

Estimation of Permeance and Activation Energy for Separation of Ethanol from Ethanol/Water Binary Mixture by Pervaporation with PDMS Membrane

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Abstract- In this study pervaporation experiments were conducted to study the permeation behavior of binary ethanol-water mixture using polydimethyl siloxane membrane. The effects of feed flow rate, feed composition, and feed temperature were examined on permeation flux and separation factor. On increasing ethanol concentration in the feed, permeation flux was enhanced and separation factor was reduced; whereas permeation flux and separation factor both were increased on increasing the feed temperature and feed flow rate.

The solution-diffusion model (combination of Henry's law of sorption and Fick's law of diffusion) is used to predict the solution-diffusion-desorption steps with a driving force term and a permeation term. The activity coefficient of ethanol and water in the binary mixture was calculated with UNIQUAC model. The activation energy of ethanol permeation was calculated by Arrhenius relationship. Permeance of ethanol varied from 10.96 to 11.62 g/m².h.bar, on increasing the ethanol concentration from 5-20 vol%. The activation energy for ethanol permeation was found to vary from 5.77 to 4.13 kJ/mol when the ethanol concentration in the feed was increased from 5% to 20 vol%.

Keywords: PDMS membrane, Pervaporation, Solution- Diffusion model, Activation energy, UNIQUAC (UNIversal-QUAsi-Chemical)

I. INTRODUCTION

Ethanol is an alternative green fuel that has received much attention over the last few decades [1]. Ethanol production from fermentation and the effects of its various by-products have been widely investigated [2]. Compared with traditional distillation, membrane separation technology has emerged as an attractive method for the separation of ethanol-water mixtures [3]. For example, pervaporation (PV) separates ethanol-water mixtures efficiently at low operational costs, as it is operated at low feed temperatures and pressures; moreover no additional chemicals are necessary for separation [4], [5].

Pervaporation is a membrane process, in which a phase change takes place across the membrane. The liquid mixture is brought in contact with one side of the membrane and the

permeate is removed as a low pressure vapor on the other side. The driving force for mass transport over the membrane is the chemical potential gradient created by applying a vacuum pump on the permeate side to lower the partial pressure of the feed liquid and thus lowering the chemical potential of the permeate stream on the downstream side [6].

One of the most applied polymeric membranes for ethanol-water separation is polydimethylsiloxane (PDMS) [2]. PDMS exhibits high selectivity and permeability towards ethanol because of its flexible structure and therefore, is preferred for the removal of ethanol from water [7]. It is well known that the pervaporation performance is not only dependent on the properties of membranes, but also the process conditions such as feed concentration, temperature, permeate pressure and feed flow rate [8]. Though a number of researchers have studied the effect of operation conditions on pervaporation performance in terms of flux and separation factor; correlating separation performance with temperature and intrinsic separation characteristics of commercial PDMS membrane remain to be an underexploited area of research [9]. Verhoef et al. (2008) demonstrated the importance of taking the combined effects of activity coefficient and saturation vapour pressure into consideration while analysing the temperature effect on a hydrophobic nanofiltration membrane (SolSep 3360) performance for separation of ethanol from ethanol-water mixture [10]. The operating parameters affect the apparent membrane performance of membranes in pervaporation. Without using the permeance as a membrane performance parameter, these effects may be simply overlooked from the flux plots. Permeance is recommended when comparing the separation performance of the membranes, since it allows distinguishing the effect of the nature of the membrane and the operating conditions [11].

The mechanism of mass transfer of permeate across non-porous polymeric membrane include two stages: sorption of liquid, and its diffusion through the free volume of the polymeric material [8]. Many models have been proposed to predict the mass transfer process, such as solution diffusion

model, pore flow model, pseudo phase change solution-diffusion model, and etc [12], [13]. Among these, solution-diffusion model is most widely used in describing pervaporation transport including preferential sorption, diffusion and evaporation steps [14], [15]. Modeling of the process of pervaporation is important not only for a quantitative understanding of the dependence of fluxes on the process parameter, but is also useful in design calculations for a pervaporation module and further optimization of the overall process [16].

Keeping this in mind, this study was designed to investigate the effect of temperature in terms of activity coefficient, saturation vapor pressure, permeance and activation energy on the performance of a commercial PDMS membrane to remove ethanol from aqueous solution by pervaporation. Pervaporation experiments were carried out to study the separation factor and mass transport flux of the system under varying operation conditions of feed flow rate, feed concentrations and feed temperature.

II. THEORY

The permeation flux through the membrane was calculated by the Fick’s law [17]:

$$J_i = \frac{m_i}{At} \quad (1)$$

where J_i is the flux of component i (g/m^2h); m_i is the mass of component i in permeate (g); A is the effective membrane area (m^2) and t is the permeation time (h).

The separation factor, α is expressed as follows [17]:

$$\alpha = \frac{y_e/y_w}{x_e/x_w} \quad (2)$$

where y_e and y_w are the weight fractions of ethanol and water in the permeate and x_e and x_w are the weight fractions of ethanol and water in the feed, respectively. The flux and separation factor are known to be strongly dependent on the operation conditions. The membrane permeance, Q can be calculated from the flux, J as follows [18]:

$$J_i = \frac{S_i D_i}{\delta} (p_{iF} - p_{iP}) = Q_i (p_{iF} - p_{iP}) = Q_i \Delta p_i \quad (3)$$

where Q_i is the permeance or pressure normalized flux which includes the sorption coefficient (S_i , thermodynamic part) and diffusion coefficient (D_i , kinetic part) of the component i in the membrane and the thickness of the membrane active layer (δ). p_{iF} and p_{iP} are the pressure of the component i in the feed (F), and permeate (P), respectively. The driving force of diffusion of component i , (Δp_i), can be considered either by the partial pressure, chemical potential difference or activity between feed and permeate side of the membrane. The driving force, Δp_i can be identified with the process parameters; feed concentration, feed temperature, and permeate pressure (P_p); and is expressed as follows:

$$\Delta p_i = x_{iF} \gamma_i P_{iF}^0 - y_{iP} P_p \quad (4)$$

The vapor pressure of the component i at feed P_{iF}^0 in mmHg is calculated with the Antoine equation and it was converted to bar in this work.

$$\ln P_{iF}^0 = \left(A - \frac{B}{t+C} \right) \quad (5)$$

where t is feed temperature in Celsius degree.

The activity coefficient of component i (γ_i) in the mixture can be calculated with models such as NRTL, UNIQUAC, Wilson, Van Laar, and etc. In this study, the activity coefficients for the ethanol and water were calculated using UNIQUAC model. PHP (Hypertext Preprocessor Language) program was used for the calculations. The Antoine constants (A , B , C) for vapor pressure and parameters used for the calculation of activity coefficients with UNIQUAC model are presented in Table I [15].

TABLE I. Parameters Used for the Calculation of Activity Coefficients and Vapor Pressure of the Components

Comp.	r	q	A_{ij}	A_{ji}	A	B	C
Ethanol	2.1055	1.972	6.5974	319.8115	8.2371	1592.864	226.184
Water	0.95	1.4			8.1962	1730.630	233.426

UNIQUAC model separates the activity of the components in the membrane into combinatorial and residual parts [19]:

$$\ln \gamma_i = \ln \gamma_i^C + \ln \gamma_i^R \quad (6)$$

$$\ln \gamma_j = \ln \gamma_j^C + \ln \gamma_j^R \quad (7)$$

Activity coefficients can be determined by [1]:

$$\ln \gamma_i^C = \ln \frac{\varphi_i}{x_i} + \frac{z}{2} q_i \ln \frac{\zeta_i}{\varphi_i} + \varphi_j \left(I_i - \frac{r_i}{r_j} I_j \right) \quad (8)$$

$$\ln \gamma_i^R = -q_i \ln (\zeta_i + \zeta_j \tau_{ji}) + \zeta_j q_i \left(\frac{\tau_{ji}}{\zeta_i + \zeta_j \tau_{ji}} - \frac{\tau_{ij}}{\zeta_j + \zeta_i \tau_{ij}} \right) \quad (9)$$

$$\ln \gamma_j^C = \ln \frac{\varphi_j}{x_j} + \frac{z}{2} q_j \ln \frac{\zeta_j}{\varphi_j} + \varphi_i \left(I_j - \frac{r_j}{r_i} I_i \right) \quad (10)$$

$$\ln \gamma_j^R = -q_j \ln (\zeta_j + \zeta_i \tau_{ij}) + \zeta_i q_j \left(\frac{\tau_{ij}}{\zeta_j + \zeta_i \tau_{ij}} - \frac{\tau_{ji}}{\zeta_i + \zeta_j \tau_{ji}} \right) \quad (11)$$

where: I_i , parameter of component $i = \frac{z}{2} (r_i - q_i) - (r_i - 1)$

z , coordination number = 10

ζ_i , area fraction of component $i = \frac{q_i x_i}{\sum_j q_j x_j}$

φ_i , volume fraction of component $i = \frac{r_i x_i}{\sum_j r_j x_j}$

τ_{ij} , parameter of interaction between components i and $j = \exp\left(-\frac{A_{ij}}{RT}\right)$

where A_{ij} and A_{ji} are UNIQUAC parameters between component i and j . q_i , q_j , r_i and r_j are molecular surface area and molecular van der Waals volume for component i and j , respectively. UNIQUAC parameters are given in cal/mol with gas constant, $R=1.9872$ cal/mol K and temperature, T in K [1].

The permeation flux depends on the feed temperature. The influence of the temperature on the permeance is described by the Arrhenius relation [20]:

$$Q_i = Q_{i,ref} \exp\left[\frac{E_{pi}}{R} \left(\frac{1}{T_{ref}} - \frac{1}{T_F}\right)\right] \quad (12)$$

The permeance of component i at the reference temperature $T_{ref} = 25$ and activation energy (E_{pi}) which includes the heat of adsorption and the activation energy for diffusion are calculated from experiments at various feed temperature [15]. The mass transfer for pervaporation is evaluated by the combination of equations, expressed as follows:

$$j_i = Q_{i,ref} \exp\left[\frac{E_{pi}}{R} \left(\frac{1}{T_{ref}} - \frac{1}{T_F}\right)\right] \cdot [x_{iF} \gamma_i P_{iF}^0 - y_{iP} P_P] \quad (13)$$

III. MATERIALS AND METHODS

A. Materials

Flat sheet PDMS membrane and membrane module were purchased from Pervatech, The Netherlands. The membrane has an effective thickness of 3-5 μm PDMS as top layer, support consisting of PET with thickness of 130 μm as the sub-layer assure the mechanical strength of the membrane and an intermediate UF membrane polyimide (PI) as the first membrane layer with thickness of 100 μm were used. Analytical grade ethanol was purchased from Fisher Scientific, Canada.

B. Membrane Swelling

Dry membrane pieces (approximately 0.3g each) were weighed and immersed in solutions of different concentrations of ethanol. The temperature was kept constant at 30°C by placing the flask that contains the membrane piece in an incubator. The membranes were removed from the solutions at fixed time intervals and the adhering solution drops were removed by wiping the membranes with tissue paper. The membranes were then weighed immediately and returned to the solution. This procedure was repeated until no further mass increase was observed as it indicated that sorption equilibrium had been reached. The ethanol concentrations of the solutions were varied from 0 to 100% ethanol and each experiment was repeated four times to ensure accurate results.

C. Equipment and Experimental Procedure

The schematic sketch of the experimental setup used in this study is shown in Fig. 1. The PDMS membrane with an estimated surface area of 89cm² was placed into the membrane module and equilibrated with ethanol for 24 hours. The prepared water and ethanol mixture was fed into the feed capacity tank (5l capacity) and it was circulated across the membrane module and back to the feed tank using the peristaltic feed pump at the desired feed flow rate in litre per hour (lph). Once the experiment began, the system was allowed to attain steady state before samples were collected. Steady state was achieved within 1 h. The feed solution was

maintained at a desired temperature with a heating blanket. A vacuum pressure of 5 ± 0.1 torr was applied on the permeate side by an oil vacuum pump. The permeating species was condensed in the condenser coil by using ethylene glycol as a coolant and collected in the bottles.

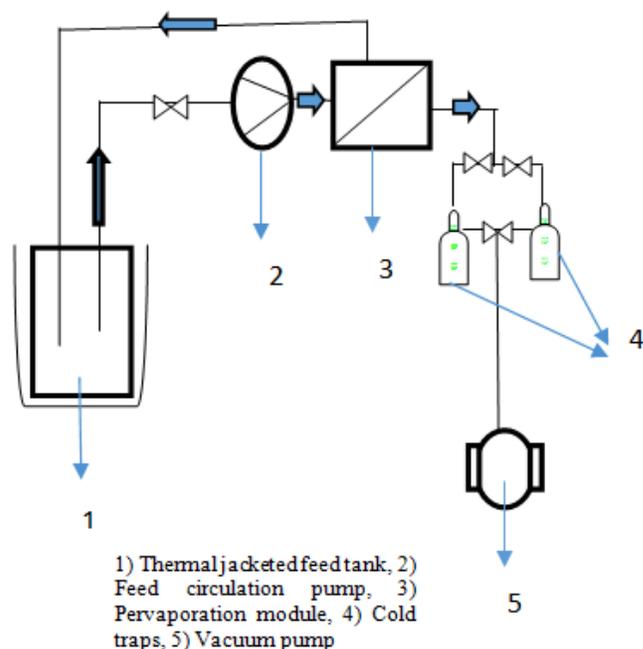


Fig. 1 Schematic diagram of pervaporation setup

D. Analytical Method

The amount of collected permeate was measured by using a mass balance and samples were taken out for ethanol concentration estimation and the average values were used for the parameter calculations.

Ethanol was analyzed by a gas chromatograph (Agilent 7890A series) equipped with a Stabilwax column (30m \times 250 μm \times 0.5 μm ; Restek Corp., USA) at 250°C, FID detector at 300°C, nitrogen at 23 psi pressure, and helium as a carrier gas.

IV. RESULTS AND DISCUSSIONS

A. Swelling Ratio of Membrane

One characteristic of pervaporation using polymeric membranes is that the membranes tend to swell. The feed liquid in contact with the membrane dissolves into it, which causes the membrane swelling. This swelling alters the membrane properties and leads to higher permeability and lower selectivity [21].

Results of the sorption experiments are shown in Fig. 2. Swelling equilibrium was reached within 5 minutes for ethanol-water mixtures. However, it took about 2 hours to reach swelling equilibrium when pure water was used. It was observed that swelling of the membrane is higher at higher ethanol concentration. The swelling ratio for pure water was

0.2 and for pure ethanol was 0.8, suggesting that the membrane has a higher affinity for ethanol than for water. Mohammadi et al. (2005) have also reported increase in swelling with increased ethanol concentration for PDMS membrane and degree of swelling of 0.5 at 5wt% ethanol [22]. Other researchers have also reported increased degree of swelling with increased ethanol concentration for PDMS membranes [1], [23].

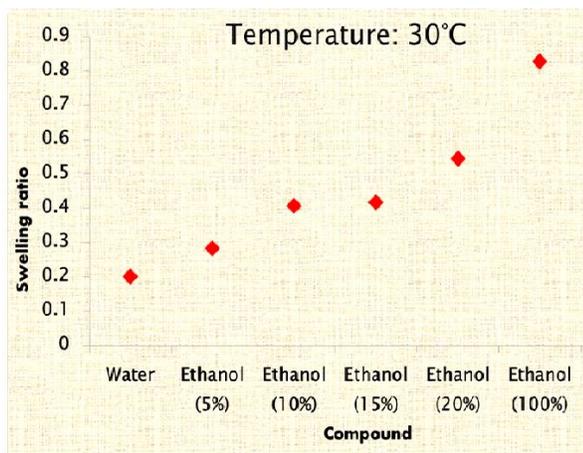


Fig. 2 Swelling ratio vs feed

B. Effect of Feed Flow Rate

The effect of feed flow rate was studied by carrying out experiments at 50 and 100 lph, keeping other experimental conditions constant at ethanol concentration: 5 vol %, temperature: 30°C, vacuum pressure: 5torr. The feed flow rate could not be increased above 100lph, as it was the maximum operating limit for the membrane module, as specified by the membrane supplier. The results are shown in Table II.

On increasing the flow rate from 50lph to 100lph, both total and ethanol fluxes were increased. The flux for ethanol, water and total flux increased from 51.6, 317.8, and 369.4 g/m².h to 77.6, 328.8, and 406.4 g/m².h, respectively, with increase in feed flow rate from 50 to 100 lph. The separation factor increased from 3.1 to 4.9, with increase in feed flow rate from 50 lph to 100 lph.

An increase in the feed flow rate decreases concentration polarization which causes increase the ethanol flux and the total flux, As a consequence, there is a better penetration of ethanol molecules, increasing the hydrophobic properties of the membrane and the process becomes more selective for ethanol [24]. Li et al. have also reported that with an increase in the feed flow rate both the total flux and the separation factor increase [25]. Sun et al. (2016) have also reported that with the increase in feed flow rate from 0.1 to 10 L/min, both flux and separation factor increased rapidly at first and then leveled off at 2 L/min for the reason of concentration polarization, using a self prepared PDMS/PES composite membrane [26].

TABLE II. Effect of Feed Flow Rate (Ethanol Conc.: 5 Vol %, Temperature: 30°C, Vacuum Pressure: 5torr)

Flow rate (lph)	Total Flux (g/m ² .h)	Ethanol Flux	Water Flux	Separation factor, α
50	369.4	51.6	317.8	3.1
100	406.4	77.6	328.8	4.9

C. Effect of Ethanol Concentration

The effect of ethanol concentration in the feed was studied by varying the ethanol concentration from 5-70 vol%, keeping other experimental conditions constant at temperature: 30°C, vacuum pressure: 5torr, feed flow rate: 100 lph. As shown in Fig. 3, on increasing the ethanol concentration from 5 to 70 vol%, both ethanol and water flux were increased. The flux for ethanol, water and total flux increased from 76.4, 328.8, and 405.2 g/m².h to 822.3, 489.9, and 1312.2 g/m².h, respectively, with increase in ethanol concentration from 5 to 70 vol%.

The effect of feed composition on separation factor is shown in Fig. 4. The highest separation factor (4.9) was achieved at the 5 vol% ethanol concentration and it was decreased to 1.1, when the ethanol concentration was increased to 70%.

An increase in the concentration of ethanol in the feed causes an increase in the flux of ethanol, increasing its concentration in the permeate stream. This phenomenon is explained as a result of the increased ethanol concentration improving the adsorption of this component to the membrane; consequently, the membrane becomes swollen, which increases permeability, but decreases selectivity [27]. Similar trend has been observed by Zhan et al. (2009) also, where they have reported the separation factor decreasing sharply with increase in ethanol concentration [28]. The obtained data indicated the PDMS membrane, used in this study was ethanol – selective at low ethanol concentrations.

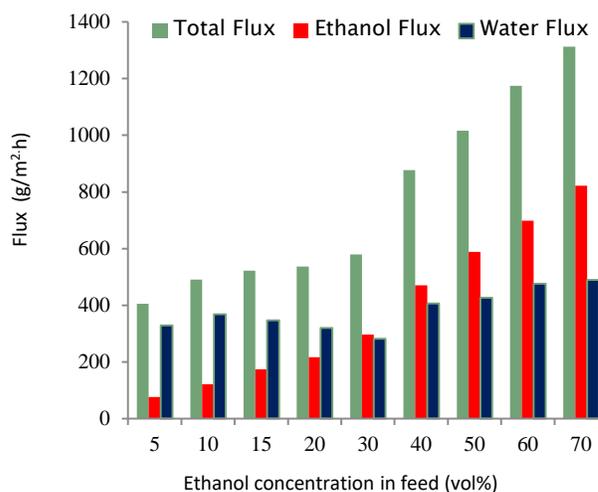


Fig. 3 Effect of feed composition on flux

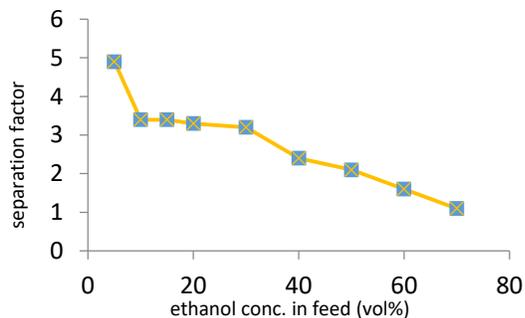
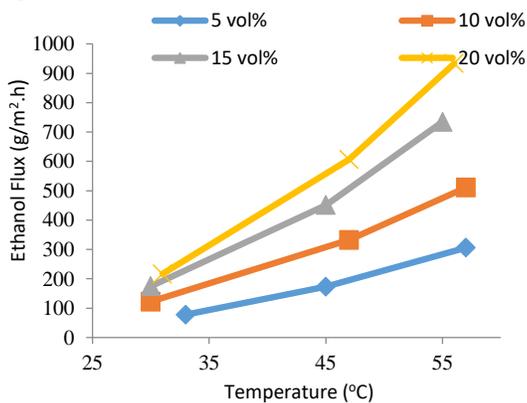


Fig. 4 Effect of feed composition on separation factor

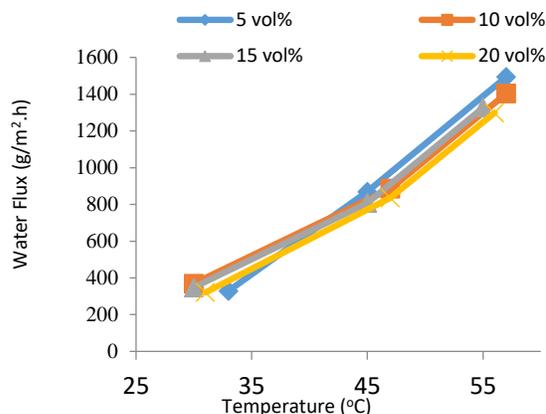
D. Effect of Feed Temperature

The effect of feed temperature was studied in the range of 30-55°C at the ethanol concentration of 5, 10, 15 and 20 vol% and vacuum pressure: 5 torr, feed flow rate: 100 lph. The temperature range selected for this study was low keeping in mind the temperature suitable for the ethanol production through fermentation. The ethanol flux, water flux, and total flux obtained in the study are shown in Fig. 5: a, b, and c respectively. On increasing the feed temperature from 30 to 55°C, ethanol flux, water flux, and total flux were enhanced from 77.6, 328.8 and 406.4 g/m².h to 306, 1493.5, and 1799.5g/m².h, respectively, at a feed ethanol concentration of 5 vol%. An increase in temperature causes an increase in the total permeate flux due to the increase of diffusion rate of individual permeating molecules by the free volumes produced because of the thermal motions of polymer chains. A higher temperature stimulates the driving force due to an increase in vapor pressure and the activity coefficient of the permeating species (and their chemical potentials) as temperature increases [17].

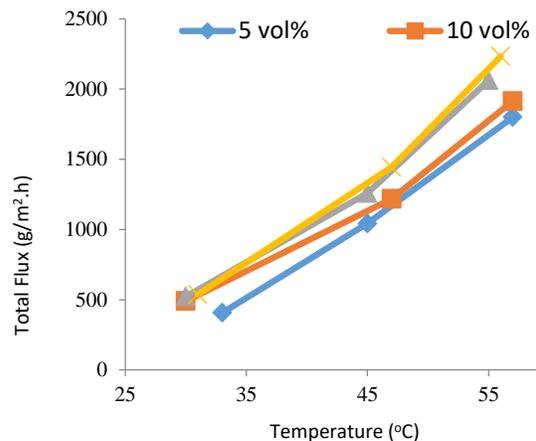
The effect of temperature on separation factor is shown in Fig. 6. The separation factor increased on increasing the temperature, and it was highest for the 5 vol% ethanol. The separation factor improved, from 4.9 to 5.5, on increasing the feed temperature from 30 to 55°C for 5 vol% ethanol feed. Similar trend for increase in the flux and separation factor have also been reported by Sun et al. [26], and Molina et al. [30].



a)



5 b)



5 c)

Fig. 5 Effect of temperature at different feed ethanol concentration on a) ethanol flux, b) water flux, and c) total flux (vacuum pressure: 5 torr, feed flowrate: 100 lph)

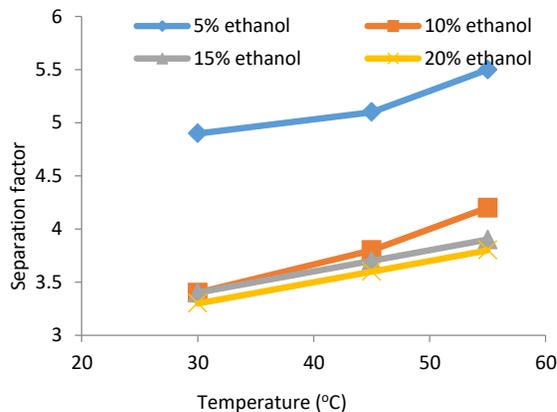


Fig. 6 Effect of temperature on separation factor

As permeance is defined as the flux divided by the driving force. The driving force combines two temperature-dependent factors γ_i and P_{iF}^0 , which are external factors outside the membrane. The activity coefficients of ethanol and water were calculated with the help of UNIQUAC equation for 5-20 vol% ethanol-water mixture at the temperature varying from

30-55°C. The results are shown in Table III. Vapor pressure of the ethanol and water were calculated at 30, 45 and 55 °C by using Antoine equation. The results are shown in Table IV. Both, the activity coefficients and vapor pressure of ethanol was found to be higher than the water.

TABLE IV. A Comparison of Ethanol and Water Vapour Pressure at Different Temperatures

Temp (°C)	Ethanol	Water
30	0.01004	0.0073
45	0.01417	0.00966
55	0.01746	0.01249

TABLE III. A Comparison of Ethanol and Water Activity Coefficients at Different Temperatures (Ethanol Concentration = 5, 10, 15 and 20 Vol%), Calculated with Help of UNIQUAC Equation

Temp. (°C)	5%		10%		15%		20%	
	Ethanol	Water	Ethanol	Water	Ethanol	Water	Ethanol	Water
30	4.49	0.0073	3.58	0.0068	2.96	0.0068	2.56	0.0069
45	4.35	0.0097	3.44	0.0101	2.85	0.0097	2.50	0.0101
55	4.28	0.0125	3.52	0.0125	2.87	0.0119	2.46	0.0122

The constants $Q_{i, ref}$ and E_{pi} of equation (12), were estimated by plotting $\ln Q_i$ versus the reciprocal of feed temperature. The ethanol permeance follows an Arrhenius function indicated by the nearly straight line as shown in Fig. 7. The permeance and activation energy of ethanol is presented in Table V. The activation energy of permeation, E_{pi} , is the sum of activation energy of diffusion (E_D) and the enthalpy of dissolution (ΔH) of the permeant [11]. E_D is generally positive, whereas ΔH is usually negative for the exothermic sorption process. Therefore, the positive value of activation energy implies that the value of positive E_D dominates over the negative value of ΔH . Or, it can be concluded that the temperature has greater effects on diffusion than sorption. Similar observation has been reported by Win et al. [15]. On increasing the ethanol concentration from 5 to 20 vol%, the activation energy was decreased from 5.77 to 4.13kJ/mol. A higher value of the activation energy indicates a more sensitive behavior towards temperature changes.

TABLE V. Permeance and Activation Energy for Ethanol at Different Feed Concentration

Ethanol Concentration, (vol%)	Permeance, (Q) (g/m ² .h.bar)	Activation Energy of permeation, (E _p) (kJ/mol)
5	10.96	5.77
10	11.18	4.72
15	11.49	4.28
20	11.62	4.13

V. CONCLUSIONS

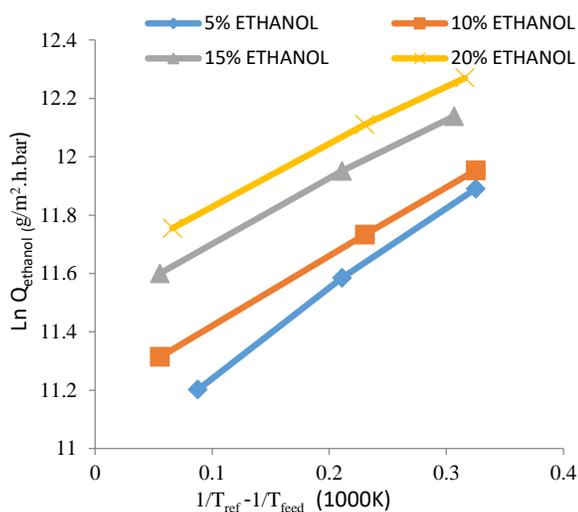
The degree of swelling for PDMS membrane was found to be higher for the pure ethanol, as compared to the water, suggesting the ethanol selectivity of the membrane. Increasing the feed flow rate has shown a positive impact on the flux, and separation factor. On increasing the ethanol concentration from 5 to 70 vol%, both ethanol and water flux were increased, whereas separation factor was decreased. On increasing the feed temperature from 30 to 55°C, both flux and separation factor were enhanced. A highest of ethanol flux, water flux, and total flux: 306, 1493.5, and 1799.5g/m².h were achieved, respectively, at a feed ethanol concentration of 5 vol% and 55 °C. Separation factor was found to be 5.5.

The ethanol permeance has shown increasing trend, on increasing the ethanol concentration, whereas activation energy has shown decreasing trend with increase in ethanol concentration.

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Fig. 7 Effect of feed temperature on ethanol permeance at different feed concentrations



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