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Pilot-scale nanofiltration system as post-treatment for fertilizer-drawn forward osmosis desalination for direct fertigation

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ABSTRACT

The integration of the fertilizer-drawn forward osmosis with nanofiltration (NF) has been investigated to evaluate the performance of NF process as a post-treatment. The primary objective of this study is to optimize the operating conditions such as feed flow rate and feed concentration, while producing fresh water including low nutrient (N) concentration can be directly used for irrigation. Investigation of operating parameters at the pilot-scale level focused on specific water flux and ammonium sulphate rejection. Results from this study showed that NF process applied as the post-treatment can effectively reject the N concentration more than 90%. Although other factors such as the applied pressure and the cross-flow rates played a certain role in the performance of the pilot-scale NF process, the influence of the feed concentration was more significant on the specific water flux and N rejection.

Keywords: Desalination; Fertilizer draw solution; Forward osmosis (FO); Nanofiltration (NF); Fertigation; Nutrient concentration

1. Introduction

A ninefold increase in freshwater use has been caused by the increase by 13-fold energy consumption and world population that has quadrupled to over 6 billion since the beginning of the twenty-first century, meaning that, prodigious amounts of water are required to match with the energy required [1]. At the same time, it has become a big challenge to provide fresh water resources for industrial and agricultural use as significantly increasing the energy consumption. Therefore, water shortages have become one of the most serious global issues at present [2].

In the past few decades and today, desalination technologies have emerged as a solution to human issues problems to produce both potable and non-potable water using seawater and brackish ground water (BGW). Reverse osmosis (RO) is one of the most well-known desalination processes to extract fresh water that is suitable for various areas ranging from human consumption to irrigation purposes. However, the critical issue in RO desalination process is energy-intensive technology because of mainly high-pressure pumping unit, it is the major contributor to the energy consumption in RO system [3,4].

In forward osmosis (FO) process, however, the energy consumption is lower than RO process because

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it drives water to pass through the membrane from a low concentration to a high concentration, which is used as the difference of osmotic pressures between two solutions [5]. Despite the great developments such desalination process as FO process, it has some limitations that hinder their application to producing drinking water [6,7]. With respect to this, a recent research has examined the possibility of applying fertilizers as a draw solution (DS) and brackish water as a feed solution (FS) to FO desalination process in order to extract water from saline water into the DS to be used for irrigation, which is called fertilizer-drawn forward osmosis (FDFO) desalination process [6–8].

Nevertheless, Phuntsho et al. [6] investigation revealed that the concentration of the diluted draw solution in FDFO desalination process was too high to apply for irrigation purpose. In addition, the challenges of FDFO process application are both fabrication and performance of FO membrane and selection of the suitable fertilizer draw solution [7]. Further to this, the concept of the integration of FDFO with nanofiltration (NF) as either pre-treatment or post-treatment has been proposed and evaluated to meet the acceptable concentration level in the final extracted water from the hybrid FDFO-NF desalination system for irrigation purpose [7].

NF is the most commonly applied process for many industries for the separation of salinity [9]. At the core of the NF process is both a semipermeable membrane capability of producing high permeability at low operating feed pressure and rejecting the dissolved salts from the feed water [10]. With respect to that, the performances of NF membrane such as permeate flux and rejection have been examined with a various feed water such as landfill leachate, recycled water, dye/salt mixtures and highly concentrated salt solutions up to seawater salinity, etc. at different experimental operating conditions [11–14].

In addition, based on the previous studies of NF membrane, Phuntsho et al. [6] identified and found that multivalent ions in the diluted fertilizer DS after FDFO desalination system were well separated by following NF membrane. Therefore, it has concluded that the final product water can have a targeted nutrient concentration for direct fertigation after NF process [7].

Pilot-scale of the integrated FDFO-NF desalination process has been investigated for further research initially. The investigation of the pilot-scale FDFO-NF was mainly related to process optimization to apply the data from this preliminary investigation for the next phase of the pilot-scale testing at Mildura region within the Murray–Darling Basin in Australia.

In this article, we report the initial results of our study on the performances of the pilot-scale NF process as post-treatment system to reduce the fertilizer concentrations after FDFO desalination using different operating conditions. In this way, the final product water can be applied directly into fertigation without further diluting the NF product water. The performance has been measured in terms of fertilizer nutrient concentrations especially nitrogen (N) in the permeate water flux.

2. Materials and methods

2.1. Theory

In this operation of NF membrane, the solution-diffusion model reviewed by Wijmans and Baker [15] was used to evaluate permeation through the NF membrane based on Darcy's law of diffusion. The driving forces for transport are generated by the differences in concentration and pressure across the membrane. The water flux is given as follows:

$$J\omega = \frac{D1C1V1}{RT\Delta x}(\Delta P - \Delta\pi) \tag{1}$$

The following equation is used to calculate specific water permeability (SWP); the term permeability refers to the diffusion of water from the bulk convective flow to the membrane surface. From Eq. (1), the specific permeability of the water through NF membrane can be expressed as Eq. (2) below:

$$P = \frac{J\omega}{(\Delta P - \Delta\pi)} \tag{2}$$

where P, $J\omega$, ΔP, and $\Delta\pi$ are the specific water permeability, the water flux, the pressure difference, and the osmotic pressure difference, respectively. The solutes rejection of membrane (R) is a measure of capability of separating salt from the FS, which is defined as follows:

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \tag{3}$$

where C_p and C_f are permeate and feed concentrations, respectively. This equation is most commonly used to calculate the rejection across the membrane. In this study, the conductivities of feed and permeate replacing with values of concentrations.

2.2. Feed solution for NF process experiments

Preliminary experiments on laboratory-scale FDFO unit have allowed selecting fertilizer for pilot-scale experiments [6,8]. In relation to this, various fertilizers were used as DS in FDFO desalination system with different types of FS such as deionized water (DI), tap water, and BGW [7,8,16]. The results from previous research have shown that the extracted water flux of ammonium sulphate (SOA) was slightly lower than others, while the reverse solute flux, which is the most common drawback for FO membrane process, was the lowest [7,8]. Therefore, SOA was employed as DS in the pilot-scale of FDFO desalination process in order to produce the diluted SOA as FS in NF process performance test. The pilot-scale FDFO desalination system was operated using tap water and 1.80 M of SOA as FS and draw solution (DS), respectively. Then, the diluted SOA after FDFO desalination process was delivered and stored in a 1000-L water tank. The pilot-scale NF process was operated with the diluted SOA with conductivity of 33.0 mS/cm as the feed water initially.

2.3. Pilot-scale of NF membrane process

Pilot-scale of NE4040–90 spiral-wound configuration (Woongjin Chemical Co., Ltd., Korea) was investigated in this study and shown in Figs. 1 and 2. The process parameters recommended by the manufacturer have shown in Table 1. As illustrated in Fig. 2, spiral-wound elements, which are manufactured from flat-sheet membranes and separated by feed spacer, consist of number of membrane envelopes attached to a centre tube that collects the product water [17]. NF membrane was initially rinsed with tap water at 10 bar for at least 1 hr in order to wash out the membrane protection chemicals and then pressurized at 25 bar using a DI to compact NF membrane for at least 1 hr. To avoid any negative effects of the results of cleaning procedure on the membrane structure, cleaning process was not conducted.

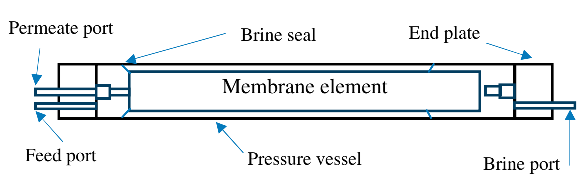

Fig. 1. Pressure vessel with NF membrane element.

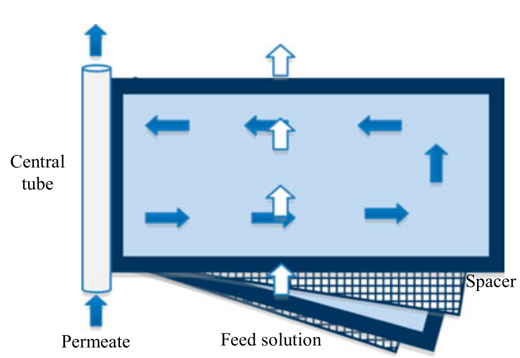

Fig. 2. Flows in NE90 spiral-wound membrane.

Table 1
Summary of the membrane characteristics (NE4040-90)

Membrane type		Thin-film composite
Materials		Polyamide (PA)
Membrane surface charge		Negative
Element configuration		Spiral-Wound, FRP wrapping
Permeate flow rate[a]		1,600 GPD (6.0 m^3/day)
Area (m^2)		7.9 m^2
pH range		3–10
Max. operating pressure		600 psi (0.41 Mpa)
Rejection (%)	NaCl (0.2%)	85.0–95.0%
	$MgSO_4$ (0.2%)	97.0%

[a]2,000 mg/L NaCl solution at 5 bar applied pressure, 15% recovery, 25˚C and pH 6.5–7.0.

2.4. Pilot-scale of NF membrane experimental set-up and operation

Pilot-scale of FDFO-NF desalination process is illustrated in Fig. 3. Pilot-scale system consists of two independent units; the FDFO desalination unit and the NF unit as post-treatment process. However, this study reports only on the performance of NF process. The pilot-scale NF unit was tested for 720 hr including the operation of FDFO desalination process. As represented in the Fig. 3, the diluted SOA after FDFO desalination unit is delivered to the NF feed tank, and then, water passes through the NF process, and thus, the final product water is collected in the fertigation tank, while the concentrated feed solution is returned to the diluted DS tank. There is no addition of feed solution and the rejected water (i.e. concentrated feed solution) fully recycled to the NF feed tank. Therefore, NF membrane can be operated without new foulants resulting in a stable water flux during the test. All sensors, pressure, flow rate, conductivity, and temperature, are connected to a computer, thereby are recorded automatically. Furthermore, increased temperature can have direct influence on the increasing

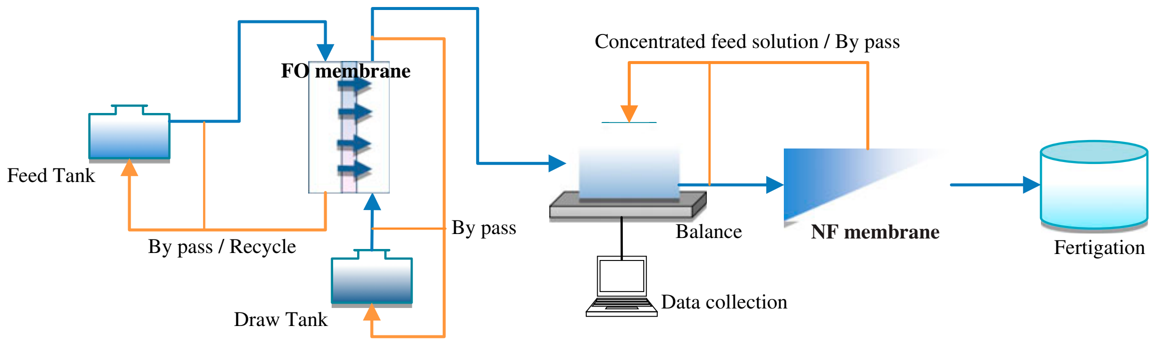

Fig. 3. Schematic diagram of pilot-scale FDFO-NF desalination process.

mass transfer of water and solutes due to an Arrhenius relation [18,19]. Nevertheless, in this study, the effect of temperature on water flux was able to be neglected because of both the short period of test and the insignificant change of temperature, which was +0.1℃ at each experiment. To determine the optimal operating conditions, the pilot-scale NF process was operated at different applied pressures, cross-flow rates, and FS concentrations; pressure applied ranges from 10 to 25 bars, the feed flow rate varied from 0.5 to 1.5 m^3/hr (from 500 to 1,500 Lh^{-1}, respectively), and the feed concentrations were 0.2 and 0.35 M. Each experiment was performed for at least 3 h and no more than 300 L of permeate was collected in any experiments. In this study, NF performance as the post-treatment was assessed by measuring the specific permeate water flux and the electro-conductivity of the feed and permeate solutions. As a result, the nutrient concentration (Nitrogen, N) in the final product water was taken as the core indicator of NF process as post-treatment. The permeate water flux was determined gravimetrically as connected to the computer, and the conductivity was measured using H270multi (HACH) conductivity meter.

3. Results and discussion

3.1. Initial performance of pilot-scale NF process using DI water and NaCl

The initial experimental study was conducted with both DI water and NaCl solution (2,000 mg/L, 100 L initial feed volume in batch mode) as NF feed water in order to compare with data shown in the relevant literature. The pressure was ranging from 2 to 10 bar at constant feed flow rate of 1.0 m^3/hr. As shown in Fig. 4, the specific permeate water was determined as an almost linear relationship between the applied pressure (bar) and water flux ($Lm^{-2}h^{-1}$). Specific water flux using NaCl as FS was lower than DI water;

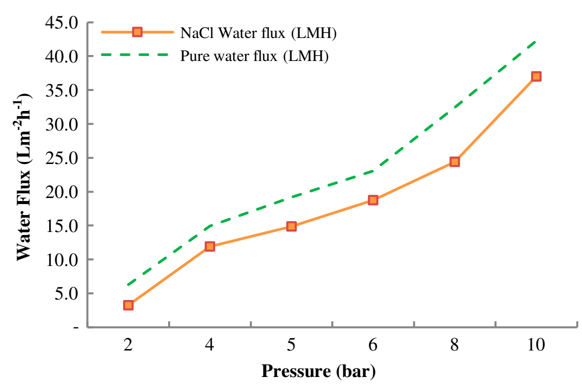

Fig. 4. Water permeability as a function of the applied pressure.

(2.88 $Lm^{-2}h^{-1}bar^{-1}$ for NaCl and 3.82 $Lm^{-2}h^{-1}bar^{-1}$ for DI water, respectively) and the water permeability of NaCl and DI water in the pilot-scale NF process increased linearly with increasing pressure as shown by other research [20]. However, the pure water permeability determined in this study for NE 90 was around 60% lower than that studied by Hilal et al. [14]. In addition to this, the initial permeate flux from the experiment carried out at NaCl concentration of 2,000 mg/L as recommended by manufacturer was lower than expected permeate flux in comparison with manufacturer's specifications, which was 6.2 $Lm^{-2}h^{-1}bar^{-1}$. Possible reasons for these differences in the initial NF performance may be due to large NF membrane area of 7.9 m^2 used and lower applied pressure used for the operation (up to 25 bar) used in this NF test, while the applied pressure was up to 40 bar in other studies [21,22].

Additionally, it is obvious from Fig. 5 that observed NaCl rejection increased with increasing pressure, it was up to 97% at 10 bar. Previous study with NE90 membrane [23] has investigated and found that the individual salts rejection order was mainly influenced by negative charged NE90 membrane. In

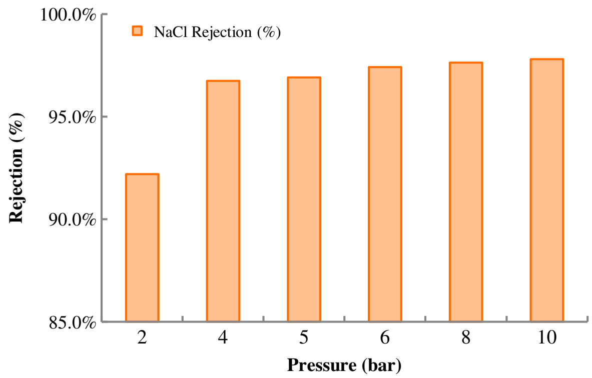

Fig. 5. Effects of applied pressure on NaCl rejection for the initial investigation.

addition, this rejection performance of NE90 process, which is promising for reducing salt concentration for direct irrigation, could be explained by the steric hindrance mechanism due to relatively small pore size of membrane [14,23]. Despite poorer permeate water flux during the test, average rejection of salt was more than 90%, which was similar to that indicated by the supplier. Therefore, in this study, we focused more on evaluating the capability of removing nutrient (N) concentration in the diluted SOA as NF feed solution to meet the requirement of suitable concentration for direct irrigation rather than investigating the amount of product water flux in NF process.

3.2. *Effects of operating conditions on performance of NF process with SOA as FS*

NF membrane performance is influenced by several factors such as feed flow rate, pressure, temperature, pH, and feed concentration [9,12,23]. In this study, the effect of different feed conditions on performance of pilot-scale NF membrane was evaluated to find out optimal operating values and identify the nutrient concentrations in the final water.

Firstly, specific water flux and SOA rejection were examined at different feed cross-flow values at 0.5, 1, and 1.5 m^3/hr, while feed pressure and concentration were constant in the range of 10–25 bars with 0.2 M SOA as shown in Fig. 6. It shows that the specific permeate flux and rejection clearly increases with increasing pressure at each experimental condition, while increasing feed flow rate had a slight effect on the specific water flux due to the increase of tangential velocity in the NF unit. Higher-cross flow rate can produce higher water flux because of the decrease in both the solute concentrations and direct absorption on the membrane surface [12]. As shown in Fig. 6 (a), however, the specific permeate flux obtained with 1 m^3/hr was higher than those obtained with 1.5 m^3/hr at applied pressure 10 and 15 bar. When the diluted SOA was passed through the NF membrane under 10 and 15 bar with 1 m^3/hr of feed flow rate, it produced a 50% higher specific water flux with 0.213 $Lm^{-2} h^{-1} bar^{-1}$. The decrease in specific flux values at higher cross-flow velocity with applied pressure 10 and 15 bar may be caused by an insufficient wetting of membrane area or/and assumption of that the solutes are accumulated within the membrane spacer in spiral-wound modules [22]. In this case, the effects of increasing the feed flow rate are insignificant because of the small changes in cross-flow rates. In addition, reduced water transfer through the membrane is caused by concentration polarization within the membrane surface [12]. As illustrated in Fig. 6(b),

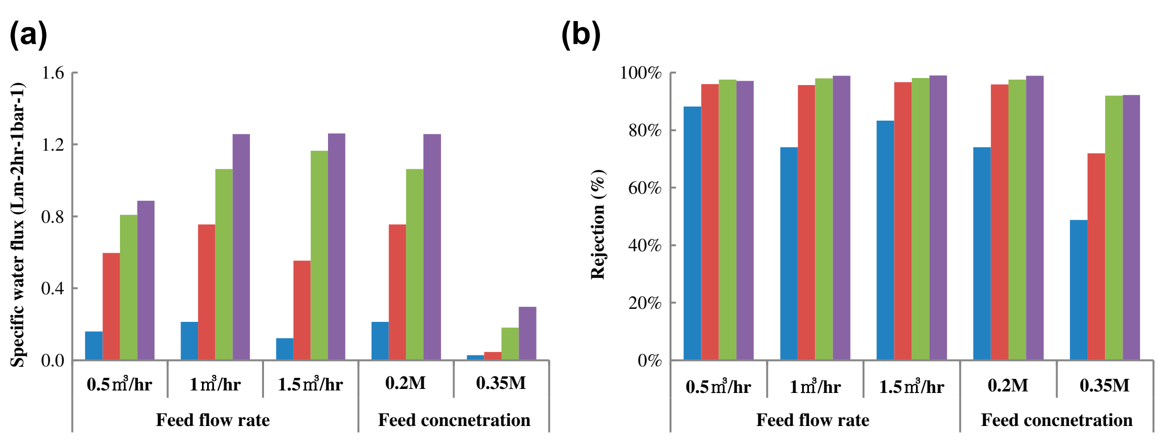

Fig. 6. Specific water flux (a) and rejection (b) with applied pressure for the diluted fertilizer (SOA) as FS for NF process at both different flow rate and concentration.

moreover, the value of SOA rejection obtained with 1 m^3/hr was about 15% lower than those obtained with 1.5 m^3/hr at 10 bar. As revealed earlier, this can be caused by the increase in adsorption or formation on the membrane surface. This effect also has resulted in the values of reduced SOA rejection with increasing flow rate at 10 bar. This seems that the specific permeate flux was less influenced by the change in cross-flow rates, but it seems to be attributable to lower rejection of dissolved solids on the NF membrane.

Secondly, specific water flux and SOA rejection were examined at different feed concentrations, 0.2 and 0.35 M, while feed pressure and feed flow rate were constant in the range of 10–25 bars with 1 m^3/hr as shown in Fig. 6. The concentration of feed solution can affect the flux reduction and rejection because the permeability of solutes is influenced by the amount of salt passing through the membrane [14]. With respect to that, the specific water flux significantly decreased to 90% of its original value with the increase in SOA concentration with values of 0.2 M and 0.35 M as illustrated in Fig. 6(a). It is clear that the higher concentration of SOA as FS in the NF process leads to significant flux reduction. In addition to higher feed concentration increases the amount of SOA near the membrane surface, which can explain the permeate flux decline due to an increase in the mass resistance coefficient. This result is in a very good agreement with other recent studies [24,25]. Furthermore, the rejection was also much lower when higher concentration of SOA used as FS with the highest rejection of 92% for pressure above 20 bar. An increase in concentration polarization layer on the membrane surface with higher SOA concentration can cause the reduction in rejection. High concentration of SOA caused a lower diffusion through the NF membrane, which led to a further decline in water flux. This also led to the deposition of SOA in the boundary layer (i.e. concentration polarization) [10]. As expected that, the poorest rejection occurred at higher feed concentration and may be influenced immediately due to significant increase in SOA solutes on the membrane surface. As a result, although the specific water flux increased when the feed cross-flow rate increased from 0.5 to 1.0 m^3/h, however, there was no significant increase in the specific water flux beyond 1.0 m^3/h. The most significant influence on the specific water flux was shown by the feed concentration. The specific water flux significantly decreased when SOA feed concentration was increased from 0.2 to 0.35 M which shows that the osmotic pressure plays a significant role in the performance of the NF process both in terms of water flux and the rejection perhaps because of the high rejection properties of the NE 90 NF membrane.

3.3. Nutrient (N) concentration in the final product water after NF process as post-treatment

In general, NF membrane process has been mainly used as a pre-treatment for desalination [10,26]; however, NF process has been installed as post-treatment for FDFO desalination process in order to achieve a targeted nutrient concentration for direct fertigation. 1.80 M of SOA was used as DS, while using tap water as FS in FDFO desalination process to make an appropriate condition of feed solution for NF process experiment. To improve the NF performance, several researchers have investigated the integration of NF with other pressure-driven membranes as pre-treatment such as microfiltration (MF) and ultrafiltration so they concluded that the outcomes depended on both the types of membrane material and the feed solutes parameters [10,27,28]. Therefore, it seems that the performance of FDFO desalination will significantly influence on NF process operation, which is the nutrient concentration in the final product water. In this part, we have assessed the nutrient concentration (N) in the final extracted water after pilot-scale of NF process.

3.3.1. The final nutrient concentration in laboratory-scale of NF process as post-treatment

As proposed earlier by Phuntsho et al. [6,7], the nutrient concentration in the extracted water from FDFO desalination system for direct irrigation was much higher than the required nutrient concentration, which is from 120 to 200 mg/L of nitrogen (N) for a targeted crop such as tomato. Results from the lababoratory-scale of NF membrane as post-treatment was summarized in Table 2.

It appeared that there was a significant improvement of the nutrient concentration in comparison of the FDFO desalination process alone and the integration of FDFO-NF process. Nitrogen (N) concentration was rejected more than 90% after NF process with lower feed solution concentration (in Table 2). In the FDFO desalination process, when BGW5 was used as FS, N concentration was significantly lower than when BGW 35 was used as FS. This can be seen the types of feed solution to FDFO desalination process also considerably influence on the final nutrient concentration in the diluted DS. Therefore, it is obvious that either further diluting the product water or lower concentration of FS is required to increase the N

Table 2
Evaluation of the final nutrient concentration in the laboratory-scale NF as post-treatment

Fertilizer	MW (g/mol)	π@1 M atm	Final nutrient concentrations (N/P/K, mg/L)			
			FDFO alone		NF as post-treatment	
			BGW5	BGW35	BGW5	BGW35
SOA	132.14	46.14	1,370/0/0	10,850/0/0	69/0/0	4,779/0/0

In FDFO desalination process, simulated brackish ground water (BGW, mixtures of Na_2SO_4, KCl, $CaCl_2.2H_2O$, $MgCl_2.6H_2O$, and $NaHCO_3$) was used as feed water.
BGW 5 and BGW35 are defined as 3,912 and 27,382 mg/L of total dissolved solids (TDS), respectively.
NF process was only operated at an applied pressure of 10 bar.

rejection in the pilot-scale NF membrane process for direct fertigation.

3.3.2. *The final nutrient concentration in pilot-scale of NF process as post-treatment*

The conductivity of the diluted SOA from pilot-scale FDFO process was gradually decreased until 33.0 ms/cm due to osmotic dilution of SOA along with the concentration of the diluted SOA slowly reduced from 1.3 to 0.2 M during the operation. Although operating FO at higher applied pressure can produce not only results in higher output and higher recovery rates but also have lower unit energy consumption in the NF process, pilot-scale FDFO desalination process was operated at the constant pressure recommended by manufacturer. The conductivity of permeate water flux after pilot-scale NF process could be predicted from the diluted SOA concentration by using the rejection indicated by manufacturer (in Table 2) or previous results [23], and thus, more than 90% of salt rejection was used. As shown in Table 3, although the expected final N concentration decreased with the decrease in the conductivity of the diluted

Table 3
The conductivity change of SOA as DS in pilot-scale FDFO desalination process and the predicted permeate flux conductivity using the diluted SOA as FS in NF process

FDFO desalination process[a]			NF process as post-treatment	
Conductivity[b] in FDFO desalination process (mS/cm)		Concentration of the diluted DS (M)	Predicted permeate conductivity after NF[c] (mS/cm)	Predicted nutrient (N) concentration after NF (mg/L)
Feed	Permeate			
205.1	162.6	1.3	16.26	4314.7
162.6	95.6	0.7	9.56	2513.7
95.1	70.5	0.5	7.05	1839.0
70.3	65.7	0.5	6.57	1710.0
65.7	61.3	0.4	6.13	1591.7
61.3	57.7	0.4	5.77	1495.0
57.7	49.0	0.3	4.90	1261.1
48.8	40.3	0.2	4.03	1027.3
40.1	34.8	0.2	3.48	879.4
34.8	33.0[d]	0.2	3.30	831.0

[a]Applied pressures on both sides of 8,040-MS-P FO membrane module were constrained by manufacturer's recommendation. The maximum pressure for draw inlet and outlet was recommended to be 0.7 bar and 0.15 bar, respectively. In FO test, 0.5 bar for draw inlet and 0.1 bar for draw outlet were applied.
[b]Conductivity was collected and monitored automatically by the HMI data logger.
[c]Based on the results from previous NF membrane test or Table 2, it was hypothesized that the rejection of SOA was about 90%.
[d]33.0 ms/cm of the diluted DS conductivity refers to the conductivity of SOA as FS in the NF process.

SOA, the result was much higher than the accepted nutrient concentration for irrigation. It can be seen that the final product water from NF process has to be recycled or diluted to achieve much lower N concentration for direct irrigation. Therefore, it is significantly important to find out the optimal operating conditions for pilot-scale NF process to avoid any additional processes.

3.3.3. The effect of operating parameters of NF process on the final nutrient concentration

The pilot-scale of NF experiments was performed with different operating parameters in order to determine suitable operation conditions so that this can help achieving N concentration expected in this study. Based on the rejection data (in Fig. 6 (b)), the conductivity of final NF permeate flux (in Fig. 7) was considerably decreased to about 85% with the increase in the applied pressure at each operation condition. The final N concentration in the NF permeate was represented in Fig. 8 using the conductivity of the product water. As a result, except for the permeate operated at 10 bar, all other NF permeate resulted in N concentration close to 200 mg/L, usually recommended for direct fertigation of crops such as tomato. However, the NF permeate flux resulted in N concentration about 4 times higher level than the acceptable N concentrations at higher SOA concentration (0.35 M). This also indicates that about 75% of the NF permeate will need to undergo second NF pass to make the final permeate acceptable for direct fertigation. In addition, when the NF was operated at higher feed concentrations and applied pressures, recovery rates of the NF process increase. This can lead to a rapid accumulation of the

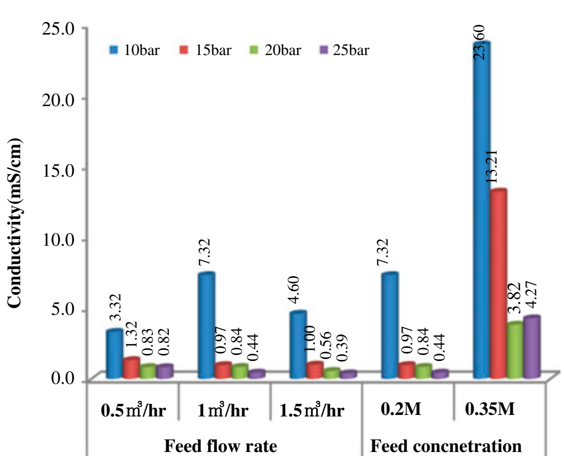

Fig. 7. Final permeate water flux conductivity of pilot-scale NF process at applied pressures.

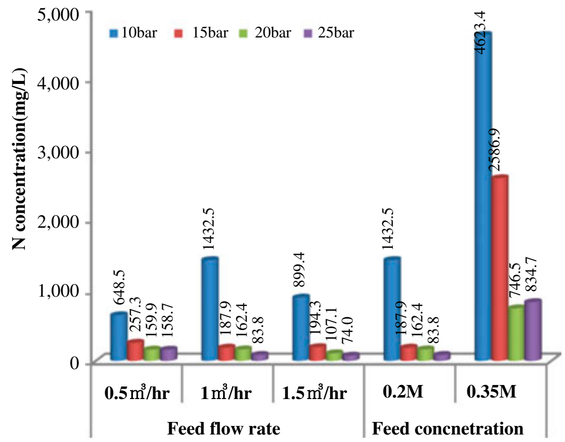

Fig. 8. Predicted N concentration in the NF permeate using the diluted SOA (DS) as FS.

solutes on the membrane surface resulting in higher conductivity of the permeate flux.

Consequently, the rejection of pilot-scale of NF process was from 92% to 99%, except for the NF operation of 0.35 M of SOA concentration at operating pressure10 and 15 bar. It can be understood that the lower feed concentration leads to lower applied pressure to remove salt thus this can contribute to less energy consumption in pilot-scale NF process. In addition, greater apparent rejection for direct fertigation is expected for feed solution containing low concentration of SOA. Therefore, this can be applied for most multivalent fertilizers due to high rejection of multivalent ions by NF membrane.

4. Conclusions

The performance of pilot-scale FDFO-NF desalination process that focuses on NF process has been investigated to optimize operating parameters and evaluate the N rejection in the final product water. Although other factors such as the applied pressures and the cross-flow rates played a certain role in the performance of the pilot-scale NF process, the effect of the feed concentration was more significant on the N rejection and the specific water flux. As a result, pilot-scale NF process applied as post-treatment after the FDFO desalination process was found to be effective in reducing the N concentration. As a way to prevent both the dilution of the final product water and second pass through NF process, the fertilizers as DS should be diluted at least 70% of initial concentration during the FDFO desalination process. From these results, we may achieve much lower N concentration in the final extracted water after NF process as post-treatment for direct irrigation and these results should be valuable in the future optimization of the entire FDFO-NF desalination plant performance to reach its full capacity.

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