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Effect of different conditions on pervaporation dehydration in CA/NYL66 blend membrane

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Abstract. In this study, cellulose acetate (CA) / nylon66 (NYL66) (95/5) blend membranes with different thicknesses were prepared by a solvent evaporation method. The effects of membrane thickness (almost 7-25μm), feed concentration (70- 95 wt.% isopropanol), and feed temperature (30-60 ºC) were investigated on the performance of membrane in the separation of isopropanol-water mixtures. With regard to the results of sorption experiments, it was found that the increase of feed temperature enhanced the overall sorption while by increasing feed concentration, the overall sorption passed through a maximum value at 70 wt. % isopropanol (IPA). The best separation factor 3080.51 was gained at high isopropanol concentration 95 wt.%, low feed temperature 30 ºC, and high membrane thickness 24.62 μm. Regarding the pervaporation separation index, the obtained results showed that proper values for the thickness of membrane, feed temperature, and isopropanol concentration in feed were 24.62 μm, 40 ºC, and 70 wt.%, respectively.

Keywords: cellulose acetate/ nylon 66; pervaporation; dehydration; isopropanol-water mixture

1. Introduction

Pervaporation is an efficient and effective technique through which the components of a liquid chemical mixture are separated on the result of difference in their partial vapor pressure through a dense semipermeable membrane (Neel *et al.* 1985). This technique has been notably developed and extensively used for a wide range of purposes including: dehydration of alcohols (Mao *et al.* 2010, Rajineekanth *et al.* 2017, Unlu 2019) and other organic solvents (Krishna Rao *et al.* 2007, Li *et al.* 2002, Smitha *et al.* 2006); removal/recovery of trace amounts of organics from dilute aqueous streams (Kujawa *et al.* 2015, Unlu and Durmaz Hilmioglu, 2016b); and more recently, separation of organic–organic mixtures especially azeotropic and closely boiling liquid mixture (Ibrahim and Lin 2016, Knozowska *et al.* 2018).

Isopropanol (IPA) is mainly employed as an effective and economical solvent in personal care products, de-icers, pharmaceuticals, inks, and surfactants, and also as a precursor for manufacturing the chemicals (Sanz and Gmehling 2006, Zafar *et al.* 2012, Araki *et al.* 2011). Accordingly, recovery of this solvent from IPA/water mixtures can be very important from the economic point of view. Most of applications need highly pure IPA, i.e. 99.5% of IPA (Xiangyi 2007). However, the purification of IPA with conventional separation techniques such as distillation or adsorption is difficult and uneconomical due to the formation of an azeotropic mixture with water at 87.9 wt.% of IPA concentration (Bruggen and Luis 2015). Pervaporation can be a good alternative because of its low energy demanding aspect and success for azeotropic separation (Xiangyi 2007).

Hydrophilic polymer membranes which are extremely used for pervaporation dehydration (Liu *et al.* 2005, Nam *et al.*1999) contain polar functional groups such as hydroxyl (-OH), amino (-NH_2), carboxyl (-COOH), and carbonyl (-CO) groups (Xiangyi 2007). These hydrophilic materials have high potential for dehydration because of relatively large differences between molecular size and polarity of an organic solvent and water (Bruggen and Luis 2015). Cellulose acetate as a hydrophilic polymer (Rajesha *et al.* 2016) (with a functional group of hydroxyl (-OH)) and nylon 66 as a mildly polar polymer (with a functional group of amide (-CONH)) can be two suitable polymer materials in order to blend for dehydration application.

In pervaporation, a liquid feed is directly placed in contact with one side of a pore-free membrane (Xiangyi 2007). The solution–diffusion model is greatly used to explain the transport through pervaporation membranes (Wang *et al.* 2013). Net mass transport of species occurs in a three-step process which encompasses sorption of the permeant on the membrane, diffusion of the permeant in the selective membrane and finally desorption of the permeant on the other side of the membrane (Tock *et al.* 1974). It is noteworthy that, the desorption step, through applying a vacuum pump or an inert gas on the downstream side of the membrane, is usually completed fast compared with the

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sorption and diffusion which are considered as rate controlling steps (Tock *et al.* 1974). Put another way, it assumes that the flux and separation factor are governed by the differences in diffusivity and solubility of the feed components permeating through the membrane (Wang *et al.* 2013). Diffusivity as a kinetic parameter is dependent on a number of physical factors such as the size and geometry of penetrant molecules, the motion of molecules and polymer chains, the available space between polymer chains, and the interactions among penetrants and also among polymeric membrane and penetrants (Wang *et al.* 2013, Crespo and Brazinha 2015). On the other hand, solubility as a thermodynamic parameter is dependent on the physicochemical character of the permeating components and the membrane material and a special affinity and/or interaction among them (Wang *et al.* 2013, Crespo and Brazinha 2015). In addition, polarity is another important factor for solubility of the permeating species through the membrane (Wang *et al.* 2013). The solubility can be qualitatively shown by solubility and interaction parameters (Xiangyi 2007, Wang *et al.* 2013) which are an approximate indicator for the description of interaction between single component and the polymer material, but they are not a precise estimate of interaction between binary mixture and the polymer material (Guo 2007).

Due to the swelling and plasticization of membrane and coupling effects in steps of the sorption and diffusion; the process of transferring the component in pervaporation is much more complex compared to that in other membrane separation methods such as gas separation (Villalueng *et al.* 2003).

Coupling phenomenon described based on thermodynamic and kinetic aspects can cause some unexpected behavior. For instance, one species, due to effect of other species, can flow without or even opposite to its own driving force (Goethaert *et al.* 1993, Qiao *et al.* 2005).

It is apparent that the extent of sorption and diffusion of components, interactions between them as well as the degree of swelling and free volume of the polymeric membrane are significantly dependent on feed temperature, concentration, and other kinds of operational parameters (Guo 2007). Accordingly, it is imperative to say that the favorable and efficient performance of pervaporation is expected to obtain under optimum operating conditions (Guo 2007).

Sajankumarji Rao *et al.* (2014) studied the effect of feed concentration in the dehydration of IPA using sodium alginate / hydroxyl propyl cellulose blend membrane. They found that the total flux increased with augmenting the feed water amount while the selectivity decreased due to the increased swelling. Mao *et al.* (2010) reported the effect of feed temperature and concentration on separation of IPA from water using a cellulose/N-methylmorpholine-N-oxide membrane and achieved excellent separation factor and acceptable flux via control of operation conditions.

The membranes based on CA have been already used for IPA pervaporation dehydration by others. Nevertheless, their sorption and pervaporation behavior in different operating conditions, especially in different feed concentrations for an aqueous solution containing IPA has investigated very limited. In the present research, the CA/NYL66 (95/5) blend membrane was used for optimizing the conditions of pervaporation experiments. The effect of feed temperature and concentration, as well as membrane thickness was investigated on the separation performance.

2. Materials and methods

2.1 Materials

Cellulose acetate (CA, molecular weight of about 30000 g/mol, 39.8 wt% acetyl content) and nylon66 (NYL66, *molecular weight* of repeat unit 262.35 g/mol) supplied by Sigma-Aldrich (USA) were employed as the polymer matrix. Analytical grade and high purity formic acid (FA) purchased from Merck (Germany) was used as the solvent. Isopropanol (IPA) with a purity of 97-99% wt. % obtained from Merck (Germany) was used without further purification for feed preparation.

2.2 Membrane preparation

In this study, the CA/NYL66 (95/5) membrane was prepared using solvent evaporation method. For this purpose, CA/NYL66/FA solution was prepared while concentration of total polymer was adjusted to be 10 wt. % and the selected weight ratio of CA/NYL66 was 95/5 since this ratio had the best pervaporation performance in dehydration of IPA.

At first step of the solution preparation, CA was dissolved homogeneously in FA as the solvent. Then, NYL66 was added to the solution which was stirred for 10 hr in temperature of 35°C.

After degassing, the polymer solution was poured onto a mirror and spread in different thicknesses by a casting knife. In the third step, the mirror was placed in an oven for 24 hr at 40°C, whereupon the solvent evaporated. Finally, the mirror was placed inside a water container and the membrane was carefully removed from the mirror surface. To completely dry the membrane from the remaining solvent and water droplets, it was placed in the oven for 6 days at 40°C.

2.3 Scanning electron microscopy (SEM)

Visual information of the cross sectional morphology of the prepared membranes was obtained using a LEO 1450 VP SEM (Germany, secondary electron (SE) detector, vacuum pressure1.33E-6 mbar, and the maximum thickness of the coated layer 4nm). Preparation of the samples was performed by breaking the membranes in liquid nitrogen, pasting them on a metal base and then coating them with a thin layer of gold. Breaking the membranes in liquid nitrogen is done to avoid damage to their cross-section and to obtain clear images of them.

2.4 Sorption and swelling experiments

To measure the extent of sorption and swelling, after complete drying of membrane (for 6 days in the oven at 40°C), at first, the membrane was weighted using a digital

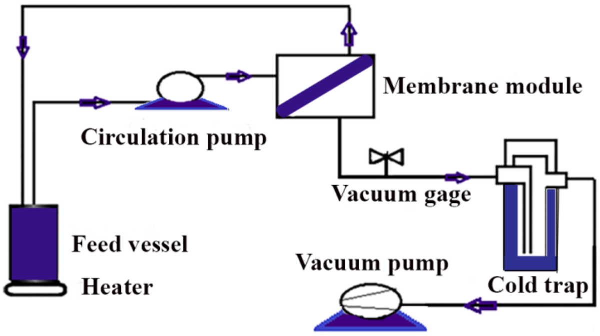


Fig. 1 Schematic diagram of pervaporation set up

scale with accuracy of 0.001 g (M_D). Thereafter, it was soaked in the feed solution for 24 h and then was removed from the solution. The droplets on its surface were carefully cleaned and its weight was immediately determined (M_S) once again. This process was repeated until the M_S attained equilibrium state as indicated by constant mass. The degree of overall sorption (swelling) was calculated using Eq. (1) (Rajineekanth *et al.* 2017)

$$DS(\%) = \frac{(M_S - M_D)}{M_D} \times 100 \quad (1)$$

The sorption tests were carried out over the whole concentration range of 0–100 wt. % IPA in feed and four levels of feed temperature 30, 40, 50, and 60 °C.

2.5 The extent of the affinity between the membrane and the solvents

The affinity of a pure solvent to a polymeric membrane in the equilibrium sorption can be expressed by a binary interaction parameter (χ_{ip}) calculated using the equation derived from the Flory-Rehner theory (Eq. (2)) (Park *et al.* 1998).

$$Ln(1-\varphi_p) + \varphi_p + \chi_{ip} = 0 \quad (2)$$

where φ_p is the volume fraction of the polymeric membrane in a liquid/membrane mixture which can be calculated from an equilibrium sorption experiment. According to this theory, a decrease in the binary interaction parameter exhibits an increase in the affinity between the solvent molecules and the polymeric membrane (Unlu and Hilmioglu, 2016a).

2.6 Pervaporation experiments

A schematic of the pervaporation laboratory set-up is illustrated in Fig. 1. The prepared membranes were characterized by dehydration of aqueous IPA solution using the set-up with an effective membrane area of 19.63 cm^2. A liquid pump was used with a flow rate of 1.6 L/min in order to circulate the feed and deliver it into the membrane module. The feed mixture was contacted with the membrane in the upper part of the module and the vapor permeated from the bottom of the module, which was under vacuum (at 13 mbar), was entered into the cold trap and liquefied.

The mass of permeate collected in cold trap was measured by an electronic balance with 0.001g accuracy. Moreover, the feed and permeate concentration was determined applying an ATAGO refractometer (Japan) and using a calibration curve of concentration versus refractive index.

Generally, the performance of pervaporation separation is evaluated by calculating the parameters of flux (J), separation factor (β) and pervaporation separation index (PSI) using Eqs. (3)-(5) (Gao *et al.* 2017)

$$J = \frac{m}{A.t} \quad (3)$$

$$\beta = \frac{(Y_i / Y_j)}{(X_i / X_j)} \quad (4)$$

$$PSI = J(\beta - 1) \quad (5)$$

where m is the total mass of permeate, A is the effective membrane area (m^2), and t is the operating time for collecting the permeate (h). Additionally, X and Y represent the weight fraction of the relevant component in the feed and permeate, respectively. The indices i and j refer to the desired (which should be separated) and second component, respectively.

Pervaporation experiments were done in four levels of membrane thickness 24.62, 22.03, 14.19, and 7.37 μm, feed concentration 70, 80, 87.9, and 95 wt. % IPA, and feed temperature 30, 40, 50, and 60 °C. In order to investigate repeatability, each pervaporation test was performed three times and the standard deviation was calculated.

3. Results and discussion

3.1 Effect of membrane thickness

The SEM images of the cross section structure of the membranes are presented in Fig. 2. As it can be seen, the thickness of the membranes is in the range of 24.62- 7.37 μm. The thicknesses were measured at four different regions of membrane, and the standard deviation was less than 3%. The experiments were performed applying a feed with a constant concentration of 80 wt. % IPA at 30°C on the membranes with different thicknesses. As can be observed from Table 1, with decreasing in the membrane thickness, IPA flux and its concentration in permeate were increased, while water flux was enhanced and its concentration was slightly decreased on the permeate side. Moreover, by declining the thickness of the membrane, a gradual increase of total flux was observed from 108 to 505

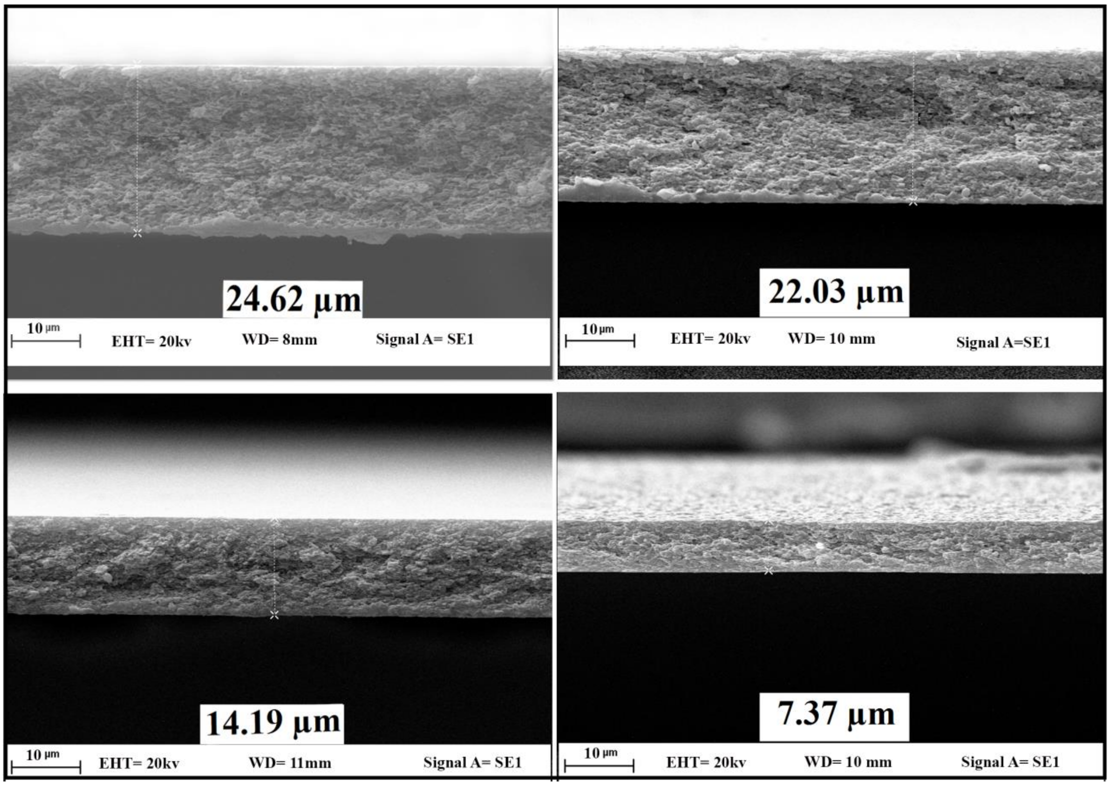


Fig. 2 Cross-sectional images of the CA/NYL66 (95/5) blend membrane with different thicknesses

Table 1 variations of different parameters with respect to membrane thickness (80 wt. % IPA concentration, feed temperature 30 °C). Standard deviation < 6% for flux and < 3.5% for permeate concentration.

Membrane thickness (µm)	24.62	22.03	14.19	7.37
Water concentration in permeate (wt.%)	99.80	98.63	97.70	96.03
IPA concentration in permeate (wt.%)	0.20	1.37	2.30	3.97
Water flux g/(m^2.h)	107.78	132.16	242.30	484.95
IPA flux g/(m^2.h)	0.22	1.84	5.70	20.05
Total flux g/(m^2.h)	108	134	248	505

g/(m^2.h). In fact, diffusion which is the determining stage in the transfer rate in pervaporation, is enhanced by decreasing the thickness of the membrane due to reduced resistance for mass transfer. In other words, with the pass of feed molecules through a short pathway to the permeate side, the diffusion can be high and accordingly, the flux increases (Sridhar *et al*. 2006).

According to Fig. 3, by decreasing the thickness in the range of 24.62-7.37 µm, the separation factor was decreased from 1952 to 97. Indeed, in membranes with high thicknesses, the passage of the interacting and smaller sized water molecules through the membrane is further, which leads to improved selectivity (Smitha *et al.* 2006). PSI values in Fig. 3 presents the best separation performance is obtained in the thickness of 24.62 µm.

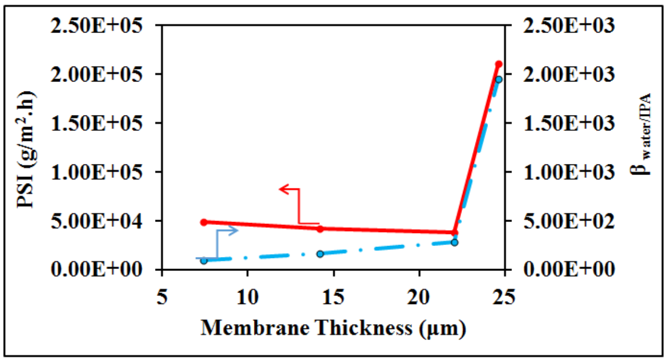


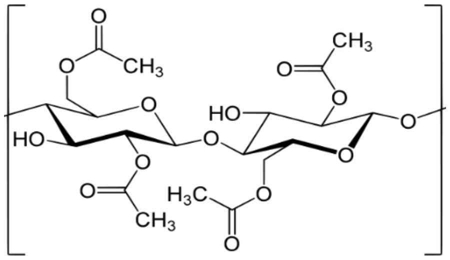
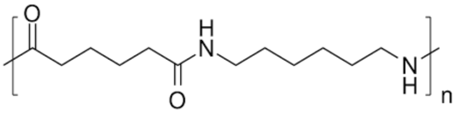
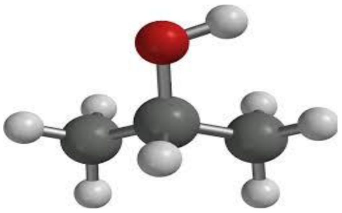
Fig. 3 Separation factor and PSI values versus membrane thickness (80 wt. % IPA concentration, feed temperature 30 °C). Standard deviation <4% for separation factor and <6% for PSI

3.2 Effect of feed concentration

3.2.1 Sorption

The sorption data for the synthesized membrane with a constant thickness of 24.62 µm at 30°C are presented in Fig. 4 for various concentrations of IPA in the feed. According to this Figure, pure component sorption experiments indicated that the DS values for pure water and IPA were 4.8 and 12.4%, respectively.

Table 2 Chemical structure and values of solubility parameter (δ, MPa$^{1/2}$), density (ρ, g/cm^3), and molecular volume (υ, A$^{o\,3}$) (Appaw *et al*. 2007, Kass *et al*. 2012, Hansen 2012, Qiao and Chung 2005)

Polymer/ Molecule	δ_d [a]	δ_p [b]	δ_h [c]	δ_t [d]	ρ	υ	Chemical structure
CA	18.6	12.7	11	25.1	1.26	-	
NYL66	17.2	9.9	16.5	25.8	1.14	-	
IPA	15.8	6.1	16.4	23.5	0.78	127	
Water	15.5	16.0	42.3	47.8	1	30	

[a]Dispersion solubility parameter; [b] Polar solubility parameter; [c] Hydrogen bonding solubility parameter; and [d]Total solubility parameter

The interaction parameter of the membrane with IPA and water was calculated using the results of pure sorption experiment and density of pure components and polymers by applying Eq. (2). Lower interaction parameter between the membrane - IPA (χ_{ip}= 1.38) than the membrane - water (χ_{ip}= 2.16), and also the proximity of total solubility parameter of IPA with both CA and NYL66 polymers (Table 2), confirms higher affinity between IPA molecule and the prepared membrane. In fact, IPA molecules and the blend membrane include both polar functional group (O-H in IPA, N-H in NYL66, and O-H in CA) and nonpolar segment (the carbon chain in IPA and the main polymer chain of NYL66 and CA) which leads to an increase in affinity of IPA and the blend membrane and promotes DS values.

Therefore, only with respect to these results, the blend membrane has more affinity toward IPA than water. It should be noted that the interaction and solubility parameters, as discussed in the introduction section, don't estimate precisely the interaction between feed mixture and membrane material. (Guo 2007).

According to Fig. 4, the DS curve almost is close to ideal behavior in the range of 0-60 and 90-100 wt. % IPA. Although it passes through a maximum value at 70 wt. % IPA in the range of 60-90 wt.% (higher than ideal state) that that can interfere by the greater interaction between water and IPA molecules in comparison with the interaction between water–water and IPA-IPA molecules.

3.2.2 Pervaporation performance

Pervaporation data as a function of IPA concentration in the feed were evaluated for the blend membrane with a thickness of 24.62 μm at 30°C, and the results are displayed in Table 3 and Fig. 5. The concentration of feed mixture affects the diffusion and solubility of each component due to differences in the chemical nature and molecular size of the components (Guo 2007). In fact, in a binary feed mixture, the composition of the components has a great effect on the interaction of membrane-components, as well as component-component, which can be significantly involved in the value of flux and selectivity (Guo 2007). Although the results of pure sorption experiments of IPA and water indicated that IPA should be considered as the component preferentially permeates the membrane, in the presence of both compounds in the feed, it was the water which passed through the membrane preferably. According to Table 3, a significant percentage of the permeability was related to water molecules in all studied concentrations and the blend membrane was water selective. This behavior may be explained by coupling effect. Thermodynamically, the sorption of one component within membrane changes due to mutual interactions of the components, as well as between components and the membrane (Qiao *et al*. 2005). Villegas *et al*. (2015) also obtained similar anomalous results of sorption and pervaporation tests in methanol-water mixture by poly (3-hydroxybutyrate) membranes. They explained that this behavior can be related to the

Table 3 Variations of different parameters with respect to IPA concentration in feed (feed temperature 30 °C, membrane thickness 24.62 μm. Standard deviation < 5% for flux and <3% for permeate concentration

IPA concentration in feed (wt.%)	70	80	87.9	95
Water concentration in permeate (wt.%)	99.90	99.80	99.69	99.39
IPA concentration in permeate (wt.%)	0.10	0.20	0.31	0.61
Water flux g/(m2.h)	139.86	107.78	84.74	54.66
IPA flux g/(m2.h)	0.14	0.22	0.26	0.34
Total flux g/(m2.h)	140	108	85	55
Normalized total flux g.μm/(m^2.h)	3446.80	2658.96	2092.70	1354.10

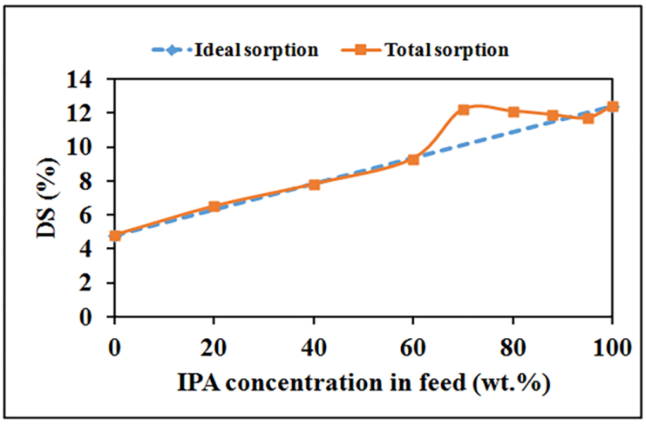


Fig. 4 DS values versus the IPA concentration in feed (membrane thickness 24.62 μm, feed temperature 30 °C)

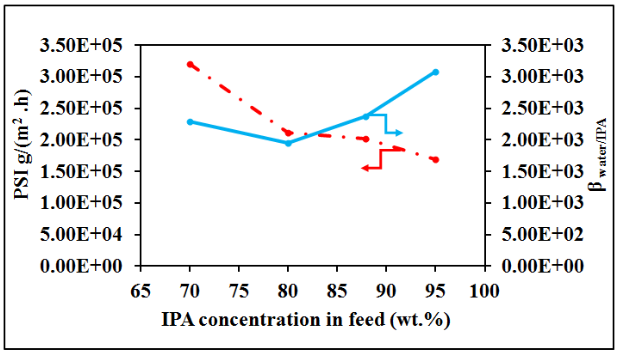


Fig. 5 Separation factor and PSI values as a function of IPA concentration in feed (feed temperature 30 °C, membrane thickness 24.62 μm). Standard deviation <3% for separation factor and <3.5% for PSI.

stronger interaction between water- methanol molecules compared to water–water and methanol – methanol molecules, causing cluster structures formation and alcohol presence as plasticizer increased water sorption and consequently water permeation. Mulder *et al.* (1986) reported that the preferential sorption in the membrane cannot be estimated from the results of overall sorption experiments.

Additionally, according to Table 2, water molecules have smaller size and shape compared to IPA molecules which leads to faster diffusivity of water molecules through the membrane than IPA molecules (Wang *et al.* 2013). Furthermore, water molecule possesses the most hydrogen bonding solubility parameter (Table 2) and the highest hydrogen-bonding ability, which suggests the strong interactions between the membrane and water molecules. Therefore, the mobility of water molecules significantly enhances while the motion of IPA molecules decreases due to cluster formation.

With increasing IPA concentration in the feed, according to Table 3, the total flux was declined from 140 to 55 g/(m^2.h). Reduction of overall flux can occur due to decrease of the driving force for passing of water molecules, which form major part of permeate flow in the blend membrane. Moreover, the increase of IPA concentration in the feed enhanced IPA concentration and decreased water concentration in permeate.

The increase of IPA concentration in feed significantly exceeded its increase in permeate flow; so that, the highest value of separation factor was obtained in feed 95 wt.% IPA (Fig. 5). It is worth noting that in the dehydration of IPA, the azeotropic point of the water-IPA mixture was successfully broken down by the blend membrane. With respect to the results obtained for PSI in Fig. 5, the best membrane separation performance was obtained for the feed with IPA concentration of 70 wt.%, having the highest water concentration and flux. Therefore, the feed with 70 wt.% IPA is selected to evaluate the effect of feed temperature on the pervaporation dehydration.

3.3 Effect of feed temperature

3.3.1 Sorption

The effect of feed temperature on DS and pervaporation output parameters of the membrane is presented in Table 4. The variation in the feed temperature causes a change in the free volume of the membrane and its distribution in the membrane (Guo 2007). As expected, the extent of DS was enhanced with increasing the temperature due to the increased thermal motion and expansion of polymeric chains (Guo 2007).

3.3.2 Pervaporation performance

The variation of flux and concentration of permeate with feed temperature is given in Table 4. When the feed temperature varied from 30 to 60°C, the partial flux of both components was increased on the result of increase in the swelling and free volume of the membrane. With any rise in the feed temperature, the swollen membrane layer would allow to pass further IPA molecules along with water molecules and the concentration of IPA was increased meanwhile the concentration of water was decreased in permeate.

Table 4 Variation of DS values and different parameters with respect to feed temperature (70 wt. % IPA concentration, membrane thickness 24.62 μm). Standard deviation < 5% for flux and <5% for permeate concentration.

Feed temperature (°C)	30	40	50	60
DS (%)	12.20	13.50	14.82	17.39
Water concentration in permeate (wt.%)	99.90	99.85	99.59	99.28
IPA concentration in permeate (wt.%)	0.10	0.15	0.41	0.72
Water flux g/(m^2.h)	139.86	262.60	328.65	466.64
IPA flux g/(m^2.h)	0.14	0.40	1.35	3.36
Total flux g/(m^2.h)	140	263	330	470
Normalized total flux g.μm/(m^2.h)	3446.80	6475.06	8124.60	11571.40

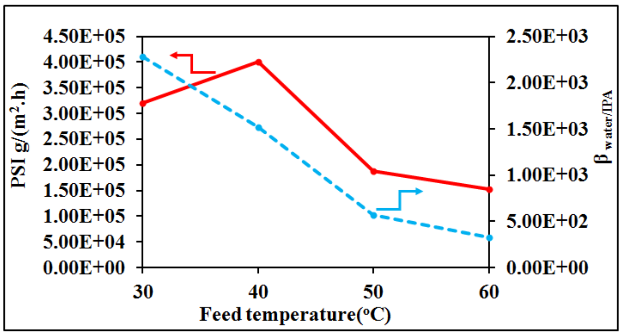


Fig. 6 Separation factor and PSI values as a function of feed temperature (70 wt. % IPA concentration, membrane thickness 24.62 μm). Standard deviation <5% for separation factor and <3% for PSI.

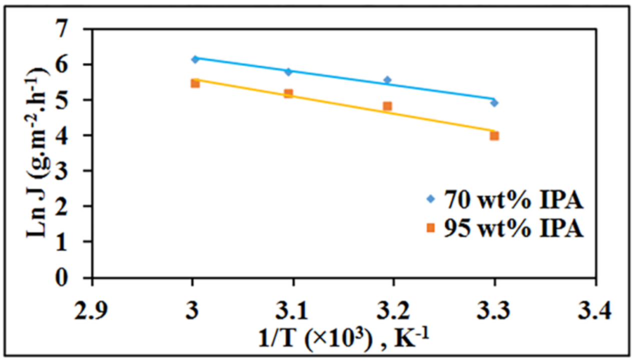


Fig. 7 Arrhenius graph of total flux versus feed temperature at two different concentrations of IPA

According to Table 4 and Fig. 6, with increasing the temperature, the overall flux was increased and the separation factor was decreased. The increase of feed temperature would accelerate the movement of the molecules and diffusion rate which consequently leads to increase of flux. On the other hand, due to the membrane swelling at higher temperatures, a greater percentage of IPA molecules along with water molecules passed through the membrane, and thus the separation factor decreased.

Figure 6 also illustrates the PSI values in terms of feed temperature. According to PSI results, the best pervaporation performance was obtained at 40°C.

The Arrhenius relationship (Eq. 6) presents the effect of temperature on flux (Huang *et al.*1999)

$$J = J_0 \exp(-\frac{E_p}{RT}) \qquad (6)$$

where J, J_o and E_p are the flux, pre-exponential constant and apparent activation energy for permeation, respectively. Furthermore, R and T are the *gas constant* and temperature in Kelvin, respectively. In fact, the E_p being sum of the activation energy of diffusion and heat of sorption can be determined from plot of the Ln (J) in terms of 1/T.

The Arrhenius plots of total flux versus the reciprocal of the feed temperature were represented in Fig. 7 at two different concentrations of 70 and 95 wt.% IPA. With increasing the content of IPA in the feed from 70 to 95 wt.%, the apparent activation energy of the membrane rises from 32.54 to 42.43 kJ/mol indicating by increasing the concentration of IPA in the feed, passing through the membrane requires more energy (Qiao *et al*. 2005).

4. Conclusion

CA/NYL66 (95/5) blend membrane was evaluated in overall sorption and pervaporation tests for IPA/water mixture. It was found that:

- The preferential transfer of components in pervaporation experiments cannot be deduced from the results of the overall sorption.
- Pervaporation tests in different levels of feed temperature, IPA concentration in feed, and membrane thickness indicated that the prepared membrane was water selective in all tested conditions.
- By increasing the membrane thickness, the flux was decreased and the separation factor was increased.
- With increasing the IPA concentration in feed, an increment in the separation factor and decrement in total flux were obtained.
- As feed temperature increased, the separation factor was declined and flux was enhanced.
- The highest PSI (almost 400476 g/(m^2.h)) was related to the feed with 70 wt.% IPA at temperature of 40°C with a membrane thickness of 24.62μm.

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