1   **Reverse osmosis treatment of condensate from ammonium nitrate production:**

2   **insights into membrane performance**

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21   **Abstract:** Ammonium nitrate is an important fertilizer and industrial explosive. The production of
22   ammonium nitrate entails the generation of a large volume of condensate laden with nitrogen that must
23   be treated before environment discharge. Results in this study show that through appropriate membrane
24   selection, over 90% rejection of ammonium nitrate can be achieved by reverse osmosis (RO) filtration.
25   Using RO (which is highly compact and efficient) to enrich ammonium nitrate in the condensate would
26   significantly reduce the size of the evaporation separator for ammonia recovery. The results also
27   highlight the importance of membrane selection for this application. Results reported here suggest that
28   a low pressure RO membrane (e.g. ESPA2) is more suitable for the dilute condensate while a high
29   pressure RO membrane (e.g. SW30) is recommended for the concentrated condensate to ensure
30   adequate ammonia and nitrate rejection. Ammonia and nitrate rejections were dependent on key
31   operating parameters including applied pressure (or water flux), temperature, feed solution pH, and
32   initial ammonium nitrate concentration in the condensate. The impact of operating conditions on
33   ammonia and nitrate rejections was more profound for low pressure (thus high flux) than high pressure
34   RO membrane. Since the condensate is a clean matrix of high nitrogen content, results from an
35   extended filtration experiment show no or little risk of membrane fouling as expected.

1. Introduction

Nitrogen fertiliser is an essential input for agricultural production [1-3]. According to the International Fertiliser Association, each year the world consumes about 100 million tonnes of nitrogen fertiliser. Nitrogen fertiliser is predominantly used in the form of calcium ammonium nitrate, urea, and liquid ammonium nitrate. The demand for fertiliser is particularly high in the Indo-Pacific given the significant economic development in this region in recent years. As an example, the fertiliser market of Vietnam is estimated at about 2 billion US$ and is growing at 4.9% over the projected period of 2021-2026. In 2020, Vietnam has begun to produce their own calcium ammonium nitrate through VinaComin, which is an industrial conglomerate on coal and mineral mining [4].

In addition to its role as a nitrogen fertiliser, ammonium nitrate is also an important industrial explosive widely used in the mining and construction industry. Ammonium nitrate is an oxidiser and can be mixed with fuel oil (or any other fuels) to form a range of customised and controllable explosive blends (called ANFO) for blasting in mining and construction projects. Ammonium nitrate is considered a safe industrial explosive. However, when incorrectly stored or handled, it can cause catastrophic accidents such as the recent deadly Beirut blast on 4 Aug 2020. In the wrong hands, ammonium nitrate can also be converted to improvised explosive devices. Thus, commercial production, trade, and usage of ammonium nitrate are carefully regulated by most authorities.

The production and usage of nitrogen fertiliser also entail significant environmental consequences. Nitrogen pollution of surface water has become a global problem [5]. When combined with adverse climatic conditions, nitrogen pollution is main cause of harmful blue green algae blooms with billions of dollar in direct and indirect economic damages [6]. The production of nitrogen fertiliser is also energy intensive. Recent introduction of the Carbon Border Adjustment Mechanism and revision of the Emission Trading System (ETS) by the European Commission are expected to significantly affect the nitrogen fertiliser industry, which is responsible for about 1% of global CO_2 emission [7]. There have been many dedicated research works to develop techniques for reducing nitrogen leaching into run-off water and thus increasing nitrogen uptake efficiency for agricultural production. Examples of these techniques include polymer coating and encapsulation to control nitrogen release [8-10], and optimise fertiliser application [11]. In addition to nitrogen leaching during crop production, the production of ammonium nitrate can also result in a significant volume of wastewater laden with nitrogen.

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68 Condensate is the most significant waste stream from industrial ammonium nitrate production. It is
69 essentially excess water after the exothermic reaction between ammonia and nitric acid in a reactor.
70 The heat simultaneously released from this exothermic reaction is harnessed to facilitate the
71 evaporation of the excess water to concentrate the ammonium nitrate solution in the reactor. The
72 evaporated steam is then condensed to form condensate. Due to the carried over effect, the
73 concentration of nitrogen in condensate water can be as high as 5 g/L ammonium nitrate and 2.5 g/L
74 as NH_3 [12]. This wastewater has little or no organic carbon content; thus, it is not suitable for
75 biological wastewater treatment unless a carbon source (e.g. methanol or sugar) can be supplied. The
76 high nitrogen content, mostly in the form of ammonia and nitrate, also provides an excellent
77 opportunity for product recovery to improve production efficiency and reduce the overall carbon
78 footprint of ammonium nitrate.

79 Recent research works have explored the potential of ion exchange and reverse osmosis (RO) to
80 recover ammonium nitrate and purify the condensate from ammonium nitrate production for
81 environmental discharge. Kauspediene and Snukiskis [13] investigated the application of cation
82 exchange resin Purolite to recover ammonia from condensate wastewater. Their technique was only
83 effective for ammonia recovery and they did not demonstrate nitrate recovery [13, 14]. In other words,
84 the nitrogen content in the form of nitrate in the effluent was not affected. Membrane contactor has
85 been used for ammonia recovery from wastewater [15, 16]. It is also not suitable for ammonium nitrate
86 condensate. In a pilot study, Noworyta et al. [17] used a three stage RO membrane system to
87 concentrate the condensate for ammonium nitrate recovery. Noworyta et al. [17] did not report detailed
88 performance data of the pilot RO plant, particularly the membrane rejection against individual ions in
89 the condensate; however, they confirmed that it was possible to achieve ammonia and nitrate in the
90 treated water of at below 35 mg/L and 50 mg/L, respectively, for environmental discharge [17]. Apart
91 from the few previous studies discussed above, there has been very little research on ammonium nitrate
92 recovery for practical application. This is in sharp contrast to the recent expansion of the nitrogen
93 fertiliser industry especially in emerging economies in the Indo-Pacific region.

94 RO membrane is modular, highly compact, and can enrich ammonium nitrate for subsequent
95 recovery by the evaporation separator. In other words, a compact RO system can significantly reduce
96 the size of the evaporation separator and improve ammonium nitrate recovery. In fact, modular RO
97 systems have been used for mineral and clean water recovery in mining and a wide range of industrial
98 applications [18-20]. Although information about ammonium nitrate rejection by RO membrane from

99   condensate water is scarcely available, their individual rejection from a diluted aqueous solution has

100   been widely reported in the literature [21-23]. Ammonium rejection by RO membrane can be lower

101   than other cations [23]. It may also be affected by the speciation between ammonia and ammonium as

102   a function of pH and operating temperature [23]. It is noteworthy that the condensate from ammonium

103   nitrate production has highly variable pH and temperature. As a major advantage, RO membrane

104   systems are highly compact, modular, and can be readily integrated or retrofitted to existing production

105   facilities to reduce the footprint of the evaporation separator used for ammonium nitrate recovery.

106   Thus, it is imperative to understand the separation performance under a range of conditions (e.g. ion

107   concentrations, pH, and temperature of the condensate) for RO membrane selection and process design.

108   This study aims to delineate the performance of a range of RO membranes for concentrating the

109   condensate for ammonium nitrate recovery and compliance with treated water discharge standards.

110   The impact of applied pressure, condensate pH and temperature, and fouling propensity were

111   systematically investigated to evaluate the recovery of ammonium nitrate from condensate water.

112   Operating conditions examined in this study are common to full-scale ammonium nitrate production

113   facilities.

114   ## 2.   Materials and methods

115   ### 2.1.   Process condensate supply and analysis

116   A simplified flow sheet of ammonium nitrate production is shown in Fig. 1 to describe the two

117   types of condensates from the process. The concentrated condensate is formed from the removal of

118   excess water to produce concentrated or dry ammonium nitrate. Because ammonia is significantly more

119   volatile than nitric acid, the concentrated condensate usually has more ammonia than nitric acid in

120   molar concentration. In some facilities, nitric acid is injected to the concentrated condensate to achieve

121   an equal molarity with ammonia so that ammonium nitrate can be recovered via an evaporation

122   separator. The evaporation separator produces an ammonium nitrate solution and a dilute condensate

123   that must be treated prior to environmental discharge.
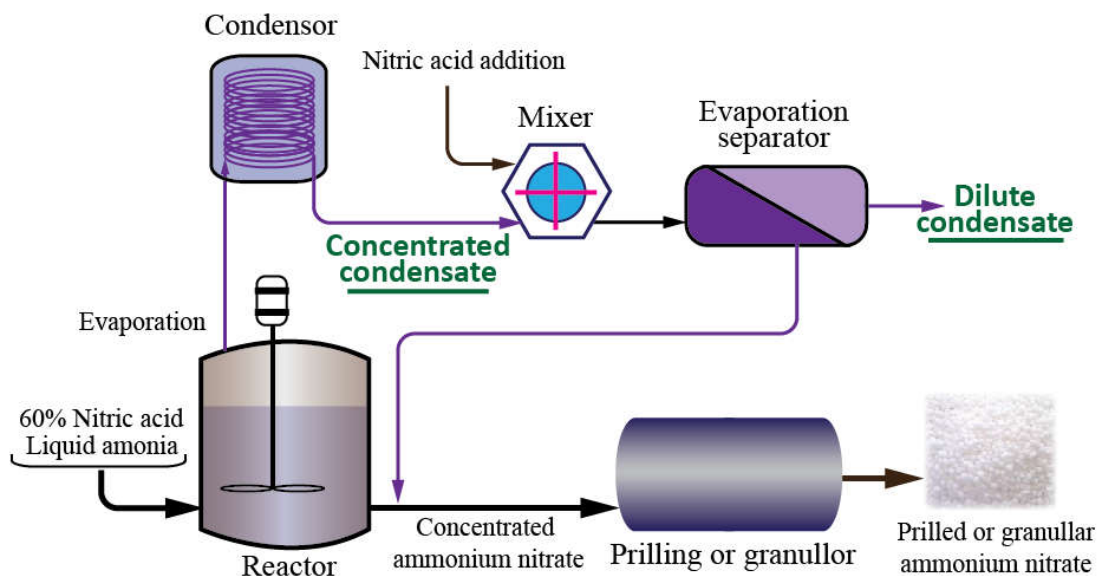
Fig. 1. The schematic diagram of an ammonium nitrate production process.

The concentrated and dilute condensates were collected from a full-scale ammonium nitrate production factory. Major characteristics of these two condensates are summarised in Table 1. As discussed above, the molar ratio of NH_3/NO_3^- in the concentrated condensate is 1.36 (i.e., more ammonia than nitrate). On the other hand, the ammonia and nitrate in the dilute condensate has the same molar concentration, resulting from the addition of the nitric acid prior to the evaporation separator (Fig. 1) for ammonium nitrate recovery.

Table 1. Major characteristics of the concentrated and dilute condensates

Major characteristics	Concentrated condensate	Dilute condensate
pH	8.98 ± 0.21	7.93 ± 0.07
Conductivity (µS/cm)	$11,040 \pm 860$	$4,689 \pm 62$
NH_3 (mg/L)	$2,460 \pm 200$	659 ± 70
NO_3^- (mg/L)	$6,590 \pm 220$	$2,360 \pm 61$

2.2. Membranes

Three flat sheet RO membranes (e.g., BW30, ESPA2, and SW30) were used in this investigation. These membranes consist of a thin polyamide skin layer on a microporous polysulphone supporting layer. The BW30 and SW30 membranes were supplied from Dow Filmtec and were stored dry in room temperature before testing. On the other hand, the ESPA2 membrane samples were extracted from a 4-inch spiral-wound ESPA2 module purchased from Hydranautics. Once extracted from the module, the

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139   ESPA2 membrane samples were stored in MilliQ water at 4 °C. It is noted that while BW30 and ESPA2

140   are destined for desalination of brackish water, SW30 is designed for seawater desalination. These

141   three RO membranes have been systematically characterized in a previous study [24] (Table 2).

142   **Table 2.** Characteristics of RO membranes used in this study (Ref: [24]).

Characteristics	BW30	ESPA2	SW30
NaCl rejection (%)	92.8	96.1	99.3
Total dissolved solid rejection (%)	92.8	95.8	99.2
Water permeability (L/(m^2.h.bar))	3.88	6.15	2.63

143   **2.3.   Filtration protocol**

144   A laboratory-scale RO unit with a stainless-steel crossflow cell was used in this study. The

145   crossflow cell had an effective membrane area of 40 cm^2 (i.e., 4 cm × 10 cm) with a channel height of

146   2 mm. The RO unit used a Hydra-Cell pump (Wanner Engineering Inc., Minneapolis, MN) capable of

147   providing pressures up to 25 bar. The temperature of the feed solution was kept constant using a

148   temperature control unit (Neslab RTE 7) equipped with a stainless-steel heat exchanger coil, which

149   was submerged directly into the feed reservoir. Permeate flow was measured by a digital flow meter

150   (Optiflow 1000, Agilent Technologies, Palo Alto, CA) connected to a computer for data logging, and

151   the crossflow rate of the feed solution was monitored with a rotameter.

152   Prior to all RO experiments with condensate feed solutions, the membrane was compacted using

153   MilliQ water at 25 bar and 25 °C for at least one hour according to a previously reported protocol [25,

154   26]. After membrane compaction, MilliQ water was completely drained from the system and a test

155   solution (i.e., with the initial volume of 7 L unless otherwise stated) was then introduced to the feed

156   reservoir. Prior to sample collection at each operating parameter, the system was equilibrated for one

157   hour. Temperature of the experimental feed solution was kept constant at either 25 °C or 38 °C. Unless

158   otherwise stated, both permeate and retentate were recirculated back to the feed reservoir throughout

159   the entire experiment. Rejection of the RO membrane with respects to conductivity, nitrate, and

160   ammonia was calculated using the equation (1) as below:

161   $$Rejection = \left[\frac{C_f - C_p}{C_f} \right] \times 100\% \qquad\qquad (1)$$

162   where C_f and C_p were the electrical conductivity or the concentration of ammonia or nitrate of the feed

163   and permeate, respectively.

164   During the RO filtration experiments, pH and electrical conductivity of the RO condensate feed

165   solution and permeate were monitored continuously using an Orion pH and conductivity meter,

166   whereas their ammonia and nitrate contents were measured by a Metrom 781 Ion Meter equipped with

167   an ammonia and a nitrate ion selective electrode. The Orion pH and conductivity meter also allowed

168   for the measurement of the RO feed solution temperature during the experiments.

169 **3.   Results and discussions**

170 **3.1.   Ammonium nitrate rejection by the three selected membranes**

171   Performance of the three selected RO membranes to enrich ammonium nitrate was assessed as a

172   function of applied pressure. As expected, ammonia, nitrate, and conductivity rejection increased when

173   the applied pressure was elevated (Fig. 2–4). However, this observation is more prominent for both

174   brackish water membranes (e.g., BW-30 and ESPA2) (Fig. 2&3) and less discernible for the seawater

175   membrane SW30 (Fig. 4). Moreover, ammonia and nitrate rejection values by the three selected

176   membranes are consistent with their nominal NaCl rejection as reported by the manufacturers. Indeed,

177   both ammonia and nitrate rejections by the SW30 membrane were notably higher than those by the

178   BW30 and ESPA2 membranes. These results are consistent with NaCl rejections by the SW30, ESPA2,

179   and BW30 (e.g., 99.3, 96.1, and 92.8%, respectively) (Table 2). Although these NaCl rejection values

180   were reported under different filtration conditions, it is evident that NaCl rejection can be used as an

181   indicator for membrane selection, should a membrane other than those studied here be considered for

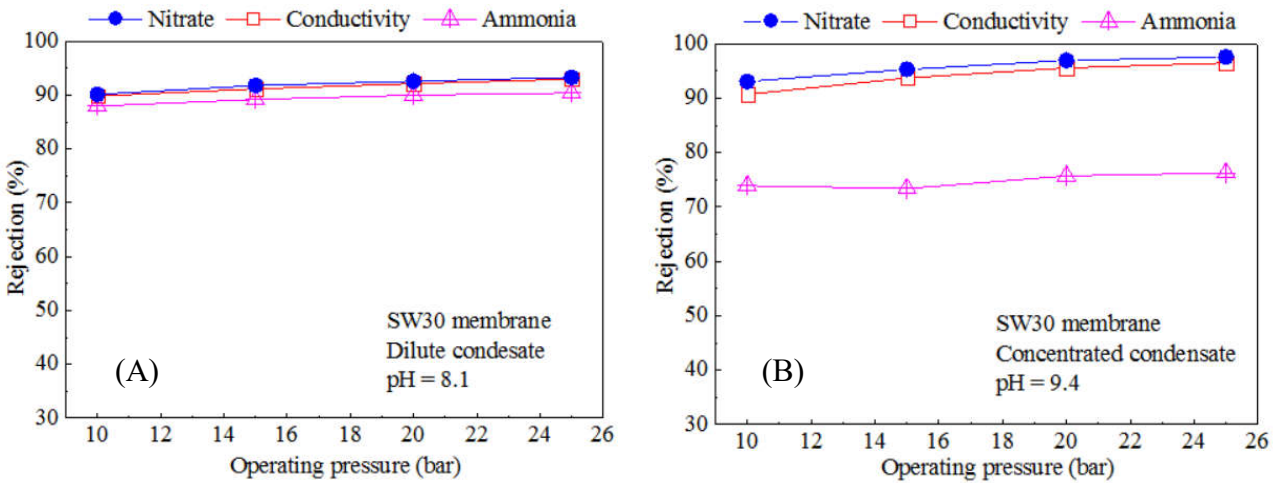182   the RO treatment of condensate from ammonia nitrate production.
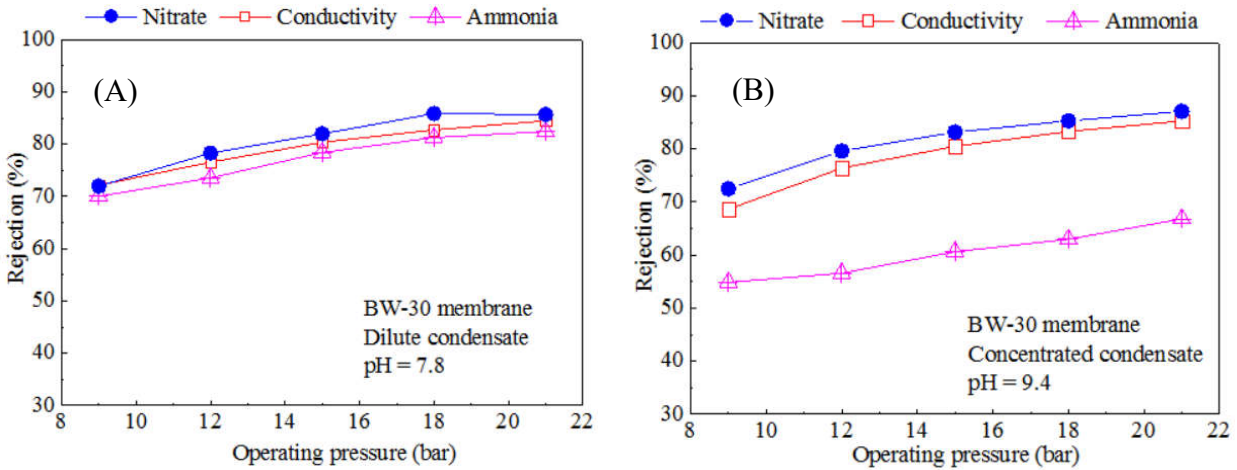
Fig. 2. Ammonia, nitrate, and conductivity rejection by the BW-30 membrane as a function of applied pressure in the RO treatment of (A) dilute condensate and (B) concentrated condensate; the condensate feed temperature was remained constant at 25 °C.
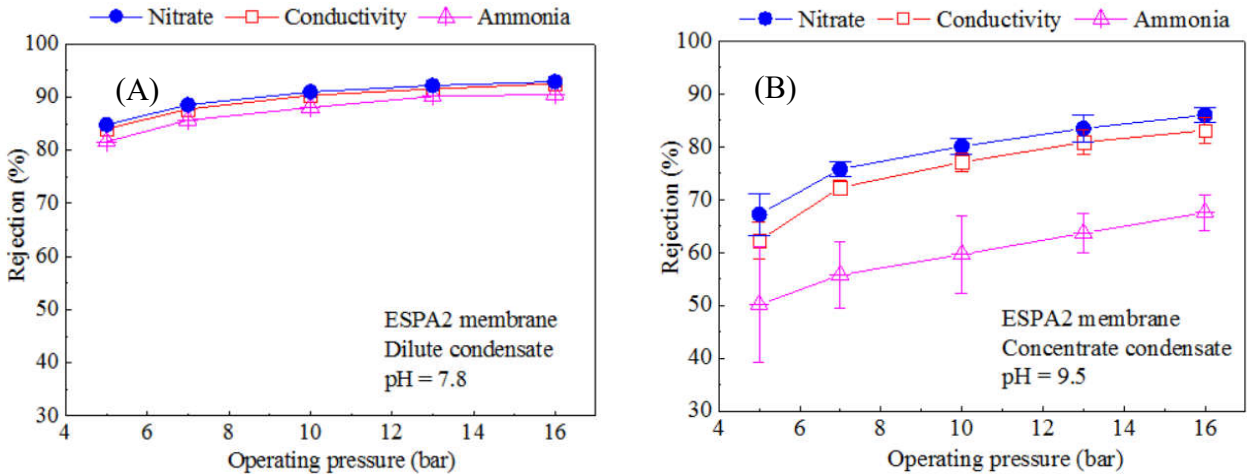
Fig. 3. Ammonia, nitrate, and conductivity rejection by the ESPA2 membrane as a function of applied pressure in the RO treatment of (A) dilute condensate and (B) concentrated condensate (error bar shows standard deviation of 3 replicate experiments); the condensate feed temperature was remained constant at 25 °C.

Of a particular note, results from Fig. 2–4 show that ammonia rejection is always lower than nitrate or conductivity rejection. Indeed, significantly lower ammonia rejection could be observed when the concentrated condensate was used as the test solution. Lower ammonia rejection can be explained by the high pH value of the concentrated condensate. Since the pKa value of ammonia is 9.2, at pH 9.4 of the concentrated condensate, ammonia (NH_3) rather than ammonium (NH_4^+) is the dominating species. As expected, RO membrane rejection of the neutral and volatile ammonia is lower than that of the cationic ammonium. It is noted that ammonia in the feed solution declined as the filtration experiments progressed, most likely due to its evaporation (i.e., confirmed by a very strong smell of ammoniac gas

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during all experiments involving the concentrated condensate). It is also noted that the concentrated condensate was approximately 4 times more concentrated than the dilute condensate. High ionic strength of the concentrated condensate (i.e., NH_4^+ and NO_3^- of 1600 mg/L and 6,540 mg/L, respectively) may also contribute to the small reduction in ammonia, nitrate, and conductivity rejection observed in the RO treatments of the concentrated condensate compared to those of the dilute one (Fig. 2–4). This contribution is small for seawater membrane (Fig. 4) but noticeable for the brackish water RO membranes (Fig. 2&3).

Fig. 4. Ammonia, nitrate, and conductivity rejection by the SW30 membrane as a function of applied pressure in the RO treatment of (A) dilute condensate and (B) concentrated condensate; the condensate feed temperature was remained constant at 25 °C.

It is noteworthy that conductivity rejection was intermediate between nitrate and ammonia rejections for all experiments conducted in this project. For the concentrated condensate, conductivity rejection was similar to nitrate rejection for all three selected membranes. Thus, the data presented in Fig. 2–4 indicate that conductivity rejection can be used as a conservative indicator for nitrate rejection during the RO treatment of condensate from ammonium nitrate production. This has an important implication for practical RO treatment of condensate from ammonium nitrate production as the measurement of solution conductivity is much more feasible than analysing solution nitrate concentration.

It is noted that data represented here were obtained from a lab-scale RO filtration set up. In a full-scale installation, a thinner filtration channel coupling with the exploitation of the spacer mesh will create a higher turbulence condition, thus mitigating the effect of concentration polarisation [27]. It is well-established that the concentration polarisation negatively affects the rejection of RO membranes

220 [28, 29]. Consequently, rejection values reported here can be slightly lower than those that could
221 potentially be realised in a full-scale RO treatment of the condensate from ammonium nitrate
222 production.

223   Experimental reproducibility of this work was examined by repeating one experiment three times
224 using the ESPA2 membrane. As can be seen in Fig. 3B, ammonia rejection was highly variable at low
225 applied pressure with a standard deviation of 10.9%. However, the variability reduced dramatically as
226 the applied pressure increased. At 13 and 16 bar, the standard deviations of ammonia rejection were
227 only 3.6% and 3.3% respectively. This result can be explained by a larger influence of the
228 hydrodynamic condition on membrane rejection at low applied pressure. Both nitrate and conductivity
229 rejections showed adequate reproducibility with standard deviation ranging from 1.3% to 3.9% (Fig.
230 3B). Overall, the experimental reproducibility with respects to ammonia, nitrate, and conductivity
231 rejections indicates that data reported in this work can be interpreted with a high level of confidence.

232 **3.2.   Effects of feed solution pH and temperature on the rejection of the membranes**

233   The SW30 membrane shown the highest ammonia and nitrate rejection. Thus, it was selected for
234 further investigation on the effects of feed solution pH and temperature on membrane rejections. While
235 feed solution pH did not impart any discernible effects on nitrate and conductivity rejections, a notable
236 reduction in ammonia rejection could be observed when the feed solution pH exceeded 8 (Fig. 5).
237 Indeed, the ammonia rejection of the SW30 membrane at pH 9 was reduced by 13% as compared to
238 that at pH 7. As explained previously, the reduced ammonia rejection of the RO membrane at the feed
239 solution pH over 8 can be attributed to the speciation of ammonia. Therefore, if pH adjustment is
240 feasible, pH less than 8 is recommended for an optimised ammonia rejection during the RO treatment
241 of the condensate from ammonium nitrate production.
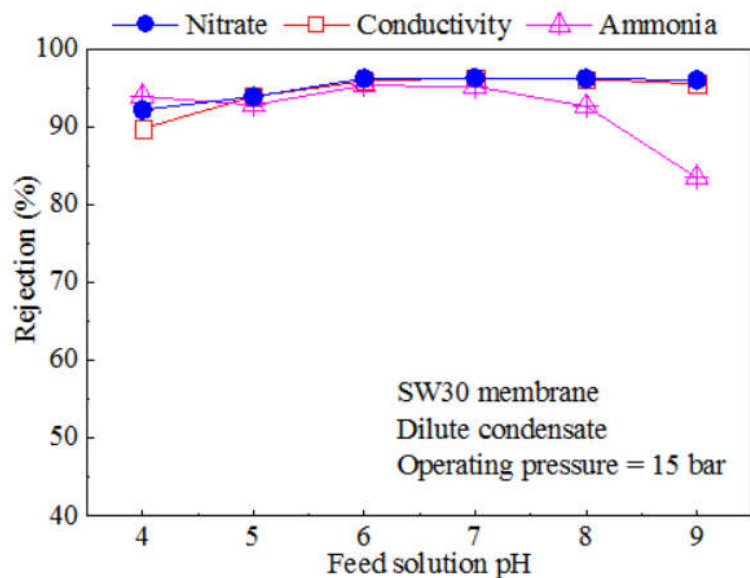
The feed solution temperature also exerted different effects on ammonia rejection and nitrate or conductivity rejection. As demonstrated in Fig. 6, a small but noticeable decrease in ammonia rejection was recorded, while nitrate and conductivity rejections were largely unaffected as the feed solution temperature was increased from 25 °C to 38 °C. This could be attributed to the smaller size of hydrated ammonium compared to nitrate in the feed solution. The sizes of hydrated ammonium and nitrate radicals have been reported to be 0.331 nm and 0.340 nm, respectively. Being a smaller hydrated ion, it is possible that ammonium could become more mobile as the solution temperature increased. Another possible theory is that at feed solution pH 8.1, there would be approximately 93% ammonium (NH_4^+) and 7% ammonia (NH_3). Indeed, NH_3 is a neutral ammonia species; therefore, its diffusion through the membrane would be much more sensitive to the feed solution temperature.
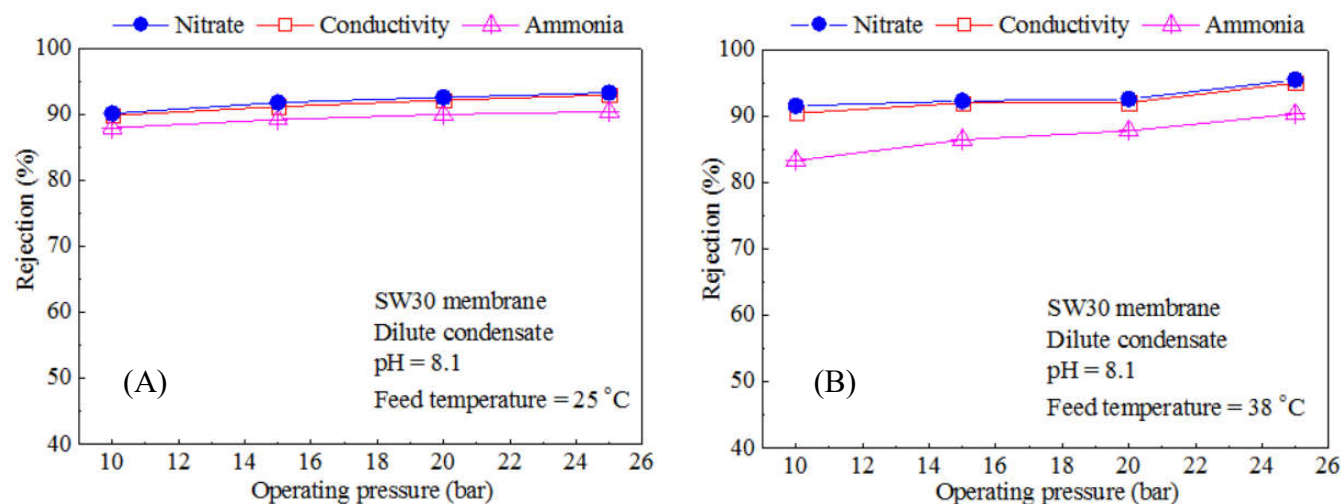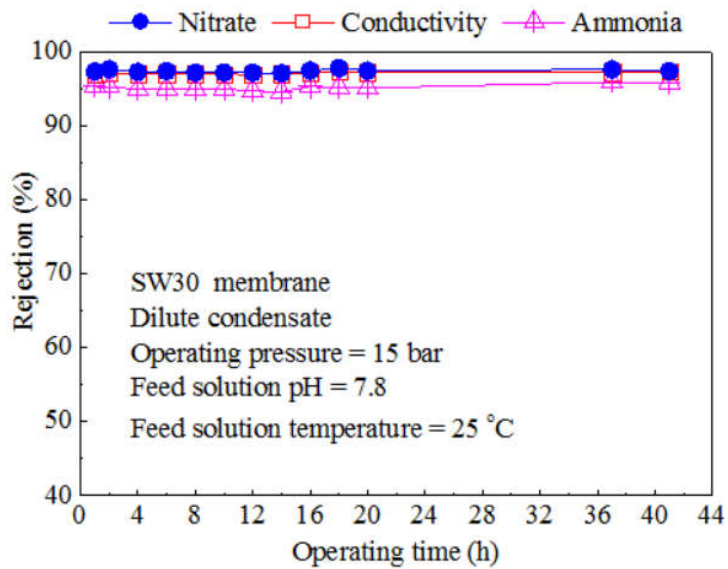
Fig. 6. Ammonia, nitrate, and conductivity rejection by the SW30 membrane as a function of applied pressure at feed solution temperature of (A) 25 °C and (B) 38 °C during the RO treatment of the dilute condensate.

3.3. Rejection stability and permeate flux of the membranes

To examine the stability of the RO filtration process, one experiment using the SW30 membrane and dilute condensate was conducted over an extended duration. No discernible variation in membrane rejection could be observed over 42 hours of operation (Fig. 7). In this experiment, the permeate was not returned to the feed reservoir to induce a gradual increase in feed concentration to simulate a condition similar to that occurs along a membrane element or vessel.

At up to 25 bar, permeate flux was proportional to applied pressure, with the highest flux recorded for the ESPA2 membrane and the lowest flux exhibited by the SW30 membrane (Fig. 8). Moreover, the RO treatment of dilute condensate exhibited higher permeate flux than that of concentrated condensate for all the three selected membranes, demonstrating the effect of feed solution salinity and osmotic pressure on the permeate flux of the RO process.

It is noteworthy that stable permeate flux was observed with all experiments conducted in this study. No evidence of membrane fouling occurred within the short time frame of RO experiments conducted. Even for one extended experiment that was conducted over 2 days, the permeate flux was constant. The risk of membrane scaling is low although further investigation may be necessary to ascertain the long-term organic fouling propensity of the condensates.

Fig. 7. Ammonia, nitrate, and conductivity rejection by the SW30 membrane as a function of operating time during the RO treatment of the dilute condensate (pH = 7.8). Other operating conditions: applied pressure = 15 bar, feed solution temperature = 25 °C.
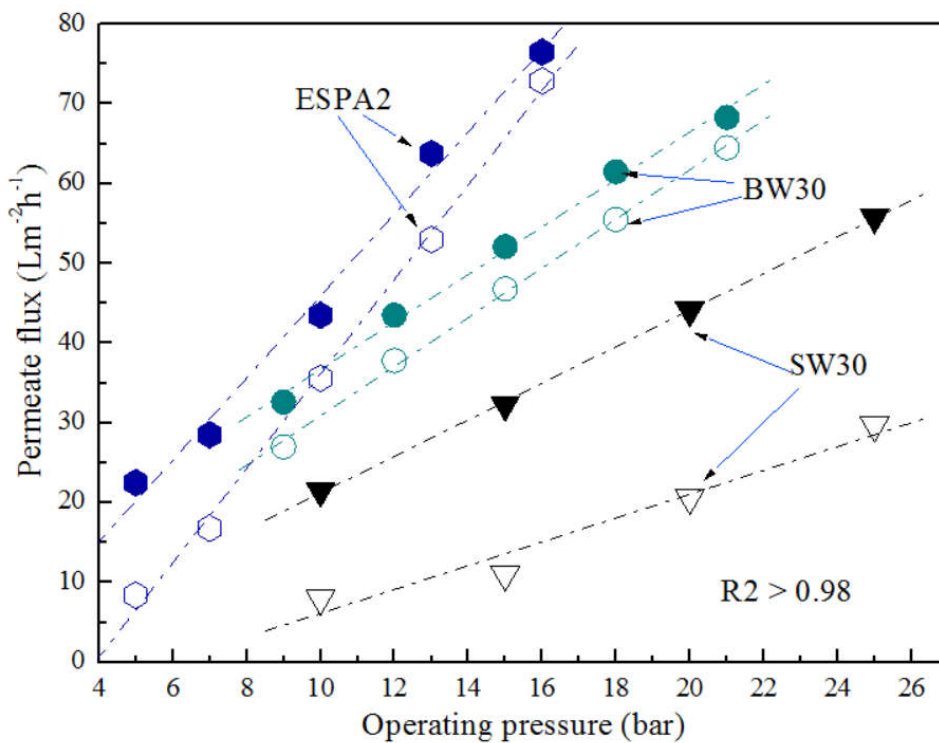
Fig. 8. Permeate flux as a function of applied pressure during the RO treatment of the dilute and concentrated condensate using the three selected membranes: the closed boxes (⬡, ●, ▼) represent the permeate flux for the dilute condensate feed while the open boxes (⬡, ○, ▽) are for the concentrated condensate feed. The feed solution temperature was 25 °C.

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4. Conclusions

This study demonstrates the potential of RO membranes for concentrating ammonium nitrate in the condensate for subsequent recovery. Ammonia and nitrate rejections were affected by operating conditions including applied pressure (or water flux), temperature, feed solution pH, and initial ammonium nitrate concentration in the condensate. The impact of operating conditions on ammonia and nitrate rejections was more significant for low pressure (thus high flux) than high pressure RO membrane. Of a particular note, ammonia rejection was lower than nitrate rejection. The difference between ammonia and nitrate rejection was more obvious for low pressure RO membrane. Results reported here suggest that a low pressure RO membrane (e.g. ESPA2) is more suitable for the dilute condensate while a high pressure RO membrane (e.g. SW30) is recommended for the concentrated condensate to ensure adequate ammonia and nitrate rejection. No evidence or significant risk of membrane fouling was observed in this study. A compact membrane system can significantly reduce the size of the evaporation separator for ammonium nitrate recovery. Results in this study are useful for membrane selection and process design to use RO for treating condensate wastewater and recovering ammonium nitrate.

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