

# Potential application of the draw solution system based on trisodium $\alpha$ -DL-alanine diacetate in forward osmosis desalination systems

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**Abstract.** This study aimed to discover the potential application of the draw solution system based on trisodium  $\alpha$ -DL-alanine diacetate (MGDA draw solution) in forward osmosis (FO) desalination systems through the optimization of some important operational parameters, the investigation of fouling behaviours after long-term operation and the efficiency of mitigating strategies, and the analytical of some key quality properties of the produced freshwater. Optimization results suggested that in the investigated range, inlet temperature was the main operational parameters that influence the osmosis performance of the MGDA draw solution. Under the optimized operational parameters (inlet pressure difference = 0.4 bar on the feed side, inlet temperature = 30°C, and feed side inlet flow rate = 250 mL·min<sup>-1</sup>), the osmosis performance obtained were  $J_w = 9.996 \pm 0.192$  LMH and  $R_{ds} = 0.3580 \pm 0.0020$  g·L<sup>-1</sup>. Furthermore, experimental results also emphasized the advantages of MGDA solution, which were low tendency of membrane fouling and the relatively ease of membrane cleaning. Finally, experiments on real brackish water samples confirmed the potential application of the MGDA draw solution in FO desalination systems, with the produced freshwater meeting key requirements as recommended in the National Technical Regulation QCVN 01-1:2018/BYT.

**Keywords:** Box–Behnken optimization; forward osmosis; long-term operation; membrane fouling; water quality

## 1. Introduction

Since ancient times, freshwater has always played an essential role in the conception and development of civilizations. Its abundance not only ensures the survival of humans, but also provides the conditions needed for the establishment of extensive farming and animal husbandry (Yevjevich 1992, Yasuda 2013). For these reasons, the United Nations General Assembly (2013) adopted resolution A/RES/64/292 recognizing safe and clean drinking water and sanitation as a human right, emphasizing that such right “is essential for the full enjoyment of life and all human rights”. Subsequently, through its 2030 Agenda for Sustainable Development, the United Nations General Assembly (2015) once again acclaimed the importance of “ensuring availability and sustainable management of water and sanitation for all”, declaring it as one of the seventeen most essential goals for the sustainable development of people around the globe.

However, due to the combined impact of many objective and subjective factors, there has been an increasing shortage of freshwater sources necessary to serve the daily needs of people, both in Vietnam and around the globe, especially during the dry months (Salehi 2022, Ngo *et al.* 2018). Faced with such pressing issues, it is inevitable that countries must seek alternative freshwater sources, including from the

desalination of brackish water, or even seawater. In recent years, the total freshwater production through desalination technologies worldwide is estimated to be over 100 million m<sup>3</sup>/day, and this number is expected to continue to increase sharply in the near future (Ahmed 2020).

However, most desalination technologies that are commonly used today (*e.g.* reverse osmosis technology and evaporation-condensation technology) all have the major disadvantage of exceedingly-high energy consumption, thus requiring the development of other alternatives that are more advanced, efficient, and environmentally friendly (Steffen 2022, Lim *et al.* 2021, Nguyen *et al.* 2021). One of such alternatives is the forward osmosis (FO) technology, which has the advantage of lower energy consumption and more stable operation, while still offering good output capacity and satisfactory quality of water production (Ahmed *et al.* 2021, Abounahia *et al.* 2023, Lambat *et al.* 2024, Teoh *et al.* 2011).

Interestingly, a review by Suwaileh *et al.* (2020) pointed out that nearly 80% of published articles on FO technology are focused on either discovering novel draw solutions and recovery methods, fabricating more functional semi-permeable membranes, or accessing the economic viability of FO designs. In contrast, the optimization of FO systems for particular draw solutions and their specific fouling behaviours are often overlooked, even though these factors hold the potential of significantly impacting the osmosis performance of all draw solutions (Hawari *et al.* 2016, Sanahuja-Embuena *et al.* 2019, Zhao *et al.* 2012, Yang *et al.* 2022). This leaves a considerable gap between theoretical research and practical application.

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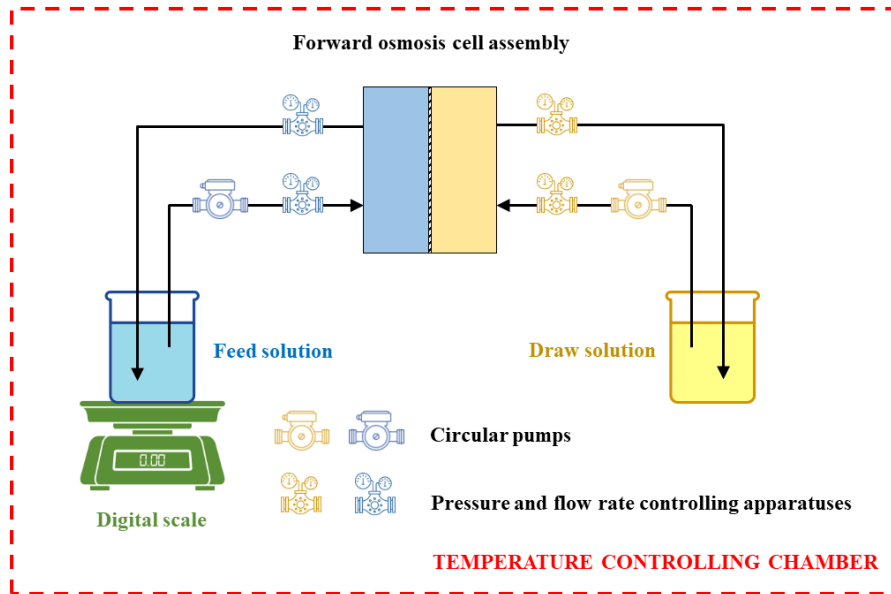


Fig. 1 Schematic diagram of laboratory-scale setup for FO experiments

Hoang *et al.* (2024) demonstrated the potential of a novel draw solution system based on trisodium  $\alpha$ -DL-alanine diacetate (MGDA solution) for water desalination applications, while also touched on the impacts of some operational parameters on the osmosis performance of the investigated draw solution. Based on such foundation, this study aimed to further optimize some operational parameters for FO systems utilizing MGDA solution as the draw solution in order to maximize water flux, while also reducing the loss of draw solutes caused by reverse osmosis flow. Additionally, this study also investigated the effects of extended and continuous operation duration on the fouling behaviours of MGDA solution, examined the efficiency of the physical cleaning method on fouling mitigation, and analyzed the overall quality of freshwater produced from the regeneration of diluted MGDA solution to assess its suitability for human consumption.

## 2. Experimental procedures

### 2.1 Materials and reagents

Trisodium  $\alpha$ -DL-alanine diacetate was obtained from BASF (Germany) under the commercial name Trilon® M Max BioBased Gran, while reagent grade sodium citrate tribasic dihydrate and citric acid were acquired from Merck (Germany). All of these chemicals were used directly without any further purification steps.

In contrast, commercial purified natural seasalt (solid contamination  $\leq 0.1\%$ ) was procured locally in Vietnam, and was dehydrated to constant weight at  $105^\circ\text{C}$  before being used. Similarly, real brackish water samples (salinity =  $10\text{‰}$ ) collected from Ha Lan estuary (So river, Nam Dinh province, Vietnam) were pre-filtered through a  $5\ \mu\text{m}$  membrane to remove large suspension particles before being utilized in FO experiments. Other solutions used in this study were prepared with deionized water freshly

produced at the laboratory utilizing a Purelab Flex-3 ultrapure water system (ELGA, UK).

Forward osmosis membranes used in this study were commercial thin-film composite (TFC) based membranes acquired from Aquaporin Asia (Singapore), while nano-filtration membranes were TFC-based TriSep TS40 membranes acquired from Sterlitech (US). Both types of membranes were originally purchased in the form of  $300 \times 300\ \text{mm}$  dry flat-sheet membranes, and later cut into appropriate dimensions before use.

### 2.2 Experimental methods

The general laboratory-scale experiment setup in this study was already described in details elsewhere by Hoang *et al.* (2024) (see Fig. 1).

In short, FO experiments were conducted on an acetal copolymer CF042D-FO laboratory scale forward osmosis cell assembly (Sterlitech, US) with an active membrane area of  $42\ \text{cm}^2$ . All experiments were conducted with feed solution stream and draw solution stream circulated through opposite sides of the forward osmosis cell assembly in opposite directions, and at average flow rates 230 and 200 mL/min, respectively. The draw solution was 25% MGDA solution, while the feed solutions were either tap water (optimization experiments), 10% artificial brackish water (long-term operation experiments), or pre-filtered real brackish water samples (water quality experiments). Additionally, all experiments were conducted with the active layer of the membrane facing the feed stream (FO mode). The initial volumes for both feed and draw solutions was roughly 5.000 mL, so that the effect of water permeation on their concentration could be minimized. The weight of the feed solution container was recorded every 15 min for a total duration of 150 min, with the first data point being recorded 15 s after turning on both pumps to ensure complete liquid filling and stable operation of the system. For long-term FO experiments, the experiment duration was

Table 1 Experimental design levels of chosen input variables in optimization of operational parameters, and the specific values for other non-investigated operational parameters

Operational parameters	Value
Feed stream	Deionized water
Draw stream	25% MGDA solution *
Inlet flow rate, draw stream	Adjusted in accordance with the changes of other variables
Relative flow direction between the feed stream and the draw stream	
Operational duration	150 minutes
* Percentage of solutes, by weight: trisodium $\alpha$ -DL-alanine diacetate = 85%, citric acid = 15%	

Variables	Factors	Levels		
		Low (-1)	Middle (0)	High (+1)
Inlet pressure difference, feed stream side (bar)	X1	0.0	0.2	0.4
Inlet temperature ( $^{\circ}$ C)	X2	20	30	40
Inlet flow rate, feed stream (mL/min)	X3	250	300	350

extended to 700 h with no periodic data recording.

Membrane cleaning experiments (after long-term operation) were conducted similar to FO experiments, however both the feed solution stream and the draw solution stream were replaced with non-circulated streams of tap water. The experiment duration was 30 min with no periodic data recording.

Nanofiltration (NF) experiments were conducted on an acetal copolymer CF042D laboratory scale crossflow cell assembly (Sterlitech, US) with an active membrane area of 42 cm<sup>2</sup>, while equipped with a 1.75 mm thick stainless-steel spacer (Sterlitech, US). NF operation parameters were as follows: feed stream pressure of 1.0 MPa, feed stream flow rate of 1.000 mL/min, and permeate stream recovery rate of 10%—which is equal to the excess water in the feed stream compared to that in the original draw solution before FO operations.

All optimization experiments were conducted at least in triplicate, so that any anomalies in experiment results would be detected. In case there were anomalies detected, another set of triplicate experiments would be again conducted, and then all data would be processed together to eliminate the anomalies. Experimental results were expressed using the measurement units of LMH (equivalent to L/m<sup>2</sup>·h) and GMH (equivalent to g/m<sup>2</sup>·h), which are standard measurement units commonly utilized in the field of osmosis study (Patel *et al.* 2021).

### 2.3 Analytical methods

The mass of solutions in their container were monitored manually using a digital scale (GeeLeaf, China) with a maximum weighing capacity of 10,000 g and an accuracy of 1 g. Likewise, the salinity and TDS of solutions were monitored manually using an Ezdo 7021 integrated salinity, TDS, and conductivity meter (Ezdo, France). The formulas for calculating water flux (J<sub>w</sub>), reverse solute flux (J<sub>s</sub>), and specific rate of draw solute loss due to reverse solute flux (R<sub>ds</sub>) from these experimental results was already described in detail elsewhere by Hoang *et al.* (2024).

Particularly, in FO systems, specific rate of draw solute

loss due to reverse solute flux (R<sub>ds</sub>) is defined as the ratio between the forward water flux (J<sub>w</sub>) and the reverse draw solute flux (J<sub>s</sub>) across the semi-permeable membrane.

The impacts of long-term operation on the surface structure of the FO membrane was evaluated by scanning electron microscopy (SEM) analysis. Immediately after the experiment was completed, the FO membrane was removed from the test apparatus, washed with deionized water to remove any solutions and other contamination still remaining on its surface, then dried with filter paper and coated with a layer of gold-palladium alloy using a sputter deposition device. Finally, SEM images of the membrane surface were acquired utilizing a Jeol SM-6510LV scanning electron microscope (Jeol, Japan) at an acceleration voltage of 8 – 12 kV and a magnification of 500 – 2,000 times.

### 2.4 Box–Behnken optimization design

In this study, 3-level Box–Behnken experimental design was applied to investigate and validate the influences of operational parameters on FO performance of the draw solution, which include J<sub>w</sub>, J<sub>s</sub>, and R<sub>ds</sub>. The variable input parameters were presented in Table 1, with specific values for each factor level determined based on preliminary experimental results. Definitions of statistical terms mentioned in this study and their significance were explained in detail by Montgomery (2001). Experimental design and results analysis were conducted automatically using the Design Expert 12 software, with optimal operational parameters being determined through utilizing the Response Surface Methodology (RSM).

## 3. Results and discussion

### 3.1 Optimizing the operational parameters

Within the scope of this study, the effects of inlet flow rate, inlet temperature, and inlet pressure difference on FO performance of the draw solution, which include J<sub>w</sub>, J<sub>s</sub>, and R<sub>ds</sub>, were investigated, with optimization done based on the

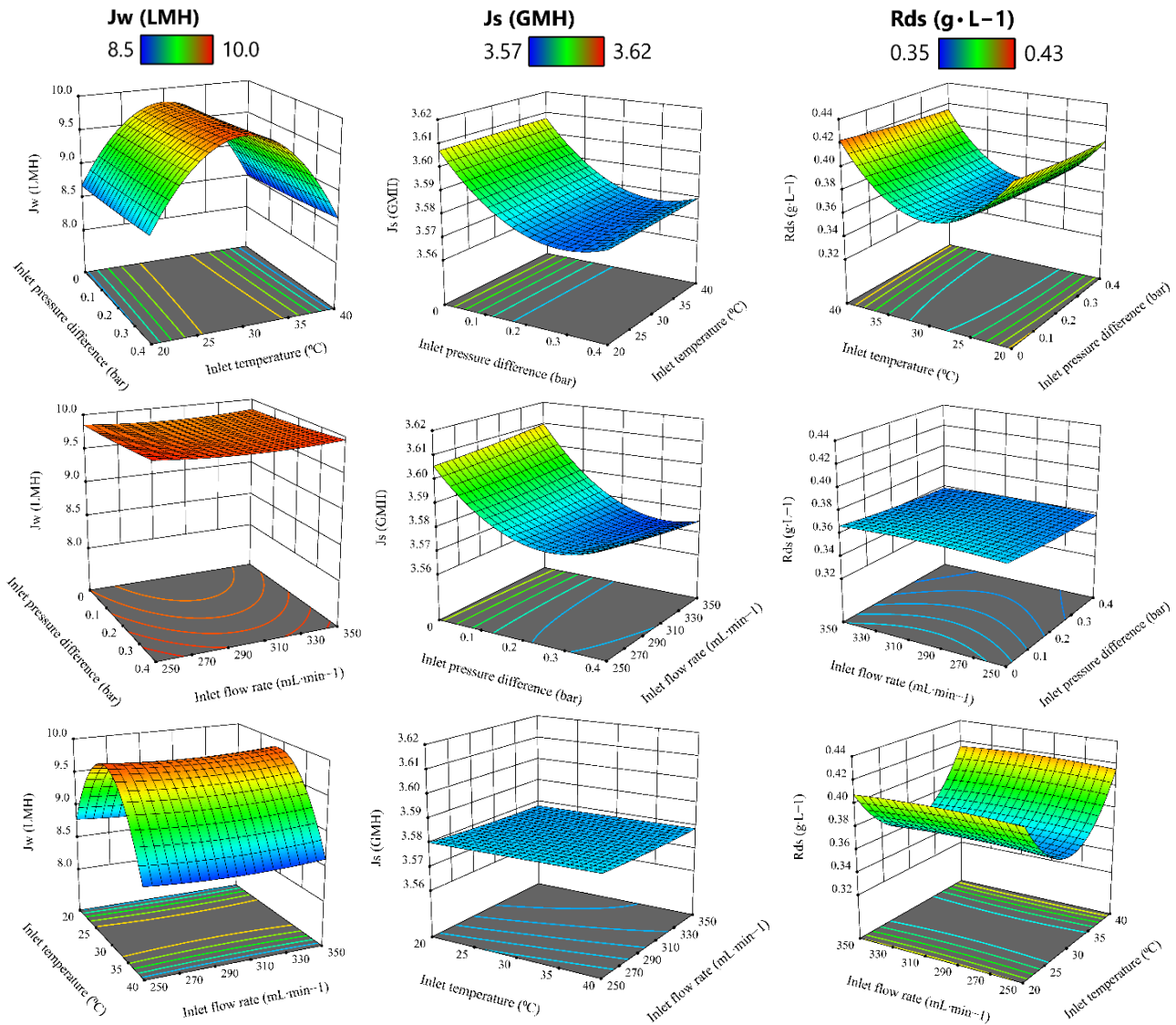


Fig. 2 3D response surfaces illustrating the influences of operational parameters on  $J_w$  (left),  $J_s$  (middle), and  $R_{ds}$  (right) values of the FO experimental system using the 25% MGDA draw solution

results received from Box–Behnken design model. The initial experimental results were presented in Table 2, while details on data fitting, model selection, and statistical analysis for model confirmation were calculated automatically on the Design Expert 12 software. The 3D response surface graphs illustrating interactions between operational parameters and FO performance of the 25% MGDA draw solution were shown in Fig. 2.

In general, analytical results suggested that for the 25% MGDA draw solution, inlet temperature was the most impacting operational parameter that affected water flux ( $J_w$ ) and the specific rate of draw solute loss due to reverse solute flux ( $R_{ds}$ ), while inlet pressure difference was the most impacting operational parameter that affected reverse solute flux ( $J_s$ ). Conversely, within the predetermined boundaries of this study, inlet flow rate demonstrated relatively little influence on FO performance of the investigated draw solution.

In particular, the  $J_w$  of the 25% MGDA draw solution exhibited significant growths when the inlet temperature

increased from 20°C to 30°C, which was consistent with existing literature and can be attributed to the influences of environmental temperature on osmosis properties of solutions. Specially, an increase in temperature would lead to the linear growths in osmotic potential of saline solutions consistent with the Van't Hoff equation for osmotic potential, since the dissolution of NaCl in water is generally unaffected by temperature as its heat of hydration almost equalizes its heat of dissolution. In contrast, salts created from weak acids and strong bases – which includes sodium salts of both N-(1-carboxyethyl)iminodiacetic acid and citric acids – have their dissolution characteristics in water highly dependent on temperature, leading to exponential growths in osmosis potential as temperature increases (Durie and Jessen 1964). Therefore, absence any other sources of influence, both the water flux and the specific rate of draw solute loss due to reverse solute flux of the 25% MGDA draw solution would increase with the increase in operational temperature (Phuntsho *et al.* 2012, Nematzadeh *et al.* 2016, Heo *et al.* 2016).

Table 2 Experimental data regarding the influences of operational parameters on FO performance of the 25% MGDA draw solution

Run	Variables			Responses		
	X1	X2	X3	Jw	Js	Rds
1	0.0	20	300	8.69	3.606	0.415
2	0.4	20	300	8.79	3.578	0.407
3	0.0	40	300	8.53	3.608	0.423
4	0.4	40	300	8.63	3.581	0.415
5	0.0	30	250	9.85	3.605	0.366
6	0.4	30	250	9.98	3.583	0.359
7	0.0	30	350	9.84	3.611	0.367
8	0.4	30	350	9.96	3.576	0.359
9	0.2	20	250	8.78	3.582	0.408
10	0.2	40	250	8.63	3.581	0.415
11	0.2	20	350	8.77	3.578	0.408
12	0.2	40	350	8.62	3.577	0.415
13	0.2	30	300	9.83	3.578	0.364
14	0.2	30	300	9.83	3.578	0.364
15	0.2	30	300	9.84	3.582	0.364
16	0.2	30	300	9.83	3.578	0.364
17	0.2	30	300	9.86	3.579	0.363

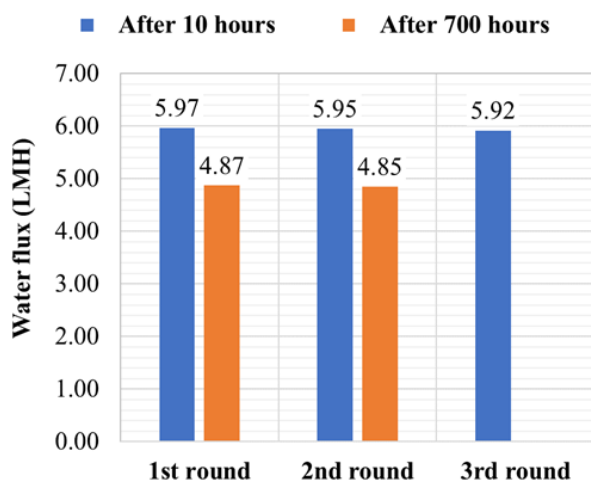


Fig. 3 Impacts of long-term operation and the use of surface cleaning procedure on water flux of 25% MGDA draw solution

However, depending on membrane design, the semi-permanent properties of the FO membrane could also change with the change in environmental temperature, leading to specific alterations in the mass transfer capability through the FO membrane of molecules and ions (Anh-Vu *et al.* 2024, Zhao and Zou 2011). For the 25% MGDA draw solution, increasing inlet temperature beyond 30°C would lead to a reduction in the Jw, indicating negative influences of environmental temperature on water permeability of the utilized FO membrane. Likewise, increasing inlet temperature would also negatively affecting solute permeability of the

semi-permanent membrane, which almost balanced out with the positive impact of temperature increasing on osmotic potential of the draw solution (Durie and Jessen 1964), leading to the apparent negligible overall influence of temperature on Js. Being the quotient received when dividing Jw by Js, it was natural for Rds to exhibit significant declines when the inlet temperature would increase from 20°C to 30°C, then significant growths when the inlet temperature would further increase from 30°C to 40°C.

Conversely, the apparent lack of impact on osmosis results of inlet flow rate can be explained by its mechanism of influence, or specially by its effects on the equalizing of concentrations within solutions. According to Hawari *et al.* (2016), at low velocities, fluids tend to flow without lateral mixing (laminar flow), leading to no cross-currents perpendicular to the direction of flow, nor swirls or eddies. Such a phenomenon would intensify the polarization of concentrations near the surface of the FO membrane, which was predominantly caused by the mass transfer of water and draw solutes across the semi-permeable membrane, reducing the overall trans-membrane osmosis drive. On the other hand, at high velocities, fluids tend to flow with much stronger swirls and eddies, promoting the homogenize of concentrations within solutions, thus alleviating negative impacts of the concentration polarization phenomenon. However, when inlet flow rate increases pass certain thresholds, the speed of concentration homogenization would significantly surpass the speed of trans-membrane mass transfer, leading to significantly reduced enhancements in osmosis results (Sanahuja-Embuena *et al.* 2019). Since preliminary experiments were conducted to determine the optimization boundaries of this study, the range of investigation for inlet flow rate would already fall within its most optimum range for desired FO performance, leading to the aforementioned experimental observed results.

Likewise, within the predetermined boundaries of this study, the apparent lack of impact on Jw of inlet pressure difference can be explained by its insignificance comparing the the main drive of water mass transfer, which was the trans-membrane difference in water osmosis pressure between the feed solution and the draw solution. Evidently, in all osmosis systems, water flux can be improved by increasing hydraulic pressure on the side of the feed solution (Gruber *et al.* 2011). However, since FO membranes are generally not designed to withstand excessive differences in trans-membrane hydraulic pressure, such increase in hydraulic pressure beyond the limits recommended by manufacturers may trigger catastrophic damages to the semi-permeable membrane. In contrast, for the mass transfer of the draw solute, such increases in inlet pressure difference on the feed side would be more significant, leading to an overall declining trend for Js, and subsequently Rds (Oh *et al.* 2014).

With the aim of maximizing water flux (Jw) while minimizing specific rate of draw solute loss due to reverse solute flux (Rds), optimization calculations were conducted, resulting in optimal levels of the investigated operational parameters as follows: inlet pressure difference = 0.4 bar on the feed stream side, inlet temperature = 30°C, and feed

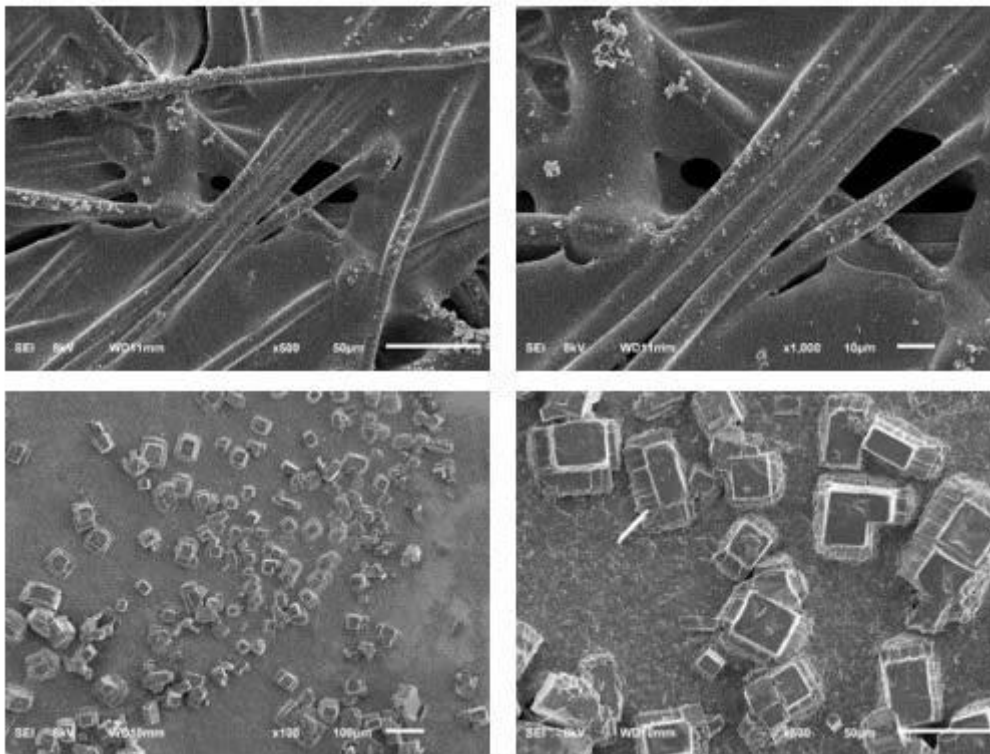


Fig. 4 SEM images of the membrane surfaces which were in contact with the 25% MGDA draw solution (top) and the feed solution (bottom)

stream inlet flow rate = 250 mL/min. At these operational parameters, the value of  $J_w$  and  $R_{ds}$  was predicted to be 9.97 LMH and 0.359 g/L, respectively.

Five additional confirmation experiments were conducted at calculated optimal operational parameters, yielding results which averaged within the 95% confident range of their predicted values:  $J_w = 9.996 \pm 0.192$  LMH, and  $R_{ds} = 0.3580 \pm 0.0020$  g/L. However, it should be noted that these optimal operational parameters were only valid within the specified range of values investigated. Additional experiments must be made to confirm any other extrapolations/interpolations built upon these experimental data.

### 3.2 Fouling behaviours of the membrane

Experiments investigating the impacts of long-term operation on fouling behaviours of FO membrane when using the MGDA draw solution were conducted at the predetermined optimum operational parameters, with the feed stream being 10% salt solution and the total operational duration being 700 hours. Experimental results (see Fig. 3) showed that after 700 hours of pseudo-continuous operation, the value of water flux significantly reduced by around 18%, from 5.97 LMH to 4.87 LMH. This phenomenon can be explained by the fact that after long durations of continuous operation, fouling would always occur on the surface and inside the porous structure of the semi-permeable membrane, hindering the mass transfer of water molecules through the membrane (Zhao and Zou 2011).

However, it should also be noted that in the case of the MGDA solution, the reduction in water flux was significantly on the lower side compared to other organic-based draw solutions. Based on SEM images of the semi-permeable membrane after 700 hours of pseudo-continuous operation (see Fig. 4), it was interesting to observe that the draw solution contacting side of the membrane was relatively clean, with almost no large-sized particles sedimentation or signs of significant microbial growth. This phenomenon can be explained by the draw solute MGDA-3Na itself being a cleaning agent, capable of preventing foulants attaching to the membrane surface.

Conversely, on the feed solution contacting side of the semi-permeable membrane, the existence of cubic crystalline particles with a size of about 10  $\mu\text{m}$  or more can be clearly observed. These particles were most likely NaCl salt crystals, formed due to the concentration polarization of the feed solution near the membrane caused by the trans-membrane movement of water molecules (Tang *et al.* 2010), which was further exacerbated by the reverse flux of  $\text{Na}^+$  cations from the side of the draw solution.

More interestingly, the membrane fouling caused by long-term FO operation using the MGDA draw solution was determined to be non-resistant to simple membrane cleaning procedures. In particular, by utilizing the surface cleaning method, it was possible to restore the water flux of the MGDA draw solution to 98% of its original value (see Fig. 3). These results clearly indicated that the membrane fouling occurred when using the MGDA draw solution as the draw solution mostly happened on the surface of the

Table 3 Key quality properties of water obtained from the regenerating of the 25% MGDA draw solution, compared to requirements by the National Technical Regulation QCVN 01-1:2018/BYT

Properties	Value		
	Analyzed	Requirements	
pH	-	6.9	6.0 – 8.5
TDS	‰	436	< 1,000
Sodium (Na)	mg/L	120	< 200
Hardness	mg/L	15	< 300
Fluoride	mg/L	0.4	< 1.5
Chloride	mg/L	0.4	0.2 – 1.0
Ammonium	mg/L	0	< 0.3

membrane, with minimal internal membrane fouling which is more resistant to surface cleaning procedures. Specially, the salt crystals formed on membrane surface were easily washed away by the combination of re-dissolving and physical removal, thus almost completely restoring the original water permeability of the semi-permeable membrane.

In conclusion, these results emphasized the advantages of MGDA solution compared to other organic draw solutions in FO desalination systems, which were low tendency of membrane fouling and the relatively ease of membrane cleaning.

### 3.3 Performance on real brackish water sample

Experimental results indicated no significant difference in the water flux generated by the 25% MGDA draw solution between two particular scenarios: when the feed solution was simulated saline water, and when the feed solution was a real brackish water sample (salinity calculated by NaCl = 10‰). In particular, the water flux generated in the two scenarios were 5.97 LMH and 5.74 LMH, respectively, representing a slight difference of only 3.85%.

The analytical results on some key properties of the water obtained from nanofiltration regeneration of the MGDA draw solution (see Table 3) showed that the draw solution can be effectively regenerated by the nanofiltration method, with the water produced meeting respective requirements as recommended in the National Technical Regulation QCVN 01-1:2018/BYT on the quality of clean water used for domestic purposes.

## 4. Conclusions

This study successfully explored various aspects of performance criteria used to determine the potentials of the MGDA draw solution in FO desalination systems, which included the optimization of operational parameters, the stability of osmosis performance in long-term operations, the effectiveness of fouling mitigation strategies, and the quality of produced freshwater. Particularly, the results

from this study highlighted the importance of managing the inlet temperature of solutions, and the advantages of MGDA solution compared to other organic draw solutions in FO desalination systems, which were low tendency of membrane fouling and the relatively ease of membrane cleaning.

In conclusion, this study bridged the gap between scientific exploration and practical application, providing valuable insights on the potential application of the draw solution system based on trisodium  $\alpha$ -DL-alanine diacetate in forward osmosis desalination systems. Findings from this study not only advanced the understanding of the MGDA draw solution, but also underscore the need to further investigate other novel draw solutions so that their potential applications can be fully explored. Future studies should expand on larger scale experiments in order to bring the MGDA draw solution closer to being utilized in real-life desalination operations.

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