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High performance pervaporative desalination of saline waters using Na-X zeolite membrane

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Abstract. A high quality Na-X zeolite membrane was synthesized on a seeded α-alumina disc by the secondary growth method. Structural characterization was done by X-ray spectroscopy, FT-IR spectroscopy, SEM and AFM imaging. The performance evaluation of the membrane was firstly tested in separation of glucose/water solutions by pervaporation process. There was obtained a separation factor 182.7±8.8, while the flux through the membrane was 3.6±0.3 kg m^{-2} h^{-1}. The zeolite membrane was then used for desalination of aqueous solutions consisting of Na^+, Ca^{2+}, Cs^+ and Sr^{2+} because of the importance of these ions in water and wastewater treatments. The effects of some parameters such as temperature and solution concentration on the desalination process were studied for investigating of diffusion/adsorption mechanism in membrane separation. Finally, high water fluxes ranged from 2 up to 9 kg m^{-2} h^{-1} were obtained and the rejection factors were resulted more than 95% for Na^+ and Ca^{2+} and near to 99% for Cs^+ and Sr^{2+}. Based on the results, fluxes were significantly improved due to convenient passage of water molecules from large pores of NaX, while the fouling was declining dramatically. Based on the results, NaX zeolite can efficiently use for the removal of different cations from wastewaters.

Keywords: desalination; NaX zeolite; membrane; pervaporation; water and wastwaters

1. Introduction

In recent years, the shortage of drinking water in many areas of the world is now a serious problem. Therefore, considerable attentions have been devoted to desalination of the natural sources of water. These considerations have become more important for increasing water consumption in industrial processes. Recycling and reuse of water have been required for these industries (Swenson *et al*. 2012). Removal of ions have been done by different methods such as Nanofiltration (NO), Reverse Osmosis (RO) and Multi-Stage Flash Distillation (MSFD) that are currently considered of famous of paths to desalination and water treatment. However, MSFD was classified as an energy consumption method, which can be affected the environment with increasing temperature (Cho *et al*. 2011, Wang *et al*. 2011, Eltawil *et al*. 2008, Borja *et al*. 2011). RO by the polymeric membrane has been used as a commercial technology, although, polymeric materials have low resistance against temperature and high concentration of organic solutions. (Swenson *et al*. 2012, Lee *et al*. 2011, Polasek *et al*. 2003). In recent years, compared with

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pressure driven processes such as RO, non-pressure driven membrane process such as pervaporation (PV) have become very efficient in removal of the dissolved solids and salts from the source water and being more resistant to certain types of fouling (Wang *et al*. 2016).

In comparison with the polymeric compositions, zeolites have greater chemical and thermal stability. These materials have alumina silicate structures, which have smoothly pore size and cavity. Specific advantages of the zeolites are shape selectivity and molecular sieving features which cause the applications of them as adsorbents and catalysts (Chen *et al*. 1979, Li *et al*. 2008). These inorganic materials are worthy of being studied in pervaporation (PV) processes because of their unique properties. In PV process, vacuum is applied as a driving force for permeation of substances that are volatile. Convenient operation of the PV method together the suit features of zeolite has been provided uses them in dehydration studies (Liu *et al*. 1996, Kittur *et al*. 2005). Pervaporation process can provide moderate conditions in highly concentrated solutions in membrane processes.

Based on information resulting in molecular dynamic (MD) simulation, the possibility of ion rejection up to 100% was confirmed for ZK-4 and NaA zeolite membranes perfectly in RO desalination (Cho *et al*. 2011, Li *et al*. 2004, Li and Wang 2010). Hydroxy sodalite was studied in sea water desalination by khajavi *et al*. and full filled salt rejections higher than 99.99% in pervaporation systems (Khajavi *et al*. 2010). Malekpour *et al*. reported the waste treatment by the NaA type zeolite membrane and they obtained the ion rejection more than 99% and water flux 1.2 $kg/m^2.h$ (Malekpour *et al*. 2008). The MFI zeolite membrane was investigated for removal of Na+ ions in the presence of different ions, where rejection decreased from 76.7 to 58.1 because of the presence of high-valance counter ions (Li *et al*. 2008, Li *et al*. 2004). The explained studies show the importance of the membrane processes in desalination and waste treatment. But the trouble blocking in membrane processes can affect the amounts of the pure substances in the separation process. In recent decades, most studies have focused on resolving this problem.

The NaX zeolite as a subset of the FAU zeolite group has large pore size (about 7.4 Å) and can be a good candidate for desalination processes with minimal fouling problems. Removal of the chromium ions from tannery wastes was studied by Covarrubias *et al* which proved the nice efficiency of FAU zeolite (Covarrubias *et al*. 2008). The application of FAU-type zeolite was also investigated in desalination of aqueous solutions through a membrane process by Liu *et al*. Molecular dynamic simulations showed this zeolitic membrane can provide ion rejection near to 100%. (Liu and Chen 2013).

This research explains the desalination of simulated saline water and nuclear waste solutions by applying Na-X zeolite membrane which synthesized by the secondary growth method in hydrothermal situation. Our final goal was the achievement of a high flux and suit rejection factor together. Before desalination experiments, separation of water from glucose was done to the qualification of the membrane.

2. Experimental

2.1 Chemical and materials

All chemicals were purchased from Merck and used as provided. SiO_2 (100%), $AlCl_3.6H_2O$ (98%) and NaOH (>98%) were used for membrane preparation. Glucose (99%), glacial acetic acid (100%) and ortho-toluidine (97.5-98%) were consumed in performance evaluation. NaCl (99%), CsCl (>99%), $Ca(NO_3)_2.4H_2O$ (>99%) and $Sr(NO_3)_2$ (99-100%) were applied to provide single and mixture ionic solutions. Double distilled water was used to make solutions which supplied by water still (HELAGO Company from model GFL -2102) through the process. Alumina disc support was supplied by Materials and Energy Research Center of Iran.

2.2 Membrane preparation

High quality NaX zeolite membrane was synthesized on a α-Al_2O_3 support with the aid of nucleation seeds from a gel synthesis mixture. A homogeneous gel mixture was prepared with a batch composition including, $NaAlO_2$:$4SiO_2$:16NaOH:$325H_2O$ at three stages (Robson 2001).

Firstly, Al-source solution was made by dissolving of $AlCl_3.6H_2O$ in NaOH solution. Sodium silicate was prepared by dissolving SiO_2 in NaOH solution. After that Na_2SiO_3 was added into the NaOH solution to achieve Si-source as another main material in the synthesis process. Finally, created solutions were combined together simultaneously and was allowed to stir by magnetic stirrer for 30 min. A porous α-alumina disc with prosity~35% ,1.3mm thickness and diameter 19 mm and with pore size 150 nm were seeded by dip-coating method (Yuan *et al*. 2011) and were put in a polytetrafluoroethylene (PTFE) disc holder. After that, the holder was put perpendicularly in polyethylene (PE) container consisting of gel synthesis. The container was fastened in a stainless steel autoclave and was placed in an oven at a temperature of 90℃ for 8 h. The autoclave was permitted to cool gradually at room temperature, alumina support was rinsed well with deionized water and dried at 100℃ for 3 h in the oven.

Membrane characterization was done by FT-IR (FT Infrared Spectroscope, JASCO, FT/IR-6300, Japan) in KBr medium in the range of 400 to 4000 cm^{-1} and XRD (X-ray diffractometer, BRUKER, D8 advance, copper anode) with K_α emission and wavelength of $1.54\AA$. SEM (Scanning Electron Microscopy, TESCAN company, from model VEGA-LMU, 15.00 kV) and AFM (Atomic Force Microscopy, DualScope, C-26, in non-contacting mode) were used to study the topological and morphological characteristics, respectively.

2.3 Procedure

Initial performance evaluation of the membrane was assessed by 3% glucose solution as a feed. Glucose concentrations in permeated substances, were determined by Atago 1310 DR-A1 Abbe refractometer and Varian UV-Vis-NIR Spectrophotometer, model EL01054609.

Sample preparation for UV-Vis was done by using the derivative method as follows: A 6% v/v solution of ortho-toluidin in glacial acetic acid as a reagent was gotten and kept in amber-colored glass away from light for a week. Permeated and standard components were mixed with the reagent with ratio 3 to 1 of reagent and glucose, respectively. After that, all provided samples were heated in boiling water bath for 10 min until the light yellow of samples converted to green color. Finally the samples were ready for determination of glucose in 630 nm (Dubowski 1962). Separation factor (α) was determined based on below equation

$$\alpha = \left(\frac{P_w / PG}{F_w / FG} \right) \tag{1}$$

Where P_W and F_W are the mole fractions of water in permeate and feed solutions, respectively. P_G and F_G are the mole fractions of glucose in permeate and feed solutions, respectively.

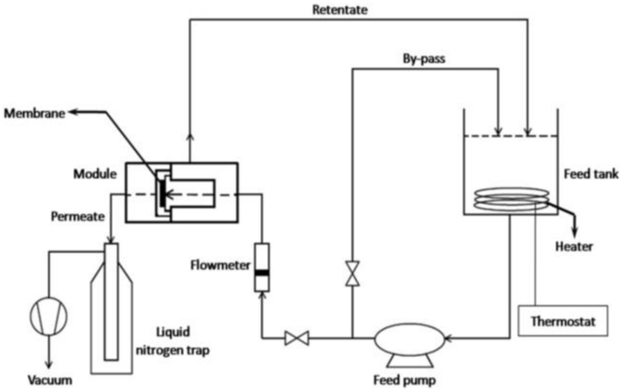


Fig. 1 Schematic of the employed pervaporation system

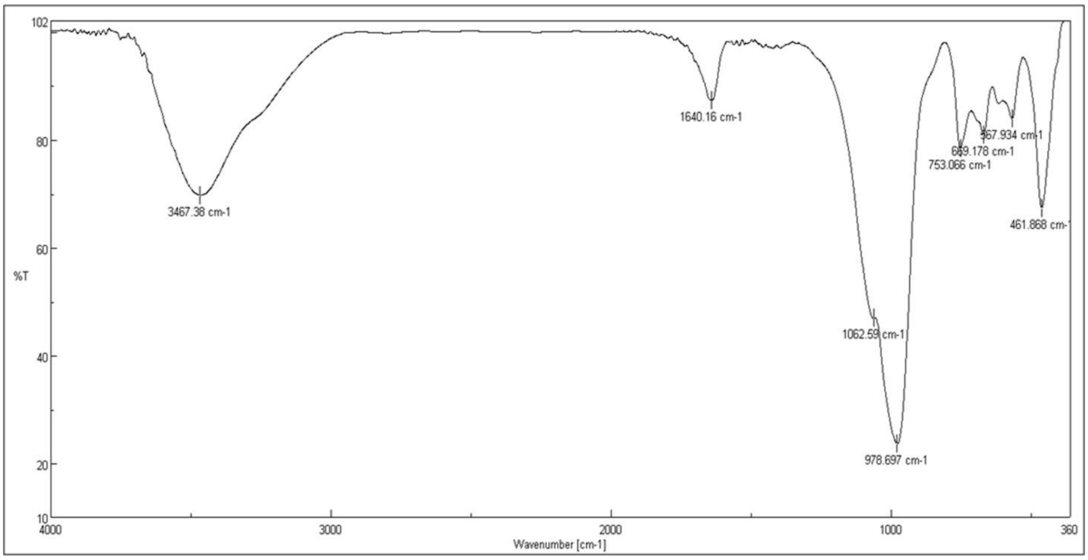


Fig. 2 Spectrum of FT-IR related to Na-X zeolite

The membrane separation performances are reported by solution flux (*F*) and rejection salt (*R*%) which are determined through following equations

$$F = \frac{Q_w}{A_{eff}.t} \tag{2}$$

$$\%R = \frac{(c_i)_f - (c_i)_p}{(c_i)_f} \tag{3}$$

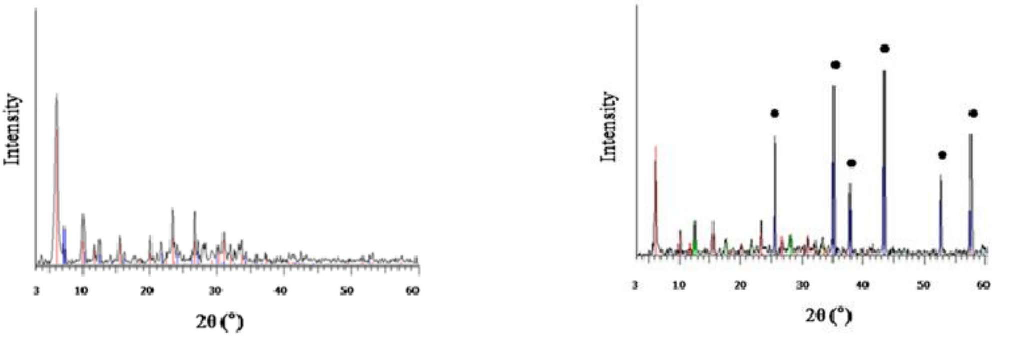
Where Q_W is the weight of the permeate substance (kg) which penetrated through the effective area of the membrane (A=1.77×10^{-4}m^2) in a specific time (t=0.83h). The concentration of ions in the feed and permeate solutions is shown by $(C_i)_f$ and $(C_i)_p$, respectively, and their molar concentrations were obtained by atomic absorption spectrophotometer (shimadzu, AA-670, Japan).

Assessment of the parameters was performed in different concentrations (0.001, 0.005, 0.01, 0.03, 0.05 and 0.1 M) and different temperatures (25, 32, 40 and 52℃) in pervaporation process. All pervaporation studies were performed on the pervaporation set-up that is illustrated in Fig. 1.

3. Results and discussion

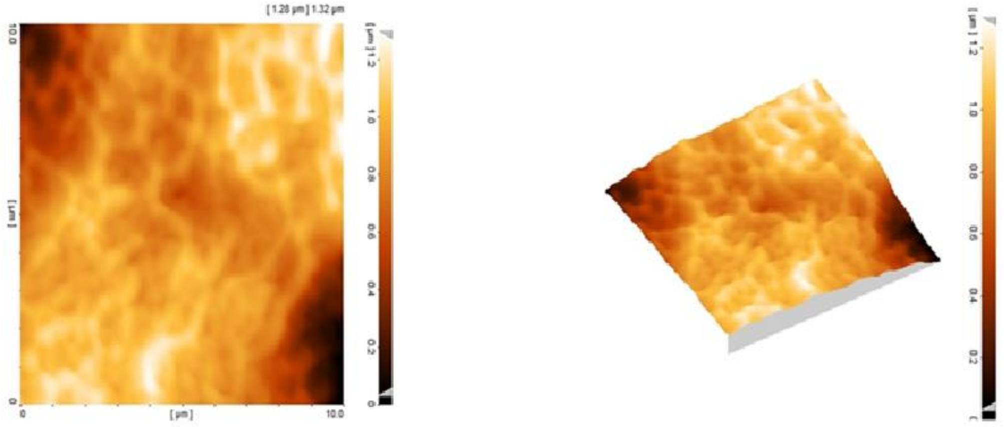
Fig. 2 shows the FTIR spectra of the prepared membrane. The symmetric and asymmetric stretching vibrations can be seen well. Resulted peak at 461.8 cm^{-1} can be shown bending vibrations and all revealed bands at 567.9, 6.1 and 753.0 cm^{-1} are related to symmetric stretching vibration of T(Si or Al)-O bonds (Bellamy 1958).

XRD patterns are shown in Fig. 3. The pattern has good compliance with standard patterns, although, minimal impurities were detected in the zeolitic phase (Covarrubias *et al*. 2008, Zhou *et al*. 2012). Comparing the two XRD patterns shows the formation of zeolite X on the surface of alumina disc. The emergence of the new peaks in Fig. 2(b) that assigns with (•) would reveal the synthesis of the zeolite membrane on the alumina support.



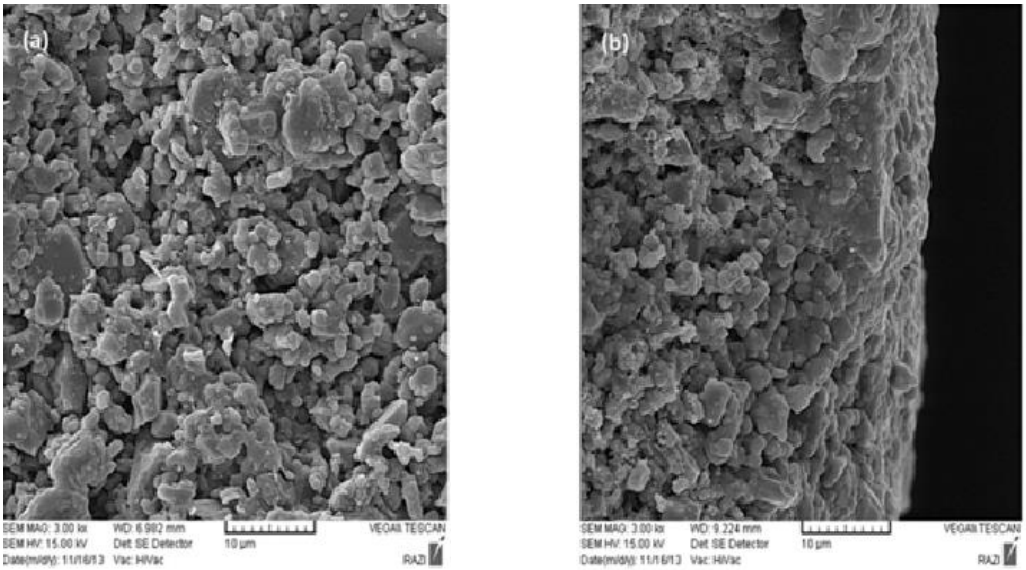
(a) Na-X zeolite powder (b) Zeolite Na-X coated on alumina disc

Fig. 3 XRD patterns of zeolite (alumina peaks are shown as (•))



(a) Two dimensional view (b) Three- dimensional view

Fig. 4 AFM images of NaX zeolite membrane



(a) Surface view (b) Cross section view

Fig. 5 SEM images of NaX zeolite membrane in two views

Table 1 Flux and separation factor for glucose/water solution in pervaporation process

Feed concentration (ppm)	Permeate concentration (ppm)	Flux ($kg.m^{-2}.h^{-1}$)	Separation factor	%Rejection
3×10^4	169.5	3.6±0.3	182.7±8.8	99.43

The AFM images in two and three-dimensional views are shown in Fig. 4. The successful application of secondary growth technique with minimal defects is obvious in prepared membrane. These images show that crystallization of zeolitic phase on the support for several times can be effective in providing continuous layer. Therefore, the membrane synthesis was repeated for three times.

Based on the SEM images shown in Fig. 5, it seemed that the size of crystals and the thickness of synthesized membrane are about 1 μm and 10 μm, respectively. The morphological information about SEM images confirms the minimal defects and the incorporation of NaX zeolite membrane on the surface of the support.

Before using of NaX membrane in desalination process, its performance was investigated by separation of glucose/water mixture in pervaporation process.

It is obvious that the 7.4 Å pore size of the membrane is not suitable for transfer of glucose molecules (8.4 Å) across the membrane, so inevitably they can pass through the non-zeolitic pores and chances have been found in permeate samples.

The primary determination of glucose in permeate was done by refractometer device and using Brix degrees and the relationship between concentration and refractive index. The glucose concentration was resulted below 0.5%. According to the description given, the glucose-orthotoluidine mixture has an absorbance in 630 nm of UV-Vis radiations.

Based on the results, separation factor was obtained 182.7±8.8 (Table 1) that shows the prevention of 99.4% glucose leakage from the permeate.

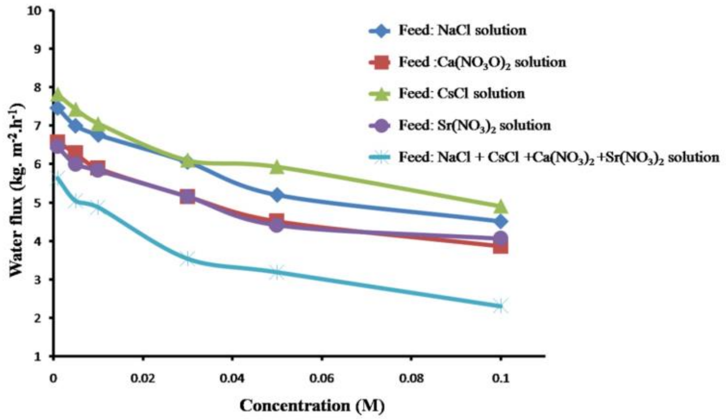


Fig. 6 Water flux as a function of concentration for single and mixed solutions 25°C

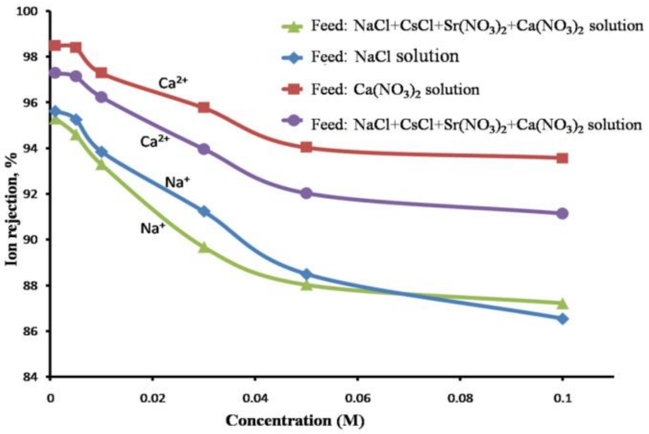


Fig. 7 Ion rejections as a function of concentration in single and mixed solutions consisting Na^+, Ca^{2+}, Cs^+ and Sr^{2+} ions at 25°C

In addition to the high separation factor, water flux 3.6±0.3 $kg.m^{-2}.h^{-1}$ also assigns well performance of NaX-zeolite membrane in the separation process.

Determination of glucose by UV-Vis spectroscopy was introduced as a simple, cheaper and in some cases more accurate than other methods such as HPLC, GC and simple gas permeation.

The ionic solutions consisting of Na^+, Ca^{2+} Cs^+ and Sr^{2+} were chosen for feed solutions. Different concentration and temperatures were applied to study of the desalination process. The size of hydrated ions could be enhanced with a charge to radius ratio in the order of $Ca^{2+}>Sr^{2+}>Na^+>Cs^+$ therefore it is expected that the rejection factors of them have the similar order, but there wasn't detected signal for Cs^+ and Sr^{2+} in permeate at different concentrations. Therefore, in this research, the effect of temperature on both Cs^+ and Sr^{2+} ions did not more studied and only variation of water flux for them is reported. Fig. 6 and Fig. 7 are shown the changing of water flux and ion rejection as functions of concentration, respectively.

With regard to the significant trend in these figures, it is clear that the reduction of water fluxes and ion rejections could be caused by increasing the concentration of ions. The main reason for the

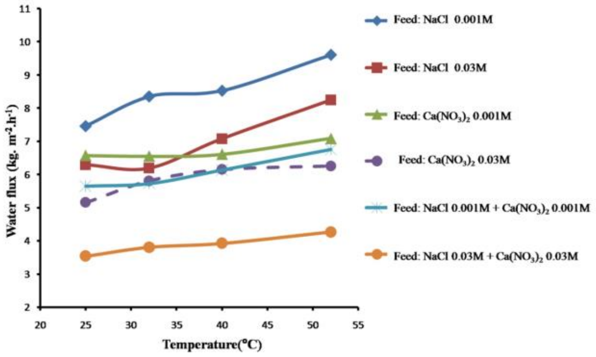


Fig. 8 Water flux as a function of temperature for single and mixed solutions in two concentration 0.001M and 0.03M

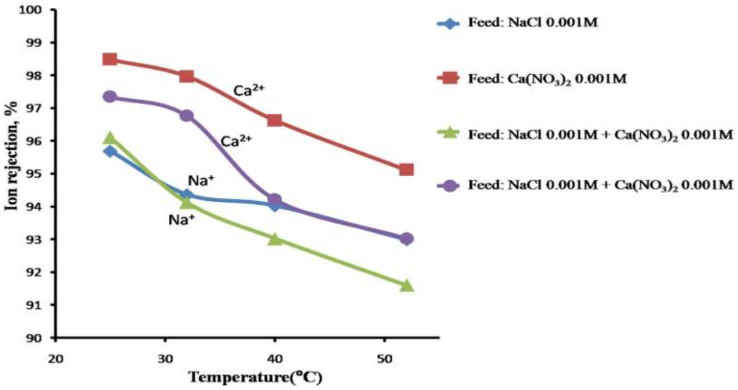


Fig. 9 Ion rejection as a function of temperature in ionic solution consisting Na^+ and Ca^{2+} in single and mixed solution

reduction with the increasing concentration is the effects of membrane fouling in the zeolite pores (Swenson *et al*. 2012, Malekpour *et al*. 2008). On the other hand the decreasing of ion rejection factors is described with double layer thickness variations. As the ionic strength increases with increasing salinity, so the double layer thickness decreases. Therefore, double layer at low concentrations can create a barrier in the diffusion of polarized species, while increasing the concentration can eliminate this positive effect in ion removal. The temperature was increased from 25 °C to 52 °C for Na^+ and Ca^{2+} in two concentrations in single and mixture solutions. The related water fluxes are shown in Fig. 8 and the ion rejection changes are shown in Fig. 9 and Fig. 10.

As seen in the Figs. 10, 11 and 12, temperature can influence the water flux and ion rejection. The flux enhancement and ion rejection decrease can occur due to increase of energy of the species (Duke *et al*. 2009). In these conditions ionic species can penetrate faster through the membrane holes and high flux and low ion rejection can be obtained. There is obvious, the decrease of ion rejection and increase of water flux. On the other hand, changes for Ca^{2+} ion is less than for Na^+ ion that is explained by the different hydrated diameter of the ions. In fact, Na^+ ion due to the small hydrated diameter probably passing from both zeolitic and non-zeolitic pores, but

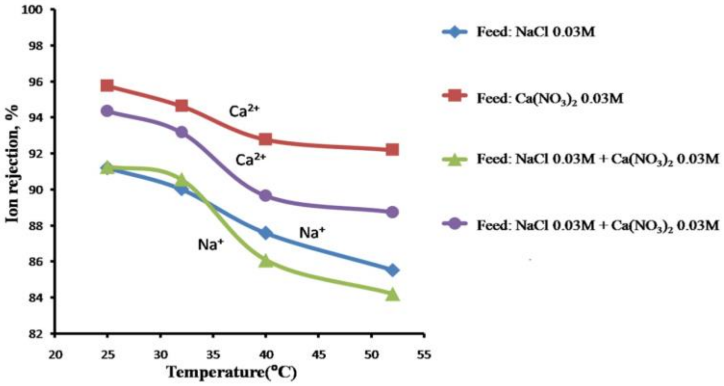


Fig. 10 Ion rejection as a function of temperature in ionic solution consisting Na and Ca in single and mixed solutions

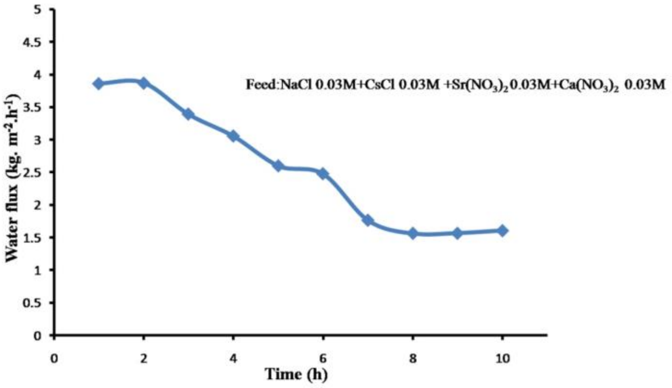


Fig. 11 Water flux as a function of operation time for NaCl solutions

Ca^{2+} ions only pass from non-zeolitic pores. Both water flux and ion rejection for the Na^+ ion solution were more influenced than solutions consisting Ca^{2+} ions with increasing temperature. Also, in the mixture solutions of ions, water flux and ion rejection can be declined rather than ion in single solutions. This effect is explained by the presence of counter ions that is obvious in Fig. 10 and Fig. 11. In fact, both the adsorption and diffusion mechanism for Na^+ and Ca^{2+} ions can affect the process.

As an integral part of this research, ion rejection and water flux were studied as functions of time. Fig. 11 shows decrease flux as a function of operation time. This phenomenon is explained due to gradual fouling of the membrane. Over time, when ions are transferred from the zeolite pores, they can precipitate as a salt form in permeate side and prevent passing other ions through the membrane pores. So fouling effect can play a positive role in increasing of ion rejection (Fig. 12) while cause appreciable reduction of water flux.

A comparative study of this work with the related work shows that the NaX zeolite membrane can resolve the fouling problems with proper water flux and suit ion rejection in desalination processes (Table 2). Although these results show usual types of zeolite materials with small pore

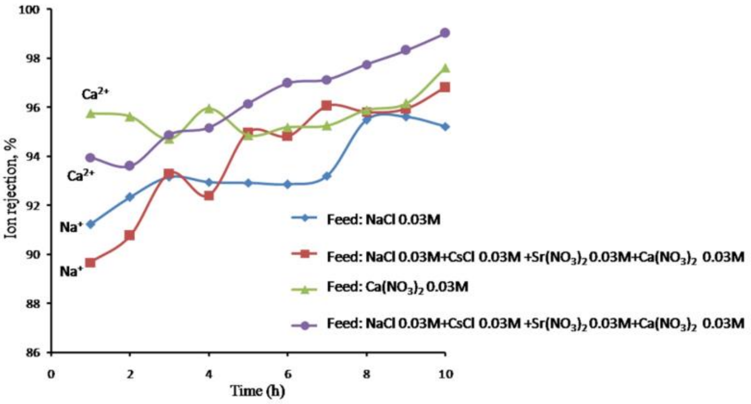


Fig. 12 Ion rejection as a function of operation time for the ionic solutions

Table 2 Recent works in application of pervaporation and reverse osmosis in desalination process

Pub. year	Membrane type	Pore size (nm)	Ions Studied	Ion rejection, %	Water flux ($kg/m^2.h$)	Membrane process	Reference
2004	MFI	0.56	Na^+,K^+,NH_4^+ ,Ca^{2+},Mg^{2+}	58.1,62.6,79.9 ,80.7,88.4	0.112	RO	(Li *et al.* 2004)
2008	NaA	0.41	Cs^+,Sr^{2+},MoO_4^{2-}	>99	1.0-5.3	PV	(Malekpour *et al.* 2008)
2008	FAU	0.74	Cr^{3+}	>95	3-8	RO	(Covarrubias *et al.* 2008)
2009	MFI	0.56	Na^+,Ca^{2+},Mg^{2+}	>99.9	0.72	RO	(Duke *et al.* 2009)
2010	Hydroxy-sodalite	0.28	Na^+	>99	3.5	RO	(Khajavi *et al.* 2010)
2011	NaA	0.41	Na^+	>99.9	1.9	PV	(Cho *et al.* 2011)
2012	MFI	0.56	Na^+	>99	11.5	PV	(Drobek *et al.* 2012)
2012	Clinoptilolite	0.44×0.72	Na^+,K^+ Ca^{2+},Mg^{2+}	>97.5 98.5,99.5	1.25	PV	(Swenson *et al.* 2012)
2013	MFI	0.56	Na^+	82	7	RO	(Zhu *et al.* 2013)
2013	Nanoporous organosilica	2	Na^+	>99.9	13	MD	(Chua *et al.* 2013)

size and different membrane processes have high ion rejection, water fluxes in all researches is low that main reason related to blocking effect in the pore of the membrane. This present work could resolve this important obstacle together, providing suit ion rejection. In subsequent work, we are using this membrane for waste treatment by the reverse osmosis process with the aim of to obtain additional water flux.

4. Conclusions

The NaX zeolite membrane was used for the removal of Na^+, Ca^{2+}, Cs^+ and Sr^{2+} from saline and wastewater solutions. The resulted information about performance and structural evaluations could prove the potential of the membrane in the desalination process. Refractometry and UV-Vis were chose as the fast and more accurate methods in characterization step of the membrane which able to use widely in industrial applications rather than another methods. The fouling problem as a main subject in membrane processes was resolved significantly using large pore size zeolite. High ion rejection was related to good efficiency of the zeolitic membrane mechanisms and suit condition of pervaporation process.

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