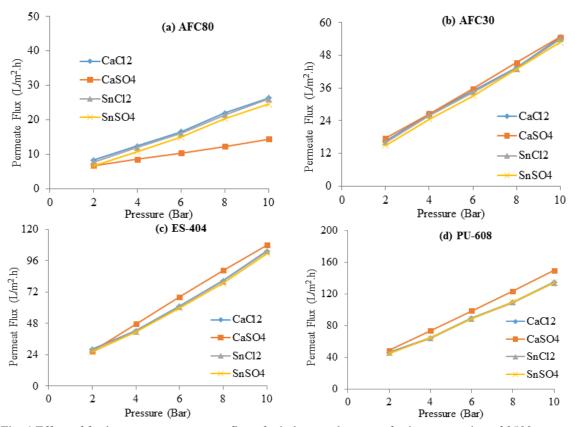


Fig. 4 Effect of feed pressure on permeate flux of tubular membranes at feed concentration of 2500 ppm and crossflow rate 1.5 LPM

rejection. From the Fig. 3, it can be comprehended that for the membranes which had better rejection of tin sulfate salts as seen in Fig. 3(b) and (d), had loose structure and their rejection was the result of Donnan exclusion and size exclusion, for nanofiltration as well as ultrafiltration membranes tested here. Whereas membranes with the comparatively tighter structure than their counterparts among nanofiltration and ultrafiltration membranes had Donnan Exclusion as the dominant rejection mechanism as can be seen here in Fig. 4(a) and (c). Fig. 4 also highlights that permeate fluxes during the experiment for each membrane seems to sustain a linear relationship with the transmembrane pressure as theory suggest.

3.3 Effect of feed concentration on rejection

Figs. 5 and 6 present the rejection and permeate flux of four salts at different feed concentrations for tubular membranes. It is noted that the feed concentration has inversely affected the salts rejection as can be seen in Fig. 5. Presence of increased amount of ions reduced the membrane surface charge through shielding effect causing less repulsion forces for coions in solution. Hence, overall rejection decreased because dominant charge effect weakened and more ions passed through membranes to maintain the electroneutrailty across the membrane. This phenomenon for nanofiltration membranes have also been reported earlier by many researchers

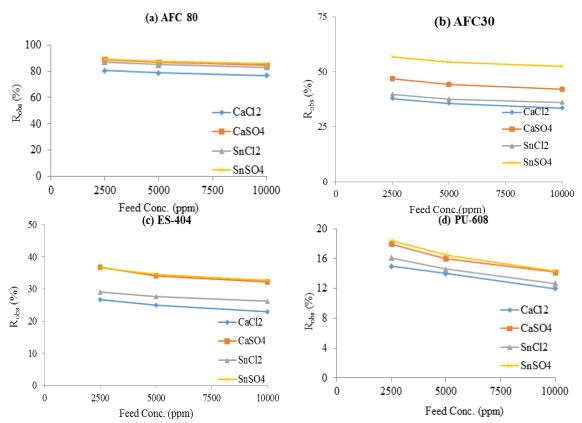


Fig. 5 Effect of feed conc. on salts rejection of tubular membranes at feed pressure of 10 Bar and crossflow rate 1.5LPM

(Shamsuddin *et al.*, Mehdipour *et al.* 2015). This phenomenon seems to be in effect here in Fig. 5, where observed rejections for all salts decreased as concentration of salts in feed is increased. It is assessed that among all four tubular membranes, AFC80 nanofiltration membrane had the least (4.8% for CaCl₂) percentage decrease in observed rejection during the feed concentration change while PU608 suffered up to 22.28% reduction for SnSO₄ in retention.

Reduction in permeate flux due to increased feed concentration is also observed here. Fig. 6, below explains the trend noted during this experiment when feed concentration was increased from 2500 ppm to 10,000 ppm for all four salts investigated here. This adverse effect of feed concentration on permeate flux is attributed to the fact that viscosity and osmotic pressure of solution soars up linearly as feed becomes concentrated. It is believed that increased osmotic pressure causes a significant reduction in net driving force of filtration at constant pressure conditions, hence resulting in less permeate flux. Also, an increase in viscosity may cause concentration polarization layer on membrane surface resulting in a non-recoverable resistance, eventually dropping the overall permeate flux of solution as mentioned by (He *et al.* 2008, Wang *et al.* 2014) in their work. It is noteworthy here, that a significant decrease in permeate flux of CaSO₄ for AFC80 membrane is unclear. But it could be the result of cake-enhanced concentration polarization layer on the membrane surface since AFC80 appears to have a relatively tighter structure as can be deduced from Table 1. Also, the less soluble nature of CaSO₄ and high

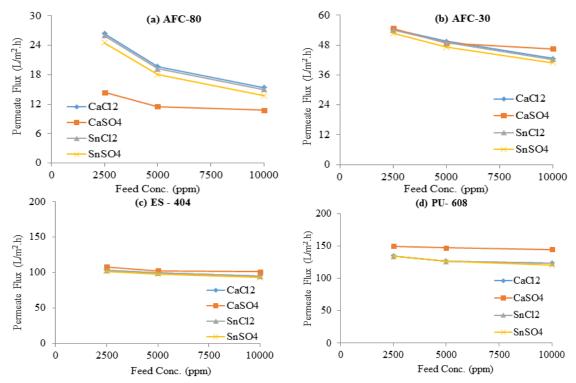


Fig. 6 Effect of feed conc. on permeate flux of tubular membranes at feed pressure of 10 Bar and crossflow rate of 1.5LPM

concentration (i.e., 2500 ppm and above) would have played role to develop this cake layer on its surface which subsequently caused the significant flux decrease in AFC80 nanofiltration membrane. The same phenomenon was observed by (Hoek & Elimelech 2003) for NaCl and LaCl $_3$ rejection studies.

3.5 Effect of anions types on rejection

Fig. 7 here shows the rejection of different anion types for investigated membranes respectively. It is noted that the presence of $(SO_4)^{-2}$ ions showed the higher rejection in comparison to chloride ions (Cl⁻) for each membrane. Higher rejection of sulphate ions is likely due to controlling phenomenon of Donnan Potential across the membranes which are negatively charged. Since, sulphate ions have bigger valence than chlorides ions, it is safe to assume that membrane charge effectively pushed the sulphate ions away from membrane consequently giving the higher rejections. In contrast chloride ions being smaller in ionic size feel lesser electrostatic force and successfully escape from the membrane surface causing lesser rejection as seen in this test and reported by (Peeters *et al.* 1998, Montalvillo *et al.* 2014, Wang *et al.* 2014) in their studies.

4. Conclusions

A performance evaluation in terms of pure water flux (PWF) and salt retention ($\%R_{obs}$) for

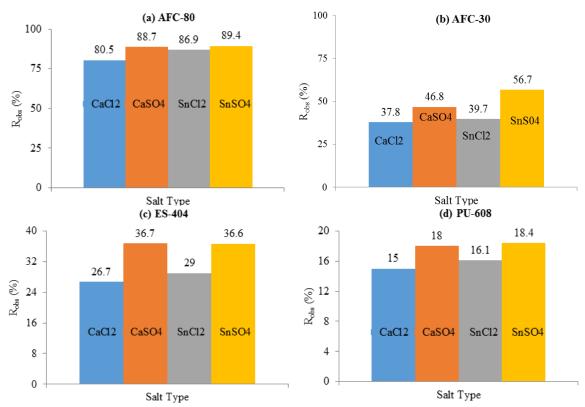


Fig. 7 Effect of anion types on salts rejection of tubular membranes at feed pressure of 10 Bar, feed concentration of 2500 ppm and crossflow rate of 1.5 LPM

commercially available tubular membranes was accomplished. Following conclusions can be drawn from the study:

- A linear relationship was observed between transmembrane pressure and fluxes as expected in such membrane separation usually.
 - Increase in feed concentration slightly reduced the rejection for these membranes.
- Rejection is dominated by Donnan Exclusion in AFC30 and AFC80 nanofiltration membranes with tight porous structure and strong surface charge.
- Separation mechanism for ultrafiltration membranes (ES404 & PU608) was mainly dominated by size exclusion mechanism.
 - Effect of ionic sizes of cations significantly increased the overall rejection of the membranes.
 - Salts with divalent anions had better rejection than the monovalent anion types.
- Rejection sequence for all tubular membranes tested here, was found to be as follows; CaSO₄≥SnSO₄>SnCl₂>CaCl₂.

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