MgCl$_2$ and MgSO$_4$ as draw agents in forward osmosis process for East Baghdad oilfield produced water treatment

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ABSTRACT

This study investigated a novel application of forward osmosis using different types of draw solutions for oilfield produced water treatment from the East Baghdad oilfield affiliated to the Midland Oil Company (Iraq). Magnesium chloride (MgCl$_2$) and magnesium sulfate (MgSO$_4$) were used as draw solutions in forward osmosis process to evaluate their effectiveness. Experiments were conducted in a laboratory scale forward osmosis system with cellulose triacetate hollow fiber membrane. In this work, sodium chloride solution was used as a feed solution with a concentration of 76 g/L (same concentration as the East Baghdad oilfield produced water) and the applied external pressure on the feed solution side was 2 bar. The impact of draw solution (DS) concentration (2.3 and 3 M) and mode of operation (batch mode and continuous mode) on the forward osmosis performance for produced water treatment were investigated on flux, recovery, feed solution concentration, and reverse salt flux. The recovery and feed solution concentration increased with increasing draw solution concentration and time, while the flux increased with increasing the draw solution concentration and decreased with time. The continuous mode of operation (t = 8 h) is more effective than the osmotic dilution mode (t = 12 h) in concentrating the feed solution. The result showed that in continuous mode with a draw solution concentration of 3 M, the feed solution was concentrated to 112.77 g/L with MgCl$_2$ as DS at which the recovery was 29.97%, while it was concentrated to 84.89 g/L with a recovery of 10.54% with MgSO$_4$ as DS.

Keywords: Forward osmosis; Magnesium chloride (MgCl$_2$); Magnesium sulfate (MgSO$_4$); Iraqi oilfield produced water

1. Introduction

The world's civilization, industrialization, and population, as well as increasing productivity, have all raised the need for clean water [1]. Desalination of produced water which is the largest amount of wastewater generated in oil and gas industries [2,3] promises to be an attractive solution for providing clean water to satisfy rising water demands. Many oil producing countries are focused on developing efficient and cost-effective treatment technologies for oilfield produced water in order to supplement their limited clean water resources [4–6]. Produced water contains dissolved organic compounds, dispersed oil droplets, dissolved inorganic compounds, and suspended solids [7]. Dissolved salts are the predominant inorganic components associated with produced water, and their concentrations range from 100 to 300,000 mg/L [8,9]. Significant pretreatment, such as skimming [10], sedimentation, coagulation/flocculation [11], and electrochemical oxidation [12] is required to reduce scaling and fouling potential before produced water can be treated using membrane or thermal methods [13]. Membrane-based separation

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techniques such as reverse osmosis (RO), nanofiltration (NF), and forward osmosis are now being used in various ways to provide fresh water from saline and polluted water. Recently, there has been an interest in developing forward osmosis (FO) technology [14].

Forward osmosis has been considered as a potential technology to solve environmental challenges due to its several benefits, including high rejection of various contaminants, and low membrane fouling [15,16]. Forward osmosis is driven by osmotic pressure rather than hydraulic pressure, as a result, it can desalinate high salinity feed waters by using simple and inexpensive low-pressure equipment [17]. In contrast to reverse osmosis, FO is not constrained by a high pressure operating limit, which corresponds to a solution total dissolved solids (TDS) concentration of around 70,000 mg/L [14]. The FO process has been proposed as a promising on-site treatment method for the treatment of produced water; however, few studies have been conducted to date to examine the performance of FO membranes for the treatment of these complicated feed streams [18,19]. In the FO process, water is transported across a semipermeable, salt-impermeable FO membrane from low osmotic pressure feed solution to high osmotic pressure draw solution [20,21]. The semipermeable membranes used in FO are generally cellulose triacetate (CTA) or thin-film composite (TFC) polyamide membranes [22].

The selection of an appropriate draw solute is a critical factor in forward osmosis process. The draw solution must have high osmotic pressure, non-toxic, also, it must have low molecular weight, highly water-solubility [23], low viscosity for easy pumping around the system and improved water flux [22], and minimal reverse draw solute flux [24]. Moreover, it should be available in large quantities at a low cost [25], and efficiently reconcentrated with minimal energy consumption [21].

Multivalent salts like $MgCl_2$, $CaCl_2$, and $MgSO_4$ have several advantages compared to monovalent salts. Because of the greater number of ionic species formed during dissociation, their larger hydration radii result in lower reverse salt fluxes when compared to NaCl, and it is possible to achieve higher osmotic pressures with the same molar concentrations [24]. In some applications where high removal is required, multivalent ions (e.g., Ca^{2+} and Mg^{2+}) solutions with lower diffusion coefficients may be preferable [26].

The osmotic pressure of a solution can be estimated from Van't Hoff equation as a function of solute concentration [27]:

$$\pi = i\phi CRT \tag{1}$$

where i is the number of the dissociating ions, ϕ is the osmotic coefficient, C is the concentration of the solute, R is the gas constant, and T is the temperature.

Among the draw solution (DS) salts, $MgCl_2$ performed the best, with no significant increase or decrease in flux performance. This is because it has three ions (Van't Hoff factor $i = 3$)) and can generate high osmotic pressure and increase water flux, whereas others only have two [1]. Magnesium sulfate has a lower osmotic pressure (Van't Hoff factor $i = 2$)) than other DS, especially at higher concentrations. The nanofiltration process, which is a low pressure membrane

process, can be used to effectively remove divalent ions with a rejection of 99% [28] and can be proposed as an alternative regeneration method for the $MgCl_2$ and $MgSO_4$ draw solutions [29,30].

On the basis of the well-known concentration polarization phenomena in pressure driven processes such as RO, the theory of FO concentration polarization has been adopted [5]. According to the orientation of the asymmetric membrane, that is, active layer facing feed solution (AL-FS) or active layer facing draw solution (AL-DS), concentration polarization may be either concentrative or dilutive and internal or external to the membrane. The term "internal" is often used to refer to the support layer, while "external" refers to the active layer [24]. There is reverse salt diffusion in FO, which is the diffusion of salt from the draw solution to the feed solution in the opposite direction of the main water flux [31].

The following equation can be used to describe the effect of internal, external concentration polarization phenomena, and reverse salt flux on flux [31]:

$$J_w = A\left(\pi_{ds} \exp\left(-\frac{J_w}{k_{sup}}\right) - \pi_f \exp\left(\frac{J_w}{k_{ca}}\right) \right)$$
$$+ B\left(\exp\left(-\frac{J_w}{k_{sup}}\right) - \exp\left(\frac{J_w}{k_{ca}}\right) \right) \text{ For AL-FS orientation} \tag{2}$$

$$J_w = A\left(\pi_{ds} \exp\left(-\frac{J_w}{k_{ca}}\right) - \pi_f \exp\left(\frac{J_w}{k_{sup}}\right) \right)$$
$$+ B\left(\exp\left(-\frac{J_w}{k_{ca}}\right) - \exp\left(\frac{J_w}{k_{sup}}\right) \right) \text{ For AL-DS orientation} \tag{3}$$

where J_w is the water flux, A is the membrane permeability of pure water, B is the salt permeability, π_{ds} is the osmotic pressure of DS, π_f is the osmotic pressure of FS, k_{sup} is the combined mass transfer coefficient for the support layer itself and the adjacent external mass transfer layer, the mass transfer coefficient for the support layer itself can be written as D/S where D is the diffusivity of salt in water and S is the structural parameter which is directly proportional to the extent of internal concentration polarization, $S = t\tau/\varepsilon$, t is the thickness of the membrane, τ is the tortuosity, and ε is the porosity, k_{ca} is the mass transfer coefficient for the channel adjacent to the active layer.

For AL-FS, $Pe_{draw} = J_w/k_{sup}$ and $Pe_{feed} = J_w/k_{ca}$ while for AL-DS $Pe_{draw} = J_w/k_{ca}$ and $Pe_{feed} = J_w/k_{sup}$, where Pe is the Peclet number. Thus, a unified relationship can be presented in Eq. (4). In moving from Eqs. (2) and (3) to Eq. (4) the opportunity has been taken to invert the terms in the second bracket, so that both terms in brackets are positive. This format emphasizes how reverse salt diffusion reduces water flux [31].

$$J_w = A\left(\pi_{ds} \exp(-Pe_{draw}) - \pi_f \exp(Pe_{feed}) \right)$$
$$- B\left(\exp(Pe_{feed}) - \exp(-Pe_{draw}) \right) \tag{4}$$

Solute builds up on FO membrane's active layer as permeate water flows from the feed solution, this is known as concentrative external concentration polarization. Simultaneously, dilutive external polarization is caused by the dilution of draw solution at the permeate membrane interface. External concentration polarization, both concentrative and dilutive, reduces the effective osmotic driving force, which results in lower water flux. However, due to the absence of hydraulic pressure in FO, the external concentration polarization is usually negligible. The internal concentration polarization, on the other hand, is an important factor in FO applications [32]. Inorganic scaling occurs when the water's soluble salts become supersaturated. These salts may be deposited on the membrane's surface, closing the pores of the membrane, resulting in obvious flux decay. The most common scaling salts in RO and FO processes are calcium carbonate, magnesium hydroxide, and calcium sulfate [33].

This work aims to study the efficiency of $MgCl_2$ and $MgSO_4$ as draw agents in forward osmosis process for simulated oilfield produced water from the East Baghdad oilfield as a feed solution. The possibility of using a CTA hollow fiber membrane with AL-FS orientation to generate pure water from the FO process will be investigated with regard to water flux and reverse salt flux.

2. Experimental work

2.1. Materials and membrane

All of the chemicals were analytic reagent grade and used without further treatment. NaCl (99%, HiMedia Laboratories Pvt. Ltd., India) to prepare feed solution has the same concentration as the East Baghdad oilfield produced water, $MgCl_2 \cdot 6H_2O$ (99%, S D Fine-Chem Limited, India) and $MgSO_4$ (99%, Central Drug House (P) Ltd., India) to prepare draw solution has different concentrations. The distilled water of conductivity 1.7 μS/cm was used to prepare the draw and feed solution. The presented work employs a cellulose triacetate hollow fiber membrane (CTA-HF) (TOYOBO, Japan) membrane. The membrane's effective surface area is 0.15 m². For AL-FS orientation (FO mode), the draw solution has been pumped to the lumen-side of the hollow fiber membrane, while the feed solution has been pumped to the shell-side.

2.2. Forward osmosis process

The experimental setup for the FO process used in this work is shown in Fig. 1. The membrane orientation was AL-FS. The experiments were conducted on counter-current flows in which the feed solution and draw solution flowed in opposite directions. The initial volumes of the feed and the draw solution in the FO process were 1,000 and 500 mL, respectively. Two diaphragm pumps (PJ-1611, China) have been used for recirculating draw solution through the HF module's lumen side, pressurizing and recirculating feed solution through the shell side. The NaCl solution with a concentration of 76 g/L was employed as FS, while $MgCl_2$ and $MgSO_4$ were employed as DS with different concentrations (2.3 and 3 M). Two rotameters were used to measure the feed solution volumetric flow rate as well as the draw solution volumetric flow rate. Feed and draw solution flow rates have been held constant at 2 and 0.1 L/min, respectively. Throughout the tests, the cooling/heating coils were immersed in the draw and feed solution containers
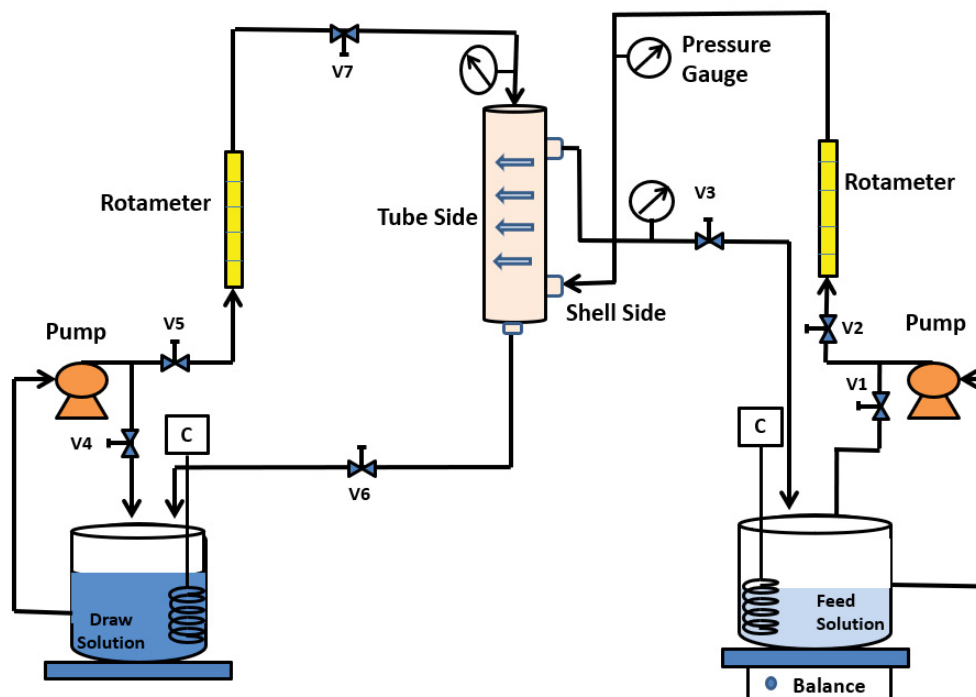

Fig. 1. The experimental setup of forward osmosis process (FO).

to control the temperature (35°C ± 1°C). The inlet and outlet pressures of the feed and draw solution were measured using a pressure gauge. The pressure of 2 bar was applied on the FS side (shell side) and it was adjusted by using valve 3. Concentrations of the draw and feed solutions were measured through a conductivity meter (EC-Meter BASIC 30, Crison, Spain). The water permeation flux (J_w) was calculated by measuring the weight change of the feed solution with a digital mass balance. J_w (L/m² h, expressed as LMH) was calculated using Eq. (5) [34].

$$J_w = \frac{\Delta V}{A_m \Delta t} \tag{5}$$

In which, ΔV is the permeate water during test duration Δt. A_m represents the effective area of the membrane.

The reverse solute flux J_s (g/m² h) of draw solution was determined by using Eq. (6) [25]:

$$J_s = \frac{V_t C_t - V_o C_o}{A_m t} \tag{6}$$

where C_t and V_t were the feed solution's concentration and volume at time t, respectively, measured at time t, and C_o and V_o were the initial concentration and volume of the feed solution.

3. Results and discussions

3.1. Batch mode

The effect of DS concentration (2.3 and 3 M) for two types of draw solution (MgCl₂ and MgSO₄) on water flux, recovery, feed solution concentration, and reverse solute flux were evaluated for 12 h. These concentrations were diluted by water transferred from the feed solution during the experiment until an approximate equilibrium between the feed and draw solutions was attained. According to

Figs. 2–4 the water flux, recovery, and FS concentration increase with DS concentration for both types of draw solution. Due to the dilution of the draw solution and the concentrate of the feed solution, the difference in osmotic pressure between the two solutions decreased, and the driving force for water permeation decreased, which resulted in flux decrease as shown in Fig. 2. These findings are supported by Blandin et al. [35].

The deviation of the flux from the linearity is due to the effects of internal concentration polarization (ICP) [Eq. (2)], which decreases the draw solution's effective driving force. The results indicate that water flux depends not only on the concentration of the draw solution but also on the type of draw solution. The flux, recovery, and feed solution concentration for MgCl₂ are greater than MgSO₄ as shown in Figs. 2–4. For example, the recovery after 12 h for MgSO₄ with concentrations of 3 and 2.3 M is lower than that of MgCl₂ for the same concentration by 62.9% and 60.2%, respectively. This could be attributed to a higher solute diffusion coefficient (D) [Eq. (2)] resulting in greater efficiency of MgCl₂ than MgSO₄. As a result, MgSO₄ salt with a bigger size than MgCl₂ salt has lower diffusion coefficients that are more affected by internal concentration polarization than MgCl₂. The diffusion coefficient values are 1.1×10^{-5} and 0.9×10^{-5} cm²/s for MgCl₂ and MgSO₄, respectively. The respective diffusion constants are 0.55×10^{-9} m²/s for Mg²⁺ ion and 1.5×10^{-9} m²/s for Cl⁻ ion, 1.07×10^{-9} m²/s for SO₄²⁻ [22,36,37]. Furthermore, MgCl₂ has a relatively high osmotic pressure according to Eq. (1). For the same concentration (M), MgCl₂ has greater osmotic pressure than MgSO₄ because the dissociating of MgCl₂ is 3 while for MgSO₄ is 2.

Fig. 2 shows that the flux in some experiments (such as at t = 1.5, 2, 5.5, and 6 h) for MgSO₄ at a concentration of 2.3 M is higher than that of 3 M. This may be attributed to the scaling and dilutive internal concentration polarization caused by the high concentration (3 M is the maximum solubility for MgSO₄) of MgSO₄.
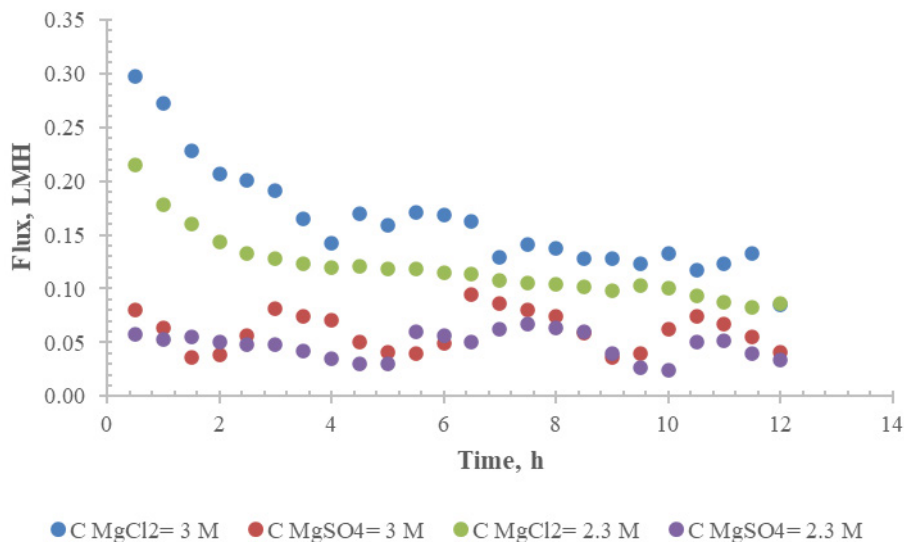

Fig. 2. Flux as a function of time for simulated PW as FS ($C_{FS,i}$ = 76 g/L) with MgCl₂ and MgSO₄ as DS for FO process.
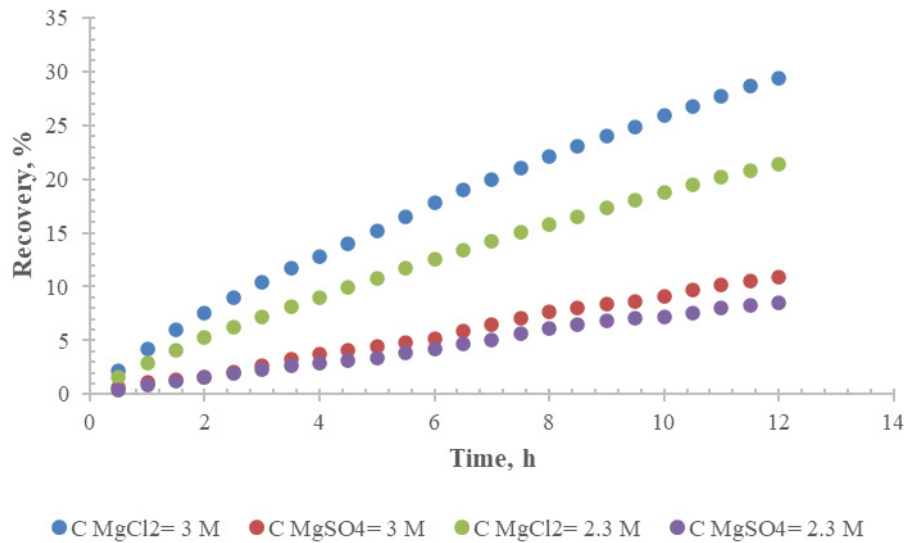
Fig. 3. Recovery as a function of time for simulated PW as FS ($C_{FS,i}$ = 76 g/L) with MgCl$_2$ and MgSO$_4$ as DS for FO process.
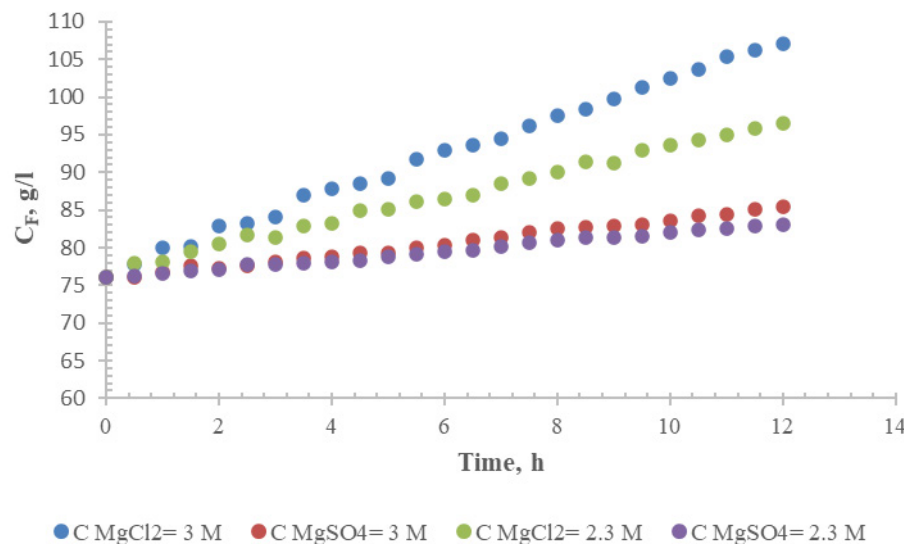

Fig. 4. Feed solution concentration as a function of time for simulated PW as FS ($C_{FS,i}$ = 76 g/L) with MgCl$_2$ and MgSO$_4$ as DS for FO process.

The reverse solute flux (J_s) was measured in FO using DS concentrations 3 M in an osmotic dilution mode for 2 h with deionized water (DW) as feed solution. Fig. 5 shows that the reverse salt flux of MgSO$_4$ is lower than that of MgCl$_2$ by 27.27%. From the values of diffusion constants mentioned above for ions, it seems that Cl$^-$ ion controlled the diffusion from DS to FS along a concentration gradient. For this reason, the reverse salt flux for MgCl$_2$ is higher than MgSO$_4$. Mineral salt scaling will also occur on the membrane surface in the case of draw solutions containing scale precursor ions (e.g., Mg^{2+} and SO$_4^{2-}$). Thus, the usage of MgSO$_4$ which is likely to cause scaling will reduce RSF. These findings are in agreement with Achilli et al. [32].

Figs. 6 and 7 show that the initial water flux and recovery of the PW as FS was significantly less than the initial flux and recovery in the baseline experiment (FS is deionized water). This is due to the interaction of high salinity, external concentration polarization, and feed solution viscosity. These findings are agreed with Hickenbottom et al. [38]. The initial water flux of the experiment with PW feed solution was lower than that of the baseline experiment by 85.7% and 96% for MgCl$_2$ and MgSO$_4$, respectively.

3.2. Continuous mode

The flux, recovery, and FS concentration were measured in FO using two concentrations of both MgCl$_2$ and MgSO$_4$ in the experiments, 2.3 and 3 M. These DS concentrations were kept constant during the experiment, and the time for each experiment was 8 h. Figs. 8–10 show the FO water flux,
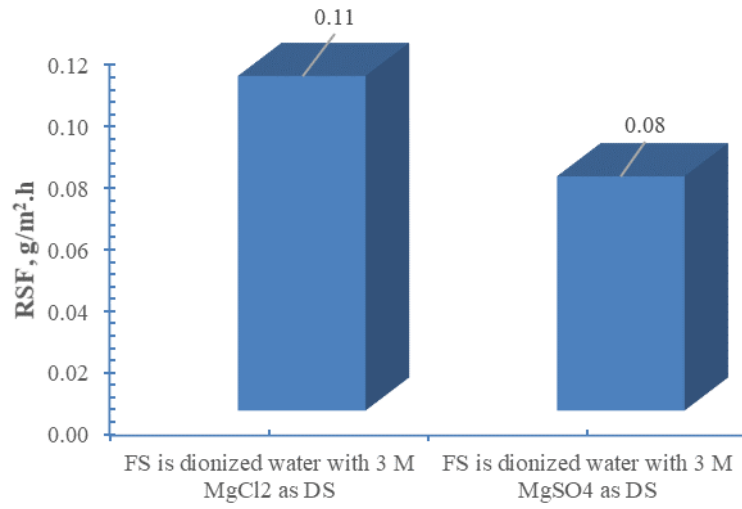
Fig. 5. Reverse salt flux of $MgCl_2$ and $MgSO_4$ for DW as FS with $MgCl_2$ and $MgSO_4$ as DS ($C_{DS,i}$ = 3 M, t = 2 h) for FO process.
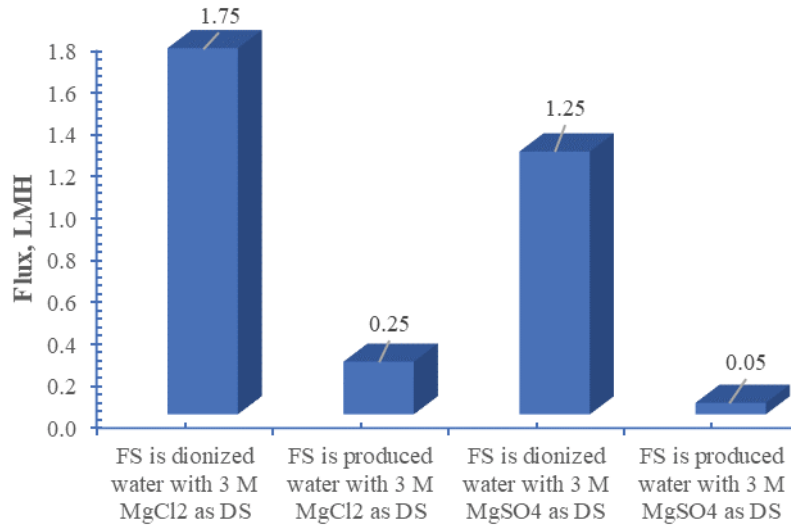

Fig. 6. Flux for PW and deionized water (DW) as FS with $MgCl_2$ and $MgSO_4$ as DS ($C_{DS,i}$ = 3 M, t = 2 h) for FO process.
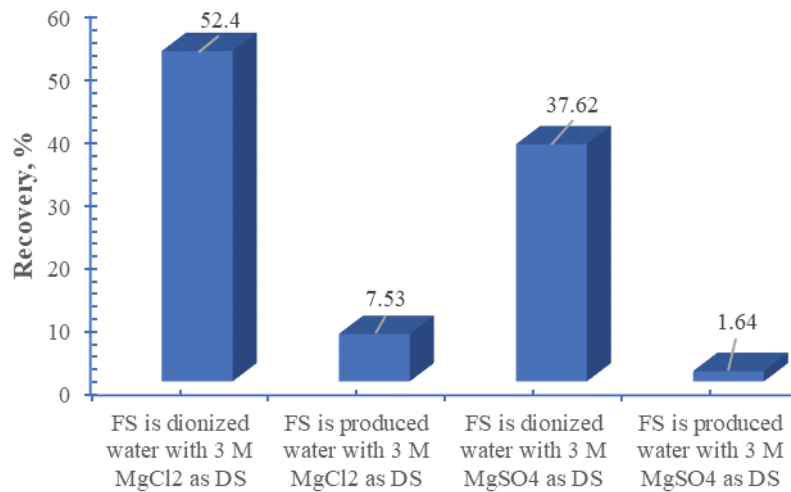

Fig. 7. Recovery for PW and deionized water (DW) as FS with $MgCl_2$ and $MgSO_4$ as DS ($C_{DS,i}$ = 3 M, t = 2 h) for FO process.
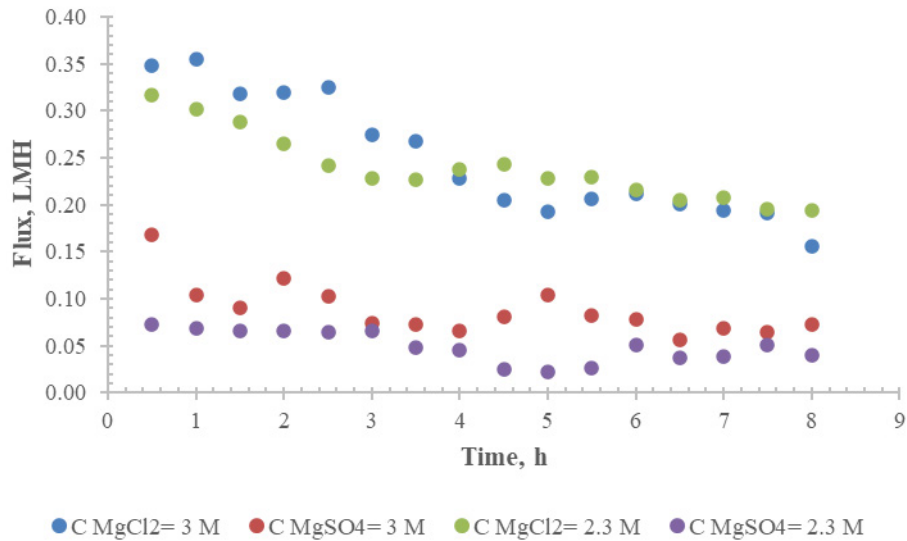
Fig. 8. Flux as a function of time for simulated PW as FS ($C_{FS,i}$ = 76 g/L) with MgCl$_2$ and MgSO$_4$ as DS for FO process.
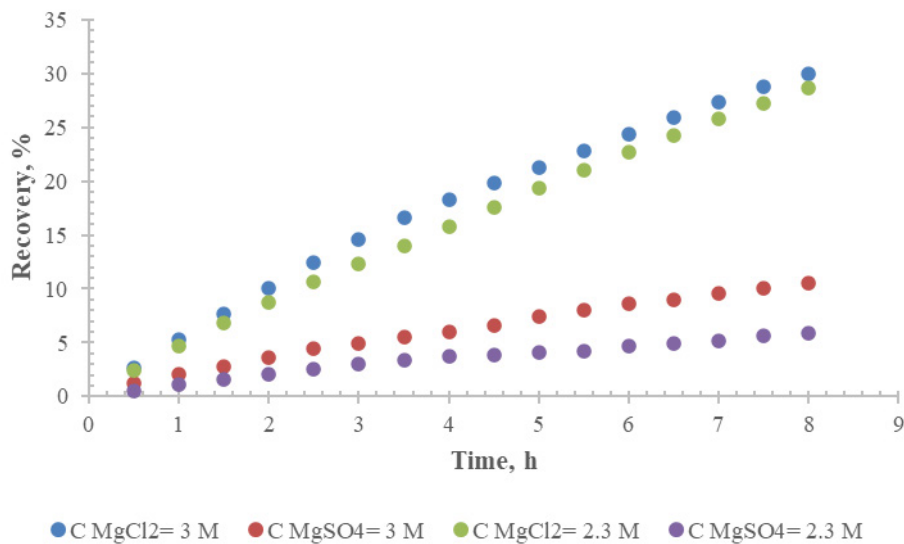

Fig. 9. Recovery as a function of time for simulated PW as FS ($C_{FS,i}$ = 76 g/L) with MgCl$_2$ and MgSO$_4$ as DS for FO process.

recovery, and feed solution concentration increases with DS concentration for both MgCl$_2$ and MgSO$_4$. The flux increases with the increase of concentration of the DS because the osmotic pressure of the DS increases which means a higher driving force. Furthermore, MgCl$_2$ gave higher water flux than MgSO$_4$ due to higher solute diffusion coefficient and lower molecular weight of MgCl$_2$ that gives higher osmotic pressure, that is, higher driving force as mentioned in the batch mode.

Water flux rapidly decreased with time due to a decrease in the osmotic pressure driving force, which corresponded to an increase in feed solution concentration, which decreased the osmotic driving force. It is, therefore, very possible that the increase of feed concentration also

increases the external concentration polarization of the active layer surface. Also, scaling caused by feed and draw solutions could also accumulate on the membrane surface and reduce flux. These findings are in agreement with Yun et al. [39], Liden et al. [40], and Jamil et al. [41].

Fig. 8 shows that the flux in some experiments (such as at t = 4.5, 5, 5.5, 7, and 8 h) for MgCl$_2$ at a concentration of 2.3 M is higher than that of 3 M. This may be attributed to the dilutive internal concentration polarization [Eq. (2)] caused by the high concentration of MgCl$_2$.

The flux of MgSO$_4$ is lower than that of MgCl$_2$ by 56.3% and 79% for DS concentrations of 3 and 2.3 M, respectively. Furthermore, the recovery of MgSO$_4$ is lower than that of MgCl$_2$ by 64.8% and 79.4% for DS concentrations of
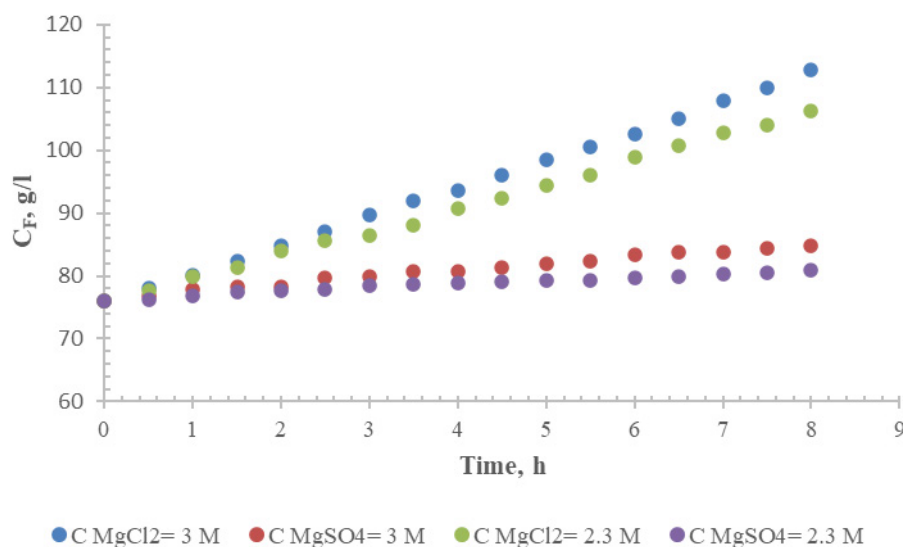
Fig. 10. Feed solution concentration as a function of time for simulated PW as FS ($C_{FS,i}$ = 76 g/L) with MgCl$_2$ and MgSO$_4$ as DS for FO process.

3 and 2.3 M, respectively. The FS concentration of MgSO$_4$ is lower than that of MgCl$_2$ by 24.7% and 23.9% for DS concentrations of 3 and 2.3 M, respectively.

4. Conclusion

The osmotic agent is considered a key factor in improving the efficiency of the forward osmosis process, especially when the feed solution has high concentrations such as produced water (i.e., East Baghdad oilfield). MgCl$_2$ osmotic agent was effective for produced water treatment in forward osmosis process compared to MgSO$_4$. MgCl$_2$ provided effective osmotic pressure than MgSO$_4$ at the same concentration, moreover, MgCl$_2$ can reach a higher concentration than MgSO$_4$ due to their high solubility, but the reverse salt flux for MgSO$_4$ is lower than that of MgCl$_2$. The flux of MgCl$_2$ and MgSO$_4$ for 3 M draw solution concentration after 8 h is 0.16 and 0.07 LMH, respectively, while the recovery is 29.97% and 10.54%, respectively. The reverse solute flux after 2 h is 0.11 and 0.08 g/m²·h for MgCl$_2$ and MgSO$_4$, respectively. Forward osmosis operated in a continuous mode can effectively concentrate feed solution 1.5 times their initial value after 8 h with 3 M MgCl$_2$ as draw solution, while forward osmosis operated in the osmotic dilution mode can concentrate feed solution 1.4 times their initial concentration after 12 h with 3 M MgCl$_2$ as draw solution.

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