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Desalination of aqueous solutions using interpenetrating polymeric membranes-integrated zeolite through pervaporation and reverse osmosis

Masoud Jafarian, Akbar Malekpour*, Gholam Ali Koohmareh

Department of Chemistry, University of Isfahan, Isfahan, 81746/73441, Iran, Tel.: +98 31 37934930; Fax: +98 31 36689732; email: a.malekpour@chem.ui.ac.ir (A. Malekpour)

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ABSTRACT

An interpenetrating polymer network (IPN) membrane and its modified form with NaA zeolite were prepared using polyvinyl alcohol/polyacrylic acid and were used in desalination of saline solutions by pervaporation and reverse osmosis. The prepared membranes were characterized using scanning electron microscopy, Fourier-transform infrared spectroscopy, tensile and swelling test. The effective parameters in the membrane preparation were investigated and optimized through experimental design. Then, Salt rejection and solution flux were selected as the criteria for the membrane performances. The pervaporation results showed that the flux increases with the feed temperature going up and the membrane thickness decrease. However, better rejection was achieved at less feed temperature and more membrane thickness. At the optimized conditions, flux 7.1 kg/h·m^2 and rejection 99% were obtained by net IPN membrane while, these parameters were changed to 11.2 kg/h·m^2 and up to 95% by IPN/NaA membrane. Reverse osmosis experiments revealed that as the pressure on the membrane increases, the amount of flux goes up, while it requires less pressure and more cross-linked membrane for having higher rejection (flux 16.2 kg/h·m^2 and rejection 90%). According to the obtained results, it was found that this composite membrane has a high ability in desalination with appropriate rejection and fluxes.

Keywords: Interpenetrating polymer network; Reverse osmosis; Pervaporation; Desalination; Composite membrane; Zeolite NaA

1. Introduction

One of the most significant human concerns is the water scarcity and not having access to healthy water. Desalination methods are one of the best ways to solve this problem [1,2]. There are many methods for desalination, among which membrane methods are considered because of high efficiency and environmentally friendly [3]. These techniques have high stability, low-cost and industrial capability [4]. Reverse osmosis (RO) is currently the best and most widely used membrane desalination method. In this process, water, under high mechanical pressure, passes through the membrane and produces low salt water [5,6].

One of the accepted transfer mechanisms in reverse osmosis is the sorption–diffusion (S-D) mechanism. According to this mechanism, both water and salt are absorbed on one side of the membrane, diffuse into the membrane, salt and impurities are trapped inside the membrane, and pure water is desorbed from the other side [7–10].

The components and quality of the membranes, should determine efficiency of them. Nowadays, nanocomposites, carbon nanotubes, graphene oxide [11,12] and polymeric membranes [13] are used frequently for membrane preparation. Beside the RO, pervaporation (PV) is another membrane method that is being considered today for desalination [14]. The advantages of this method include, low energy consumption, flexibility and operational simplicity [15,16].

* Corresponding author.

The PV process is a membrane separation method in which the feed solution is in contact with a semipermeable membrane and the other side of the membrane is being vacuumed. A chemical potential difference occurs on both sides of the membrane and separation occurs [17].

In desalination a hydrophilic membrane that can be a polymer or an inorganic porous in needed [14]. One of the common monomers that used in the preparation of desalination membranes is polyvinyl alcohol (PVA) because it is non-toxic, has good physical properties and is hydrophilic [18]. To control the swelling of PVA in aqueous solutions, we need some modifications such as cross-linking or combining it with some materials [19].

Interpenetrating polymer network (IPN) membranes are among the polymer membranes that used in the desalination and membrane processes. These polymers comprise two or more polymeric networks that are locked together but do not form a chemical bond. These polymers have high mechanical properties and high-pressure tolerance. They swell in water without being dissolved. They have high phase and temperature stability and are transparent [20]. The properties of IPN can be improved by adding some materials such as zeolites [21], carbon nanotubes [22], MOFs [23] and graphene [24,25].

These membranes have the advantages of both agents, namely the separation and mechanical properties of mineral particles (zeolites) as well as the easy synthesis, availability, low-cost and variety of polymers [26]. Zeolites are mineral particles that have a special three-dimensional structure and their cavities can be a place to absorb some materials such as water [27]. These particles have properties such as high thermal and chemical stability, high degree of dehydration, high adsorption volume and ion exchange properties [28]. One of the most widely used zeolites in water sorption is NaA zeolite. Its cavities are such that only water molecules can enter them.

In the present work, an IPN membrane, which is a combination of cross-linked polymer networks of PVA and polyacrylic acid (PAA), was used for desalination of saline solution by pervaporation and reverse osmosis methods. This IPN membrane is very hydrophilic and because both the polymer networks in it have cross-linked chains (full IPN), it has good strength and resistance in the test conditions. Also, in order to improve the performance of this membrane, we add NaA zeolite to its texture. This composite membrane, in addition to having the advantages of IPN, will also have the characteristics of zeolite. In all stages of the work, we used the experimental design method (CCD), which has more advantages and speed than the old methods such as one at the time. The best conditions for membrane performances was found by solution fluxes and salt rejections.

2. Experimental set-up

2.1. Materials

Polyvinyl alcohol (PVA) with molecular weight 125,000 was prepared from Merck Co. and no purification was done on it. Accordingly, acrylic acid (AA), as a guest monomer, and ethylene glycol dimethacrylate (EGDMA) as a cross-linker and benzoyl peroxide (BPO) as an initiator of polymerization were provided from Merck Co. Zeolite NaA, glutaraldehyde (GA) and HCl (37%) were used as filler, cross-linker and catalyst, respectively. NaCl and $MgCl_2$ were prepared from Merck Co. and in order to neutralize the acidic property, NaOH was used.

2.2. Membrane preparation

2.2.1. Cross-linked PVA membrane

With the purpose of preparing 5% PVA solution, PVA is mixed for 2 or 3 h in DI water at 80°C. After reach to ambient temperature, 0.2 mL of GA as a cross-linker and 0.5 g of HCl as a catalyst were added and mixed for 20 min. It is important to mention that it should be poured in a molding container before the solution goes to be stiff completely. It should be kept in ambient temperature for 24 h. After that, in order to make sure to cross-link, the membrane was put in the oven with 100°C for 5 h. Consequently, NaOH solution is used to wash the membrane and to remove excess HCl. Moreover, the membrane is washed by water several times to remove excess salt. Finally, the membrane was dried in lab temperature.

2.2.2. PVA/PAA IPN membrane

Corresponding aforementioned section, 10% PVA solution is prepared. Then, AA and BPO are added to this solution as an initiator and EGDMA as PAA cross-linker. After that, it should be mixed in 70°C for 12 h. It ought to be noted that other next stages are like steps which mentioned in the earlier steps.

2.2.3. IPN composite membrane

To prepare the IPN/NaA membrane, before the PVA was added to water at 70°C, the filler was entered the water and completely was dispersed in it. Then PVA was added to the water and all the steps of membrane synthesis such as IPN membrane preparation were repeated.

2.3. Characterization of membranes

With the aim of identifying membranes structurally, attenuated total reflection Fourier-transform infrared spectroscopy (ATR-FTIR) by Jasco-6300 instrument was being recorded. For tensile testing, two membranes were being examined by Santam-20 with rectangular section and a gage with 20 mm length. This test was done in room temperature and 5 mm/min extension speed. Furthermore, different amount of mechanical resistance was obtained based on the ingredients of each membrane. For investigating the amount of hydrophilic of membranes contact angle method was used. The more hydrophilic is the membrane, the amount of spreading water drop increases. Moreover, the contact angle reduces between water and membrane. To study the membrane morphology, membranes examined by scanning electron microscopy (SEM) Quanta FEG-450 and in 10 kV and high vacuum atmosphere. Swelling of each membrane could be obtained on the basis of the following formula in which W_w equals the weight of swollen membrane in water for 48 h and W_d is the weight of dried membrane.

$$\text{Swelling percentage}\% = \frac{\left(W_w - W_d\right)}{W_d} \times 100 \quad (1)$$

2.4. Set-up

It should be mentioned that for pervaporation method, a module is required for membrane setting. This module consists of two separated parts which were clasped together through some clamps. One part of the module has been linked to vacuum pump, and brings the outlet to a trap which kept in low temperature. Additionally, the other part of the module is connected to the rotary pump which linked to the feed solution. The vacuum of the vacuum pump was almost 20 kPa. To explain more, the feed tank is attached to a heater and thermostat which it can be used in the temperature programming. The length of each experiment was different from 15 to 30 min.

Since there was a need to high pressure (up to 80 bar), it is important to have an appropriate module for RO. In fact, there is a high-pressure pump which joins feed to the module. Moreover, the other part of the module is connected to a trap and gathers the outlet solution from the membrane. This pump is also linked to a governor for pressure programming. Because of high pressure of inlet solution to module, it is necessary to put a sponge before the membrane in order to partly control the initial pressure on the membrane.

2.5. Flux

Flux (J) is considered as the amount of passing water through the membrane which can be obtained by the following formula. In this formula, Q is the amount of passing solution; Q/A. t (kg/m^2·h)= J; Q = the amount of passing solution (kg); A = area of the membrane (m^2); t = the time of experiment based on hour (h).

2.6. Membrane rejection

Atomic spectrophotometer (Shimadzu AA-670) is used to measure the membrane rejection. Thus, the standard samples of each ions were brought in the instrument. After plotting the calibration curve, the amount of salt in solution passing through the membrane was obtained based on curve equation.

3. Results and discussion

3.1. Swelling of IPN, cross-linked PVA and IPN/NaA membranes

The swelling curve for the membranes was obtained on the basis of cross-linker percentage (Fig. 1). As it is evident, with increasing cross-linker, due to the tightening of the membrane structure, the swelling decreases, while at concentrations higher than 5% of cross-linker no change in swelling was observed. Even though IPN membrane is more hydrophilic, the amount of membrane swelling of IPN was less than PVA membrane significantly, it seems due to having two mixed cross–linked networks the structure of polymer was more rigid. In composite membrane with increasing zeolite percentage, due to the increase in hydrophilic sites in membrane, swelling was increased (Fig. 2).

3.2. Contact angle

As is clear in the images obtained (Fig. 3), due to the presence of two hydrophilic polymeric networks, IPN membrane has a lower angle of drop on its surface (83.79) and this indicates more hydrophilicity of this membrane than PVA membrane (85.96). This difference can be due to the presence of acrylic acid polymer inside the polyvinyl alcohol network as well as more hydrogen bonds of this network than the PVA membrane. Due to its hydrophilic zeolite, the composite membrane interacts more with the water droplet, resulting in a smaller angle with the water droplet (72.59).

3.3. FTIR spectra

The spectrum of IPN exhibits a strong transmittance at 1,650–1,700 cm^{-1}, which can be assigned to the C–O stretching of carbonyl group of polyacrylic acid. Peaks between 920–850 cm^{-1} are assigned to the characteristics transmittance s of syndiotactic and isotactic PVA chains. The strong

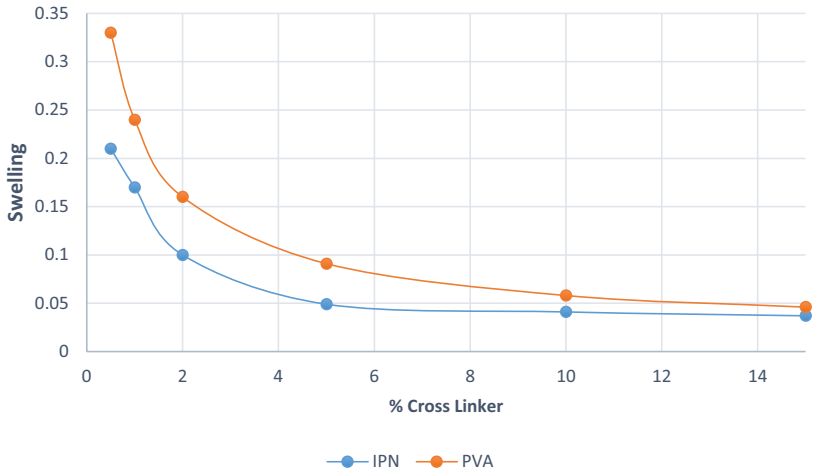

Fig. 1. Swelling test of IPN and cross-linked PVA membrane.

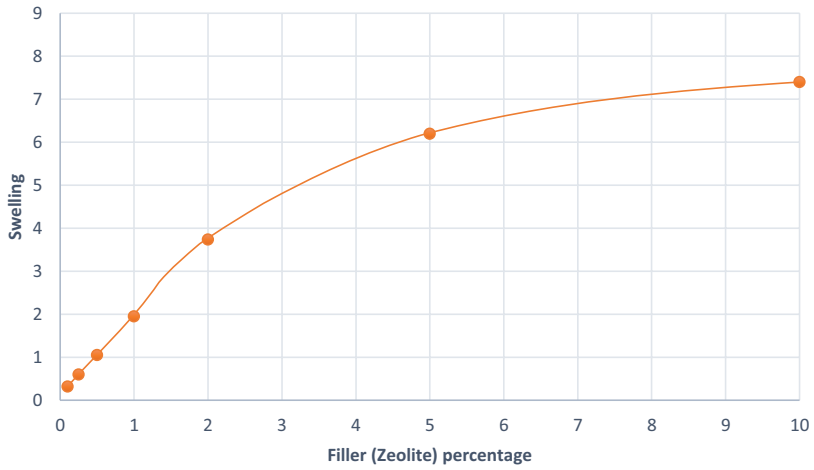

Fig. 2. Swelling test of composite membrane.

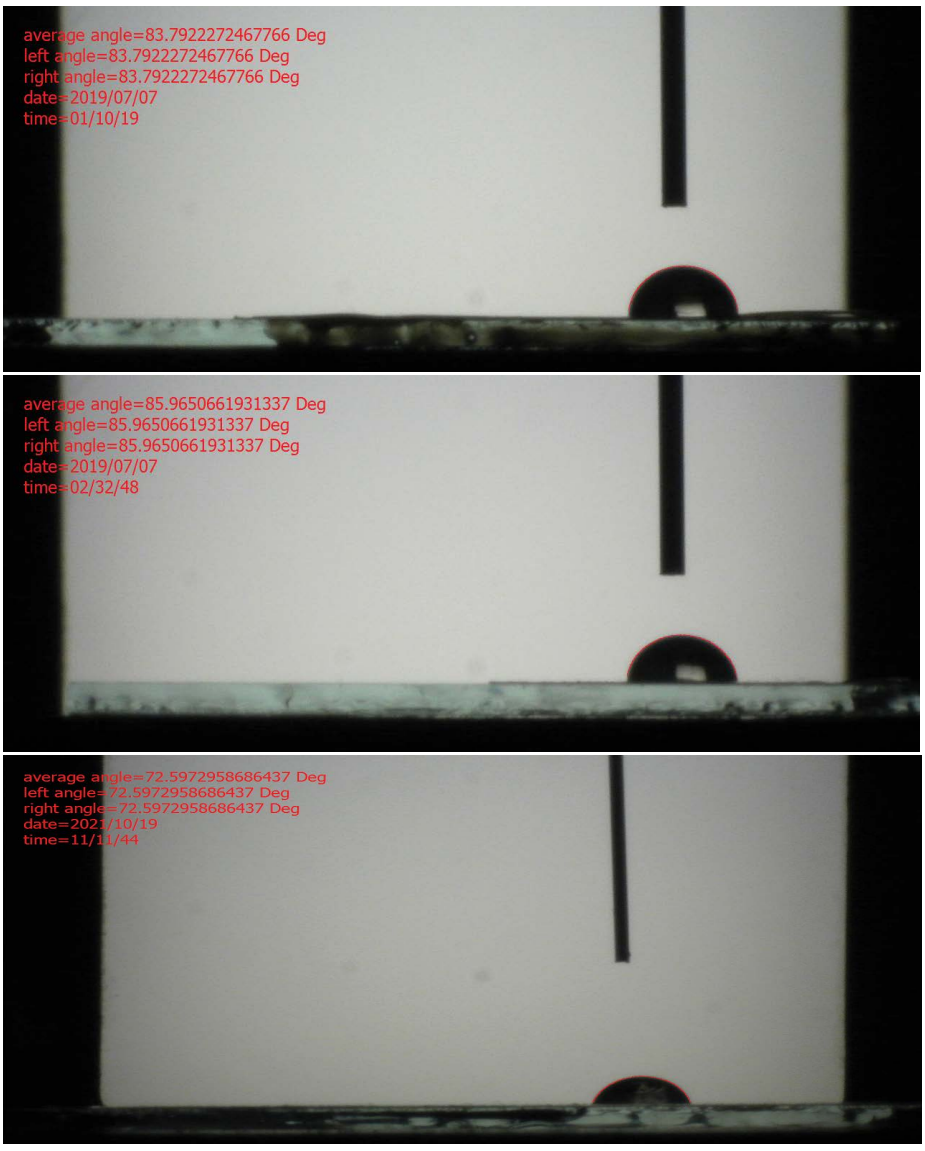

Fig. 3. Contact angle test, (a) IPN membrane, (b) PVA membrane, and (c) composite membrane.

transmittance at 3,400 cm^{-1} is assigned to the Hydrogen-bonded O–H stretching mode. All the major bonds of PAA and PVA are also present in the FTIR spectrum of IPN, so it is compatible with and IPN structure (Fig. 4). In the composite membrane FTIR spectrum, as it turns out, the 1,150 cm^{-1} peak is related to the internal vibration of T–O asymmetric stretching. There is also a strong peak in the 700 cm^{-1} that is related to internal vibration of T–O symmetric stretching. According to these cases, the presence of NaA zeolite in the membrane is confirmed.

3.4. Tensile test

As shown by the diagram (Fig. 5) of the tensile test, the IPN membrane has a much higher tensile strength than the PVA cross-linked membrane because of its rigid structure due to the existence of two interpenetrated networks with cross-linking agent. As can be seen from the results, the maximum force that the IPN membrane endured was 77.41 Newton, while the PVA membrane only tolerated 31.64 Newton. At this force, IPN membrane had a 2.17 mm elongation but PVA membrane had an elongation of 3.17 mm. It seems that due to the presence of zeolite particles inside the tissue of the composite membrane, the arrangement of the IPN network is slightly disturbed and as a result the resistance of the membrane is reduced. The maximum force that the composite membrane endured was 66.39 Newton and at this force, composite membrane had a 2.07 mm elongation.

3.5. Scanning electron microscopy

As shown in the pictures (Fig .6) zeolite particles are well dispersed in the membrane tissue and the dimensions of the zeolite particles are almost equal except in small parts. The fine particles seen in the IPN image are related to BPO particles that remain unreacted during the membrane preparation process. Based on SEM images, the zeolite particle size is estimated at about 100 to 200 nm.

3.6. Optimization of parameters of membrane preparation

In order to optimizing of IPN membrane parameters including the amount of cross-linkers, the amount of guest

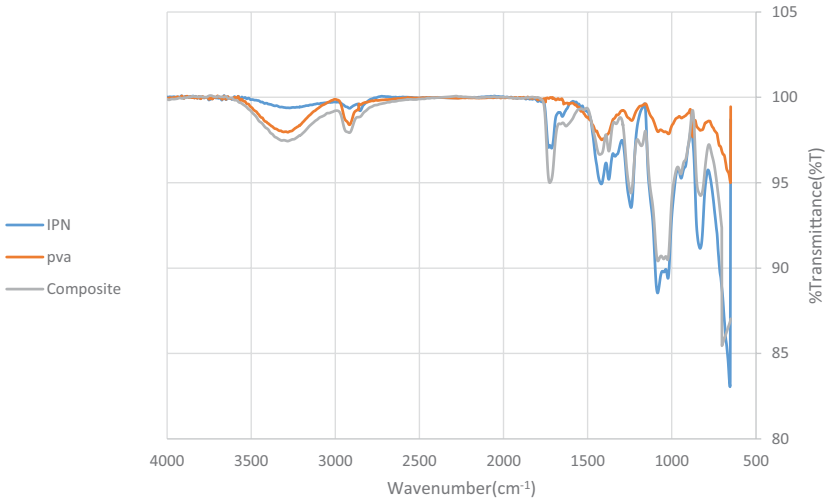

Fig. 4. ATR-FTIR spectrum of PVA, IPN and composite membrane.

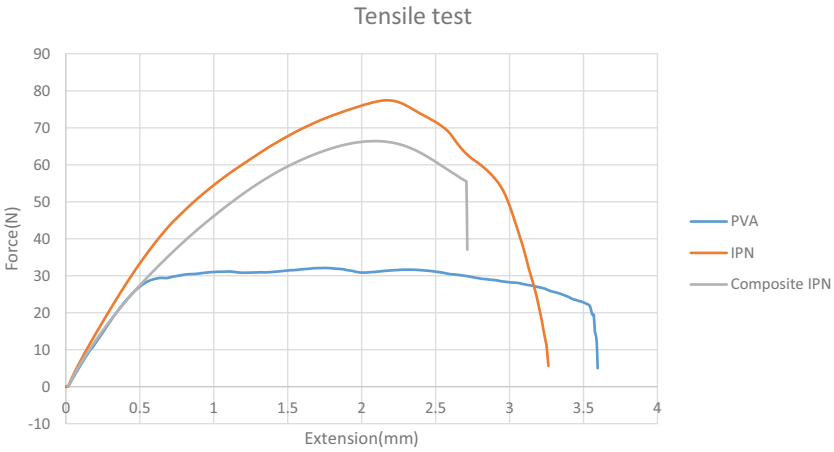

Fig. 5. Tensile test of IPN, PVA and composite membrane.

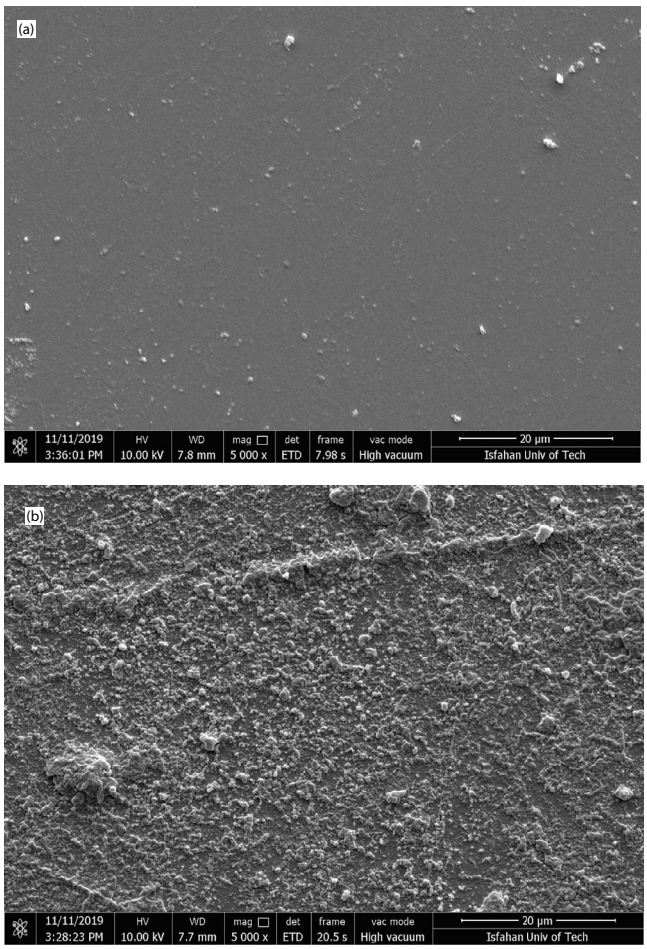

Fig. 6. SEM image of membrane, (a) IPN membrane and (b) composite membrane.

monomer and membrane thickness, an experiment was designed. The desired response is regarded as to be the amount of flux which is the amount of water across membrane in this experiment. It should be mentioned that the amount of PVA is 5% (w/w) and was done through pervaporation method. According to data obtained from experiment designing and ANOVA table, the lack of fit does not occur, moreover, corresponds to the expected data (Fig. 7). The effective parameters in preparing and optimizing IPN membrane are percentage of acrylic acid, PVA cross-linker, the thickness of membrane and square root of acrylic acid percentage in membrane. Consistent with such data, it could be concluded that as the amount of AA goes up, the amount of flux increases because of hydrophilic of membrane accumulating. Moreover, if the amount of PVA cross-linker goes up over the specific range, the amount of flux decreases due to concentration made in membrane. Also, the amount of flux increases as the thickness of membrane and concentration of polymeric layers decrease (Fig. 8). In order to have an appropriate flux in accordance with optimized data, the amount of the followings should be: AA = 22.6%, AA cross-linker = 1.78%, PVA cross-linker = 4.26%. It needs to be mentioned that the amount of membrane volume must be 3.09 mL to reach flux over 4.32. In the following section, in an attempt to prepare membrane and investigating the application of membrane, these amounts are used.

The membrane prepared in the previous step was used to make the composite membrane. The parameters used in the optimization of this membrane are: percentage of zeolite, time of adding zeolite to the solution and time of stirring the solution. To achieve the highest possible flux, after experimental designing, it was found that to achieve flux 8.1, we should add 4.41% of zeolite to water that which PVA has not yet been added and mix it for 53.41 min while it is still on heater.

3.7. Application of membrane

3.7.1. Pervaporation method

NaCl standard solution was used for investigating the function of membrane in pervaporation method. Consequently, an experimental design was applied to examine this method which is considered as a comparison between IPN, composite and PVA cross-linked membranes. The examined parameters were operating temperature, pH, feed concentration and the type of membrane. Furthermore, two responses of flux and the amount of membrane rejection were obtained. Therefore, the obtained data, which designed in ANOVA table, does not have a significant lack of fit. These data correspond to the expected data, as well (Fig. 9). In the section of optimizing the amounts, if the aim of the experiment is higher flux, it goes up by increasing the temperature which caused by increasing molecular mobility, and the more distance among polymeric layers. On the other hand, the more the feed concentration goes up, the less flux could be seen as more cell membrane are closed (Fig. 10). Conversely, if the purpose is higher rejection, so it can have higher rejection through decreasing the temperature which caused by decreasing molecular mobility and getting the membrane more rigid in lower temperature. The more the feed concentration increases, which led by more numbers of salt molecules and more salt passage, the less rejection could be seen (Fig. 11). If the appropriate amount of two responses of flux and rejection are needed, the optimum value must be considered for parameters. In all these states, the superior membrane is PVA membrane. The following conditions are required to reach rejection 93% and flux 7.7. Temperature: 60°C, Feed concentration: 413 mg/L, Neutral pH and IPN membrane.

To test the performance of the composite membrane, another experimental design was performed with the same parameters as before. After performing tests related to the experimental design, it was found that the designed model did not have a significant lack of fit. Also, the predicted values match the actual values. According to the data obtained from ANOVA, the effective parameters in this membrane are temperature, feed concentration, membrane type and interaction between temperature and membrane type. If maximum flux is considered, in the process of pervaporation, a composite membrane with temperature and feed concentration of 64.8°C and 282.4 mg/L, respectively, and in an environment with pH 4.23 should be used. Under these conditions, flux 14.5 kg/h·m^2 and salt rejection 91.18% are obtained. Also, if maximum salt rejection is desired, IPN membrane with feed temperature, concentration and

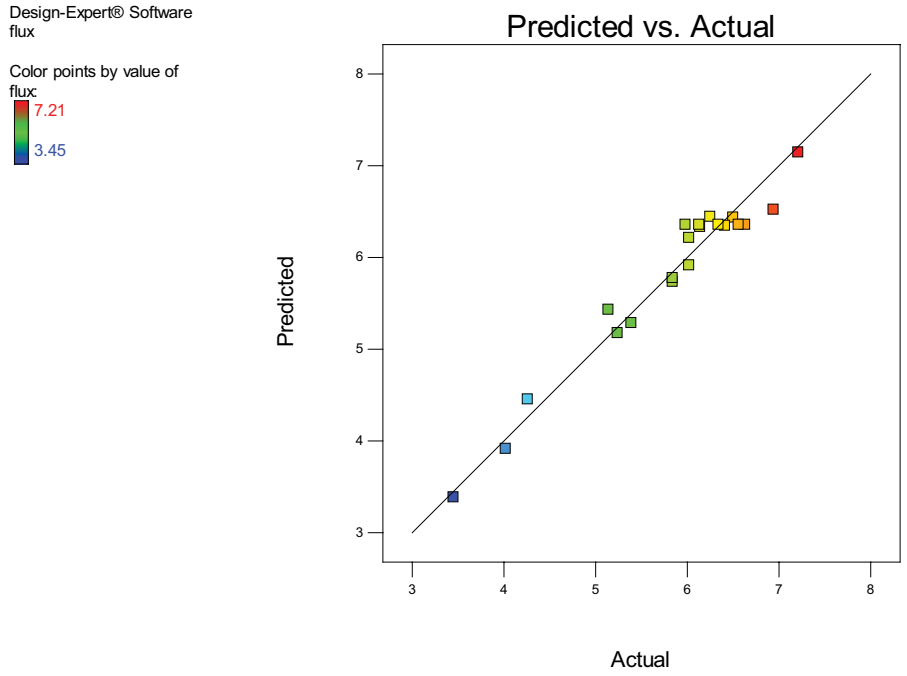

Fig. 7. Predicted vs. actual data from experimental design for IPN membrane by pervaporation.

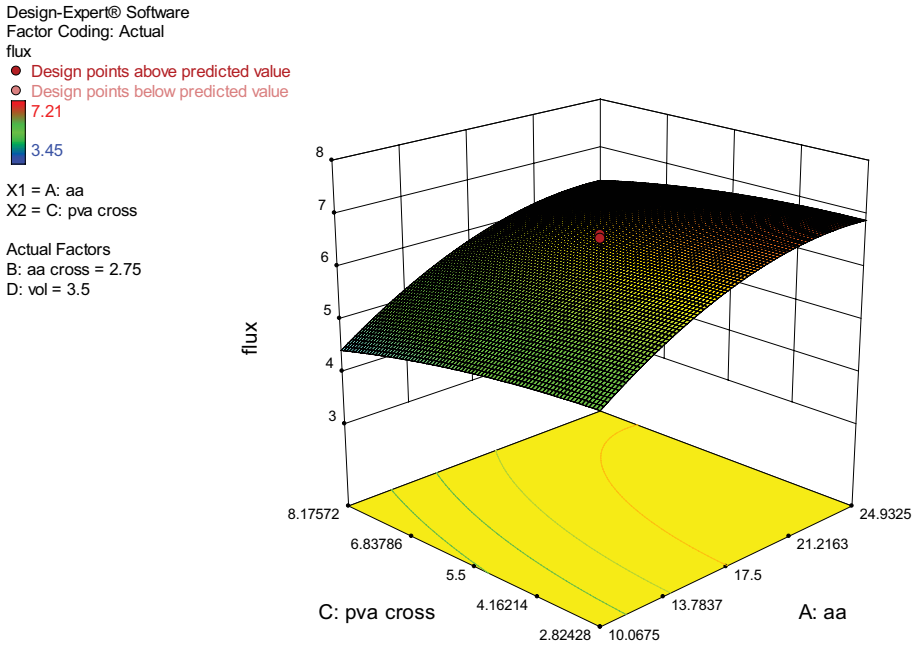

Fig. 8. Flux vs. AA and PVA cross-linker for IPN membrane.

pH, 35.3°C, 340.08 mg/L and 10.52, respectively, should be used in the pervaporation process. Under these conditions, flux 9.26 kg/h·m^2 and salt rejection 95.18% are obtained. If a good salt rejection and flux are considered at the same time, a composite membrane with temperature, feed concentration and pH of 36.4°C, 282.42 mg/L and 10.77, respectively, should be used in the pervaporation. Under these conditions, flux 11.59 kg/h·m^2 and rejection 94.44% are obtained.

3.7.2. Application of IPN and composite membrane in salt mixture by pervaporation method

As a matter of fact, some mixtures were made with various percentage of sodium and magnesium salts in obtained optimum conditions in previous stages. Flux and salt rejection were obtained as well (Tables 1 and 2). Based on such data, the more the percentage of magnesium increases in

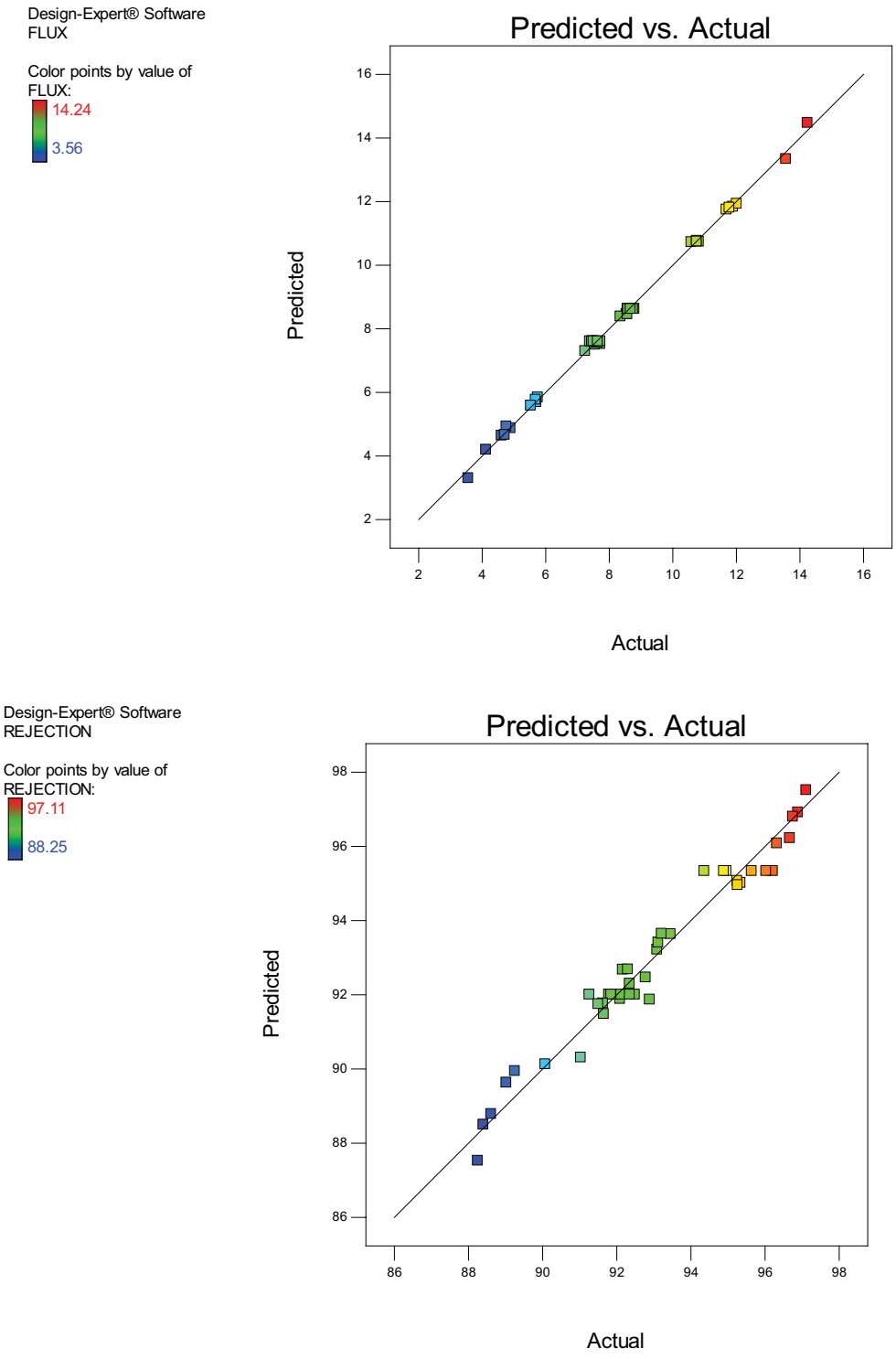

Fig. 9. Predicted vs. actual data from experimental design in pervaporation for, flux and rejection.

the mixture, the less flux could be seen which being bulkier compared to sodium could be its reason which result in cell membrane closure and make no more transition permission. Additionally, as the compound of each type of salt increases in the mixture, the membrane rejection decreases compared to it.

3.7.3. Reverse osmosis method

In order to investigate the function of IPN membrane and compare to cross-linked PVA membrane, an experiment was made to examine pressure parameters, pH, feed concentration and the type of membrane. Consequently, the

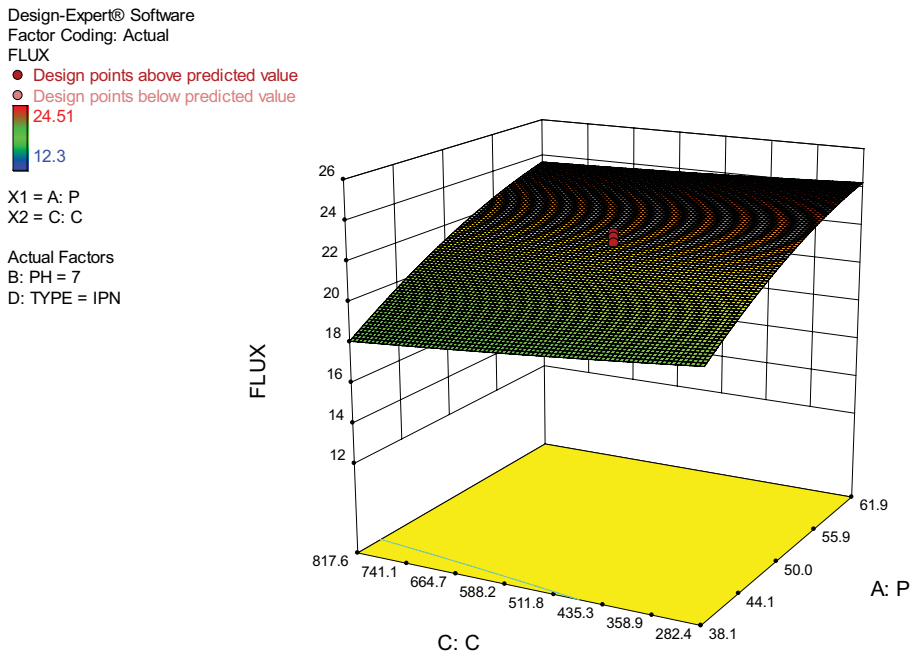

Fig. 10. Flux vs. *T* and *C* in pervaporation for IPN membrane.

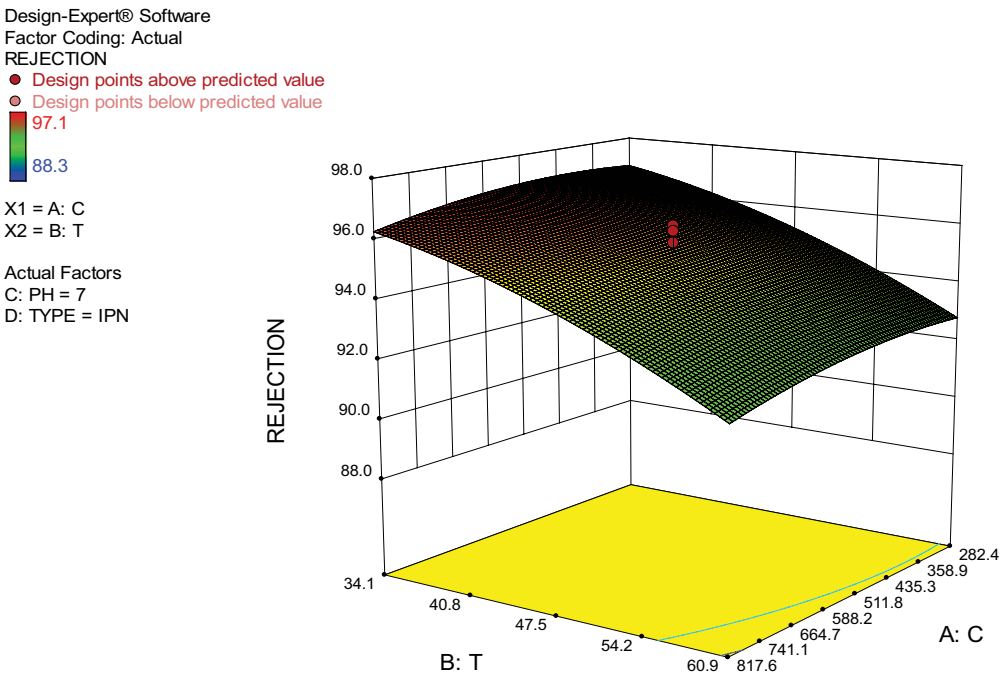

Fig. 11. Rejection vs. *T* and *C* in pervaporation for IPN membrane.

desired responses are flux and membrane rejection. So, based on the obtained data from designing, there is no significant lack of fit. These data correspond to the expected data completely (Fig. 12). It should be noted that the amount of R^2, modified R^2 and expected R^2 are absolutely close to each other. In optimizing stage, in order that two responses of flux and membrane rejection to be placed in desirable condition, pressure must be about 54 atm, pH = 7.9 and salt concentration = 282 mg/L, consequently, flux 16.22 and rejection 87.3% occur. It should be mentioned that the preferred membrane is IPN. If the flux needs to be maximum, pressure and pH must be increased while concentration should be decreased. In addition, rejection and flux must be 85.8% and 17.5%, respectively. Alternatively, if the rejection needs to be maximum (89.55%), pressure and concentration must be decreased, and neutral pH should be used. In this case,

Table 1
Application of IPN membrane in salt mixture by pervaporation method

Na (mg/L)	Mg (mg/L)	Na rejection	Mg rejection	Flux (kg/m²·h)
500	0	96.4	100	8.3
400	100	97.01	99.1	7.7
300	200	98.25	98.12	7.1
200	300	98.94	97.02	6.5
100	400	99.63	95.89	5.9
0	500	100	94.65	5.1

Table 2
Application of composite membrane in salt mixture by pervaporation method

Na (mg/L)	Mg (mg/L)	Na rejection	Mg rejection	Flux (kg/m²·h)
500	0	92.5	100	10.7
400	100	93.1	95.1	9.2
300	200	93.9	94	8.5
200	300	94.6	93.1	7.9
100	400	95.7	92.3	7.1
0	500	100	91.6	6.6

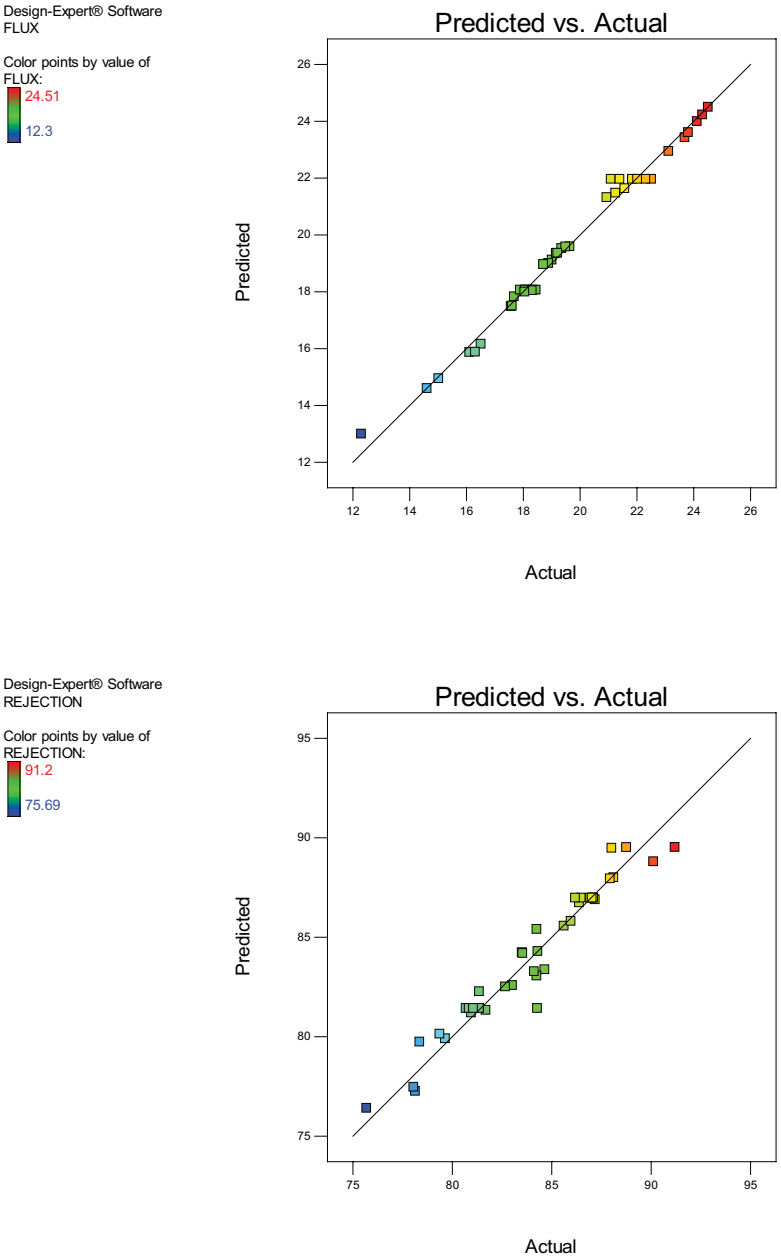

Fig. 12. Predicted vs. actual data from experimental design in reverse osmosis for, flux and rejection.

flux will be 19.5. In keeping with obtained data, more molecules could be passed through the membrane by increasing the pressure to the membrane. Consequently, the flux increases. Moreover, if there are more salt particles in water, the amount of membrane fouling decreases and it makes the passage of water easier. In addition, the flux goes up. IPN membrane is preferred due to more rigid and stronger structure compared to PVA membrane because of higher pressure tolerance (Fig. 13). The reason of decreasing rejection by increasing pressure is that molecules can be passed through the membrane in higher pressure, so more salt particles could be passed through the membrane. Additionally, by increasing the concentration as there are more salt particles in the solution and reach the membrane, and because

Table 3
Application of IPN membrane in salt mixture by reverse osmosis method

Na (mg/L)	Mg (mg/L)	Na rejection	Mg rejection	Flux (kg/m²·h)
500	0	86.32	100	15.3
400	100	87.69	89.32	14.9
300	200	88.26	87.78	14.5
200	300	89.31	86.25	14.1
100	400	91.02	85.01	13.6
0	500	100	83.86	13.2

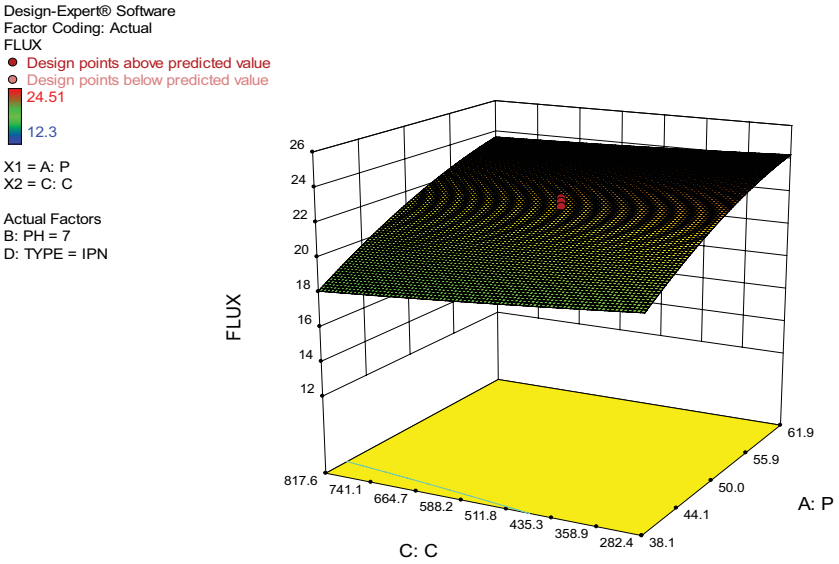

Fig. 13. Flux vs. *P* and *C* in reverse osmosis for IPN membrane.

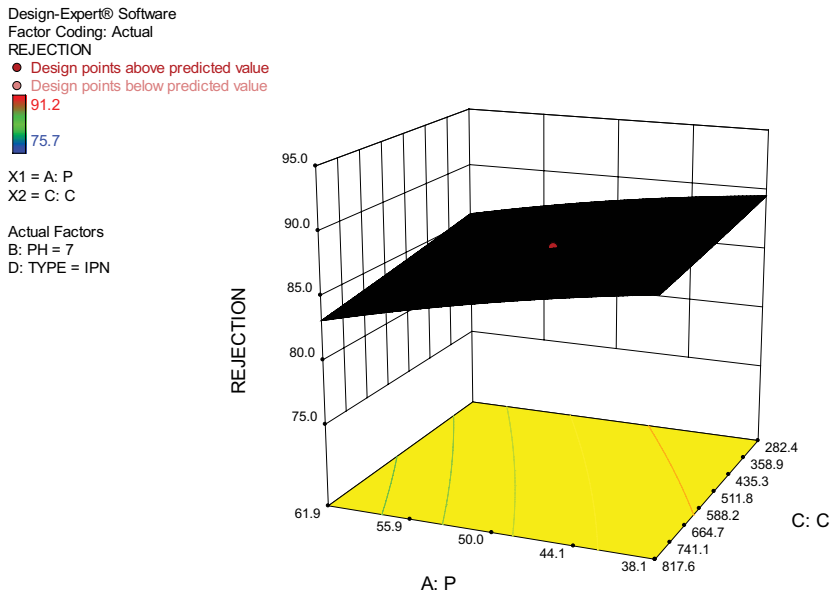

Fig. 14. Rejection vs. *P* and *C* in reverse osmosis for IPN membrane.

Table 4
Compare this work with other same works

Membrane type	Flux (kg/m^2·h)	Salt rejection (%)	Rejected salt	Method
PVA TFC/PSf	2	84	NaCl	Pervaporation [29]
PI/GO	36.1	99.9	NaCl	Pervaporation [30]
PVA/Maleic anhydride/Silica	6.9	99.5	NaCl	Pervaporation [31]
CNT/polyamide polymer	28.05	90	NaCl	Pervaporation [32]
PVA/PAA IPN	7.7	93	NaCl	Pervaporation (This work)
PVA/PAA/NaA zeolite composite	10.3	91	NaCl	Pervaporation (This work)
PVA/PAA IPN	15.6	87.3	NaCl	Reverse osmosis (This work)

of high pressure, more passing salt particles can be seen. The percentage of rejection is decreased, as well (Fig. 14).

3.7.4. Investigation of the application of IPN membrane in salt mixture by reverse osmosis

The mixture of two sodium and magnesium salt with different percentage in the conditions which made for IPN membrane, was investigated by use of reverse osmosis (Table 3). Subsequently, the amount of flux is reduced by increasing magnesium in the mixture. In fact, magnesium is bigger which resulted in membrane closure could be the reason. Moreover, the more percentage of each salt in the mixture, the less membrane rejection to it. Because more particles reach the membrane. And they could be passed through the membrane because of high pressure.

3.7.5. Reproducibility in both pervaporation and reverse osmosis with IPN and composite membrane

Reproducibility (n = 6) of flux and rejection in pervaporation method and in central point of experiment designing is 0.7 and 0.5, respectively with IPN membrane and 0.6 and 0.4 with composite membrane. On the other hand, in reverse osmosis, the reproducibility of flux and salt rejection in central point of experimental designing is 1.7 and 0.9, respectively. It is highly important to note that these numbers indicate high reproducibility of these two methods.

3.7.6. Compare this work with same works (Table 4)

According to Table 3, the IPN and composite membrane prepared in this work has favorable conditions in terms of performance. This membrane is less efficient than some membranes and methods and much higher than others.

Therefore, considering the simpler preparing method and materials needed for synthesis, it can be said that this membrane has a high efficiency.

4. Conclusion

Polyvinyl alcohol and polyacrylic acid and NaA/IPN composite membrane were prepared by sequential synthesis. Glutaraldehyde and ethylene glycol dimethyl acrylate were used as cross-linking agents. FTIR, SEM, swelling test, hydrophilicity test and mechanical tests were performed to identify the properties of this membrane.

Subsequently, the membranes were used for pervaporation and reverse osmosis. Results showed that the IPN membrane, due to its stronger structure and more hydrophilic properties than the base polymers, gives higher flux in both methods and has higher salt rejection. Also, when composite membrane is used, due to the more hydrophilicity of this membrane, the amount of flux increases significantly, but it seems that due to the presence of zeolite particles in the membrane tissue and reducing the order of the polymer network arrangement, the rejection rate decreases. We argue that this impressive ability is largely due to interpenetrating of two hydrophilic polymers as well as incorporation of zeolite particles into the membrane structure. Zeolite particles may force the polymer chains to take up further distance and lead to better water fluxes.

If the target is higher rejection and the flux rate is not very favorable, the pervaporation method is superior, but if the flux rate is considered, reverse osmosis is the better method. Overall data confirmed that the combination of the two polymers to preparation of IPN could lead to utilization of benefits of both of them especially for increasing of water flux.

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