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Multi-walled carbon nanotube doped PVA membrane for desalination

B. Yilman, F. U. Nigiz, A. Aytaç and N. D. Hilmioglu

ABSTRACT

In this study, multi-walled carbon nanotubes (MWCNT) loaded polyvinyl alcohol (PVA) membranes were synthesized and applied for desalination by means of pervaporation technique. Membranes were characterized using scanning electron microscope (SEM). Sorption studies were carried out to determine the degree of swelling of the MWCNT/PVA membranes into different types of salt solution. Pervaporation experiments were carried using the pristine and MWCNT loaded membranes. The effect of MWCNT concentration and salt concentration on the swelling, flux and salt rejection were investigated at the constant temperature of 40 °C. As a result, greater than 92% of rejections were obtained using all membrane. The highest flux was obtained using 0.3 wt% MWCNT loaded membrane with the highest rejection of 98.8%. SEM analysis confirmed that the excess amount of particles caused agglomeration and negatively affected the desalination performance.

Key words | desalination, MWCNT, pervaporation, PVA membrane

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ABBREVIATIONS

CNT	carbon nanotube
GO	graphene oxide
MD	membrane distillation
MWCNT	multi-walled carbon nanotube
PVA	polyvinyl alcohol
RO	reverse osmosis
SEM	scanning electron microscope

SYMBOLS

S_d	degree of swelling
W_s	weight of swollen membrane
W_d	weight of dry membrane
J	flux
R	salt rejection
W	weight of permeate
A	effective membrane area
t	experiment period
C_f	feed concentration
C_p	permeate concentration

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INTRODUCTION

Global water demand is constantly increasing due to population growth and economic development. Therefore, various methods have been developed to remove the saltiness of seawater in order to obtain fresh water. It has been observed that the number and size of desalination projects worldwide have increased by 5–6% per year since 2010 (Voutchkov 2018). Desalination technologies are divided into two main categories as thermal technologies and membrane technologies. Thermal processes are based on the evaporation and condensation of the seawater. Although the installation of thermal processes, pre-treatment, and

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operating costs are low, it has some disadvantages such as high energy requirement and needs to increase heat transfer efficiency (Daer *et al.* 2015; Liu *et al.* 2018). Membrane technologies are currently considered the most effective techniques for the desalination process depending on having an energy saving process route, efficient desalination performance and high operational stability compared to thermal methods. Currently, reverse osmosis (RO) is known as a widespread membrane-based process in Europe and all around the world. In the RO process, fresh water is separated from the saline water by applying higher feed pressure to defeat the osmotic pressure of the water. The main limitations of desalination with RO technology are brine discharge, low performance for higher salinity feed, high-pressure requirement, structural failure of membranes to contamination and require pre- and post-treatment techniques following the one-stage reverse osmosis process (Wang *et al.* 2016b; Chaudhri *et al.* 2018). Membrane distillation (MD) is a promising but not commercial technique which can be used to purify high salinity waters. However, this technique is limited by the pore wetting and biological fouling tendency of the hydrophobic membranes (Li *et al.* 2017).

Most recently, the new pervaporative desalination technology has been used to obtain fresh water from the saline water. Pervaporation is an emerging-promising membrane separation process which has long been used for alcohol dehydration, desulfurization, fuel dehydration, aroma recovery, and volatile organic compound removal from the water. In the pervaporation method, one of the components is removed from the medium according to the membrane-solvent interaction, affinity, hydrogen bonding capacity and other factors related to the membrane material. In this system, the liquid mixture passes through as a vapor phase on the downstream and condenses. The driving force is the chemical potential gradient between the upstream and downstream sides of the selective membrane (Wang *et al.* 2016a). Contrary to the distillation method which requires seriously high heat energy, pervaporation is carried out at the mild operation conditions and the only energy requirement is consumed by vacuum requirement. Therefore, the energy consumption is less than compared to the thermal-based separation method (Gao 2016; Cheng *et al.* 2017). There are crucial parameters that affect the performance of the pervaporation. Particularly, the temperature is the most effective factor to increase the permeate flux. However, in the literature, it is mostly reported that the increasing temperature causes a slight decrease in selectivity and separation factor (Sae-Khow & Mitra 2010). Trans-membrane pressure, membrane thickness, the composition of the mixture, the structure of the membrane material, filler type and concentration also directly affect both the permeate flux and selectivity.

Recently, pervaporation has been used for desalination of saline water. The type of membrane used for the pervaporation is hydrophilic membranes which have advantages as they provide a superior antifouling property by preventing the adsorption of hydrophobic organic pollutants from the feed solution (Chaudhri *et al.* 2018). The productivity of the pervaporative desalination method is characterized by water flux and the efficiency of the system is directly related to the rejection capacity of the membrane. The concentration of the constituent in the saline water, the temperature of the operation, the quantitative amount of the pressure difference between the sides of the membrane, condensation efficiency are main factors to determine desalination performance. Beside the operating conditions, structural properties of the membrane play an important role in an effective purification. Recently, pervaporative desalination studies are gaining importance depending on the higher rejection capacity of dissolved salt ions. Several years, lots of promising results have been reported related to pervaporative desalination. Singh *et al.* (2015) synthesised a cetyltrimethylammonium bromide (CTAB)–silica membrane and desalinated a synthetic seawater including 40.000 ppm NaCl at 25 °C. They obtained 99.9% salt rejection with the changing flux in the range between 1–2.6 kg/m^2 h. Chaudhri *et al.* (2018) prepared a poly(vinyl alcohol)–silica (PVA-SiO_2) hybrid material on porous polysulfone hollow fibers and they obtained 99.9% of salt rejection and 20.6 kg/m^2 h water flux for the desalination of synthetic seawater containing 2,000 ppm salt at 333 K. Li *et al.* (2017) reported that they prepared a graphene oxide (GO) containing PVA/PVDF composite membrane and carried out a pervaporative desalination study. They found that the 0.1 wt% GO containing membrane gave the better desalination results with the flux of 28 L/m^2 h and greater than 99.9% salt rejection. Liang *et al.* (2015) also

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used GO as a functional filler and incorporated into PAN matrix to fabricate a composite pervaporation membrane. They reported greater than 99.99% rejection results and flux values of 14.3 L/m^2 h and 65.1 L/m^2 h at the operating temperature of 30 °C and 90 °C, respectively.

For an effective water desalination, inorganic particle incorporated polymeric membrane; in other words, mixed matrix membranes (MMM) are preferred. Mixed matrix membranes not only exhibit higher desalination performance but also contribute operating stability under the harsh conditions. Most recently, carbon-based materials such as carbon nanotube (CNT), graphene nanoplate, GO materials exhibit superior desalination performance when they are used as filler (Cohen-Tanugi & Grossman 2012; He *et al.* 2015; Homaeigohar & Elbahri 2017). Carbon derivatives contribute to increasing salt rejection and microbial fouling resistance. In the literature, incorporation of carbon-based materials – even the concentration are low – is improved the stability durability of the membrane for desalination purposes. Besides them, multi-walled CNT provides superior separation performance depending on its nano-sized separation hole that allows selective water passage (Wan Azelee *et al.* 2017).

In this study, desalination experiment was performed with multi-walled carbon nanotubes (MWCNTs) loaded polyvinyl alcohol (PVA) membrane. Since MWCNT has been discovered, it has attracted interest for a lot of applications due to its durability, electrical conductivity, high adsorption properties (Aqel *et al.* 2012). Carbon nanotubes have been used in areas such as sensors, adsorbents, membranes, catalysts (Sarkar *et al.* 2018). In the literature, the CNT is also studied for the dehydration of isopropyl alcohol (Shirazi *et al.* 2011; Amirilargani *et al.* 2013), 1,4-dioxane (Ong *et al.* 2011), ethylene glycol (Hu *et al.* 2012). It has been reported that the functional and non-functional types of CNT improved the pervaporation performance by increasing membrane productivity and selectivity.

It has also been investigated in applications of desalination and it has been seen that it has advantages such as increasing water flow, maximizing salt separation and preventing membrane contamination and prolonging life (Rashid & Ralph 2017). Zhao *et al.* (2014) prepared a MWCNT doped aromatic polyamide membrane and used in the reverse osmosis. They reported that they achieved 76% salt rejection accompanied with 0.71 L/m^2 h and they improved the performance of the pristine membrane by adding of CNT (Zhao *et al.* 2014). Another study performed by Bhadra *et al.* (2013), COOH functionalized nanotube was incorporated into PVDF membrane and used for MD purposes. The authors reported a 99% of salt rejection and 19.2 kg/m^2 h flux value. Although CNT is not used in pervaporative desalination yet, it is used in systems such as reverse osmosis, advanced osmosis, MD, and ultrafiltration, and successful results have been obtained (Daer *et al.* 2015). MWCNT has mostly been used in a hydrophobic membrane and never been used for pervaporative desalination. Therefore, in this study, MWCNT was first time used to prepare a hydrophilic poly(vinyl alcohol) (PVA) based membranes and purify the saline water by means of pervaporation. PVA polymer was selected due to the good fill forming capability, higher hydrophilicity caused by the hydroxyl and carboxyl group in its structure (Shirazi *et al.* 2011). It is important to use a hydrophilic membrane for deed seawater desalination to prevent the surface fouling across the biological contaminating. The usage of PVA as a membrane minimizes the biological-microbial degradation resulted by the humic and fulvic acid (Bolto *et al.* 2009).

In the present study, pervaporative desalination experiments were performed with MWCNT loaded PVA hybrid membrane. Model seawater solutions including constant amount of KCl (200 ppm), $MgCl_2$ (200 ppm) NaCl (2,000 ppm) have been prepared and desalination experiments were carried out at the constant temperature of 40 °C. Prior to desalination experiment, the degrees of swelling measurements were done for each membrane to determine the behavior of the pristine and MWCNT loaded membrane in saline water medium. The effects of MWCNT concentration (in a ranging from 0.1% to 0.5% with respect to the weight of dry polymer), salt concentration on the degree of swelling, water flux, and salt rejection values were evaluated.

MATERIALS AND METHODS

Materials

MWCNTs were supplied from Grafen Co. with diameters ranging from 10 to 30 nm and lengths ranging from 10 to

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30 μm. Polyvinyl alcohol (MW = 125,000 g/mol) was supplied from Aldrich Chemistry, Turkey. Hydrochloric acid was supplied from J. T. Baker Inc. 2-propanol and magnesium chloride hexahydrate (MW = 203,3 g/mol) were supplied from Merck Chemicals, Turkey. Sodium chloride was supplied from Carlo Erba Reagents. Potassium chloride (MW = 74,55 g/mol) was supplied from Sigma Aldrich, Turkey. Glutaraldehyde 50% aq. sol. was supplied from Alfa Aesar, Turkey.

Preparation of hybrid membranes containing MWCNTs

MWCNT doped PVA mixed matrix membrane was prepared using the solution-casting technique. 3 wt.% of PVA-water solution was prepared and stirred for 3 h at 80 °C. In order to distribute MWCNT particles into the PVA matrix, particles were firstly distributed separately in small amounts of water. MWCNT-water solution was mixed in an ultrasonic bath for 1 h. Then, the polymer mixture was slowly added to the MWCNT-water dispersion. Membrane solution was cast onto a poly(methylmethacrylate) surface and allowed to evaporate under the room conditions overnight. After a membrane had formed, it was peeled off from the surface and cross-linked in a cross-linking solution including glutaraldehyde, acetone, water, and hydrochloric acid. Following the cross-linking, membranes were taken from the bath, washed rapidly and dried prior to use in the desalination experiment. Because the membrane thicknesses were different from each other, the flux values were normalized to 100 μm.

Degree of swelling experiment

In order to investigate the swelling properties of the membranes in the different concentration of saline solutions, membranes were cut into pieces with an area of 2 cm^2. Membranes were immersed in 2,000 ppm NaCl-water, 200 ppm KCl-water, 200 ppm $MgCl_2$-water solutions. At a 1 h interval, membranes were rapidly taken from the solution and the weighs were recorded. Before weighing, the free water particles on the membrane surface was removed by a filter paper and the exact amount of water adsorbed by the membrane was determined as a function of time. Swelling analysis was carried out for 24 h. The degree of swelling (Sd) values of the membranes were calculated from in following Equation (1)

$$\mathrm{Sd} = \left[\frac{\mathrm{Ws} - \mathrm{Wd}}{\mathrm{Wd}}\right] \times 100 \quad (1)$$

where W_s is the weight of swollen membrane, and W_d is the weight of the dry membrane.

Pervaporation experiment

Synthetic saline water desalination experiments have been performed using constant concentration of KCl (200 ppm), $MgCl_2$ (200 ppm) NaCl (2,000 ppm). Experiments have been conducted in a laboratory-scale pervaporation unit that has an effective membrane area of 19.6 cm^2 and a volume of 250 mL. Pervaporative test unit is covered with a temperature controlled oven to provide the desired temperature. Experiments have been performed over 5 h. An hourly interval, permeate sample is taken from the downstream and the conductance of the sample is analyzed using a conductivity meter. The experiments were carried out once, and the analyzing of the water was repeated several times.

Water flux (J) is calculated from the weight of the permeate sample (W) that is permeated through the effective membrane area (A) per hour (t) as follows:

$$J = W/A.t \quad (2)$$

Ion rejection (R) is calculated from the feed concentration (C_f) and permeates concentration (C_p) of water as follows:

$$R(\%) = ((Cf - Cp)/Cf) * 100 \quad (3)$$

RESULT AND DISCUSSION

Membrane characterization

Scanning electron microscopy micrographs of the MWCNT loaded PVA membrane is seen in Figure 1. Where the light particles represented the MWCNT particles, dark and continuous phase shows the PVA matrix. In Figure 1(a) and

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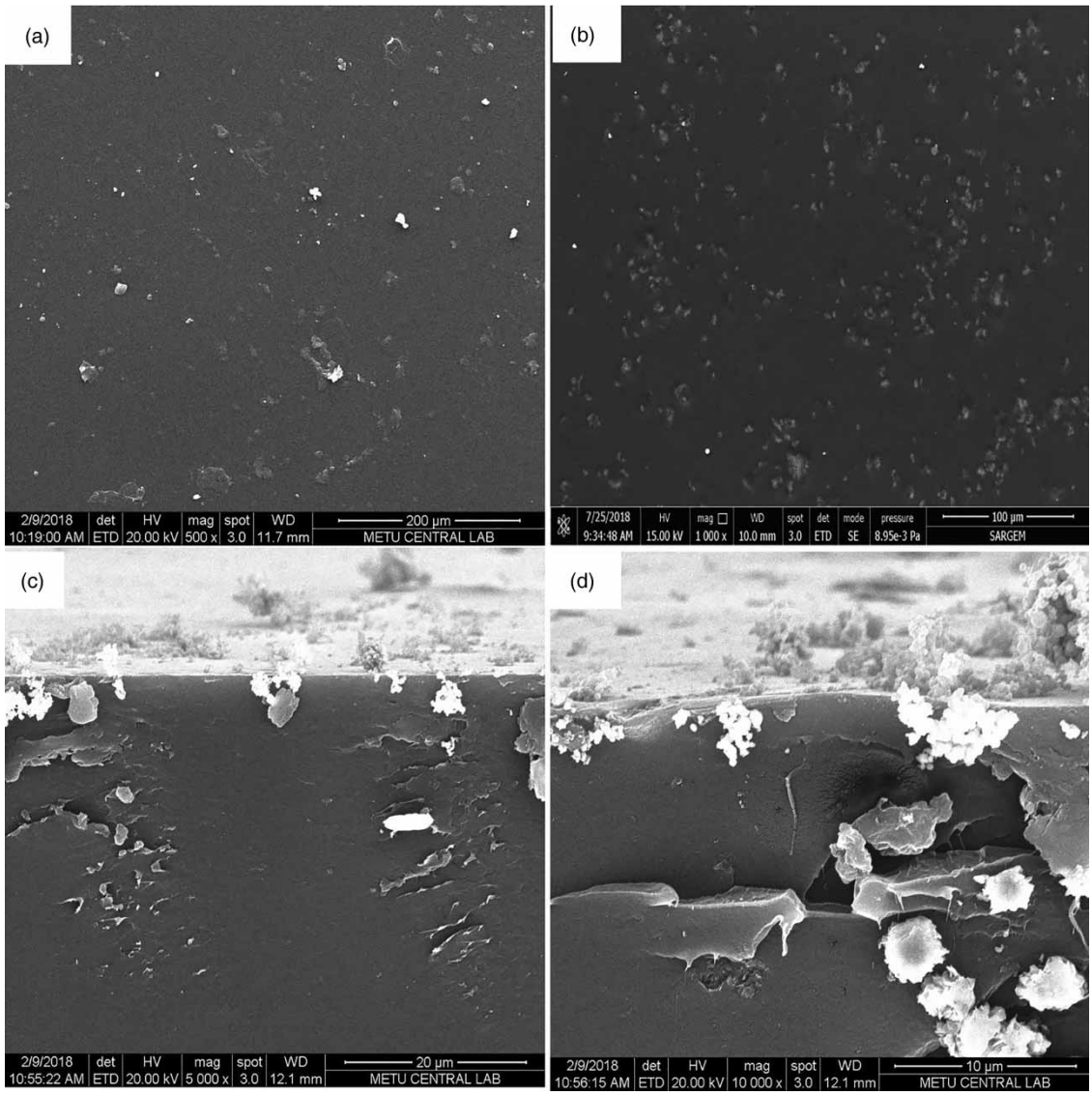


Figure 1 | Surface SEM micrographs of the 0.1 wt% MWCNT loaded (a) and 0.5 wt% MWCNT loaded (b) PVA membrane, cross-sectional micrographs of 0.1 wt% MWCNT loaded (c) and 0.5 wt% MWCNT loaded (d) PVA membrane.

1(b), the dense, homogeneous and non-porous structure of the 0.1 wt% MWCNT-PVA and 0.5 wt% MWCNT-PVA membranes were clearly seen. There was not any contact-free region between the MWCNT and PVA. The particles were homogeneously distributed on the surface of the PVA membrane. In Figure 1(c) and 1(d), cross-sectional micrographs of 0.1 wt% MWCNT and 0.5 wt% MWCNT incorporation PVA membranes were seen. The excess amount of MWCNT and agglomerations were seen both in the surface and structure of the membrane.

Degree of swelling results

Time dependent degrees of swelling values of the membranes at different concentration of CNT in different salt solutions are given in Figure 2.

From the graphics, it is seen that 0.3 wt% MWCNT loaded membrane showed a high degree of swelling values in all solutions. In the first plot, the highest degree of swelling values of 350% was obtained when the 0.3 wt% CNT loaded membrane was immersed in 200 ppm KCl-water

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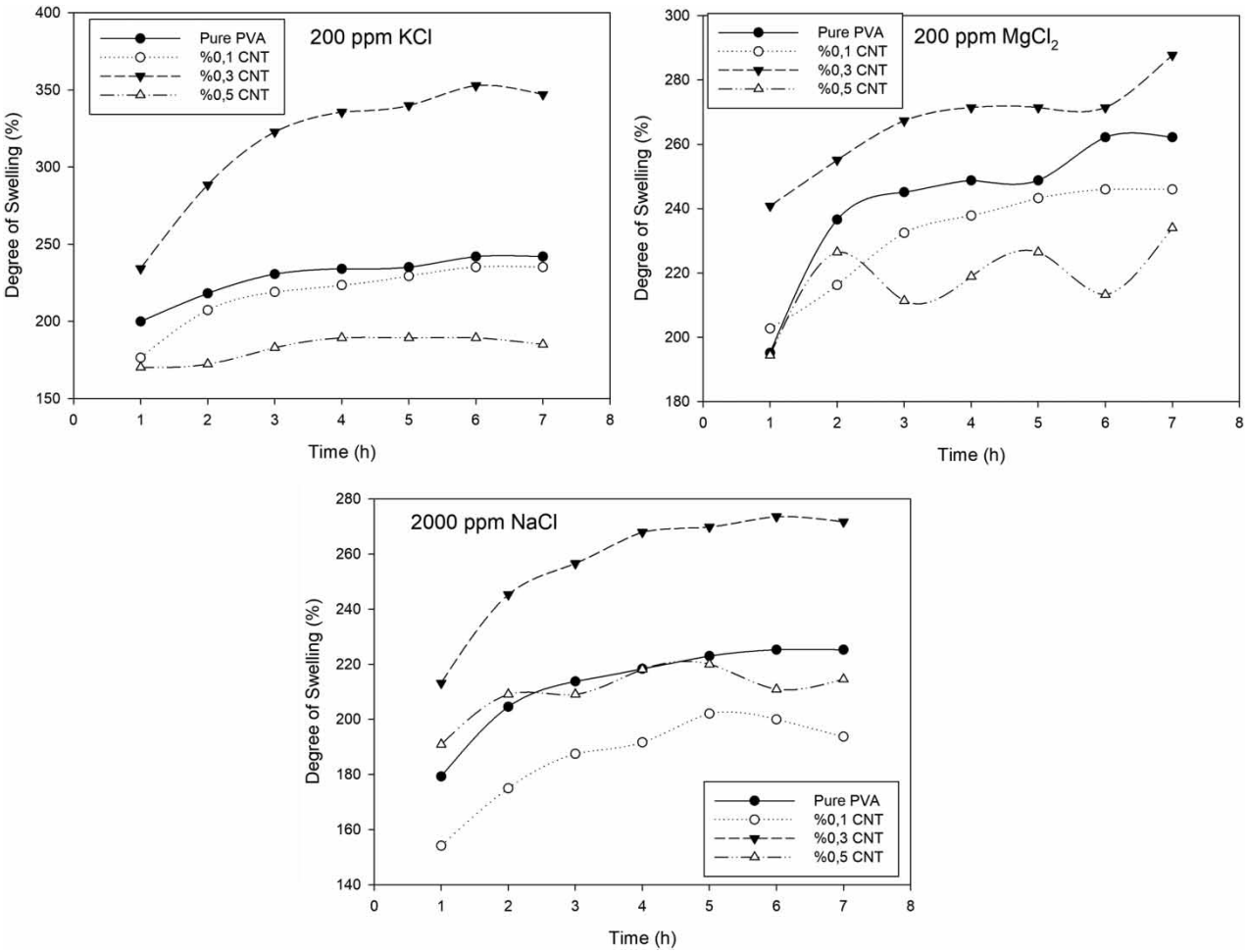


Figure 2 | Swelling degrees of the membrane immersed in different types of salt.

solution. In the second plot, the highest degree of swelling 288% in 200 ppm $MgCl_2$-water solution and in the third plot 275% was obtained in 2,000 ppm NaCl-water solution. The membrane with 0.5 wt% CNT loaded membrane showed a very low degree of swelling compared to the others. Pure membrane and 0.1 wt% CNT loaded membrane showed similar values. Incorporation of the MWCNT into the polymeric matrix causes an increase in the amount of the total amorphous region within the polymers and a decrease in the amount of crystalline structure. The amorphous region within the membrane is responsible for the swelling. Therefore, the increase in swelling should be attributed to the decrease in crystallinity which was confirmed by the DSC finding in our previous study (Yılman *et al.* 2018). The similar increasing tendency has also been reported by Panahian *et al.* (2015). However, the swelling degree decreased as the MWCNT concentration increased from 0.3 wt.% to 0.5 wt.%. It is known that the carbon-based material shows agglomeration tendency. The excess amount of MWCNT caused agglomeration and resulted in the formation of continuous polymer and continuous carbon phases. This situation was also seen in scanning electron microscope (SEM) analysis (SEM analysis of the surface of the membrane with 0.1 wt.% and 0.5 wt.% MWCNT incorporation). While the continuous polymeric phase showed the swelling tendency is the same as the pristine PVA membrane, the continuous MWCNT phase showed a resist to swell due to the hydrophobic character of MWCNT. Therefore, because the agglomerated region did not swell, the total degree of swelling decreased when

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the excess amount of MWCNT was incorporated into the PVA matrix (Qiu *et al.* 2010).

Desalination results

Figure 3 indicates the flux and rejection results of the synthetic saline water prepared by varying concentration of NaCl (1,000, 1,500, and 2,000 ppm) and constant concentration of $MgCl_2$ (200 ppm) and KCl (200 ppm). Desalination experiments were carried out at the constant temperature of 40 °C. As seen in the figure, greater than 92% of rejection results were obtained with all membrane types. As the increasing salt concentration in the synthetic saline water, rejection results enhanced. As seen in the figure, the flux values are generally increasing as the ratio of MWCNT increases. Due to the fact that the water molecules can pass through the hole of MWCNT with less resistance compared to the PVA matrix. Additionally, increasing amount of amorphous region can contribute to enhancing flux (Choi *et al.* 2009; Qiu *et al.* 2010). Another observation from Figure 3 is that the flux difference between the membrane with MWCNT and without MWCNT increased when the NaCl concentration increased from 1,000 ppm to 2,000 ppm. This should be attributed to the structural change in free volume of the PVA. The PVA polymer exhibits a more rigid behavior in the presence of salt. Therefore, because the membrane has a higher crystallinity rate in its pure state, it has lower flux values than the composite membrane at high NaCl concentration. The decrease in flux values as NaCl increases in the feed solution has been reported in many studies (Liang *et al.* 2014; Chaudhri

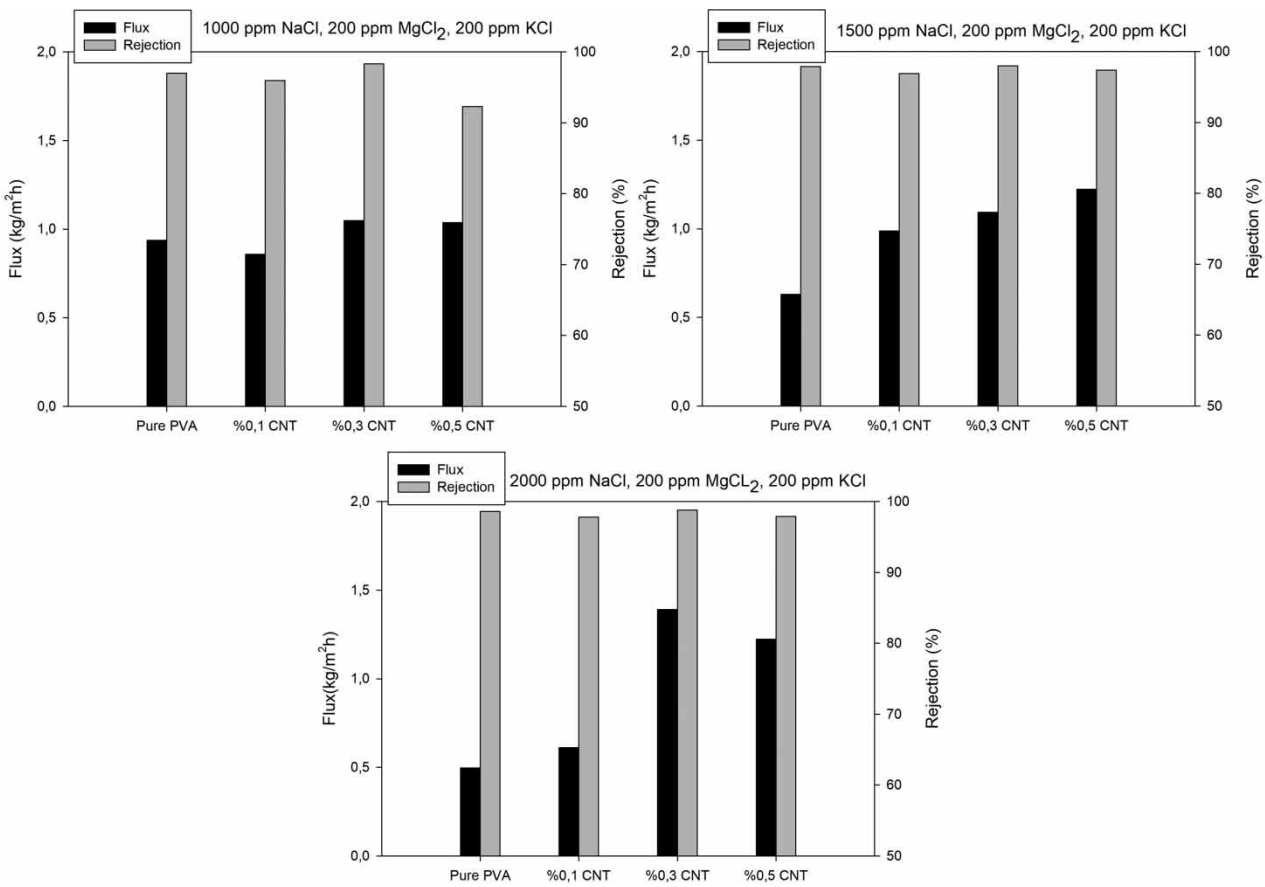


Figure 3 | Flux and rejection results of the membrane at varying concentrations of NaCl.

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et al. 2018). As seen in Figure 3, this decrease is prevented by adding MWCNT.

In Figure 3, an increment–decrement depending on MWCNT addition is also seen when the NaCl concentration is 2,000 ppm. In the literature, a similar increment–decrement relationship was also obtained by Panahian et al. (2015). In the study performed by Panahian and co-workers, CNT incorporated membrane was also prepared and used for dehydration of an alcohol. They prepared both the pristine and MWCNT loaded membrane. CNT particles were used with and without functionalization. The results were compared and reported that the rejection drastically decreased after the optimal loading of the non-functionalized MWCNT filler. The reason for this situation was explained by the agglomeration tendency of non-functionalized MWCNT particles. They reported that the free volume was created around the particles and a convective permeation might occur throughout these regions. In the present study, the lowest rejection results were obtained by the 0.5 wt% MWCNT incorporated membrane resulted from the agglomeration of MWCNT particles which was also observed in SEM results. In order to prevent the trade-off, strong acid can be used to functionalize the MWCNT to fix the compatibility of the filler with the membrane.

CONCLUSIONS

In the present study, MWCNT-PVA membranes were synthesized with varying concentration of MWCNT and applied for desalination of synthetic seawater. Sorption studies were carried out to determine the degree of swelling into different types and varying concentration of the salt solution. Effect of MWCNT concentration and salt concentration on flux and salt rejection were investigated at the constant temperature of 40 °C. The main findings obtained in the study are as follows:

- 0.3 wt% MWCNT loaded membrane showed the highest of swelling.
- Greater than 92% of rejections were obtained using all membrane.
- The highest rejection of 98.3% was obtained using 0.3 wt % MWCNT loaded membrane.
- The highest flux was obtained using 0.3 wt% MWCNT loaded membrane.
- SEM analysis confirmed that the excess amount of particles caused agglomeration and negatively affected the desalination performance.

Consequently, swelling results showed that there was not any relationship between the swelling and pervaporation study. The excess amount of MWCNT exhibited agglomeration tendency and caused a sharp decrease in rejection. It could be better to use functionalized nanotubes to obtain higher desalination performance.

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