



Nitrate removal from aqueous solution by direct contact membrane distillation using two different commercial membranes

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ABSTRACT

In the present work, the removal of nitrate from aqueous solution by direct contact membrane distillation (DCMD) using flat sheets polypropylene (PP) and polyvinylidene fluoride (PVDF) membranes was studied. Effect of operating parameters, such as feed temperature, feed flow rate, initial nitrate concentration, feed ionic strength, and competing co-existing anions on permeate flux and nitrate rejection, was investigated. In all DCMD experimental runs, an almost complete nitrate rejection was achieved (higher than 99.90%) and the permeate nitrate concentration was largely below the maximum permissible level in potable water. Under the same operating conditions, PVDF hydrophobic membrane showed a higher permeate flux of 37.21 L/m²h than PP membrane with a permeate flux of 4.12 L/m²h. For both the membranes, feed temperature is the important operating parameter which enhanced exponentially the permeate flux. Likewise, a positive effect on permeate flux was found when feed flow rate was increased. However, no significant effect was found by varying initial nitrate concentration, feed ionic strength, and the presence of co-existing anions on DCMD permeate flux or nitrate rejection efficiency.

Keywords: Nitrate removal; DCMD process; Permeate flux; Nitrate rejection; Hydrophobic membrane

1. Introduction

Surface, brackish, and groundwater, in many parts of the world, are not suitable for direct consumption due to high salt concentrations or in some cases due to naturally occurring hazardous contaminants such as arsenic [1,2], fluoride [3,4], and nitrate [5,6]. The

presence of high levels of nitrates (NO₃⁻) in water is mainly due to intensive agriculture activities and wastewater discharges. Excessive nitrate levels in drinking water can lead to taste and odor problems and even contribute to a potential risk to human health such as methemoglobinemia, cancer, and tumors [7]. The World Health Organization (WHO) has recommended the maximum nitrate level in

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drinking water to be 50 mg/L [8]. Thus, their removal from contaminated water gains more and more attention.

Nitrate is a stable and highly soluble anion with low potential for co-precipitation or adsorption. These properties make it difficult to remove using conventional water treatment technologies such as coagulation, filtration, and disinfection [9]. Currently, there are several technologies used for treating nitrate-contaminated water, including ion exchange [10], adsorption [11], biological denitrification [12], catalytic reduction [13], reverse osmosis [14], nanofiltration [15], and electrodialysis [16]. However, each one of the above methods presents some drawbacks. For example, the need of regeneration, the environmental impact due to the use of chemical compounds, and the loss of the adsorbent capacity with time are some issues related to adsorption or selective exchange resins techniques.

Recently, membrane distillation (MD) has been investigated as a possible alternative membrane process for the treatment of waters contaminated by nitrate. MD is an emerging non-isothermal membrane separation process. It is a thermally driven process that involves transport of water vapor through non-wetted porous hydrophobic membrane [17], while all non-volatile compounds (like nitrate contaminant) are retained at the feed compartment. The main advantage of MD is the ability to operate at lower hydrostatic pressure than conventional pressure-driven membrane processes and a lower operating feed temperature, considerably below its boiling point, than conventional distillation.

Direct contact membrane distillation (DCMD) is the simplest and the best known configuration of MD, in which the hot feed side and the cold permeate solution are maintained in direct contact with the two sides of the hydrophobic membrane [18]. Previous studies have widely demonstrated that DCMD could be effectively applied for desalination of different sources of saline water [19–22], but few studies have

been undertaken on DCMD used for removal of contaminants such as fluoride [23,24] and arsenic [25–28].

Nitrate removal using MD has not been studied much previously. In this work, the feasibility and performance of DCMD process to treat aqueous solution with high nitrate contaminant using two kinds of hydrophobic membranes [polypropylene (PP) and polyvinylidene fluoride (PVDF)] was studied. DCMD was carried out under relevant parameters such as feed temperature, feed flow rate, initial nitrate concentration, feed ionic strength, and the presence of different competing anions.

2. Materials and methods

2.1. Membrane and DCMD setup

Two commercially available hydrophobic microporous membranes made of PVDF and PP polymers were used in our DCMD pilot scale. Table 1 shows the principal characteristics of used membranes as specified by manufacturers and completed by others' analysis techniques, including atomic force microscopy (AFM), contact angle measurement, and differential scanning calorimetry (DSC), which is presented in Fig. 1.

AFM image can give an idea on the surface roughness which is an important property of polymeric membranes [29]. The roughness parameters, obtained with AFM analysis software, characterize the surface of studied membranes, and the values of the average roughness and maximum roughness for PP membrane were 3.233 and 16.08 nm, respectively, and for PVDF membrane were 31.73 and 145.99 nm, respectively. The thermal properties of polymer samples can be realized by DSC analysis. DSC thermogram gives the enthalpy of melting and the melting point of studied membranes; their values for PP membrane were 123.766 J/g and 164.64°C, respectively, and for PVDF membrane were 24.275 J/g and 163.49°C, respectively. The values of contact angles for two commercial

Table 1
Characteristics of PP and PVDF membranes

| Material | Polypropylene | Polyvinylidene fluoride |
|---------------------------------------|-----------------------|-------------------------|
| Contact angle (°) | 114.3 | 127.3 |
| Nominal pore size, μm | 0.064 | 0.22 |
| Thickness, μm | 25 | 125 |
| Porosity, % | 55 | 75 |
| Manufacturer | Hoechst-Celanese, USA | GVHP, Millipore |
| Liquid entry pressure, kPa | 200 | 204 |
| Effective membrane area, m^2 | 4.2×10^{-3} | 4.2×10^{-3} |

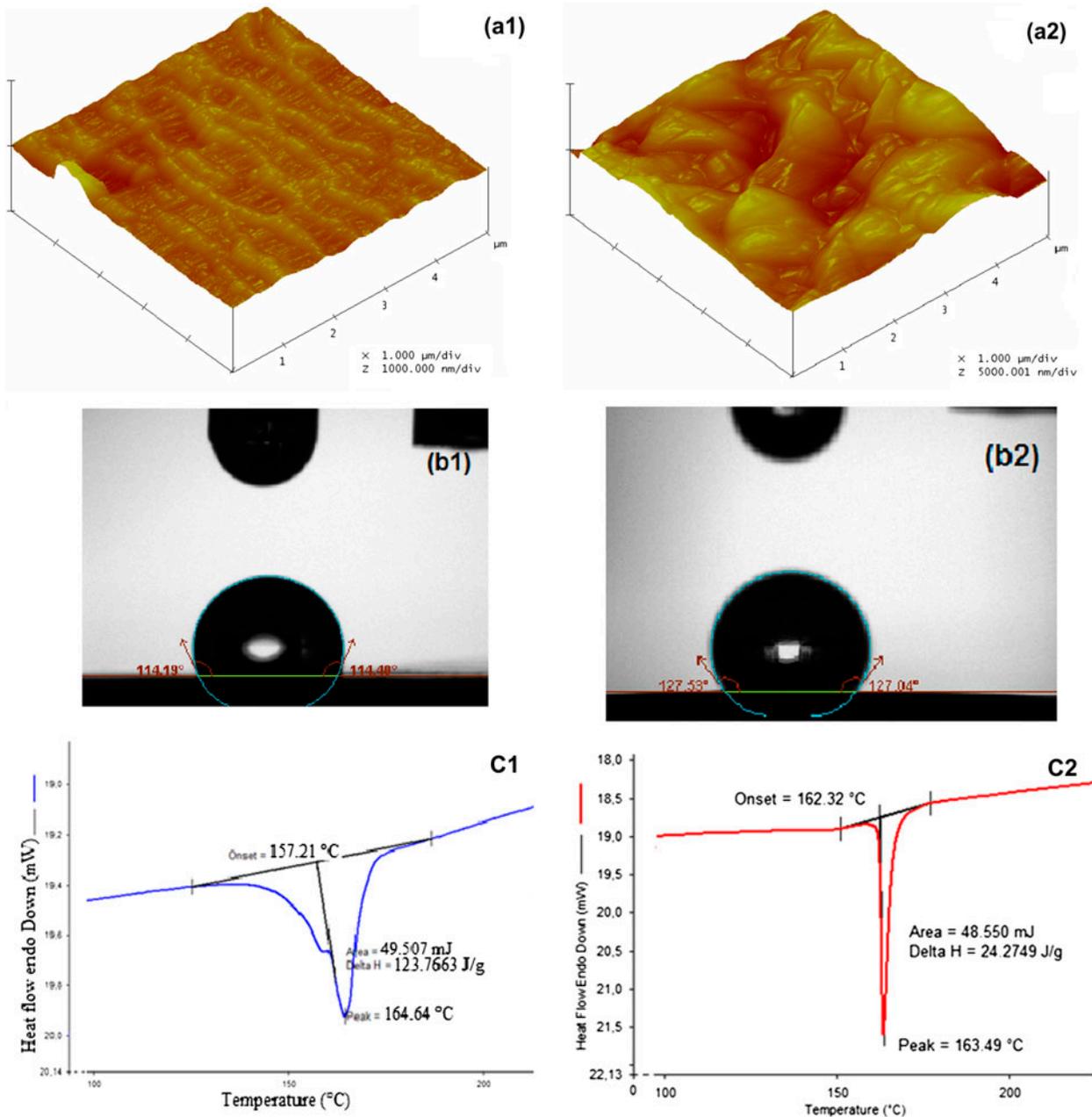


Fig. 1. (a) AFM, (b) contact angle measurement, and (c) DSC of both (1) PP and (2) PVDF commercial membranes.

membranes were 114.2° for PP membrane and 127.3° for PVDF membrane, which give the information about the surface hydrophobicity of two kinds of membrane.

The schematic diagram of the laboratory-scale flat sheet DCMD process designed, built, and tested for this study is shown in Fig. 2. The laboratory pilot consisted of two thermostatic cycles (feed and permeate) that were connected to a membrane module which was made by Plexiglas. The feed compartment, which

is connected to a heating system, is maintained at a hot temperature and the permeate compartment, which is connected to a cooling system, is maintained at a cold temperature. The hydrophobic membrane was placed between the two compartments with spacer material. The effective membrane area is 0.0042 m^2 . The bulk feed and permeate temperatures were measured inside each compartment by digital thermocouples with an accuracy of $\pm 0.1^\circ\text{C}$. Two peristaltic pumps were used to circulate the hot feed

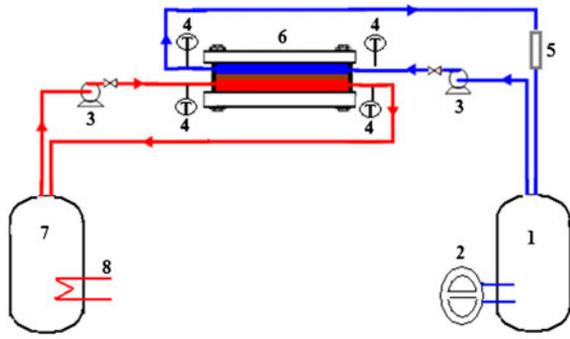


Fig. 2. Schematic diagram of DCMD setup: (1) permeate tank, (2) cooling element, (3) peristaltic pumps, (4) thermocouples, (5) conductometer, (6) flat sheet module, (7) feed tank, and (8) heating element.

and the cold permeate in batch mode operation. The ionic strength of the feed and the permeate solution were measured using a conductivity meter.

2.2. Permeate flux equation

During experimental runs, the permeated liquid was collected in a graduated cylinder and the volume was measured at regular time intervals; the permeate flux ($L/m^2 h$) was calculated by the following equation:

$$J = \frac{\Delta V}{S \Delta t} \quad (1)$$

where ΔV is the volume of permeate (L), S is the effective membrane area (m^2), and Δt is the sampling time (h).

The permeate flux (J) of water vapor diffusing through the dry porous membrane is proportional to the vapor pressure difference across the membrane, and can be expressed by Darcy's law [27]:

$$J = B_m(P_f - P_p) \quad (2)$$

where B_m is the membrane coefficient, P_f and P_p are the vapor pressures at the feed and permeate vapor/liquid interface, respectively. P_f and P_p at the temperatures T_f and T_p , respectively, are related to the activity of the solution by:

$$P_i = a_{wi} P_i^0 \quad i = f, p \quad (3)$$

where a_{wi} is the water activity and P_i^0 is the pure water vapor which can be evaluated by using Antoine equation:

$$P_i^0 = \exp\left(23.238 - \frac{3,841}{T_i - 45}\right) \quad (4)$$

P_i^0 in Pascal and T_i in Kelvin.

The vapor pressure composition can be estimated using the Raoult's law, which can be written in case of dilute solutions:

$$P_i = (1 - x_i) P_i^0 \quad (5)$$

where x_i is the mole fraction of the solute at the membrane interface.

2.3. Reagents and analysis methods

Different concentrations of nitrate, as $NaNO_3$, were prepared by diluting the stock solution with distilled water. The effect of co-existing anions on nitrate removal efficiency was determined using NaF , Na_2SO_4 , and Na_3PO_4 reagents. The effect of feed ionic strength on nitrate removal efficiency and DCMD permeate flux was determined by using $NaCl$ salt at different concentrations. The entire required chemical materials were purchased from MERCK in analytical grade.

Different feed and permeate water solutions were collected at definite intervals of all the experiments, and were analyzed. Electric conductivity was measured using a conductivity/pH meter (Consort C561). Nitrate analysis was performed at an absorbance of 229 nm by a Lambda 25 UV-Vis Spectrophotometer (PerkinElmer, USA). The pH solution was adjusted in the range 6–8 by addition of small quantities of $NaOH$ (0.1 M) or HCl (0.1 M) as needed.

3. Results and discussion

3.1. Effect of feed temperature

MD is a non-isothermal membrane separation process, in which feed temperature (T_f) was considered as primordial operating parameter that affects permeate flux. Fig. 3 shows the effect of feed temperature in the range of 40–80°C on DCMD permeate flux for both membranes, PVDF and PP. The results show that permeate flux increased significantly with increasing feed temperature for two kinds of membranes' polymers. Increasing feed temperature

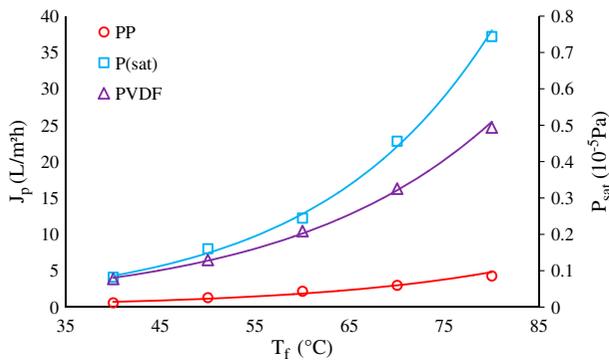


Fig. 3. Effect of feed temperature on permeate flux and feed vapor pressure ($[\text{NO}_3^-]_0 = 200 \text{ mg/L}$, $T_p = 20^\circ\text{C}$, $Q_f = 20 \text{ L/h}$, and $Q_p = 20 \text{ L/h}$).

from 40 to 80°C increases the permeate flux from 0.60 to 4.29 L/m² h (7.15-fold) for PP membrane and from 4.12 to 37.21 L/m² h (9-fold) for PVDF membrane. The exponential increase in DCMD permeate flux with temperature in the two cases can be estimated by Antoine equation (Eq. (4)) [30], which expresses the relationship between the vapor pressure difference, as driving force for DCMD process, and feed temperature.

It can also be seen that the permeate flux for PVDF membrane is higher than PP membrane, which can be explained by the larger pore size and the higher porosity of PVDF membrane. Likewise, the higher permeate flux can be attributed to the rougher surface of PVDF membrane which helps in mixing at the membrane interfaces [27].

From Fig. 3, it was also observed also that the vapor pressure difference and the permeate flux as function of feed temperature at initial nitrate concentration of 200 mg/L are almost on the same order of magnitude, which confirm the relationship between P^{sat} and T_f .

It can be noted that a nitrate rejection rate higher than 99.90% factors was found for all the experiments with PP membrane and PVDF membrane, and was not influenced by the feed temperature changes.

3.2. Effect of feed flow rate

The effects of feed flow rate (Q_f) on DCMD permeate flux using two types of hydrophobic membranes are presented in Fig. 4. The permeate flux was increased when the feed flow rate increased from 5 to 55 L/h for both PP membrane and PVDF membrane. The benefit of working at high flow rate was to increase the Reynolds number that in turn enhances the heat transfer coefficient and thus reduces the effect

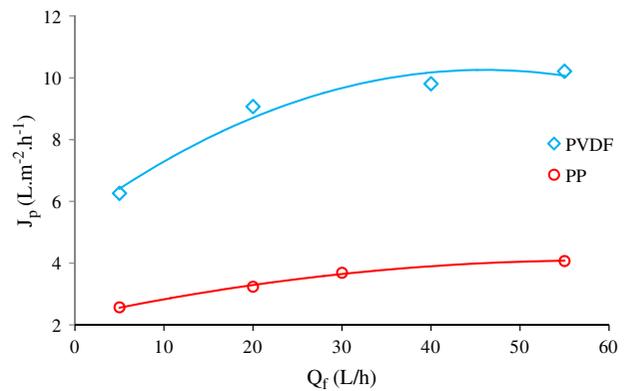


Fig. 4. Effect of feed flow rate on DCMD permeate flux ($[\text{NO}_3^-]_0 = 200 \text{ mg/L}$, $T_f = 50^\circ\text{C}$, $T_p = 20^\circ\text{C}$, and $Q_p = 20 \text{ L/h}$).

of both temperature and concentration polarization phenomenon [27]. This causes a larger driving force for mass transfer through the micro-porous membrane and consequently enhances the DCMD permeate flux. The effect of feed flow rate on permeate flux is less than that of the feed temperature. For higher feed flow rates, the permeation flux tends to reach an asymptotic value. This is due to the reduction in the boundary layer's thickness when the Reynolds number increases, approaching a limiting value [31].

In this part, nitrate rejection factor was around 99.98% throughout all experiments, and there is no relation between this factor and feed flow rate.

3.3. Effect of initial nitrate concentration

The influence of nitrate feed concentration on permeate flux and permeate nitrate concentration for PP and PVDF membranes has been tested in the range of 50–1,000 mg/L. Fig. 5 shows that increasing feed

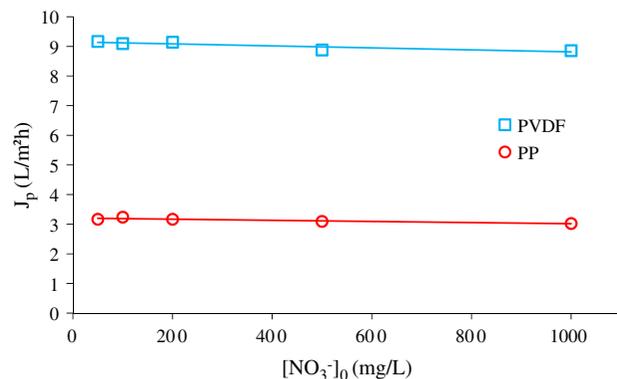


Fig. 5. Effect of initial nitrate concentration on DCMD permeate flux ($T_f = 50^\circ\text{C}$, $T_p = 20^\circ\text{C}$, $Q_f = 20 \text{ L/h}$, and $Q_p = 20 \text{ L/h}$).

nitrate concentration leads to a slight decrease in permeate flux. The effect of feed nitrate on permeate was not as significant as that of feed temperature or feed flow rate in the studied range. The permeate production decreased only by 3.4% for PVDF membrane and by 9.5% for PP membrane, when the initial nitrate concentration was increased from 50 to 1,000 mg/L. The decrease in DCMD permeate is more pronounced when the nitrate concentration is higher, because of the increases in concentration polarization phenomenon. Similar results were found for fluoride removal from contaminated groundwater [25].

Fig. 6 shows the effect of increasing feed nitrate on permeate conductivity, using PP and PVDF membranes. It can be found that the permeate nitrate concentration increased slightly when the initial nitrate concentration increased from 50 to 1,000 mg/L for two studied membranes. For PP membrane, the permeate nitrate concentration was kept under 0.2 mg/L, which means almost 99.96% of nitrate rejection, and for PVDF membrane, the permeate concentration was kept under 0.4 mg/L, which means 99.98% of nitrate rejection. For all experimental runs, the nitrate concentrations were found largely below the admissible value (50 mg/L) recommended by the WHO. These results can be attributed to a partial wetting phenomenon which occurred on membrane surface at high feed concentration. Furthermore, increasing feed concentration could increase the boundary layer thickness which causes the membrane wettability on the feed side.

3.4. Effect of ionic strength

The effect of ionic strength, adjusted by adding sodium chloride salt, on permeate flux at constant

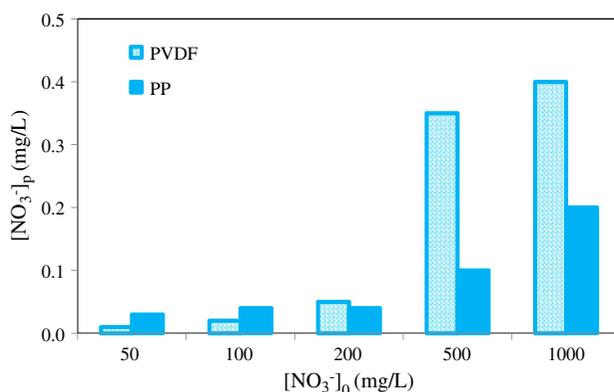


Fig. 6. Effect of initial nitrate concentration on DCMD permeate conductivity ($T_f = 50^\circ\text{C}$, $T_p = 20^\circ\text{C}$, $Q_f = 20\text{ L/h}$, and $Q_p = 20\text{ L/h}$).

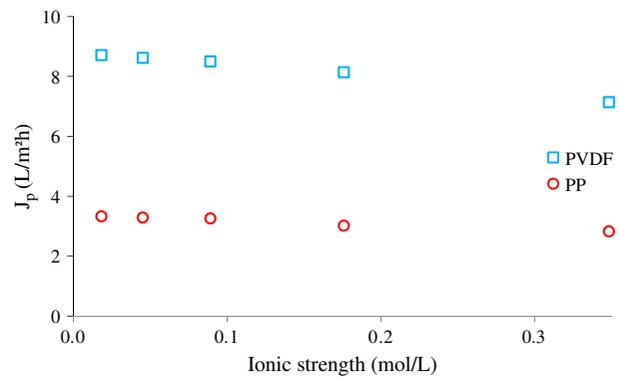


Fig. 7. Effect of initial ionic strength on DCMD permeate flux ($[\text{NO}_3^-]_0 = 200\text{ mg/L}$, $T_f = 50^\circ\text{C}$, $T_p = 20^\circ\text{C}$, $Q_f = 20\text{ L/h}$, and $Q_p = 20\text{ L/h}$).

nitrate feed concentration is illustrated in Fig. 7. The results reported in this figure indicate that the DCMD flux decreases slightly with increasing feed ionic strength from 1.83×10^{-2} to $34.34 \times 10^{-2}\text{ M}$ for both hydrophobic membranes. The permeate fluxes were decreased from about 15.8 and 10.7%, respectively, for PP and PVDF membranes. The reduction in permeate flux can be attributed to the fact that the addition of salt reduces the partial vapor pressure of feed water according to Raoult's law, (Eq. (4)), and as a consequence reduces the driving force.

Increasing ionic strength, using NaCl salt, leads to an increase in the boiling point of the solution. This indicates that less water vaporization can occur at the membrane surface, which diminishes the amount of vapor transported through the membrane pores [32].

Also, while the ionic strength of the feed solution increases, the dynamic fluid will change as a result of increasing viscosity and then the concentration polarization should be added to the temperature polarization, which reduces the imposed DCMD driving force and then the permeate flux [33].

For all experiments performed in this section, DCMD provides nitrate rejection rate more than 99.98%. DCMD technique can produce a high quality of free nitrate potable water from different sources of nitrate-contaminated water.

3.5. Effect of competing anions

Nitrate removal by DCMD, using two kinds of hydrophobic membranes, was investigated in the presence of other anions such as F^- , SO_4^{2-} , and PO_4^{3-} . The anionic solutions' composition used in these experiments contained 200 mg/L of NO_3^- and 200 mg/L of other co-anions. The results of this study are listed in

Table 2
Effect of competing anions on DCMD performance

| Solution | <i>I</i> (mM) | PP membrane | | PVDF membrane | |
|------------------------------------|---------------|----------------------------|----------------------------|----------------------------|----------------------------|
| | | $[\text{NO}_3^-]_p$ (mg/L) | J_p (L/m ² h) | $[\text{NO}_3^-]_p$ (mg/L) | J_p (L/m ² h) |
| $\text{NO}_3^- + \text{NO}_3^-$ | 2.4 | 0.04 | 1.43 | 0.05 | 9.05 |
| $\text{NO}_3^- + \text{F}^-$ | 3.6 | 0.09 | 1.32 | 0.2 | 8.98 |
| $\text{NO}_3^- + \text{SO}_4^{2-}$ | 3.3 | 0.02 | 1.24 | 0.02 | 9.02 |
| $\text{NO}_3^- + \text{PO}_4^{3-}$ | 4.6 | 0.12 | 1.27 | 0.18 | 8.93 |

Table 2. The permeate fluxes as functions of feed composition are almost stable around 1.3 and 9 L/m² h for PP and PVDF membranes, respectively. For two kinds of hydrophobic membranes, the variation of competing anions in the range of their studied concentration has no effect on DCMD permeate flux. With regard to the nitrate removal, it was found to be higher than 99.90 for both the membranes and for all the experimental runs. Likewise, the variation in competing anions has no noticeable effect on nitrate rejection factor. The independent effect of competing anions on DCMD performance can be attributed to the fact that the separation mechanism in MD is based principally on the difference in volatility [34]. The studied solutions containing a variety of non volatile solutes, independently of the type of solute, only water vapor flows through the hydrophobic membranes and theoretical retention of the non volatile components approaches to 100%.

4. Conclusion

Nitrate, which is the most widespread groundwater contaminant in the world, must be reduced to keep it below the maximum permissible level (50 mg/L). This study showed that DCMD is able to eliminate nitrate from aqueous solution using two types of hydrophobic membranes: PP and PVDF. The effect of relevant operating parameters, including feed temperature, feed flow rate, initial nitrate concentration, feed ionic strength, and the presence of competing anions, was studied. PP and PVDF membranes achieved a very high nitrate rejection above 99.90% for all the experimental runs. The permeate flux was increased exponentially with increasing feed temperature, and under the same operating conditions, PVDF membrane showed the higher permeate flux of 37.21 L/m²h than the PP membrane with a permeate flux of 4.29 L/m²h. The permeate flux was also affected by feed flow rate. Hence, increases in feed flow rate are accompanied by an increase in permeate flux. With the increase in initial nitrate concentration and feed ionic strength, the

permeate flux showed a slight decrease. The presence of competing anions, such as F^- , SO_4^{2-} , or PO_4^{3-} , had no significant effect on permeate flux and nitrate rejection. These experimental results confirm that DCMD could be operated most efficiently for nitrate removal from synthetic water, and should be examined for natural contaminated water.

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