

## PDMS/PSF 중공사 분리막의 시간 의존적 에탄올-물 분리 거동 연구

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### A Transient Separation Behavior of PDMS/PSF Hollow Fiber Membrane Modules for Ethanol-Water Mixtures

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**요약:** 알코올의 탈수를 위하여 에너지 다소비 공정인 증류 공정을 투과증발 막 공정으로 대체하려는 연구가 많이 진행되어 왔다. 대표적인 투과증발 막인 PDMS 분리막에 대한 시간의존적 분리 거동은 분리 메커니즘의 이해에 매우 중요하다. 따라서 본 연구에서는 50 wt% 에탄올-물 혼합용액에 대하여 50°C에서 막면적 1194 cm<sup>2</sup>인 PDMS/PSF 분리막 모듈의 시간의존적인 투과증발 분리 거동을 고찰하였다. 총 유속과 에탄올/물 분리계수는 투과증발 시간이 증가함에 따라서 초기에 증가하다가 다시 감소하였다. 초기 분리성능의 증가는 건조한 PDMS 분리막에 에탄올이 용해되는데 시간이 걸리기 때문이며, 후기 분리성능의 감소는 주입 탱크의 에탄올 농도가 시간에 따라서 감소하기 때문에 나타나는 현상이었다. 따라서 본 연구로부터 PDMS 분리막을 통한 에탄올의 투과는 용해-확산 메커니즘에 의해 발생된다는 것이 재확인되었다.

**Abstract:** Many studies on pervaporation (PV) for the separation of dilute alcohols as an alternative to conventional energy-intensive technique of distillation have been conducted earlier. The pervaporation transition behavior of ethanol-water mixtures through the PDMS/PSF membrane is important, in order to understand the mechanism of diffusion process. Therefore, in the present work, transient PV behavior for 50 wt% EtOH/H<sub>2</sub>O mixture at 50°C was investigated by using 1194 cm<sup>2</sup> PDMS/PSF hollow fiber membrane modules. The overall total flux and the separation factor of all the membrane modules increased initially and then gradually decreased with respect to PV time. The initial increase can be attributed to fact that membrane fibers were dry and it took time to dissolve into the membrane surface, but the subsequent decrease is due to the depletion of ethanol concentration in the feed. Therefore, it was confirmed that the ethanol permeation through a PDMS membrane is governed by the solution-diffusion mechanism.

**Keywords:** PDMS membrane, pervaporation, transient behavior, permeation mechanism

#### 1. Introduction

Solvents such as isopropyl alcohol (IPA), ethanol, butanol, benzene, and toluene are commonly produced and discharged into wastewater from industries such as the food industry, fermentation industry, chemical industry, etc. The most frequent solvent utilized in per-

vaporation is ethanol, since the separation of ethanol from aqueous streams is particularly essential from an industrial standpoint[1]. Global ethanol consumption was \$89 billion in 2019, with a CAGR of 4.8 percent projected from 2020 through 2027. The use of ethanol as a biofuel has raised the demand with a high purity grade more than 99.8 wt%. As a result, it must be

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well purified for the use of transportation fuels.

Over the last few decades, pervaporation technology has been intensively explored for the separation of organic-organic, water-organic, and organic-water mixtures[2-6]. Furthermore, it has been proposed as a practical and effective technological substitute for traditional methods, such as the distillation process utilized for the separation of azeotropic mixtures[7-11]. PV has major benefits over traditional processes, including economic effectiveness, environmental friendliness, high selectivity, and simplicity of modifying process parameters[12]. These considerable advantages of pervaporation technology have prompted the adoption of PV in a variety of applications, such as PV-distillation, PV-reaction hybrid processes, and PV-fermentation[13]. After that, the use of PV to separate organic solvents has become more common.

In pervaporation, the most common membrane materials studied for the past few decades are polydimethylsiloxane (PDMS), poly (1-trimethylsilyl-1-propynen (PTMSP), polysulfone (PSF), polyether sulfone (PES), polyacrylonitrile (PAN), polyvinylidene fluoride (PVDF), polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), cellulose acetate (CA) and Polyamide (PA). Despite the fact that there are many materials available, still none of these materials have not fulfilled the industrial requirements i.e, these materials are poor in ethanol selective properties. From the literature, polydimethylsiloxane (PDMS) is the most investigated material for evaluating ethanol pervaporation performance[14,15].

The permeation of a molecule through a PDMS membrane is influenced by many factors such as composition of feed mixtures, type and shape of membrane module, and processing conditions. Among physicochemical properties of feed mixtures, the polarity of components plays an important role in the mass transport through the hydrophobic PV membranes. Hydrophilic membranes have higher affinity for the polar components of the mixture and will allow it to pass through the membrane. On the other hand, the hydrophobic membrane such as PDMS will have less affinity for

polar components. Furthermore, penetrant-polymer interaction governs the permeation, in other words the diffusion through the separation layer. Therefore, it is very complicated to understand the mass transfer mechanism of organic-water mixtures through a membrane[16,17].

W. Zang *et al.* investigated the effects of temperature and concentration on the pervaporation performance of PTFE supported PDMS membranes for dilute organic solutions. It was observed that increasing the temperature, the overall flux rises initially and subsequently declines, and selectivity increases up to 40 °C. Moreover, by increasing the feed concentration, the flux increases but the selectivity decreased[13]. S.J. Lue *et al.* studied PDMS/polyurethane (PU) blend membranes and revealed that when the PU amount increased, diffusion rate decreased due to the monotonically drop of the free volume of the membrane. It was also determined that the trade-off between selectivity and flux was optimized by controlling the blending ratio[18]. T. Mohammadi *et al.* studied PV performance of a PDMS membrane for different alcohol mixtures such as methanol/water and ethanol/water. It is observed that methanol had a higher flux than ethanol. This is due to the smaller size of methanol, which enables it to interact less with the membrane, resulting in high flux. L. Li *et al.* investigated the transport behaviors of symmetric PDMS membranes under various conditions. They elucidated that the boundary-layer transfer coefficient rose exponentially with temperature and was proportional to the Reynolds number. The Arrhenius correlation of the membrane mass transfer coefficient was observed to be independent of flow states such as laminar, transient, and turbulent flows[20].

From aforementioned literatures, it is evident that, most of the studies focused on the separation behavior based on varying the type and concentration of feed solvents, additives to PDMS, PV operation conditions such as temperature and time, but there are no systematic study on the transient PV behavior. Therefore, in this study transient separation behavior of PDMS/PSF hollow fiber membrane modules has been investigated for 50 wt% ethanol-water mixture at 50°C.

## 2. Experimental

### 2.1. PDMS hollow fiber membrane modules

PDMS hollow fiber membrane module used in this study was manufactured and supplied by Airrane Co., Ltd. It was prepared by placing 950 hollow membrane fibers coated with PDMS on the surface of Polysulfone hollow fiber in an aluminum membrane module case and both ends were sealed by potting process. Test membrane modules used for this study had a surface area of 1194 cm<sup>2</sup> per module. Fig. 1 shows hollow fiber membrane module coated with PDMS on the surface of polysulfone manufactured by Airrane and hollow fiber bundle before potting.



**Fig. 1.** Images of pristine PDMS hollow fiber membranes and module.

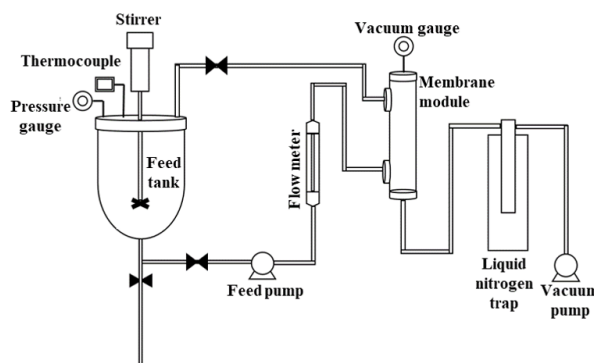
### 2.2. Characterizations

For SEM analysis, the PDMS/PSF hollow fibers were immersed in the liquid nitrogen for about 3 min and then cut while keeping it in the liquid nitrogen in order to have clear cross-section images. Field emission scanning electron microscopy (FE-SEM, Hitachi S-4800) connected with energy-dispersive X-ray spectroscopy was used for the membrane surface and cross-sectional thickness measurements.

### 2.3. Pervaporation studies

Pervaporation tests were performed using PDMS/PSF hollow fiber membrane modules to evaluate the separation behavior of EtOH/H<sub>2</sub>O mixtures. The schematic diagram of the pervaporation system employed in this work for EtOH/H<sub>2</sub>O separation is shown in Fig. 2.

The feed concentration of 50wt% EtOH/H<sub>2</sub>O mixture was prepared by combining deionized water and absolute ethanol (99.9% Conc.). The feed-tank had a total volume of 2 liters, and it was kept at 50°C, with constant stirring (200 rpm). Initially, the membrane was given a stabilization time for 1.5 hr in order to achieve proper wetting and 50°C temperature so that it can perform at the desired conditions. This was achieved by allowing the feed solution to circulate at a constant flow rate of 0.5 L/min across the membrane module. A rotary pump was used to pump the feed at a con-



**Fig. 2.** Schematic presentation of pervaporation test system.

stant rate of 0.5 L/min, and a vacuum was applied to the permeate side. The sample was collected using liquid nitrogen traps to measure the EtOH/H<sub>2</sub>O separation compositions. The permeate composition was analyzed using a gas chromatography (GC) fitted with a thermal conductivity detector (TCD). The retentate was recovered by injecting it back into the feed tank, and the feed tank overall temperature was maintained at 50°C. The total flux ( $J$ ) and the separation factor ( $\alpha$ ) was calculated by

$$J = \frac{m}{(A \times t)} \quad (1)$$

$$\alpha \left( \frac{EtOH}{H_2O} \right) = (W_i/W_j)_{perm} / (W_i/W_j)_{feed} \quad (2)$$

Whereas  $W_i$  and  $W_j$  are the mass fractions of the compounds  $i$  and  $j$  on the permeate side,  $m$  is the weight

of defrosted traps (kg),  $t$  is the time (min) and  $A$  is the membrane area ( $m^2$ ) respectively.

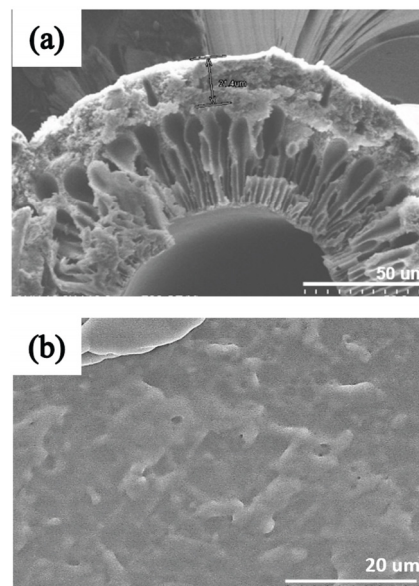
### 3. Results and Discussion

#### 3.1. PDMS/PSF hollow fiber membrane

Fig. 3 shows a surface and cross-sectional SEM-images of a single PSF supported PDMS hollow fiber membrane. Fig. 3(a) depicts that the structure of the PSF-support is in the microstructure region and the selective PDMS separation layer, has a thickness of 28  $\mu m$ . Fig. 3(b) exhibits the defect-free and uniform surface of PDMS/PSF membrane.

#### 3.2. Pervaporation performance of PDMS/PSF membrane modules

Table 1 shows the PV transient behavior of PDMS/PSF hollow fiber membrane Module 1 for a 50 wt% EtOH/H<sub>2</sub>O mixture. The concentration of ethanol on the feed side reduced from 47.75 to 38.9 wt%, while the concentration in the permeate has decreased from 87.8 to 84.7 wt%. Moreover, the separation factor initially increased followed by gradual decrease and finally remains stable. Similarly, in the case of Modules 2, 3, 4, and 5, shown in the Tables 2, 3, 4 and 5, the ethanol concentration on the feed side decreased from 49.15 to 40.1 wt%, 47.8 to 41.8 wt%, 47.8 to 39.6 wt%, 45.1 to 41.1 wt%, and 45.19 to 41.18 wt%. Also, on the permeate side, the same phenomena of decrease in the ethanol concentration was observed. In



**Fig. 3.** SEM images of PDMS/PSF hollow fiber (a) cross-section (b) membrane surface.

the case of water concentrations on the feed side increased from 50.69 to 59.52 wt% as shown in Table 1. This increase in the water concentration clearly suggested a decrease in the concentration of ethanol on the feed side. Whereas, on the permeate side the concentration of water increased from 12.14 to 15.30 wt%. The percentage increase in the concentration of water on the permeate side was very low as compared to the concentration increase on the feed side because the PDMS/PSF hollow fiber membrane is hydrophobic in nature and has more affinity towards ethanol molecules to adsorb. The same trend of percentage increase of

**Table 1.** Pervaporation Behavior of Module 1 Evaluated at 50°C for 50 wt% EtOH/H<sub>2</sub>O Mixtures

Time (hr)	Flux (Kg/m <sup>2</sup> .hr)			Feed conc. (%)		Permeate conc. (%)		Separation factor ( $\alpha$ )
	Total	Water	Ethanol	Water	Ethanol	Water	Ethanol	Ethanol/Water
0.25	1.98	0.24	1.74	50.67	47.75	12.14	87.86	7.68
0.5	1.89	0.24	1.66	51.50	46.91	12.50	87.50	7.69
0.75	2.17	0.30	1.87	56.92	41.50	14.00	86.00	8.42
1	2.08	0.32	1.76	54.11	44.31	15.25	84.75	6.79
1.25	1.87	0.26	1.61	56.16	42.26	14.03	85.97	8.14
1.5	1.73	0.26	1.47	57.67	40.75	15.20	84.80	7.89
1.75	1.69	0.26	1.43	59.52	38.90	15.30	84.70	8.47

**Table 2.** Pervaporation Behavior of Module 2 Evaluated at 50°C for 50 wt% EtOH/H<sub>2</sub>O Mixtures

Time (hr)	Flux (Kg/m <sup>2</sup> .hr)			Feed conc. (%)		Permeate conc. (%)		Separation factor ( $\alpha$ )
	Total	Water	Ethanol	Water	Ethanol	Water	Ethanol	Ethanol/Water
0.25	1.72	0.19	1.53	49.27	49.15	11.10	88.90	8.03
0.5	1.93	0.19	1.75	50.86	47.56	9.59	90.41	10.09
0.75	1.90	0.29	1.62	50.94	47.47	14.99	85.01	6.08
1	1.93	0.29	1.64	52.42	46.00	14.81	85.19	6.55
1.25	1.86	0.27	1.59	54.95	43.47	14.61	85.39	7.39
1.5	1.90	0.30	1.60	56.32	42.09	15.62	84.38	7.23
1.75	1.69	0.27	1.41	58.32	40.10	16.14	83.86	7.56

**Table 3.** Pervaporation Behavior of Module 3 Evaluated at 50°C for 50 wt% EtOH/H<sub>2</sub>O Mixtures

Time (hr)	Flux (Kg/m <sup>2</sup> .hr)			Feed conc. (%)		Permeate conc. (%)		Separation factor ( $\alpha$ )
	Total	Water	Ethanol	Water	Ethanol	Water	Ethanol	Ethanol/Water
0.25	1.94	0.27	1.67	50.57	47.85	13.96	86.04	6.51
0.5	2.06	0.30	1.75	51.20	47.22	14.66	85.34	6.31
0.75	2.04	0.31	1.73	52.76	45.66	15.01	84.99	6.54
1	1.80	0.28	1.52	53.35	45.07	15.36	84.64	6.52
1.25	1.69	0.25	1.44	54.01	44.40	15.07	84.93	6.86
1.5	1.96	0.29	1.67	55.16	43.26	14.63	85.37	7.44
1.75	1.94	0.30	1.64	56.58	41.84	15.35	84.65	7.46

**Table 4.** Pervaporation Behavior of Module 4 Evaluated at 50°C for 50 wt% EtOH/H<sub>2</sub>O Mixtures

Time (hr)	Flux (Kg/m <sup>2</sup> .hr)			Feed conc. (%)		Permeate conc. (%)		Separation factor ( $\alpha$ )
	Total	Water	Ethanol	Water	Ethanol	Water	Ethanol	Ethanol/Water
0.25	1.82	0.23	1.60	50.55	47.87	12.44	87.56	7.43
0.5	1.48	0.17	1.31	51.33	47.09	11.48	88.52	8.40
0.75	1.75	0.21	1.55	52.35	46.07	11.76	88.24	8.53
1	1.78	0.21	1.57	52.96	45.46	11.76	88.24	8.74
1.25	1.79	0.25	1.55	53.26	45.16	13.76	86.24	7.39
1.5	1.88	0.27	1.61	54.16	44.26	14.57	85.43	7.17
1.75	1.72	0.25	1.47	58.74	39.68	14.79	85.21	8.53

water concentrations on the feed side and permeate side was observed in the other Modules 2, 3, 4 and 5 exhibited in Table 2, 3, 4 and 5 respectively.

In Table 1 the total flux varies from 1.98 to 1.69 Kg/m<sup>2</sup> hr. Even though it shows an overall decrease, in the middle i.e., at 0.75 hr and 1 hr it exhibits a high total flux of 2.17 and 2.08 Kg/m<sup>2</sup> hr respectively,

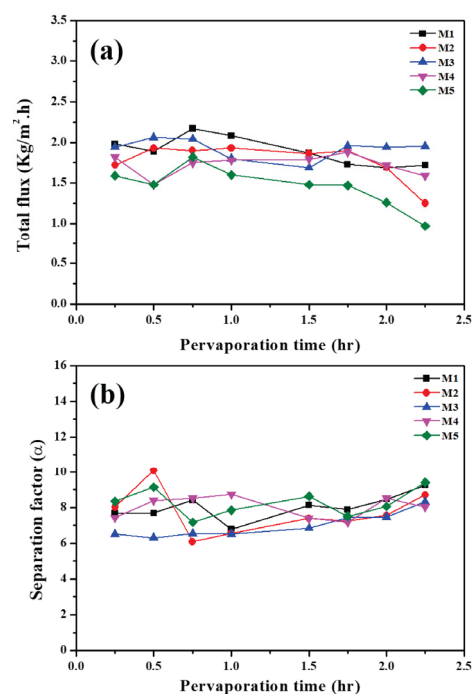
which is a deviation from the normal behavior. The separation factor ( $\alpha$ ) also followed the trend similar to flux, it fluctuated from 7.68 to 8.74. Table 2 summarizes the PV performance of Module 2 in which the total flux changes from 1.72 to 1.69 Kg/m<sup>2</sup> hr and the separation factor ( $\alpha$ ) from 8.03 to 7.56. After 0.5 hr separation factor ( $\alpha$ ) suddenly increased to 10.09 then

**Table 5.** Pervaporation Behavior of Module 5 Evaluated at 50°C for 50 wt% EtOH/H<sub>2</sub>O Mixtures

Time (hr)	Flux (Kg/m <sup>2</sup> .hr)			Feed conc. (%)		Permeate conc. (%)		Separation factor ( $\alpha$ )
	Total	Water	Ethanol	Water	Ethanol	Water	Ethanol	Ethanol/Water
0.25	1.59	0.20	1.40	53.23	45.19	12.36	87.64	8.35
0.5	1.48	0.17	1.31	53.92	44.50	11.71	88.29	9.14
0.75	1.82	0.25	1.57	52.49	45.93	13.74	86.26	7.18
1	1.60	0.21	1.39	53.76	44.66	13.30	86.70	7.85
1.25	1.48	0.19	1.29	54.98	43.43	12.79	87.21	8.63
1.5	1.47	0.22	1.25	56.29	42.13	15.10	84.90	7.51
1.75	1.26	0.18	1.07	57.24	41.18	14.71	85.29	8.06

dropped to 6.08 followed by a further increase up to 7.56. Module 3 shows the pervaporation performance which is somewhat similar to the behaviors observed in the case of Module 1 and 2. Initially it increased and the maximum total flux obtained was 2.06 Kg/m<sup>2</sup> hr at 0.5 hr and separation factor ( $\alpha$ ) increased from 6.51 to 7.46. In this case, the trend observed was different as compared to first two Modules 1 and 2. In Module 1 and 2 the trend was more inconsistent but in this Module 3 the separation performance was more consistent and gradually increased as pervaporation time increased as exhibited in Table 3. In addition, the PV transient behavior of Module 4 presented in Table 4, the total flux obtained altered from 1.82 to 1.72 Kg/m<sup>2</sup> hr and separation behavior ( $\alpha$ ) observed was similar to Module 1 and 2 and values varied from 7.43 to 8.53. Module 5 showed overall low performance in terms of total flux as compared to all other Modules (1, 2, 3 and 4) but in case of separation factor it followed the pattern of Module 1, 2 and 4 and the maximum value obtained was 9.14 at 0.5 hr.

Total flux and separation factor of all the PDMS/PSF hollow fiber membrane modules are displayed in Fig. 4. In terms of total flux the maximum performance was showed by the Module 1 and 3 and the lowest performance was shown by Module 5. However, similar pattern was not observed in terms of separation factor. In this case, Module 5 shows the better separation performance than the other modules. Moreover, Module 3 showed a gradual increase in the

**Fig. 4.** Pervaporation performance of pristine PDMS hollow fiber membranes Modules (1, 2, 3, 4 and 5) as a function of time for 50 wt% EtOH/H<sub>2</sub>O mixtures at 50°C.

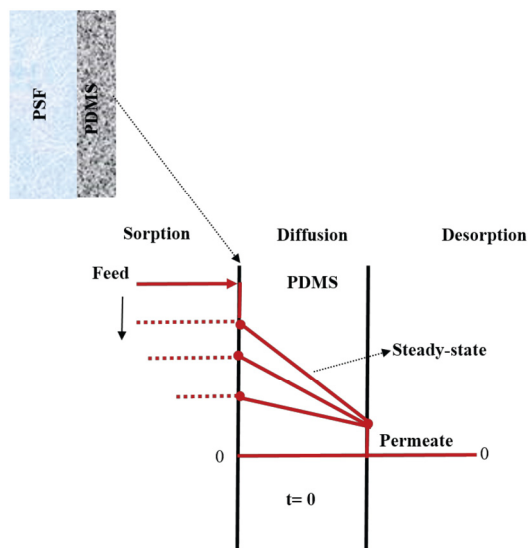
performance instead of fluctuating performance as observed in the Modules 1, 2, 4 and 5.

As time passes, flux begins to decrease, as seen in Fig. 4, with the exception of Module 3, which exhibits a slight increase, nevertheless this increase may be attributed to the aforementioned factors. Overall, all modules exhibit a drop in total flux, Apart from that, it could also happen due to cluster formation by a decrease in the ethanol concentration on the feed side.

This type of effect is not visible at lower concentrations, but in this study, the concentration is sufficiently high, i.e., 50 wt%, to produce this type of phenomena. During cluster formation, ethanol and water create hydrogen bonds with one other, increasing the overall size of the molecule. Therefore, aggregates into the polymer matrix and the diffusion coefficient decreases[23].

This kind of PV transient behavior give us an idea about the transport phenomena of PDMS/PSF hollow fiber membrane. Fig. 5 depicts the schematic representation of solution-diffusion mechanism which explains the separation behavior according to the results observed from the modules. The mass transport of non-porous PDMS membrane is recognized by so-called solution-diffusion mechanism which generally involves three steps: (a) Diffusion of the mixture species at the feed/active surface; (b) diffusion through the membrane; and (c) transport across the membrane permeate interface[17]. In the Fig. 5 the red color line represents the concentration of ethanol with respect to time. It shows high concentration of ethanol on the feed side followed by the saturation of the membrane surface, then decrease in the concentration of ethanol on the feed side. There are various factors that influence the solution diffusion mechanism that can be considered in order to explain such a kind of PV transient behavior and some of them are discussed below.

The transient behavior of flux and selectivity might have occurred for the following reasons: Specifically, because of the high concentration polarization at the feed-membrane interface rather than the permeate-membrane interface. The concentration is high in the upstream side, and it is nearly zero in the downstream side. Furthermore, as this process was operating at a higher temperature of 50°C, so, it brings up another possible factor that could be considered as a contributor to changes in flux, named “membrane plasticization”. At high operating temperature the membrane swells, causing the free volume to increase, hence the flux increases[21,22]. Another possible explanation for the increase in flux, is that the ethanol and water sorp-



**Fig. 5.** Schematic representation of solution-diffusion mechanism.

tion is an endothermic process, so, as the temperature increases flux increases.

Additional possible reason for the PV transient behaviors might be due to the dimerization of the ethanol-water molecules. The definition of dimerization is “an addition reaction in which molecules are linked by either strong or weak interactions”. Because of the hydrogen bonding, the ethanol-water dimer clogs the channel for both molecules, causing the diffusion coefficient to drop and, as a result, influencing the fluxes and separation factor. The high separation factor in Module 2, can be understood by the degree of dimerization. If the dimerization of ethanol-water molecules increases, the average size of the ethanol molecule increases, and the diffusion coefficient decreases, resulting in a negative impact on membrane performance. On the contrary, when the dimerization of ethanol-water molecules decreases, the average size of ethanol also decreases, and hence the diffusion coefficient of ethanol increases. In case of Module 2 performance, the dimerization might be decreased, which has subsequently increase in separation performance[23].

All the aforementioned possible reasons i.e., concentration polarization (due to high ethanol con.), plasticization (due to temperature i.e., 50°C), cluster for-

mation (due to hydrogen-bonding) and dimerization (due to additional reaction) broadens the understanding of the solution-diffusion mechanism. Which helps to understand the transient behavior observed during the PV of ethanol/water mixture through PDMS/PSF hollow fiber membranes. In this study, the concentration polarization factor mainly influence the solution diffusion mechanism, as the concentration of ethanol decreases on the feed side and increases on the permeate side because of the selective adsorption of ethanol on PDMS/PSF hydrophobic membrane.

#### 4. Conclusion

In this study, transient pervaporation behavior of 50 wt% EtOH/H<sub>2</sub>O mixture through PDMS/PSF hollow fiber membrane module was investigated. Initially the hollow fiber modules were dry and upon the interaction with the ethanol/water mixture (1:1) it started to dissolve into the separation layer of the membrane. So, it caused an increase in the performance but eventually with the passage of time it started to decline especially in case of permeate flow rate. Moreover, with the passage of time the increase in the concentration of ethanol on the feed side becomes low as compared to water concentration. The water concentration on the permeate side gets very low as compared to the concentration increase on the feed side because the PDMS/PSF hollow fiber membrane is hydrophobic in nature and has more affinity towards ethanol molecules to adsorb. As a result, it was confirmed that the solution-diffusion mechanism governs the ethanol permeation across a PDMS membrane.

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